ENCYCLOPEDIA OF EXPLOSIVES AND RELATED ITEMS

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VOLUME 1

BY

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TABLE OF CONTENTS

	Page
Preface	
Introduction	I
Nomenclature	II
Physical Tests Used to Determine Explosive Properties	VII
Abbreviations, Code Names, and Symbols	Abbr 1
Abbreviations for Books and Periodicals	Abbr 66
Descriptive Text of Encyclopedic Items	A1 to A673
Table I. Comparison of US, British and German Sieve Series	A674
Table II. Calibers of US and Foreign Ammunition	A675
Index of Subjects as Alternate Names of Items	A677

LIST OF FIGURES AND ILLUSTRATIONS

	Page
Sand Test Bomb	XXII
Taliani Test Apparatus	XXV
Vacuum Stability Test Apparatus	XXVI
Apparatus for Determination of Acetylene	A75
Flow Diagram for Synthesis of Ammonia	A298–9
Flow Diagram for Production of Ammonium Nitrate	A316–7
Apparatus for Determination of Moisture	A371
Nitrometer Apparatus for Determination of Nitrogen	A374
Apparatus for Determination of Nitrobenzene in Aniline	A416
Anvil of a Percussion Primer	A473
Lead Azide Normal Crystals	A567
Lead Azide Needle Shaped Crystals	A568
Apparatus for Lead Azide Content by US Ordnance Corps Gasometric Method	A569
Apparatus for Determination of Lead Azide Content	A582
Apparatus for Determination of Solidification Point	A613

PREFACE

The widespread interest in explosives during and since World War II has resulted in the need for a comprehensive coverage of the field of explosives and related items. In 1941-1944, Dr B. T. Fedoroff in collaboration with G. D. Clift had published a "Laboratory Manual of Explosives" in four small volumes (Lefax Co), for which there were numerous requests. Since the printed editions had been exhausted and the plates were no longer available, Dr Fedoroff decided to write a revised edition. As the work progressed, it became evident that additional help would be needed, not only because of the tremendous expansion of the literature, but also because it was decided to broaden the scope of the work

This Encyclopedia is intended to cover the following items: a)Military and industrial explosives, explosive compositions, propellants and pyrotechnic compositions b)Explosives and explosive compositions which have not been used for military or industrial purposes c)Analytical procedures for the more common explosives, propellants and pyrotechnic compositions d)Compounds which deflagrate or may possibly explode because of the presence of plosophoric groups e)Ammunition items, such as projectiles, bombs, grenades, detonators, fuzes, etc f)Calibers of weapons and projectiles used in the US and foreign countries g) Brief definitions of ordnance terms h)Names of scientists who made important contributions in the fields of explosives, ammunition and weapons

Over the years a number of works, including dictionaries, have been published in the field of explosives, propellants, etc, which are of general or limited scope. None of these has attempted to include in one work a comprehensive coverage of the broad field of items listed above

The authors hope that this Encyclopedia will be of value not only in saving many hours of library work but also in reducing the need for much laboratory work in order to obtain information already available. It is hoped that some of the data and even lack of data may stimulate additional work in the fields covered

In compiling this work, the authors have freely consulted with, and had the cooperation of so many individuals that a listing would be impractical. Any attempt to do so would surely result in some embarrassing omissions. We therefore take this opportunity to thank all those who have been consulted or who have helped in other ways in the preparation of this work. Throughout the Encyclopedia information received from individuals is acknowledged in the text. Picatinny Arsenal Technical Information (Library) reference works, such as journals, periodicals books and unclassified reports, were made available through the cooperation of all Library personnel. The complete copy of this manuscript was Vari-typed by Miss Margaret Dee, Mrs Sylvia Griffin and Mrs Bertha Kelly with the cooperation of Mr Joseph Farkas & Mr John Noonan, whom we consulted freely (all of the Technical Publications Unit, Feltman Research and Engineering Laboratories). Special acknowledgement is due to the officials (both military and civilian) of Picatinny Arsenal for encouraging this work and for obtaining its financial support

Although considerable effort has been made to present this information as accurately as possible, mistakes and errors in transcription do occur. The interpretations of data and opinions expressed are the responsibility of the authors and are not necessarily those of the Department of the Army or of Picatinny Arsenal. This report has been prepared for information purposes only and the Department of the Army or Picatinny Arsenal shall not be responsible for any events or decisions arising from the use of this information

INTRODUCTION

The user of this Encyclopedia is urged to read this Introduction to obtain an understanding of the authors' way of treating and covering the subject matter. Because of the broad scope of the work and the vast amount of material available, discussions of most items are brief. Theoretical and physico-chemical aspects, except for a few constants, are usually covered by the references. Items of distinct military or commercial importance are discussed in more detail. References to all sources of data, as well as references for broader coverage, are given following each item. No claim is made to complete coverage, but the authors believe that, in general, few pertinent references have been omitted

No attempt has been made to cover the large volume of material included in classified reports and no information from such reports has been used. However, for the benefit of those who have the right of access to classified information, some references to these sources may be given. Army regulations declassifying most of the classified reports originating before January 1, 1946, were not issued in time to permit review of the large number of these reports for information which would have been included under the letter A and covered in this first volume. Subsequent volumes may include subject matter from these declassified reports. In some cases, reports listed here as classified, may subsequently have been declassified

As was mentioned in the preface we have listed not only compounds which have been reported as explosive, but also compounds which have been or may be prepared and which, because of the presence of plosophoric groups, may possibly be explosive. This has been done because, unfortunately, many workers who have prepared compounds which may be explosive, have not made tests for explosibility. Many substances ordinarily not considered explosive, have exploded accidentally or been caused to explode experimentally

Because of the potential hazard from compounds containing plosophoric groups we have included compounds which, in our opinion, contain a sufficient percentage of such groups to make them dangerous under certain conditions. Such compounds may possibly find use as components of explosive mixtures, fuse compositions, etc. In the case of nitro compounds, this percentage was arbitrarily set at about 14% NO_2 and/or NO nitrogen, although some compounds with lower nitrogen content have been exploded. High nitrogen compounds on combustion yield large volumes of gas which may contribute to the ballistic potential of a propellant composition containing such compounds. If these compounds are not in themselves exothermic, their endothermicity may be of value in reducing the flash of propellent compositions. We have, therefore, decided to include compounds which contain about 40% or more nitrogen

In addition to a given explosive compound, we have included references to what may be considered as the parent compound of azido, nitro or nitroso derivatives. This is done because some information concerning the parent is usually needed for the preparation of explosive derivatives. References (mostly Beilstein) to intermediate non-explosive derivatives are included for the same reason. For example, naphthalene and its various mononitro and dinitroderivatives, which are not explosive are listed and references given

In order to make the Encyclopedia as compact as possible we used abbreviations, many of which are the same as used in Chemical Abstracts except that periods after abbreviations are omitted. A list of abbreviations symbols, code letters and special designations of items connected with explosives, propellants, pyrotechnics, ammunition and weapons is included in this work. This list is placed immediately before the Encyclopedia proper (see Abbreviations, pp Abbr 1-59) and also includes abbreviations and code letters for various Ordnance establishments, industrial installations and scientific institutions, both US and foreign. Some additional abbreviations are given in a supplementary list (see Abbreviations, pp Abbr 59-65). Wherever we have been able to do so and are permitted by security regulations, the meaning of code letters on ammunition, weapons and other military items is briefly explained

Following the above lists, the journals, books, and other reference sources most frequently used are given, together with our abbreviations for them (See pp Abbr 66-76). Journal abbreviations, not included in our list, are the same as given in the "List of Periodicals Abstracted by Chemical Abstracts," Ohio University, Columbus 10, Ohio (1956), except that we do not use periods and leave no space between abbreviated words. Although the French, Italian, Spanish and Portuguese books and journals do not capitalize the words in titles (except the first word) we use captials, as is the practice in abbreviating US and British journals

Nomenclature

Since most organic compounds can be designated by several names, it was necessary in each case to decide under which name to list a particular compound. Not only are different names used by different writers, but frequently the Abstractor for Chemical Abstracts used another name and in the Chemical Abstracts Indexes the compound is often indexed under a still different name. In general, the Chemical Abstracts Index name is here given preference. However, when a trivial name or an older name is used, the compound may be listed under one of these names. In every case where more than one name has been used to designate a compound, the others are also listed. In addition, alternative names are or will be listed and the various names cross-indexed. The reader will thus usually have little difficulty in locating the desired item under which the compound is discussed in this book. In most cases, when a compound is described in the German literature, the German name is also given. This should be of help to those who seek information on the compound in the German literature, particularily Beilstein and Gmelin

Azido, nitramino, nitro and nitroso derivatives are listed under what may be considered as the parent compound. Thus all the mono-, di-, and trinitrotoluenes will be discussed under toluene. For example, nitraminotetrazoles are discussed under aminotetrazole. With this system the various azido, nitro, etc derivatives included above are kept together and are not scattered throughout the Encyclopedia. Since these derivatives of a given parent compound are usually of some related interest from the point of view of properties, preparation and references, we believe that this arrangement is the most convenient

While most azido, nitroso, and nitro derivatives are listed under their parent compounds, the amino, azo, azoxy, etc derivatives are listed as parent compounds, themselves, either individually or as a group. Similarly, alkyl, phenyl and other

derivatives are listed under their corresponding alkyl, aryl, etc names, eg amyl derivatives under amyl, etc. Bis- compounds in general will be listed under B, and tris- compounds under T. Halogen containing compounds will be found under the appropriate halogen; eg chlorobenzene under C etc. Salts, peroxides, hydroperoxides, etc of organic compounds are listed under the corresponding organic parent compound, while metallic salts are discussed under the appropriate acids, such as chloric, nitric, perchloric, etc. An exception is Ammonium Nitrate which is discussed separately in this volume and not under Nitric Acid. Normal, iso-, tertiary-, etc, isomers of alkyl compounds are listed under the corresponding alkyl group. Thus isobutylperoxide will be found under butylperoxides

When the position of a substituent group has not been established or is in doubt, the doubtful position may be indicated by x or followed by (?). In some cases the probable positions are given in parentheses, eg 4(or 7)-aminobenzotriazole. Where two ways are commonly used to indicate the position of groups or elements in a molecule, one of the alternative ways is usually placed in parentheses directly after the letter or symbol designating the position, eg 2(or o), and α (or 1H). This is done to avoid repeating the whole name

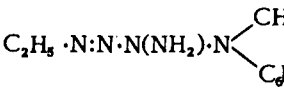
The nomenclature, particularly of complex compounds, is not always satisfactory in spite of the good work of the nomenclature commissions of the International Union of Pure and Applied Chemistry. Occasionally, a competent chemist would not be able to write the correct structure based on a given name. This is particularly noticeable in the naming of open chain and cyclic polynitrogen compounds and especially when the molecule contains both types of structures. As a result of our work in this field we have evolved a system which we have been using and which has some advantage in reducing ambiguity. Where it has seemed advisable, an additional name has been added, based on the system described below. This system was worked out in collaboration with Drs H. Matsuguma and H. Walter of Picatinny Arsenal and is essentially a combination of those systems used in Beilsteins Handbuch der Organischen Chemie and Chemical Abstracts

The open chain polynitrogen groups include:

Diazene	–HN·N<
Triazene	–N:N·N<
Tetrazene(1)	>N·NH·N:N– [called herein *isotetrazene* as has been done by F.L.Scott et al, JACS **75**, 5310(1953)]
Tetrazene(2)	>N·N:N·N< (which herein will be called simply *tetrazene)*
Pentazadiene	–N:N·NH·N:N–
Hexazadiene	–N:N·NH·NH·N:N–, etc

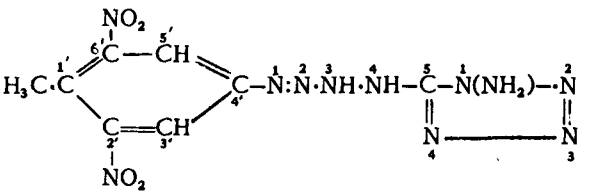
In these groups Beilstein designates the nitrogen at one end of the chain as N and the one at the other end as N′, but there is no provision for naming the compound if some organic radical is attached to one of the nitrogens not at an end of the chain. The system used in US Chemical Abstracts consists of designating the nitrogens by the numerals 1,2,3,4 etc but there is no provision for differentiating these numerals from those used in the ring structures (such as benzene, pyridine, triazole, tetrazole etc) which may be attached to one of the nitrogens of the open chain compounds. None on the above systems provides a ready, unequivocal indication of the position (in the ring) to which the nitrogen of the open chain compound is attached

In the system we have adopted the nitrogens of open chain compounds are designated as N^1,N^2,N^3,N^4,N^5,N^6, etc. For instance, the compound $C_2H_5 \cdot N{:}N \cdot NH \cdot NH \cdot C_6H_5$ would be called (N^1-ethyl, N^4-phenyl)-isotetrazene and the compound



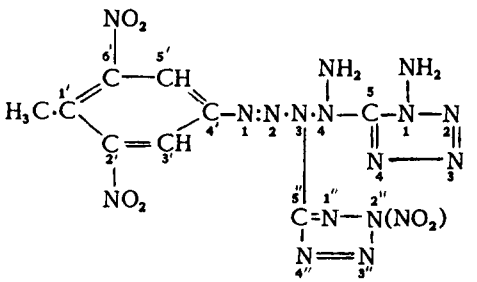
would be called (N^1-ethyl, N^3-amino, N^4-methyl, N^4-phenyl)-isotetrazene. Slightly more difficult would be the naming of a compound in which one (or several) hydrogens in an attached ring or in the straight nitrogen chain is substituted by one (or several) radicals, such as NH_2, NO_2, C_6H_5, etc

Let us name, according to our system, the compound:



. We have adopted the rule that a ring attached to the left N of the open chain nitrogen compound has the ring atoms designated 1′,2′,3′,4′, etc arranged *counterclockwise* and the atoms of the ring attached to the right N of the open chain nitrogen compound numbered 1,2,3,4, etc arranged *clockwise*, then the name of the compound would be [*N^1-(2′,6′-dinitrotoluene-4′), N^4-amino-α-tetrazole-5)*]-*isotetrazene*. Here, –4′) indicates that N^1 is attached to position 4′ in the 2′,6′-dinitrotoluene ring. The -5) indicates that N^4 is attached to position 5 in the tetrazole ring. The α-indicates the type of tetrazole to distinguish it from the β-tetrazole. In the α-compound the hydrogen atoms are in positions 1 and 5, whereas in the β-compound they are in the 2 and 5 positions. Currently Chemical Abstracts uses the designations 1H and 2H to indicate the ring atom to which a hydrogen is attached in the parent tetrazole ring. Since the NH_2 group is in an α-tetrazole it can only be attached to the N in the 1-position and it is therefore unnecessary to indicate the position

In rare cases in which a third ring compound is attached to one of the intermediate N atoms of an open chain nitrogen compound, its substituents would be numbered *clockwise* using double primes, as 1″,2″,3″, etc. In some cases the groups NH_2, NO_2, etc may be attached to intermediate N atoms of the open N chain. Following is the formula of a complicated hypothetical compound:

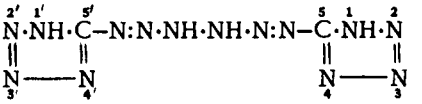


. According to our system its name would be: [*N^1-(2′,6′-dinitrotoluene-4′), N^3-(2″-nitro-β-tetrazole-5″), N^4-amino, N^4-(1-amino-α-tetrazole-5)*]-*isotetrazene*

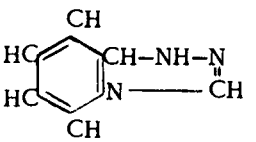
By adopting the proposed system it would be easier to arrive at a name corresponding to a given formula or to write a formula corresponding to a given name

than by using the systems described in Beilstein, Chemical Abstracts or British literature

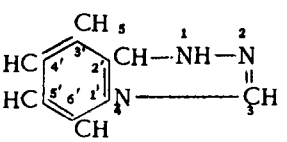
As another example may be cited the compound



which we would name [N^1,N^6-*bis(α-tetrazolyl-5)*]-*hexazadiene*. Let us now find a simple unequivocal name for the compound:



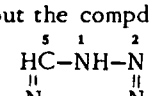
which has been variously named: a)pyrido-[2,1-c]-s-triazole b)triazolo-pyridine c)2,3-diazopyrrocoline d)benztriazole and e)1,2,3-benziso-triazole. Of these, only the 1st name would allow writing the correct structural formula once one learns the significance of the letter c. This system, however, not only uses numerals for indicating the position of atoms but also uses letters, a,b,c,d,e,etc for each side of a ring compound. On examining the above formula it is evident that the compound is a pyrido-s-triazole, but since there may be several such compounds, it is necessary to indicate the position at which the pyridine is connected to the s-triazole. If we adopt the system of numbering each ring separately, and if the group on the right side of the formula has the numerals 1,2,3, etc starting from NH and counting clockwise, while the group on the left side has the numerals 1′,2′,3′, etc starting from N and counting counterclockwise, then the formula numbering would be



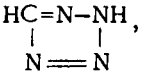
and the proposed name is *(pyrido-1′,2′)-s-triazole-4,5*.

If this compound had an amino group in position 4′ of the pyridine ring and a methyl group in position 1 of the triazole ring, then the name would be (4′-aminopyridine-1′,2′)-(1-methyl-s-triazole-4,5)

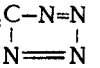
In the case of **tetrazoles** we use the Chemical Abstract system of numeration, but the compd



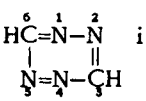
, known as 1H-tetrazole we call *α-tetrazole* and the compd



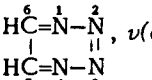
known as 2H-tetrazole we call *β-tetrazole*. We retain the name *isotetrazole* for the compound



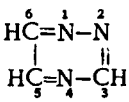
In the case of **tetrazines**, the compound

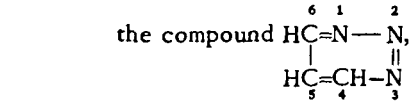
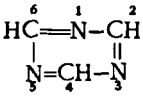


is called by us *s(or sym-)tetrazine* and the compound

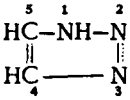


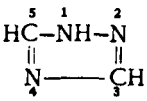
, *v(or vic-)-tetrazine*. The as-tetrazine is not known

In the case of **triazines**, the compound  is called by us *as-triazine,*

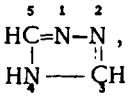
the compound  , *v(or vic-)-triazine* and the compound  ,

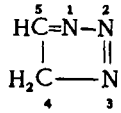
s(or sym-)-triazine

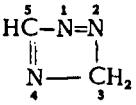
In the case of **triazoles**, the compound  is called *v(or vic-)-triazole*

and the compound  , *s(or sym-)-triazole.* If H is in position 1, we add

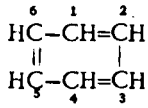
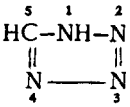
α-in front of s- or of v- and if H is in position 2, the letter *β*- is used and in the case of the compound

 , our name is *γ-s-triazole*

In the case of **isotriazoles,** the compound  is called by us *v(or vic-)-*

isotriazole, and the compound  *s(or sym-)-isotriazole*

In order to avoid the use of rings (which have to be drawn by hand) as much as possible, we adopted a system similar to that used in Beilstein for such compounds as benzene, pyridine, tetrazole, etc. For example, benzene is written as

 and *α*-tetrazole as  . It should be noted that in these

formulas, as written, position 1 is always in the middle of the upper line

PHYSICAL TESTS FOR DETERMINING EXPLOSIVE AND OTHER PROPERTIES OF ITEMS DESCRIBED IN THIS WORK*

Abel's Test(KI Heat Test or KI-Starch Test) (Épreuve d'Abel, in Fr.) A brief description is given on pA2 of this volume. The test is also described in TM 9–1910(1955),57–60

Ability to Propagate Detonation; Transmission of Detonation or Extent of Propagation of Explosion. The property of an expl to conduct detonation, which has been started by an initiator, to neighboring layers is dependent on the props of the expl mainly velocity of deton and on some other factors(such as confinement, thickness of layer, diam of charge, density of packing, and temp) (Ref 1,3,4,5 & 6). In some substs(such as AN), the deton wave might easily die out(or dampen) if the optimum conditions for propagation are not fulfilled

Munroe(Ref 2) described several tests for detn of propagation of deton in AN. For these experiments, use was made of a wooden trough, appr 5½" square in cross-section and 10' long, which rested on the ground. Such a trough held ca 125 lbs of AN. Use was made of both warm nitrate(52°) and of cold nitrate(21°). The warm nitrate was used to insure deton and was placed at the initiating end of the train. The effect was detd by the size and extent of the crater produced beneath the box. As the initiation of AN could not be produced by detonators(such as electric tetryl detonator), it was necessary to use sticks of blasting gelatin. With this initiation complete detonation of AN could be obtained, except in cases when AN was 1" or smaller in diameter. Warm AN detond easier than cold AN

The propagation test may also be conducted similarly to the *pin method* described by Cook(Ref 6)

Refs: 1)Colver(1918),639 2)C.E.Munroe, ChemMetEngrg **26**, 541(1922) 3)J.L.Sherick, ArOrdn **24**, 329 & 395(1924) 4)G.W.Jones, ArOrdn **5**, 599(1924) 5)D.B.Gawthrop, ArOrdn **6**, 47(1925) 6)Cook(1958),29–31

Absorption of Moisture by Explosives, Propellants and Pyrotechnic Compositions. See Hygroscopicity

Action of Light on Explosives, etc. See Light, Action on Explosives, etc

Aptitude à l'inflammation, Essai. Same as Capability to Inflame Test

Armor Plate Impact Test(Shell Impact Test). This test was developed during WW II to provide an additional sensitivity test for HE's commonly used, and to supplement data obtained by impact and rifle-bullet tests. The test is supposed to duplicate the conditions which take place when a HE projectile hits a hard surface

For this test a modified 60 mm Mortar Shell is loaded with the test expl, drilled about ½ inch, and closed with a steel plug screwed into the shell to give a close fit between the plug base and the charge. The igniter and propelling charges are loaded into the "gun" through a simple breech plug. The loaded shell is fired from a 5 foot length of Shelby steel tubing against rigidly-mounted mild steel plates. Velocities of the shells are measured electronically and whether or not flash occurs on impact is determined by observation. The value reported is the velocity in ft/sec at which the expl is unaffected in 50% of the trials. *Refs:* 1) OSRD 5146(1945),2–3 & 11 2)PATR 1740, Rev 1(1958)

Available Energy. Same as Maximum Available Work Potential

Ballistic Mortar Test(Ballistische Mörser, Probe in Ger) (Essai au mortier éprouvette, in Fr) is a measure of power. It is used in the US in preference to the Trauzl Test which is standard in some European countries. The Ballistic Mortar Test consists of firing various charges of test explosive in a heavy steel mortar, attached to a pendulum bar and suspended on knife edges, and comparing the degrees of swings produced with that obtained on detonation of 10 g samples of TNT. From the values for TNT and from the weights of charges of sample producing nearly the same deflections as 10 g of TNT, the amt of sample producing exactly the same deflection as 10 g of TNT is calculated. Then the Ballistic Mortar Value is derived from the formula:

$$\text{BM Value} = \frac{10 \times 100}{\text{Sample Weight}} \text{ \% of TNT}$$

(See also Mortar Test). *Refs:* 1)US BurMinesBull **346**(1931),46–9(Ballistic Mortar, DuPont Type) 2)W. Taylor & K.Morris, TransFaradSoc **28**, 545–58(1932) 2a)Vennin, Burlot & Lecorché(1932),189 3)Stettbacher (1933),370 4)OSRD Rept 803(1942),19–21 5)Picatinny Arsenal Testing Manual No 7–2(1950) 6)TM 9–1910 (1955),69(Described as Ballistic Pendulum) 7)PATR 1740, Rev 1(1958)

Ballistic Pendulum Test(Essai au pendule balistique, in Fr) (Ballistische Pendel Probe, in Ger). This is the Official Test for the power of coal mine explosives, both in the US and GtBtitain. The pendulum used at the US BurMines Testing Station, Bruceton, Pa consists of a mortar(12.2" diam and weighing 31600 lbs), attached to a pendulum, as described in Ref 3, p 43 . The test expl is loaded (8 oz) in a borehole (2¼" ID & 2½" deep) of a steel cannon (24" OD × 36" long) and the charge is stemmed(tamped) with 2 lb of clay. The cannon is then moved on a track to within $^{1}/_{16}$" of the mortar and after adjusting the bore exactly opposite the mortar-muzzle the charge is fired by means of an electric detonator. The impact of products shooting from the cannon against the mortar causes deflection(swing) of the pendulum. The swing is measured and compared with that produced by the same amt(8 oz) of 40 per cent straight Dynamite(NG 40, $NaNO_3$ 44 woodpulp 15 & $CaCO_3$ 1%), which is designated as PTSS(Pittsburgh Testing Station Standard) Dynamite. Its swing is 2.7 to 3.1"

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* For meaning of abbreviations, see pp Abbr 1 to Abbr 76, which follow

By means of trial and failure the weight of the sample(W) that gives approximately the same swing as 8 oz of the standard is then detd, and three shots are fired with this wt. The exact swing of the sample(S_x) is calcd from the formula:

$$S_x = \frac{W \times S_D}{8}$$, where S_D is swing given by 8 oz of the standard(Refs 1,2,3 & 4)

The pendulum in use at the British testing station at Rotherham weighs 5 tons and is suspended by steel rods from an overhead axle having roller bearings. The bore-hole of the cannon(gun) is 30" long and 1⅞" diam. The charge consists of 4 oz of expl well-rammed with 2 lbs dry clay as stemming. The cannon is moved to within 2" of the mortar-muzzle and the charge is fired by an electric detonator. The swing is read and compared with 3.27" which is the swing produced by 4 oz of 60% Gelignite(NG 60, CC 4, KNO_3 28 & woodmeal 8%) (Ref 2,p 183–4)

The ballistic pendulum test is also used in GtBritain for determining performance of military explosives. For this a 10 g sample of expl is detonated in a loose condition under light confinement at the center of a heavy hollow cylinder, closed at one end, and suspended so as to form a pendulum. The swing is compared with that produced by 10 g of picric acid. The results are expressed as percentages of the performance of picric acid(Ref 5) *Refs:* 1)Marshall 2(1917),473 2)Barnett(1919),182–4 3US BurMinesBull 346 (1931),40–6 3a)Vennin, Burlot & Lécorché(1932),269 4)Stettbacher(1933),368 5)Blatt,OSRD 2014(1944)

Behavior Towards Heat Tests. See Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc Tests, as well as Burning Tests, Combustion Tests and Idex of Inflammability Tests

Bergmann-Junk Test has been used widely in Europe and to some extent in the US for testing the stability of NC. In this method NC is heated at 132° for 2 hours which action causes the evoln of some nitrogen oxide fumes. The fumes are absorbed in water giving a soln of nitrous and nitric acid. The nitrogen content of the soln is detd by the Schulze-Tiemann method. More detailed description will be given in Vol II,under B *Refs:* 1)E.Bergmann & A.Junk, ZAngChem 17,982,1018 & 1074(1904) 2)Reilly(1938)83–5 3)Kast-Metz (1944),218–20 & 312 4)PATR 1401,Rev 1(1950),19–25

Bichel Bomb or Bichel Pressure Gage is a device for measuring the press of an expln and for collecting and examining the gaseous, liq, and solid products formed. The apparatus consists of two stout cast steel horizontal cylinders, one of 15 l and the other 20 l capacity. Each cylinder can be closed with heavy lids provided with lead gaskets and secured in place by heavy stud bolts and an iron yoke. Three(or more) small diam holes are drilled through the upper part of cylinders: the 1st hole is connected to the tube of a vacuum pump, the 2nd accommodates an insulated plug that provides a means for conducting the electric current to the electric detonator inside the bomb and the 3rd is connected to a press gage provided with a registering drum

For examination of an expl a charge(usually 50 to 300 g) is placed inside the bomb, and, after closing the lid, the air is evacuated by means of a vacuum pump. Then the chge is fired electrically and the pressure diagram is obtained. Method of computation of results is given in Ref 2. The result thus obtained is termed the *"maximum pressure of the explosive in its own volume"*

This apparatus also affords a means for collecting and examining the products formed on expln. The method of sampling is described in Ref 2, pp 92–3.*Refs:* 1)C.E.Bichel, "New Methods of Testing Explosives," Griffin,London(1905) 2)US BurMinesBull 346(1931),84–95 3)Vivas, Feigenspan & Ladreda, v 4(1944),98–104(Under the name "Medidor de presion de la casa Carbonit")

Blast Effects in Air, Earth and Water will be described in Vol II, under B.*Refs:* 1)TM 9–1910(1955),72–6 2)Cook(1958),322 3)Ordnance Proof Manual OPM 80–12(1959)

Blasting Caps and Detonators, Initiating Efficiency. See under Initiating Efficiency of Primary Explosives, Blasting Caps and Detonators

Bomb Drop Tests(Bomb Functioning Test) are usually conducted using bombs assembled in the conventional manner, as for service usage, but provided with either inert or simulated fuzes. The target is usually reinforced concrete.*Refs:* 1)Ordnance Proof Manual, Aberdeen Proving Ground, Nos 9–11(1949) & Nos 10–80(1957) 2)PATR 1401,Rev 1(1958)

Booster Sensitivity Test involves measuring the relative sensitivities of various expls to an arbitrary graded series of boosters. This test was designed to classify expls on the basis of their ease of deton by boosters. The source of the shock consists of tetryl pellets of varying weights which may be degraded by wax spacers of Acrawax B. The booster charge is initiated by a No 8 detonator. The weight of tetryl reported as a final value is the min wt which will produce 50% detonations through the thickness of wax in inches, as indicated

This test is considered as one of the **Detonation by Influence(Sympathetic Detonation) Tests.** (See also Gap Test, Halved Cartridge Gap Test and Wax Gap Test). *Refs:* 1)OSRD Rept 5746(1945) 2)PATR 1740, Rev 1(1958)

Brisance or Shattering Effect(Brisance, in Fr) (Brisanz, in Ger) (Poder rompedor or Brisancia, in Span) (Potere dirompente, in Ital) can be approximately measured by the following methods: a)**Compression Tests**

[such as Lead Block Compression Test(Hess Test), Copper Cylinder Compression Test(Brisance Meter of Kast Test) and Compression of Small Lead Blocks] b)*Fragmentation Test*(qv) c)*Fragment Density Test*(qv) d)*Nail Test*(qv) e)*Plate Tests*(qv) (Cutting or Denting using various metals, such as brass, copper, iron, lead and steel) f)*Quinan Test*(qv) g)*Sand Test*(Sand Crushing Test)

Brisance can also be calculated from the formula of Kast. This gives *Brisance Value*, called in Ger Brisanzwert(Refs 1&4). A detailed discussion on brisance will be given in Vol II, under B. (See also under Compression Tests).*Refs:* 1)H.Kast, SS 8, 88(1913) 2)Marshall 2 (1917),495 3)Barnett(1919),184 4)H. Kast, SS 15, 181(1920) 5)Stettbacher(1933),49–50 6)Reilly(1938),68 7)Davis(1943),3 8)Vivas, Feigenspan & Ladreda, 4(1944),58–62 & 118 9)Belgrano(1952),39–41 10)TM 9–1910(1953),60–3 11)Cook(1958),17 & 34

Bulk Compressibility and Bulk Modulus is one of the important constants of an elastic solid. Bulk modulus is defined as the ratio of stress to strain when the stress is a pressure applied equally on all surfaces of the sample and the strain is the resulting change in volume per unit volume. The reciprocal of bulk modulus is called bulk compressibility. One apparatus for the direct exptl measurement of the dynamic bulk modulus of a solid was developed at the NOL, White Oak, Md(Ref 1). Some data obtained, on several HE's, using this apparatus are given in Refs 2 & 3. *Refs:* 1)NAVORD Rept No 1534(1950) 2)NAVORD Rept No 4380(1956) 3)PATR 1740,Rev 1(1958)

Bullet Impact Sensitiveness Test or Rifle Bullet Test(Essai au choc des balles, in Fr) (Beschuss-Sicherheit Probe, in Ger) (Prueba al choque de las balas, in Span) will be discussed in Vol II, under B. *Refs:* 1)Vennin, Burlot & Lécorché(1932),215 2)OSRD Repts 803 & 804(1942),15 3)Meyer(1943),374 4)Vivas, Feigenspan & Ladreda 4, (1944),115 5)OSRD Rept 5745(1945) 6)Ohart(1946),31 7)E.Burlot, MAF 23, 185(1949) 8)L. Médard & Cessat, MAF 23, 195(1949) 9)A.LeRoux, MP 33, 283(1951) 10)TM 9–1910(1955),49(described under "Sensitivity to Frictional Impact")

Burning Rate Tests are discussed in US BurMinesBull 346(1931),30–1

Burning Tests. As surplus expls are usually destroyed by burning, it is desirable to know their burning characteristics before proceeding to burn them on a large scale. A number of different methods have been designed at the US BurMines. One of the tests, designated as V–466 is as follows: Paper is placed on the ground, the cartridges, the wt of which should not exceed 2 lbs, are opened and the contents spread in a thin layer on the paper. After saturating the expl with kerosene, a charge (ca 25 g) of black blasting pdr is placed on the edge of the layer and an igniter, connected to a firing machine placed at a distance of not less than 60 ft, is placed in contact with blk pdr. The duration of burning of the expl is recorded. (See also Combustion Tests, Index of Inflammability Test and under Sensitivity to Flame, Heat, Spark, Electrostatic Discharges, etc Tests). *Ref:* US BurMinesBull 346 (1931),31

Calorimetric Tests for Explosives, Propellants and Pyrotechnic Compositions. These tests include determinations of *heat of combustion*(designated as Q_c), *heat of explosion*(Q_e), *heat of formation*(Q_f), *heat of fusion* (Q fusn), *heat of sublimation*(Q subln) and *heat of vaporization*(Q vapzn)

This subject will be discussed in Vol II, under C. *Refs:* 1)Marshall 2 (1917),440–2 2)Barnett(1919,197 3)US BurMinesBull 346 (1931),100–4 4)Vennin, Burlot & Lécorché(1932),60–7 5)Stettbacher(1933),83 6)A.Schmidt, SS 29, 259 & 296(1934) 7)OSRD Rept 293(1941) 8)OSRD Rept 702(1942) 9)OSRD Repts 803 & 804(1942),32 10)Vivas, Feigenspan & Ladreda 4 (1944),73–84 11)Physico-Chemical Unit Rept No 52–HI–595(1952),PicArsn,Dover,N.J. 12)F.D.Rossini, "Experimental Thermochemistry," Interscience,NY (1955) 13)H.W.Sexton, "The Calorimetry of High Explosives," ARDE Rept (5) 4/56, Apr 1956(Conf) (Not used as a source of information) 14)Parr Oxygen Combustion Bombs. Description of bombs and operation procedures may be obtained from the Parr Instrument Co,Molline,Ill

Capability to Burn Tests. See Burning Tests

Capability to Inflame Tests(Épreuves de l'aptitude de l' inflammation or Épreuves de combustion, in Fr). See Combustion Tests, Index of Inflammability Test and also under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc Tests

Cavity Charge Performance. See Shaped(or Hollow) Charge Efficiency in this section

Cholon Test permits simultaneous detn of the brisance and potential. It is discussed by Pepin Lehalleur (1935),64

Characteristic Product of Berthelot(Product characteristique de Berthelot, in Fr) will be discussed in Vol II, under C. *Ref:* Marshall 2 (1917),417

Closed Vessel Test(Essai en vase clos or Épreuve à la bombe, in Fr) will be described in Vol II, under C. *Refs:* 1)H.Muraour, "Poudres et Explosifs," Presses Universitaires de France, Paris(1947),73–4 2)H. Muraour et al, MAF 22, 517–93(1948)

Coefficient'd Utilisation pratique(CUP or cup) (Épreuve de travail spécifique). It is a modification of Trauzl Test(qv) designed by Dautriche and used as an official French test Briefly the test consists of packing the cavity(25 mm diam & 125 mm deep) of Trauzl lead block(200 mm diam & 200 mm high) with 15 g of crystalline PA(picric acid) in such a manner that the height of charge is exactly 38 mm. In an identical block is placed such an amt of expl to test, that it would produce an expansion of cavity as close as possible to that produced by 15 g of PA. After slightly compressing the charge and inserting a perforated cork with a No 8

detonator, the cavity is filled to the top with dry sand and the same is done with the chge of PA. After firing the charges, the expansions of cavities are detd and compared. If expansion(V' cc) produced by C' g of sample is not exactly identical with the expansion(Vcc) produced by 15 g PA, but does not differ much, the exact wt of expl(C g) necessary to produce expansion Vcc can be found from the equation $\left(\frac{C}{C'}\right)^{1.25} = \frac{V}{V'}$ Then the value of CUP is obtained from the formula $\frac{50 \times 100}{C}$, where C is wt of expl necessary to produce the same expansion as produced by 15 g of PA(Ref 6). It has been claimed that this method gives more reliable results than the regular Trauzl test. *Refs:* 1)Marshall 2, (1917),472 2)Vennin, Burlot & Lécorché(1932),171 3)Stettbacher(1933),363 4)Pepin Lehalleur(1935),66 6)L.Médard, MP 33, 344(1951)

Combustion Tests(Épreuves de combustion ou de l'Aptitude à l'inflammation, in Fr). French official combustion tests for expls and propellants include: a)Combustion en gouttière de 20 mm(Combustion in a trough of 20 mm), known also as Épreuve de propagation dans une gouttière de 20 mm(Propagation test in a trough of 20 mm) and b) Combustion en tas conique(Combustion in a conical pile), known also as Épreuve de sensibilité a l'inflammation(Sensitivity to ignition test) are described by L.Médard, MP 33, 329–30(1951)

Compression Tests, such as *Copper Cylinder Compression or Crusher Test*(Brisance Meter of Kast Test), *Lead Block Compression or Crusher Test*(Hess Apparatus Test) and *Compression with Small Lead Blocks* will be discussed in Vol II, under C. *Refs:* 1)Marshall 2, (1917),495–501 2)US BurMinesBull 346 (1931), 106–8 3)Stettbacher(1933),365–7 4)Pepin Lehalleur(1936),63 & 78

Concrete Test will be discussed in Vol II, under C. *Ref:* Marshall 2, (1917),273

Cook-off Test is briefly discussed in this volume, under Ammonium Nitrate, A354, Note a. *Ref:* Spencer Chemical Co, "Safety Data," Feb 4, 1960

Copper Cylinder Compression (or Crushing) Test. Same as Brisance Meter of Kast Test will be discussed in Vol II, under B

Cratering Effect or Earth Cratering Test(Essai dans la terre, in Fr) will be discussed in Vol II, under C. *Refs:* 1)Pepin Lehalleur(1935),67 2)Meyer(1943),379–80 3)Vivas, Feigenspan & Ladreda 4, (1944),117 4)H.Muraour, "Poudre et Explosifs," Paris(1947),80–1 5)Belgrano(1952),28–30 6)TM 9–1910(1955),76–8

Crawshaw-Jones Apparatus for testing coal mine explosives for permissibility will be discussed in Vol II, under C. *Ref:* US BurMinesBull 346, (1931),95

Crusher(Crushing) Tests, such as Copper Cylinder Crushing(Compression) Test and Lead Block Crushing (Compression) Test are used for estimation of the brisance of explosives

CUP or cup Test. See Coefficient d'utilisation pratique in this section

Dautriche Method for Determination of Velocity of Detonation will be discussed in Vol II, under D, as one of the Detonation Velocity Tests

Deflagration Test or Deflagration Temperature Test. See Ignition Temperature Test in this section

Deliquescence Test. See Hygroscopicity Test in this section

Density Determinations will be discussed in Vol II, under D

Detonation by Influence or Sympathetic Detonation Tests(*Transmission of Detonation at a Distance Test)* (Aptitude à transmettre la détonation à distance, Essai or Coefficient de self-excitation, Essai, in Fr) (Detonationsübertragung Probe or Schlagweite Probe, in Ger) (Determinación de la sensibilidad a la iniciación por simpatía, in Span) (Distanza di esplosione per simpatia, Prova, in Ital) include the following methods: a)*Booster Sensitivity Test*(qv) b)*Four-Cartridge Test*[See PATR 2510(PB 161[illegible]) (1958),p Ger 52] c)*Gap Test*(qv) d)*Halved Cartridge Test*(qv) and e)*Wax Gap Test*(qv). *Refs:* 1)Marshall 2 (1917),430 2)Barnett(1919),212 3)US BurMinesBull 346, (1931),59 4)Pérez Ara(1945),112 5)L.Médard, MP 33, 342 (1951) 6)Belgrano(1952),43

Detonation Pressure is, according to Cook(Ref), a property of great importance in detonation technology. Its direct measurement cannot be made due to its transient nature and its exceedingly high magnitudes, at least in condensed explosives. The detonation pressure is, however, accurately defined by the hydrodynamic equation given on p 32 of the Ref. *Note:* The "detonation pressure" must not be confused with "Pressure of Gases Developed on Detonation"(qv). *Ref:* Cook(1958),32

Detonation Rate Determination or Velocity of Detonation Test(Mesures de vitesse de détonation, in Fr) (Detonationsgeschwindigkeit Probe, in Ger) (Medida de la velocidad de detonación,in Span) (Determinazione della velocità di detonazione, in Ital), can be approx calcd, but more reliable results are obtained experimentally by one of the following methods: a)Mettegang b)Dautriche c)Rotating Drum Streak Cameras d)Rotating Mirror Cameras(such as Bowen RC–3; Cook-Doering-Pound; Beckman & Whitley, Inc; AEC-Bowen Type, etc cameras) e)Grid-Framing Camera of Sultanoff f)O'Brian & Milne Image Dissector g)Pin-Oscillograph Method h)Microwave Method i)Miniature Charge Techniques, etc. *Refs:* 1)Marshall 2, (1917), 477 2)Barnett(1919),185 3)US BurMinesBull 346, (1931),160 4)Vennin, Burlot & Lécorché(1932),158–61 5)Stettbacher(1933),53–61 6)Reilly(1938),68–9 7)OSRD Repts 803 & 804(1942),22–3 8)Davis(1943),14–18 9)Vivas, Feigenspan & Ladreda 4, (1944),62–72 10)PATR 1465(1945) 11)L.Médard, MP 33, 352(1951) 12)Belgrano(1952),30–9 13)TM 9–1910(1955),41 14)Cook(1958),22–35 & 41–2

Detonators and Blasting Caps, Initiating Efficiency. See under Initiating Efficiency of Primary Explosives, Blasting Caps and Detonators

Distribution of Shell Fragment Masses was detd at ERL, Bruceton, Pa by firing shells in a Fragmentation Pit filled with sawdust. The fragments were recovered by a magnetic separator. Details of procedure are given in OSRD Rept 5607(1945). See also OSRD Repts 5606 and 5608

Drop Test. Same as Impact Sensitivity Test

Earth Cratering Test. See Cratering Effect Test in Vol II, under C

Erosion of Gun Barrels[Erosion(ou usure) des bouches à feu, in Fr] (Erosion der Gewehrläufe; Bohrabnutzung, or Bohraubrennung, in Ger) Test will be discussed in Vol III, under E. *Refs:* 1)Marshall 2 (1917),315 2)Vennin, Burlot & Lécorché(1932),274 3)Marshall 3, (1932),93 4)Stettbacher(1933),211 5)Pepin Lehalleur (1935),102 6)H.Muraour, "Poudres et Explosifs," Paris(1947),115–16 7)PATR 2510(PB161270) (1958), p Ger 43

Esop's Test for Efficiency of Detonators, devised by K.Esop of Austria, in 1889, consisted of the following operations: A 50 g sample of uniform grain PA(picric acid), was made into a cartridge and, after inserting a test detonator, it was placed on a steel plate covering two *small lead cylinders*(crushers), set vertically on a steel base. After the chge was fired, the compression of the cylinders was measured and this served as a std value indicating complete detonation. This same type of detonator was then tested in mixts of PA with varying amts of cotton seed oil and the max amt of oil still permitting complete deton was detd. The larger this amt, the more efficient was the detonator(Ref 1)

This test was investigated in Europe after WWI and found to be more reliable than other tests, especially the sand test(Refs 1&2). In a modification of the test devised at the Chemisch-Technische Reichsanstalt, in Berlin(Refs 3&4) mixts of TNT with paraffin wax and later of TNT with talc compressed into pellets, served as inert expls for testing detonators. Completeness of deton was judged by firing the pellet with the test detonator in a small lead block(Trauzl test) and measuring the enlargement of cavity(Compare with Grotta's Test and Miniature Cartridge Test). *Refs:* 1)Marshall 2 (1917),532 2)H.Kast & A.Haid, SS 18, 166(1924) 3)Jahresber CTR V,112(1926) & VI,121(1927) 4)Marshall 3 (1932),163–4

Explosion by Influence(or Sympathetic Detonation) Tests. See Detonation by Influence Tests

Explosion(or Ignition) Temperature Test. See Ignition(or Explosion) Temperature Test, in this section

Explosion(or Ignition) Time Test(at Constant Temperatures). See Ignition(or Explosion) Time Test(at Constant Temperature)

Extent of Propagation of Explosion. See Ability to Propagate Detonation in this section

Exudation(or Sweating) Tests(Exsudation Essais, in Fr) (Ausschwitzungsproben, in Ger). The purpose of the exudation test is to determine whether expls, such as dynamites, DNT, TNT, etc would release any liq in storage, especially in hot climates

Following tests are described in Ref 1, pp 25–7: a)*Centrifuge Test* b)*Forty-Degree Test* and c)*British Test.* Of these the centrifuge test was adopted by the BurMines as being best suited for ascertaining the liability of dynamites to exude

The French test, called *exsudation par étuvage,* is conducted as follows:

Weigh to the nearest mg a tall Kraft paper container 30 mm in diam and 0.2 mm thick. Pack the container with the sample(100 g) and reweigh. Tare to the nearest mg a piece of porous cardboard 5×5 cm square and 2 mm thick. Place the container on the cardboard and insert the ensemble in an oven, thermostatically maintained at $40 \pm 1^\circ$ or $50 \pm 1^\circ$. After 21 days, reweigh the cardboard, the sample + container, and the empty container

If the loss in wt of sample is P, increase in wt of container p and increase in wt of cardboard is p', then (p+p') is the wt of exudate and P–(p+p') the loss in volatiles. Multiply the wts by 100 to express in percentage(Ref 5)

Another French method is described in Ref 3a

A German method for detn of exudation is described in Ref 7

Some tests have been investigated at Picatinny Arsenal, but exudation has been observed or induced in loaded items of ammunition by subjecting them either to alternate periods of heating and cooling or to continuous heating at $160^\circ F(71^\circ C)$. There is no record of TNT exudation from US shell smaller in caliber than 105 mm or from Composition B loaded shell stored at ambient temp. However, most TNT and TNT-contg expls can be caused to exude by storing the loaded components at $160^\circ F(71^\circ C)$ or in temps which fluctuate sharply. In one series of tests to induce exudation and to obtain exudate samples for analysis and examination of properties, unfuzed shell were placed in an inverted position in temps up to $160^\circ F(71^\circ C)$ and the exudate was collected in a tared vessel. A summary of the pertinent results from exudation studies conducted at PA has been prepared by Stein(Ref 6). *Refs:* 1)Marshall 2 (1917),419–22 2)Barnett(1919),215 3)US BurMinesBull 346 (1931),25–7 3a)Pepin Lehalleur(1935),61 4)Reilly(1938),65 5)L.Médard, MP 33, 328(1951) 6)PATR 2493(1958) 7)PATR 2510(PB 161270) (1958),Ger 45

Falling Weight Test. Same as Impact Sensitivity Test

Fl Test(*Figure of Insensitiveness Test*). The degree of sensitiveness exhibited by an expls detonated on mechanical shock is important because sensitiveness largely determines the precautions necessary in manufacturing, handling, and applications of the explosive. The difficulties sometimes attributed to determining sensitiveness by impact or drop-weight machines were reportedly overcome by an apparatus designed by Dr Rotter of the Research Dept, Woolwich, England. By this procedure reproducible accurate results were obtained, not by personal observation of sound or flash produced, but by a quantitative measurement of the gas produced at different heights of fall when the falling weight delivered sufficient energy to cause decompn of the expl

The results are compared to a standard expl, such as picric acid, gunpowder or mercury fulminate, and expressed as a ratio known as *figure of insensitiveness*. This ratio represents the relative energies of the impact required to produce explosions of equal degrees of completeness from initial decomposition to complete detonation. Picric Acid is taken as 100, and explosives giving higher numbers are less sensitive while those giving lower numbers are more sensitive than picric acid. *Ref:* R.Robertson, JCS 119 I,15(1921)

Fire Resistance or Fire Tests are described under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc

Flame Test(Length and Duration of Flame Determination) (Grösse und Dauer von Sprengstoff-Flammen Probe, in Ger) (Medición de la longitud y la duración de la llama, in Span). The test is based upon the belief that the greater the length of the flame an explosive emits and the longer the time during which that flame endures, the greater are the chances that such a flame when shot into the atmosphere of a coal mine will ignite inflammable or expl mixts of mine gas and air; of coal dust and air; or of mine gas, coal dust, and air

The flame-test apparatus used at the US BurMines Explosives Experiment Station at Bruceton, Pa consists essentially of a cannon in which an explosive is fired or detonated. The cannon, identical with that employed for the ballistic pendulum,is mounted vertically on a concrete foundation located in a dark building. By means of a photographic camera equipped with suitable devices to cut off all extraneous light rays, the flame is continuously observed such that its apex is in the field of view. The flame is recorded on a sensitized film wrapped about a drum that revolves at a predetermined rate of speed. The length of each flame is indicated by its height in the photograph, and the duration by the length of photograph

When testing detonating expls, cartridges 1¼" in diam are used, the wt of charge being 100±0.5 g, including the wrapper. When testing black blasting powder or other burning expls, the chge is tamped in the borehole and the igniter is imbedded centrally in the top of the charge. *Refs:* 1)US BurMinesBull 346, (1931),67 2)Stettbacher(1933),65–8 3)Reilly(1938),69 4)Vivas, Feigenspan & Ladreda 4 (1944),108–11 5)Pérez Ara (1945),125

Flash Point Test. See Ignition(or Explosion) Temperature Test, described in this section

Flash Test for Caps. See Optical Method for Testing Caps

Forty-Degree Test. See under Exudation Tests

Four-Cartridge Test is the Ger detonation by influence test. It is described in PATR 2510(PB 161270) (1958), p Ger 52

Fragmentation Test(Splitterprobe, in Ger). The fragmentation test consists of static functioning of the expl filler of a projectile, rocket, bomb or mine for the purpose of determining the number and weight grouping of the recovered fragments. The test gives a measure of the brisance and efficiency of an expl as well as the efficiency of the test item

Tests used abroad are briefly discussed in Refs 1,3,4&9, while the tests used in the US are described in Refs 2,5,6,7,8&9

There are four general types of fragmentation tests used by US Ord Dept: a)*Closed Chamber or Pit Test*, b)*Open Pit Test*, c)*Panel Test*, and d)*Velocity Measurement Test*. The Closed Chamber Test, as conducted at Picatinny Arsenal, is described in detail in Ref 7. The other three tests, as conducted at Aberdeen Proving Ground, are described in Ref 6. More information on fragmentation will be given in Vol III, under F *Refs:* 1)Stettbacher(1933),50–1 & 218–19 2)L.V.Clark,IEC 25, 1389–90(1933) 3)A.Majrich & F.Sorm, SS 30, 298–9(1935) 4)A.Stettbacher, Protar 8, 90(1942) 5)Ohart(1946),33 & 213 6)Ordnance Proof Manual OPM 40–23(1947) 7)PicArsn Testing Manual 5–1(1950) 8)TM 9–1910(1955),63–4 9)PATR 1740, Rev 1 (1958) 10)PATR 2510(PB 161270) (1958),p Ger 52

Fragment Density-, Fragment Concentration- or Density of Splinters Test(Splitterdichteprobe) is described in PATR 2510(1958),p Ger 52

Fragment Gun was originally developed by the British as a convenient instrument for imparting high velocity to controlled fragments in order to study their performance. The "gun" consists merely of a steel tube into which is inserted a flat slug, of any desired shape, cast in a Wood's metal matrix. A cylindrical charge of expl is inserted into the other end of the tube so that it fits smoothly against the disc of slug and Wood's metal surround. The charge is then detonated, from the end opposite to the slug, using a tetryl booster and suitable detonator. With a given type of tube and slug, the velocity imparted to the slug is a measure of a property of the expl closely related to *brisance*. The velocity is determined by sending the slug through three wire screens and determining the time intervals by means of a Mettegang recorder or other suitable device. *Refs:* 1)OSRD Rept 803(1942),27–8 2)OSRD Rept 804(1942),27–8

Fragment Velocity Measurement of statically detonated projectiles provides data for analysis of the effectiveness of projectile fillers and shell design. Evaluation of the lethality of fragments also depends upon the detn of fragment velocity

The basic technique for detg fragment velocity consists of firing a model shell (such as 3") filled with a test HE against a mild steel panel(such as ¼" and ⅜" thick) and photographing the fragments with a motion picture camera which also records elapsed time. By comparing the time with the distance traveled, an av velocity is obtained(Ref 2). In tests conducted at ERL, Bruceton, Pa, 18 expls were investigated using the "Bruceton Fragment Retardation Apparatus." Damage to steel panels was also detd. *Refs:* 1)OSRD Rept 5622(1946) 2)Ordnance Proof Manual OPM 80–16(1957)

Freezing Tests(for Dynamites). The freezing of expls, such as dynamites, leads as a rule to an increase in the rigidity and brittleness of the cartridges, as evidenced by cracks in the sample. The extent of the rigidity has been measured at the US BurMines by means of an apparatus called the "Crusher Board." This consists of a wooden base with a superimposed aluminum plate provided with a hole at each of its four corners by which it may be made to slide up or down on vertical brass guide rods which are attached to the corners of the wooden base. The sample of expl to be tested(a section of a cartridge, previously frozen at desired temp, 10 cm long cut from the center minus wrapper) is placed on its side in the central position of the wooden base and the aluminum plate is slipped over the guide rods so as to rest on the sample. Both ends of cartridge are observed for the cracks. If none appears in either end in 10 secs a 100 g weight is added to the center of the Al plate and if no cracks appear in 10 secs an addnl 100 g wt is added. These operations are continued until the first crack is seen on either end of the sample. The total wt on top of the plate plus the wt of the plate(911 g) is taken as a measure of the compressive strength

Expls that do not freeze when exposed to temps as low as 35°F(+1.67°) are called LF(low-freezing), those not freezing at 0°F(−17.78°) are ELF(extra low-freezing) and those not freezing at lower temps are NF(non-freezing). *Ref:* US BureauMinesBull 346, (1931),27–29

Frictional Impact Sensitivity Test. See under Friction Sensitivity Test

Friction Pendulum Test. See under Friction Sensitivity Tests

FRICTION SENSITIVITY TESTS(Reibungsempfindlichkeit Proben , in Ger) (Essais à la friction, in Fr) (Pruebas al sensibilidad al rozamiento, in Span). The tests may be divided into qualitative and quantitative types

A. Qualitative Friction Tests: a)*French Test No 1.* Place a gram of expl in a porcelain mortar and rub with a pestle. Report if it detonates or deflagrates(Refs 7 & 10) b)*French Test No 2.* Place a small sample on a tile or on an anvil and strike a *glancing blow* by means of a wooden hammer. Report the results(Refs 6 & 8) *Note:* Expls sensitive to these tests are considered as dangerous to handle and if used in mining should not be tamped even with a wooden tamper c)*German Test* of Imperial Railway Commission consisted of rubbing a small quantity of expl in a small unglazed mortar with an unglazed porcelain pestle(Refs 1,2 & 6) d)*British Tests of Dupré* are glancing blow tests. They are described in Ref 6, p 79

B. Quantitative Friction Tests. a)*Rathsburg Test* uses an apparatus which consists of two steel discs about ½" in diam with polished surfaces. The lower disc is stationary, while the upper rotates at 80 rpm. Loads ranging from 1 to 20 kg can be placed on the upper disc, thus maintaining it at various pressures. The explosive to be tested is mixed with finely pulverized sand and then a small portion is placed on the lower disc. A 20 kg load is placed on the upper disc, the disc is lowered to touch the sample and the rotation is started. If the sample does not explode after 20 revolutions, it is removed from the apparatus and a new portion is tested in the same manner. If no detonations occur after 6 trials, the expl is considered insensitive to friction. If any detonations are observed with a 20 kg load, the tests are repeated using successively smaller loads until the wt is reached with which no expln takes place The av number of revolutions required to detonate an expl when tested 6 times with the minim pressure is then recorded(Refs 5 & 11)

b)*US Bureau of Mines Pendulum Friction Apparatus* was devised in 1911 by C. E. Munroe, C.Hall & S.P. Howell and three models of different sizes were built at that time. The apparatus consists of a pendulum to the lower end of which is attached a 20 kg shoe, with an interchangeable face of steel or fiber. It is also possible to use other types of shoes, such as a wooden one with/or without carborundum cloth attached to its striking surface. The shoe is permitted to fall from a height of 1 m and to sweep back and forth on a steel anvil, the polished face of which is provided with three grooves ca ⅛" deep cut at right angles to the line of swing. The grooves are designed to prevent the sample of expl, spread upon the anvil for testing, from being brushed off the anvil by movement of the shoe across it. The pendulum is adjusted, before placing the sample on the anvil, to swing 18±1 times before coming to rest

With a steel shoe raised to the height of 1 m, spread a 7 g sample of the explosive evenly in and about the grooved portion of the anvil, and allow the shoe to strike the sample with glancing blows until it comes to rest. Clean the anvil and shoe, place another portion of the same expl and continue the test Repeat test a total of 10 times and report the number of snaps, cracklings, ignitions, and/or explosions. If the sample detonates after 1 or 2 trials, discontinue the test in order to prevent damage to the apparatus. Such an explosive is considered as not passing the permissibility test. If the expl remains unaffected in 10 trials with the

steel shoe, it is considered to pass the test. If some trials with the steel shoe produce burnings or cracklings, repeat the test using the hard fiber shoe. If in 10 trials with this shoe there is no more unfavorable result than an almost undistinguishable local crackling, the expl is considered as passing the test for permissibility(Refs 3,6,8,12,13,14 & 15) c)*Laboratory Model of Pendulum Friction Apparatus.* As the regular BurMines apparatus is expensive and requires large samples of expls. Taylor & Rinkenbach used a smaller model(ca ¼ the size of the B of M app). The shoe weighed 74 g and samples 0.005 to 0.05 g. One of these models is at the B of M Testing Station and another at PicArsn. This small model is considered particularly suitable for testing iniating expls(Ref 4) d)*Impact-Friction Pendulum*, used by the Spencer Chemical Co(Ref 16) consists of a hammer with a 9 ft handle and a 400 lb head. The hammer delivers energy to a sample of explosive by falling a specified distance before striking the sample. Variation in energy delivered is achieved by adjusting the height from which the hammer is released. The hammer is drawn to this height in an arc, by an electric winch and released from a distance by a lanyard connected to a trigger e)*Rifle-Bullet Sensitivity Test* is described in Ref 15, p 49 as one of the tests for "Sensitivity to Frictional Impact''. *Refs:* 1)Marshall 2, (1917),423 & 437 2)Barnett(1919),216 3)US BurMines Tech Paper 234, (1919) 4)C.A.Taylor & W.H.Rinkenbach, JFrank Inst 204, 369(1927) 5)H.Rathsburg, ZAngewChem 41, 1284(1928) 6)US BurMinesBull 346, (1931),79–84 7)Vennin, Burlot & Lécorché(1932),212 8)Marshall 3, (1932),125 9)Stettbacher(1933),370–1 10)Pepin Lehalleur(1935),75–6 11)R.Wallbaum-Wittenberg, SS 34, 162–3(1939) 12)OSRD Repts 803 & 804(1942),16 13)Pérez Ara(1945),107 14)PicArsn Testing Manual 7–1(1950) 15)TM 9–1910(1955),47–49 16)Spencer Chemical Co, "Safety Data,'' Kansas City, Mo(1960)

Fuse Test is one of the Fire Resistance Tests described under Sensitivity to Flame, Heat, Sparks, etc Tests

Galleries for Testing Permissible Explosives (Galeries d'essai, in Fr) (Schlagwetter-Versuchstrecken, in Ger) are described in the following *Refs:* 1)Marshall 2, (1917),585–95 2)Barnett(1919) 3)US BurMines Bull 346, (1931),49 4)Vennin, Burlot & Lécorché(1932),235–43 5)Stettbacher(1933),248–52 6)PATR 2510(1958) (PB 161270),p Ger 215(Versuchsstrecke Dortmund-Derne)

Gap Test is one of the *detonation by influence(sympathetic detonation)* tests. The purpose of this test is to determine the sensitivity of a charge of expl to initiation by another charge located a certain distance from the 1st chge. The larger the distance, the more sensitive is the expl

In the test used in France, two cartridges 30 mm diam, each weighing 50 g, are placed upon two lead plates supported on two vertical steel cylinders. The cartridges are placed a known distance apart with axis coinciding(in line), and then one of the cartridges is detonated. After finding the max distance at which there are 3 successive detonations by influence of the 2nd cartridge, the min distance is detd at which there are 3 successive failures. The mean of the two distances is designated as *CSE*(coefficient de self-excitation) Refs 1, 3 & 4)

The US BurMines uses the following test, known as the *Halved-Cartridge Gap Method:* By means of a wooden device(such as a skewer), punch a cavity in the center of one end of a 1¼" diam cartridge to receive a No 6 electric detonator, which will be inserted when the operator is ready to fire. Cut the cartridge at right angles to its axis so that the column of expl at the end of the cartridge in which the cavity has been punched is 4" long. If the expl runs freely, place over the cuts small pieces of thin paper and fasten them in place with rubber bands. With the two cut ends facing each other, space the two halves of the cartridge the required distance apart by rolling them on a flat surface in a piece of manila paper 0.005 to 0.0055" thick, cut to such a length that each gap mark is 4" from the end of the paper and to such a width that it will wrap exactly 3 times around the cartridges. Hold the tube thus formed in place by means of carpet tacks and bring the temp of sample to 22±5°C. Insert the detonator and fire it

The greatest distance between the halves at which both of them detonate in four shots is termed the "sensitiveness'' and is expressed in cms; the min distance at which no explosion occurs in four trials is also detd. Finally, by four trials at each intermediate distance, the number of "explosions'' and "no explosions'' that occur is noted and recorded

In the *gap test* described in Ref 5, p 68, cylindrical cartridges of expl 8" in length and 1.25" in diam are prepd by pressing or casting equal wts of the expl into paper shells. Two of these are suspended vertically so that there is an air space between them and their axial lines are coincident. The space between the faces of cartridges is always a multiple of 1". A detonator is embedded axially in the lower end of the lower cartridge and used to initiate detonation in this cartridge. By repeated tests with varying air spaces, there is detd the max distance at which the upper cartridge can be detonated by the lower. This will be 1" less than the min distance at which three successive tests fail to detonate the upper cartridge

The interposition of solid barriers such as wood or concrete decreases the max distance for sympathetic deton and this effect is very significant when small charges are involved (Ref 5)

Application of the gap test to detn of efficiency of detonators is described by Clark as one of the "Propagation Tests" (Ref 2a). For this test a 0.50g chge of DADNPh (diazodinitrophenol) is placed in a No 8 detonator shell and pressed under a reënforcing capsule at 3400 psi. In a similar manner detonators contg 0.50 and 1.00 g chges of MF(mercuric fulminate) are prepd. The test detonator is placed centrally in a cylindrical oaken shield, with its long axis parallel to and coinciding with the long axis of the shield

and with its base flush with the end of the shield. A cartridge of 40% straight dynamite with its cut end facing the detonator across an air gap of known length, is wrapped together with the detonator in three turns of heavy paper. The max gap over which detonation can be transferred with certainty from the detonator to cartridge of dynamite is detd by four trials. (See also Booster Sensitivity Test and Wax-Gap Test). *Refs:* 1)E.Burlot, MAF **9**, 799(1930) 2)USBurMinesBull **346** (1931), 59 2a)L.V.Clark, IEC **25**, 668 (1933) 3) M.Dutour, MP **31**, 74 (1949) 4)L.Médard, MP **33**, 342-4(1951) 5)TM 9-1910(1955),67-8

132° German Test (Erhitzungsprüfung bei 132°, in Ger). This test, originally designed to be conducted at 135° to determine the stability of NC and propellants, was used to a considerable extent in Germany and in other countries. A 2.5g sample of NC or of smokeless propellant is placed in a test tube 350 mm long, 16 mm ID and 19 mm OD. A strip of blue litmus paper is inserted so that it is 25 mm above the expl. The tube is loosely closed with a cork, and inserted in one of the orifices in the cover of the bath contg boiling xylene and provided with a reflux condenser. The orifices are in the form of tubes 11 cm long, closed at the bottom and contg glycerin. The time of heating required to turn the litmus paper red is taken as an indication of stability. Then heating is continued until the appearance of brown fumes and may be further continued until explosion occurs. *Refs:* 1)Reilly(1938), 82 2)Kast-Metz(1944), 233-4

Granulation Test. The purpose of this test is to detemine particle-size distribution. For this superimpose the required number and sizes of US Standard Sieves as required by the specification, in the order of decreasing size, placing the largest mesh on top and a receiving pan at the bottom. Place a 50 g portion of the sample on the top sieve,cover it, and shake mechanically (at 300±15 gyrations and 150±10 taps) or by hand, for 5 mins. Weigh the material retained on each sieve and calculate the percentage of the sample through each sieve and, if required, that retained on any sieve *Ref:* Spec MIL-STD-286(1956), Method No 506.1

Grotta's Test for Detonators, described by B.Grotta, IEC **17**, 134-8(1925) consisted of firing the charges of an insens expl(such as a mixt of TNT 92 with iron oxide 8%), placed on a lead plate 1.5 × 1.5" and 0.25" thick, with various strength detonators and observing the damage caused to the plate. It was found that the so-called "Reënforced Booster Type Detonators" (compd detonators contg MF,HgN_3+TNT) produced complete detonations in 100% of tests, while simple detonators (contg only MF) gave 90% of misfires (Compare with Esop's Test and Miniature Cartridge Test)

Halved Cartridge Gap Test. See under Gap Test

Heat of Combustion (Q_c), **Heat of Explosion** (Q_e) **and Heat of Formation** (Q_f) will be discussed under Calorimetric Tests in Vol II.

Heat Tests (Thermal Stability Tests) include among others the following: 1)Abels' or KI Test (qv) 2) American Test at 65.5° or 80° (See Ref 2 p 80 and under Surveillance Tests in this section) 3)Angeli's Test (See Ref 2, p 90 and p A403 of this volume) 4)Bergmann-Junk Test (qv) 5)Brame's Test (Ref 2, p 88) 6)Brunswig's Test (Ref 2, p 86) 7)Chiaraviglio & Corbino's (Ref 2, p 88) 8)Conductivity Method (Ref 2, p 91) 9)Continuous Stability Test (Ref 2, p 70) 10)Desmaroux Test (Ref 2, p 90) 11)Dupré's Vacuum Test (Ref 2, p 87) 12)Dutch Test (Ref 2, p 85) 13)German 132° Test (qv) 14)Guttmann's Test (Ref 2, p 78) 15)Haid, Becker & Dittmar's Test (Ref 2, p 92) 16)Heat Tests at 100°, 120° & 134.5° (qv) (Compare with German 132° Test) 17)Hess' Test (Ref 2, p 78) 18)Hoitsema's Test (Ref 2, p 78) 19)Horn-Seifert's Test (Ref 2, p 79) 20)International 75° Test (qv) 21)Jensen's Test (Ref 2, p 80) 22)Marqueyrol's Test (Ref 2, p 90) 23)Meerscheidt-Hüllessem's Tests (Ref 2, pp 85 & 89) 24)Methyl Violet Test (Ref 2, p 79) 25) Mittasch's Method (Ref 2, p 87) 26)Moir's Test (Ref 1, p 224) 27)Obermüller's Method (Ref 2, p 87) 28) p H Measurements (Acidity Measurements) are made on a 5g sample of expl after heating from 75° to 132° according to the nature of the expl, and the change in pH is noted 29)Pollard's Test (Ref 2, p 80) 30) Resistance to Heat Test (qv) 31)Silvered Vessel Test (qv) 32)Simon Thomas' Test (Waltham Abbey Test) (Ref 1, p 225 & Ref 2, p 80) 33)Spica's Test (Ref 2, p 78) 34)Surveillance Tests at 65° or 80° (qv) 35) Sy's Test (Ref 2, p 85) 3)Talliani Test (qv) 37)Taylor's Test (Ref 2, p 82) 38)Tomonari's Test (Ref 2, p 91) 39)Vacuum Stability Tests (qv) 40)Vielle's Test (Ref 1, p 224 & Ref 2, p 78) 41)Warm-lagermethode 75° (Ref 2, p 81) 42)Will's Test (Ref 1, p 225 & Ref 2, p 86) 43)Zinc Iodide Test (Ref 2, p 77) *Refs:* 1)Barnett (1919), 217-225 2)Reilly (1938), 70-93 3)PATR 1401, Rev 1 (1950), 12-18

100° Heat Test is one of the US standard stability tests. Transfer four weighed 0.60g portions of sample of known moisture content to each of four test tubes, 75 mm long and 10 mm diam, two of which have been tared. Place all tubes in an oven maintained at 100±1°. After 48 hrs remove two tared tubes, cool in a desiccator and weigh each tube. Replace the tubes in the oven and heat for the 2nd 48 hr period. Cool in a desiccator and weigh. Calculate the percentage loss in wt during each 48-hr period of heating and subtract the known percentage of moisture from wt lost during the 1st 48 hr period. Allow the untared test tubes to remain in the oven for 100 hrs of continuous heating and note if ignition or expln takes place. *Refs:* 1)PATR 1401, Rev 1 (1950), 13 2)TM 9-1910 (1955), 55-6

120° and 134.5° Heat Tests. The 120° test is used for testing the stability of HE's & double-base propellants, while the 134.5° test is used for single-base propellants and for nitrocellulose. In either case, weigh five 2.5g portions of the sample and place each in a heavy Pyrex test tube, 15 mm ID, 18 mm OD and 290 mm long. Insert a piece of std, normal methyl violet paper 70 mm long and 20 mm wide, vertically in each tube so that

the lower edge of paper is 25 mm above the sample. Stopper each tube with a cork through which a hole 4 mm in diam has been bored to prevent build-up of pressure inside the tube. Place the tubes in the appropriate const temp bath at 120.0±0.5° or 134.5±0.5°, which is so designed that no more than 7 mm of the tube projects above the cover. The bath is in the form of a cylindrical tube, provided with a perforated cover and reflux condenser. The bath is filled with aq glycerin d ca 1.21 for 120° and d 1.24 for 134.5° bath. Examine at 5 mins intervals each tube by lifting one-half of its length and replacing quickly. Record the time at which the test paper in any of the 5 tubes changes to a *pink salmon color.* Continue heating until any of the tubes become filled with *red fumes.* Heating may be continued further to det whether sample in any of the tubes *explodes*. *Refs:* 1)PATR 1401, Rev 1 (1950), 16-17 2)TM 9-1910(1955), 243 & 245

Hemispherical Iron-Dish Test is one of the Fire Resistance Tests described under Sensitivity to Flame, Heat, Sparks, etc Tests

Hess' Brisance Test. See under Brisance (Shattering Effect) in Vol II

High Speed Optical Devices Used for Measuring Detonation Rates are mentioned under Detonation Rate Determination. *Ref:* Cook (1958), 22-35

High-Speed Radiography may be subdivided into: 1)Flash Radiography and 2)Continuous (or Streak) Radiography. The 1st has been very useful in studing the behavior of the collapsing liner and the jets from shaped charges, while the 2nd has been used in the study of solid explosives. *Ref:* Cook(1958), 35-6

Hollow Charge Efficiency Test. See Shaped Charge Efficiency Test

Hopkinson's Pressure Bar Test. The quantitative measurement of the press developed by expls over small intervals of time, which is a measure of expl violence, was made possible by application of the method conceived by Prof B. Hopkinson. The application of Hopkinson's principle to a wide field of research; such as initiation of deton, properties of the deton wave, and the design of detonators & fuses; and the design of a variety of instruments were carried out by physicists of the Res Dept, Woolwich, England

The principle on which the determination of press is based depends on the fact that when a charge is fired against the end of a cylindrical steel bar ballistically suspended, a wave of compression travels along the bar and is reflected at the far end as a tension wave. In order to investigate the properties of the wave, a short length of the bar farthest from the charge is cut off, the ends are surfaced, and the pieces are joined by a film of vaseline. The compression wave travels unchanged through the joint into the short bar (known as the time-piece), but the film is unable to transmit the tension wave. Hence, when the amplitude of the reflected tension wave reaching the joint becomes greater than that of the original compression wave, the time-piece is projected with a momentum which depends on the expl press developed and the time to traverse the short bar. By the use of time-pieces of different lengths, it is possible to approximate the maximum pressure developed, and to calculate mean pressure values over various time intervals. In order to protect the instrument it is necessary to interpose a pellet of standardized material between the test expl and the pressure bar. *Refs:* 1)B. Hopkinson, PhilTrans **213A**, 437(1914) 2)R.Robertson, JCS **119** I, 19-24(1921) 3)J.L. Sherrick, ArOrdn **24**, 330(1924) 4)Marshall **3** (1932), 155-7

Humidity Test. See Hygroscopicity Test

Hygroscopicity (or Humidity) Test (Absorption of Moisture Test) (Reprise d'humidité, Essai, in Fr) (Hygroskopizität- or Feuchtigkeit Probe, in Ger). The hygr nature or property of a material to absorb moisture from its environment must be known if an expl is to be considered for military or commercial use. Absorption of moisture can have an adverse effect on the sensitivity, stability, or reactivity of expls. This property should be negligible to very low absorption for most expls

In one test procedure a weighed sample of known granulation, if solid, is exposed to predetermined conditions of temp and humidity until equilibrium is attained. In cases where either the rate of absorption is very low, or large amounts of water are picked up, the sample is exposed for a stated time, for example, 24 hrs. The exposed sample is then reweighed and the moisture absorbed is expressed as % hygroscopicity

French hygroscopicity tests, known as: a)Reprise d'humidité de l'explosif en vrac (Taking up moisture by an explosive in bulk) and b)Reprise d'humidité en atmosphère saturée d'eau des explosifs encartouchés (Taking up moisture by cartridged explosives in atmosphere saturated with water) are described in Ref 4 *Refs:* 1)Marshall **2** (1917), 416-9 2)Barnett(1919), 214 3)Davis(1943), 313 4)L.Médard, MP **33**, 325-7 (1951) 5)TM 9-1910(1955), 10-2 6)PATR 1740, Rev 1(1958)

Ignition (or Explosion)Temperature Test; Ignition Point Test *(Deflagration Temperature or Flash Point Test)* (Entzündungstemperatur; Explosionstemperatur or Entzündungspunkt Probe, also called Verpuffungstemperatur Probe, in Ger); (Essai de déflagration, in Fr). Heat causes the decompn of all expls at a rate which varies with the temp. Almost all expls have a *critical temperature* below which the rate of decompn is small to negligible. One measure of the relative sensitivity of expls to heat is detd by means of the ignition or explosion temperature test

The procedure, in one test, is to immerse to a fixed depth in a bath of Woods' metal, a metal blasting cap containing 0.02 g expl sample. The molten bath is maintained at a controlled temp by means of an electric furnace. A number of tests is made with the bath at various temps so as to produce flashes or explosions over a range of 2 to 10 seconds. The data so obtained are plotted as a time-temp curve and from this curve is found the temp to cause ignition or explosion in 5 seconds (Refs 5,7,8,9,10&11)

Another method is to place an expl sample directly on the molten Wood's metal bath or other metal surface heated at a controlled temp. The temp of the metal surface is increased until a value of 0.1 sec for ignition or explosion is estimated by the observer as an almost instantaneous interval of time(Refs 9,10 & 11). Other methods are described in Refs 1,2,3,3a & 4). *Refs:* 1)Marshall 2, (1917),435–7 2)Barnett (1919),213 3)M.M.Kostevitch, SS 23, 156(1928) 3a)Vennin, Burlot & Lécorché(1932),211 4)Stettbacher (1933),373 5)L.V.Clark, IEC 25, 668 & 1389(1933) 6)Reilly(1938),66 & 83 7)Davis(1943),21 8)OSRD Rept No 1986(1943) 9)PATR 1401, Rev 1(1950), 10)TM 9–1910(1955),50 11)PATR 1740, Rev 1(1958)

Ignition(or Explosion) Time Tests(at Constant Temperatures). In some cases the explosive is maintained at const temp and the time to explosion is measured. Same type of apparatus can be used as for Ignition Temperature Test. Patterson(Refs 1&3) detd the relationship between time to ignition and temp and showed that the lower the temp the longer was the time reqd for ign of propellants. Wiggam & Goodyear(Ref 2) have made a critical study of the explosion–time test. *Refs:* 1)G.W.Patterson, SS 5, 49(1910) 2)D.R. Wiggam & E.S.Goodyear, IEC,AnalEd 4, 77(1932) 3)Reilly(1938),83

Impact-Friction Pendulum Test is briefly described, p A354, Note d), under Ammonium Nitrate *Ref:* Spencer Chemical Co, "Safety Data," Kansas City, Mo(1960)

Impact Sensitivity or Shock Sensitivity Test *(Drop Weight or Falling Weight Test)* (Sensibilité au choc du mouton, in Fr) (Stossempfindlichkeit or Fallhammer-Probe, in Ger) (Prueba al choque or Prueba de la caída de un peso, in Span) (Determinazione della sensibilità all'urto or Saggio alla Berta, in Ital). This test was designed to determine the sensitivity(resistance) of expls to shock(impact). The information obtained by this test is considered most valuable, as it gives assurance of safety of handling, transportation and use

Essentially, the test consists of placing a small amt of expl on a surface of a stationary steel block(anvil) and then striking the sample by allowing a certain load(called hammer) to drop on it from a designated height. The max height that a substance can withstand without exploding or deflagrating is considered the impact value. The greater the fall of the hammer of the same wt, the less sensitive to impact is the expl

It seems that one of the first successful impact machines was constructed in Germany by *F. Lenze*, who described it in Ref 1. This apparatus is also briefly discussed by Marshall(Ref 2). Stettbacher(Ref 8) p372) gives a brief description of the apparatus called *Fallhammer nach Kast*(See also Ref 11)

The US Bureau of Mines constructed several models, small and large(Refs 5,13&15), which seem to be similar to the German machines. With the *Bureau of Mines Small Impact Apparatus*, tests are conducted as described in Refs 5,13 & 16. One of such machines is installed at Picatinny Arsenal. The max wt of the hammer is 2 kg and the maximum height is 100 cm. This app uses unconfined samples of expl, each weighing 0.02 g. This machine gives consistent results for fairly sensitive expls, but for expls such as TNT, the results are not very reliable. *Note:* Considerable efforts were made during WWII to place impact testing on a more fundamental basis both from the suitability of the apparatus used to give reproducibility and the reliability and mathematical interpretation of results(See Refs 10,13 & 17)

More consistent results than some obtained with the B of M machines are obtd with an apparatus used for many years at Picatinny Arsenal(Refs 4,13,15 & 16). With this machine, known as *Picatinny Arsenal Impact Apparatus*, samples are tested under confinement and results are reported in inches. The usual weight hammer is 2 kg for HE's and smaller wts of 1 lb or 8 oz for initiating expls. For a description of apparatus and procedure, see Refs 13,15 & 16

Both the US B of M and PA Impact Machines have been used at Picatinny Arsenal for the testing of liquid expls, with a modification in sample handling required only in the case of the BM apparatus(Refs 12a & 16)

Dr Rotter of the Research Dept, Woolwich, England, designed an apparatus and a method of testing(Refs 3 & 6), which is claimed to be more accurate than other known methods. This is now an official British test and the value obtained is known as *FI(Figure of Insensitiveness)* (qv)

The French tests, known as *essais au choc du mouton*(described in Refs 7,9 & 14) are subdivided into: a)*essai au choc du petit mouton*, which uses small wts, such as 2 kg and b)*essai au gros mouton*, which uses large wts, such as 30 kg. A detailed description of these tests is given in Ref 14. *Refs:* 1)F.Lenze, SS 1, 287–93(1906) 2)Marshall 2 (1917),423–4 3)R.Robertson, JCS 119, 16–18(1921) 4)H.S.Deck, Army Ordn 7, 33–7(1926) 5)US BurMinesBull 346, (1931),71–8 6)Marshall 3, (1932),127 7)Vennin, Burlot & Lécorché(1932),213–15 8)Stettbacher(1933),371–3 9)H.Muraour, MAF 12, 560–1(1933) 10)OSRD Repts 803 & 804,(1942),3–14 10a)Vivas, Feigenspan & Ladreda 4, (1944),105–7 11)Pérez Ara(1945),104–7 12)H.Muraour, "Poudres et Explosifs," Paris(1947),81–3 12a)PATR 1738(1949) 13)PATR 1401, Rev 1 (1950),2–6 14)L. Médard, MP 33, 330–4(1951) 14a)Belgrano(1952),49–51 15)TM 9–1910(1955),43–7 16)PATR 1740, Rev 1(1958) 17)Cook(1958),38–40 & 332–4

Index of Inflammability. This is a measure of the likelihood that a bare charge will catch fire when exposed to flames. The test is made by bringing an oxyhydrogen flame in contact with the sample. The maximum time of exposure which gives no ignition in 10 trials and the minimum exposure which gives ignition in each of 10 trials are determined. The index of inflammability is 100 divided by the mean of the two times in seconds. The most inflammable substances have high indices, such as 2.50. (See also Sensitivity to Flame, Heat, Sparks, etc Tests). *Ref:* PATR 1740, Rev 1(1958)

Inflammability Test. See Index of Inflammability Test and under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc

Influence Tests. See Detonation by Influence Tests

Initial Velocity(Muzzle Velocity) Determination. See under Ballistics, External in Vol II

Initiating Efficiency (or Strength) of Detonators by the Miniature Cartridge Test. This test developed at the US BurMines, consists of loading, at a const packing density, a 5 g charge of an insensitive expl(such as a homogeneous *TNT-Iron Oxide Mixture*) into a paper cartridge ½" ID and 2¾" long. After inserting the detonator to test into the cartridge, the ensemble is fired in the center of 1000 g of Ottawa std sand placed in a steel bomb of 3" ID. The crushed sand which passes through a No 30 US Std Sieve(see Table I, p A674) is weighed and from this is subtracted the value for detonator alone(blank), which is obtained by similarly firing a miniature cartridge contg 5 g of pure iron oxide and the same type of detonator. The difference in crushed sand thus derived represents the initiating efficiency of the detonator. *Ref:* US BurMinesTechPaper 677(1945)

INITIATING EFFICIENCY(INITIATING VALUE OR STRENGTH) OF INITIATING EXPLOSIVES, BLASTING CAPS AND DETONATORS, DETERMINATIONS(Essais des amorces, in Fr) (Grenzinizialen, in Ger). Initiating efficiency(strength or value) can be expressed in terms of min wt of primary(or initiating) expl or in smallest No of blasting cap or detonator required to cause max detonation of a HE. This can be detd by one of the following methods: a)*Esop's Test*(qv) b)*Gap Test*(qv) c)*Grotta's Test*(qv) d)*Lead Plate Test* (see under Plate Tests) e)*Miniature Cartridge Test*(see previous item) f)*Nail Test*(qv) g)*Optical Method*(qv) h)*Sand Test*(see next item) i)*Small Lead Block Compression Test*(see Esop's Test) j)*Small Lead Block Expansion Test*(see under Trauzl Tests) k)*Sound Test*(qv) (Compare with *Sensitivity to Initiation by Initiating Explosives, Detonators and Boosters Tests*). *Refs:* 1)Marshall 2, (1917),530–2 2)H.Kast & A.Haid, SS 19, 146 & 165(1924) 3)L.Wöhler, SS 20, 145 & 165(1925); SS 21, 1, 35, 55, 97 & 121(1926) 4)W.Friederich & P.Vervoorst, SS 21, 51(1926) 5)L.Wöhler et al, SS 22, 95(1927) 6)B. Cserneczky, SS 24, 169–72(1929) 7)A.Haid & H.Koenen, SS 25, 393, 433 & 463(1930) 8)Marshall 3, (1932),163–4 9)Stettbacher(1933), 361 10 Reilly(1938),69–70 11)Pérez Ara(1945),121–3 12)PATR 1401, Rev 1(1950),12 13)TM 1910(1955),64(Initiating Value)

Initiating Efficiency(or Strength) of Primary Explosives by Sand Test. Using 0.400 g charges of tetryl and 0.400 g of the initiating explosive under test, det the wts of sand crushed by initiator alone and by initiator + tetryl. Subtract from the last value, the amt crushed by 0.400 g of initiator and record this as the value crushed by tetryl. Repeat the test with initiator + tetryl a total of 5 times and compare the results. If the values for tetryl do not vary by more than 3.0 g and the av of these is within 2.0 g of the av value for tetryl obtained with 0.300 g of LA as an initiator, consider it as the maximum. Repeat the test using smaller amts (0.350 g, 0.300 g etc) of initiator(and always 0.400 of tetryl) until the minimum is reached. (Compare with Sensitiveness to Initiation by Detonators, etc). *Refs:* 1)L.V.Clark, IEC 25, 666(1933) 2)PATR 1401, Rev 1(1950),12

Initiating Power. Same as Initiating Efficiency

Initiating Strength. Same as Initiating Efficiency

Initiating Value. Same as Initiating Efficiency

Initiation Sensitivity by Electrostatic Discharges or by Sparks. See under Sensitivity to Flame, Heat, Sparks and Electrostatic Discharges

International 75° Test is one of the common stability tests for expls. Place two samples of 10 g each in tared weighing bottles 35 mm diam and 50 mm high, cover them and weigh. Heat the loosely covered bottles for 2 hrs at 75°, cool in a desiccator and reweigh. Calculate the loss of wt as % of volatility of sample. Note if the material undergoes decompn or is markedly volatile as indicated by discoloration, the appearance of brownish fumes, etc. *Refs:* 1)Reilly(1938),80 2)PATR 1401, Rev 1(1950),13 3)TM 9–1910(1955),55

Iron Oxide-TNT Test for Detonators. See Grotta's Test and the Initiating Efficiency of Detonators by the Miniature Cartridge Test

Kast Brisance Meter is an apparatus for measuring the brisance by compression(crushing) of a copper cylinder. It will be discussed in Vol II, under Brisance Meter of Kast

Kast Value or Brisance Value of Kast will be discussed in Vol II, under Brisance Value of Kast

KI-Heat Tests or KI-Starch Tests. See Abel Test, p A2 of this volume

Kraftzahl(KZ) Probe(Strength Number Test) is a German modification of Trauzl Test. *Ref:* PATR 2510 (PB 161270) (1958), p Ger 102

Lead Block(or Cylinder) Compression(or Crushing) Test(Lead Block Test or Hess Test) (Stauchprobe nach Hess, in Ger) (Épreuve au block de plomb, in Fr) is one of the German tests for brisance, also called percussive force. It will be described in Vol II, under Compression Tests

Lead Block Expansion Test. See Trauzl Test

Lead Plate Test. See under Plate Tests

Length and Duration of Flame Test(Grösse und Dauer von Sprengstoff-Flammen Probe, in Ger). See Flame Test

Maximum Available Work Potential is discussed in Cook(1958),36–7

Maximum Potential Work is the same as Brisance Value of Kast

Maximum Pressure of Explosion(Maximum Pressure of Gases Developed on Explosion) (Maximale Explosionsdruck or Gasdruck, in Ger). See under Pressure of Explosion

Mechanical Shock may be of two types: shock due to friction and shock due to impact. These tests are described under Friction Tests and under Impact Tests. *Ref:* Barnett(1919),208

Miniature Cartridge Test. See Initiating Efficiency of Detonators by the Miniature Cartridge Test

Miniature Charge Techniques for detn of detonation velocity is briefly discussed in Cook(1958),41–2

Mortar Test(Épreuve de tir au mortier or Essai au mortier éprouvette, in Fr) (Mörserprobe, in Ger). A device used both in England and France essentially consists of a large cast-iron, solid cylindrical block securely fixed on a concrete foundation and set with its axis at elevation of 45°. The upper face of the block contains a cylindrical bore, 125 mm diam and 85 mm deep, and on the bottom of this bore is a smaller bore serving as a receptacle for 10 g charge of expl to test. A 15 kg cylindrical cast-iron shot 123 mm diam and 125 mm high, perforated in the center to allow the passage of fuse, is inserted in the bore above the charge of expl and the fuse is ignited. Instead of a fuse an elec detonator with wires passing through the perforation in the shot can be used. The distance that the shot is thrown is measured and compared with that obtained with 10 g of a std expl of the same nature. With this device blasting gelatine gave 240 meters, gelatine dynamite 188 and Brit permitted expls 80 to 120 m(Ref 1)

Same method as in France has been used in Spain and the device is called *mortero probeta*(Refs 3 & 4). According to Ref 3, the test with morter permits calculation the *travail utile de l' explosif*(useful work of the explosive). This is called in Ref 4 *medida del potencial o efecto útil de un explosivo*

Vennin et al(Ref 2) do not recommend this test for brisant expls. (Compare with Ballistic Mortar and with Ballistic Pendulum Tests). *Refs:* 1)Barnett(1919),181–2 2)Vennin, Burlot & Lécorché(1932),189 3)Pepin Lehalleur(1935),66–7 4)Vivas, Feigenspan & Ladreda, Vol 4(1944),116–17 5)Pérez Ara(1945),120

Munroe-Neumann Effect Test. See Shaped Charge Efficiency Test

Muzzle Velocity(Mündungsgeschwindigkeit, in Ger) or **Initial Velocity Determination** will be discussed in Vol II under Ballistics, External

Nail Test(Essai au clou, in Fr) (Nadel Probe, in Ger) (Prueba de la puntilla, in Span). A simple, cheap, and accurate test to determine the relative efficiency of detonators and one suitable for use in the field is called the nail test. In this test a wire nail is fastened to the side of a detonator suspended horizontally in the air and the detonator is fired. The angle to which the nail is bent is measured to the nearest 0.25° and the average of five tests is the computed result. Four-inch wire finishing nails of approximately the same length, gage and weight are used in the test. *Refs:* 1)US BurMinesBull 59, (1913),25 2)US BurMinesBull 346, (1931),113 3)Stettbacher(1933),354 4)Davis(1943),421 5)Pérez Ara(1945),123

Normal(or Specific) Volume. See under Volume of Gases Evolved on Detonation(or Explosion)

Optical Method for Testing Caps consists in photographing the flashes produced on explosion. (Compare with Flame Test). *Refs:* 1)H.E.Brownsdon, JSCI 24, 381(1905) 2)W.D.Borland, JSCI 25, 241(1906) 3)Reilly (1938),70

Pendulum Friction Device Test. See under Friction Sensitivity Tests

Percussive Force of an Explosive can be defined as the capacity of an explosive to produce compression or disruption when it explodes under atmospheric confinement only. Percussive force is manifest only in HE's and can be measured by detonating unconfined expls on top of a steel plate covering a small lead block. The compression of block is approx proportional to propulsive force(Ref 1). According to Refs 2 & 3, the "relative percussive force" is identical with "relative brisance." *Refs:* 1)US BurMinesBull 346, (1931), 106–7 2)Vennin, Burlot & Lécorché(1932),190 3)L.V.Clark, IEC 25, 1389(1933)

Permissibility Tests in Galleries are described in Refs listed under Galleries For Testing Permissible Explosives. The US BurMines Tests are described in Bull 346, (1931),49–59 and other publications of the B of M

Plate Denting Tests, although not actually involving shattering by expls, are used as measures of *brisance.* In these tests the effect of a cylinder of expl when detonated in contact with a *steel plate,* is detd under such conditions that the more powerful expls depress and dent but never puncture or shatter the plate, while less powerful expls merely dent or bend it

Plate denting tests used at ERL, Bruceton, Pa have been conducted by two methods:

Method A. A 20 g charge of expl is cast or pressed in a copper cylinder of ¾" ID and $^{1}/_{16}$" wall. The loaded tube is placed vertically on a 4" square piece of cold-rolled steel plate, $^{5}/_{8}$" thick, supported by a short length of heavy steel tubing, placed in a vertical position. The expl charge is boostered by a 5 g pellet of tetryl which in turn is initiated by a No 8 detonator(Refs 1,3 & 4)

Method B. A modification of Method A involves firing a $1^{5}/_{8}$" diam by 5" long *uncased* charge of expl on a lightly greased 1¾" thick, 5" square cold-rolled steel plate, with one or more of similar plates used as backing. The charge is initiated with a No 8 detonator and two $1^{5}/_{8}$" diam, 30 g tetryl pellets as boosters(Refs 2 & 4)

The depth of dent in both test methods is measured within 0.001 to 0.002" and a measure of the *relative brisance* or *plate denting value*, X, is calculated from the formula

$$X = \frac{\text{Test Sample Dent Depth}}{\text{Dent Depth for TNT}} \times 100$$

Data obtd by Method A are summarized in Refs 1,2,3 & 4and data obtd by Method B are given in Refs 2 & 4. *Refs:* 1)OSRD Rept 804(1942),29–31 2)OSRD Rept 5746(1945),20–2 3)TM 9–1910(1955),61–2 4)PATR 1740, Rev 1(1958)

Plate Tests include *Plate Cutting and Plate Denting Tests*(Plattenproben or Durchschlags- und Strahlungs-Proben, in Ger) and use brass, copper, iron, lead and steel as materials for plates. The tests are used either for detn of *brisance* of expls or for detn of *efficiency of detonators*. Essentially the tests consist of detonating a test item in the center of a plate supported at both ends. This may either dent the plate or cut it(puncture), and these damages serve as criteria of brisance or efficiency. When testing a detonator on a lead plate the strength is judged not only from the size of dent or hole made, but even more from the number and depth of the striations on the surface of the lead made by the minute particles of metal(such as copper) from the capsule of detonator (Ref 1a) (See also Refs 2a & 6)

The Steel Plate Denting Tests used in the US are described above, while other plate tests are discussed in the following references: *Refs:* 1)Marshall 2, (1917),501(French method involving detonation of a charge 100 to 200 g of expl in the middle of a soft steel plate 500×500×25 mm thick, resting on two supports 400 mm apart, and measuring the depth of dent produced) 1a)Marshall 2, (1917),530(Testing of detonators by lead plate test) 2)B.Grotta, ChemMetEngrg 26, 1126–32(1922) (The lead plate test as applied to commercial detonators) 2a)H.Kast & A.Haid, SS 18, 166(1924) 3)L.V.Clark, IEC 25, 1386–7(1933) (Same lead plate test as in Ref 2) 4)Stettbacher(1933),361(Brief discussion on uses of iron, brass and copper plates) 5)Vivas, Feigenspan & Ladreda 4, (1944) (Lead plate tests called by them in Span "Pruebas sobre las planchas de plomo'') 6)Pérez Ara(1945), 121–4(Lead plate test called by him in Span, "Prueba de la placa de plomo,'' described in detail) 7)Stettbacher(1948), 89(Plate test using 10 mm thick iron plate) 8)Stettbacher(1952),115 & 141 9Belgrano(1952),51–2(Steel plate test, called in Ital, "Prova della piastra di acciaio'')

Potential(Potentiel or Effet utile, in Fr). According to definition given in Refs 1 & 2 it is equal to Q×425 kg-m/kg, where Q is heat of expln in Kcal/kg and 425 is mechanical equivalent of heat. This unit is identical with W which is the *maximum quantity of work* that can possibly be done by a unit weight of the explosive

A slightly different definition is given by Muraour(Ref 3): the *potentiel de l' explosif* is equal to Q×428, where Q is the heat evolved on decomposition of 1 kg of explosive and 428 is the mech equiv of heat. *Refs:* 1)Marshall 2, (1917),469 2)Pepin Lehalleur(1935),43 & 64 3)H.Muraour, "Poudres et Explosifs,'' Paris(1947),76

Power of Explosive is defined by Barnett(Ref 2) as "its capacity for doing useful work.'' Power may also be defined as energy×time. The value called in France *rendement pratique or effet utile* corresponds approx to "power.'' The Fr value can be calcd as has been done in France either as *potentiel*(potential) or as *produit characteristique de Berthelot*(characteristic product of Berthelot)

Power is usually defined in terms of one or several of the following experimental methods: a)*Ballistic Mortar Test*(qv) b) *Ballistic Pendulum Test*(qv) c)*Cratering Effect Test*(qv) d)*Mortar Test*(qv) e)*Quinan Test*(qv) f)*Trauzl or Lead Block Expansion Test*(qv), and its modifications *CUP* and *Kraftxabl.* *Refs:* 1)Marshall 2, (1917),463 2)Barnett(1919),178 3)Vennin, Burlot & Lécorché(1932),166–89 & 192–3 4)Marshall 3, (1932),143 5)OSRD Repts 803 & 804(1942),18–21 6)Vivas, Feigenspan & Ladreda 4, (1944), 111–15 7)Belgrano(1952),23–8

Pressure-Bar Apparatus of Hopkinson. See Hopkinson Pressure-Bar Apparatus

Pressure of Gases Developed on Combustion of Propellants or Explosives can be detd either by calcn or by combustion in a closed vessel(combustion en vase clos, in Fr), as described by H.Muraour, "Poudres et Explosifs,'' Paris(1947),73–4

Pressure of Gases Developed on Explosion or Detonation is an important factor because it serves as a measure of the capacity of an expl to do work, although the character of the work is detd by the rate at which this pressure is built up

Pressure of gases can be either calcd or approx detd experimentally by detonating a sample in one of the following devices: Bichel Bomb, Krupp Bomb, Noble and Abel Recording Pressure Gage, Petavel Recording Manometer, Piezoelectric Gages, etc. (See also Bichel Bomb and Detonation Pressure). *Refs:* 1)Marshall 2, (1917),444–7 2)US BurMinesBull 346, (1931),84 3)Marshall 3, (1932),133 4)Vennin, Burlot & Lécorché, (1932),50–3 & 72–86 5)Stettbacher(1933),69 6)Vivas, Feigenspan & Ladreda 4, (1944),20–29, 85–6 & 98–104

Primary Explosives, Initiating Efficiency. See under Initiating Efficiency of Primary Explosives, Blasting Caps and Detonators

Produit Characteristique(Fr). See Characteristic Product

Propagation of Detonation. See Ability to Propagate Detonation, in this section

Propagation Test. Under this name is described by L.V.Clark, IEC 25,668 & 1389(1933) an application of the *gap test*(qv) for testing the efficiency of detonators

Propulsive Force. According to L.V.Clark, IEC 25, 1388(1933), the *relative propulsive force* can be detd either by ballistic pendulum test or by Trauzl test, the usual tests for detn of *power of explosives*

Quickness(Vivacité, in Fr) of Burning of Propellants, Determination is described by H.Muraour, "Poudres et Explosifs," Presses Universitaires de France, Paris(1947),90–92, as well as in his other papers published in MP and MAF

Quinan Test permits determination simultaneously the brisance and the work performed on detonation of an expl. This value is called in Fr "potential" and may be considered as being approx proportional to power. The apparatus consists of two cylindrical steel blocks placed one on top of the other and guided vertically by four steel rods imbedded in the base. The top of the lower block is provided with a central cavity to hold 1–2 g of expl and a small electric detonator. The upper block is perforated in the center to allow the passage of elec wires and it is also provided with a device(such as a ratchet) permitting the block to be held in any position, but not interfering with its movement upwards. A steel plate is placed on top of the base and a small solid lead cylinder(crusher) is inserted betw the plate and the bottom of the lower steel block. After inserting the charge with detonator, the upper steel block is lowered on top of the lower block and the charge is fired. The pressure of expln will push the lower block down compressing the lead cylinder located underneath and will lift simultaneously the upper block to some position in which it will be held by the ratchet. The diminution in height of lead cylinder is taken as a measure of brisance and the height to which the upper block is lifted as a measure of potential or work, which is practically the same as power. *Refs:* 1)Vennin, Burlot & Lécorché(1932),192–3 2)Pepin Lehalleur(1935),63 3)Pérez Ara(1945),118

Rate of Detonation Tests. See Detonation Rate Tests

Red Iron Test is one of the US BurMines "Fire Resistance Tests." See under Sensitivity to Flame, Heat, Sparks, Etc

Relative Percussive Force of an Explosive. See Percussive Force of an Explosive

Relative Propulsive Force of an Explosive. See Propulsive Force of an Explosive

Reprise d'humidité, Essai(Fr). See under Hygroscopicity Tests

Resistance to Heat Test(Épreuve de la résistance à la chaleur). This Fr "Official" test for detn of stability of NC or of smokeless propellants is conducted in a thermostatically controlled oven, "type d'Arsonval." NC is tested at 108.5° and propellants at 108.5° and 109°, both with std blue litmus paper. The test is conducted in the following two stages: a)*Test to the First Red*(Épreuve du premier rouge). Place in each of five clean test tubes, near the bottom, a rolled strip of blue litmus paper, followed by a 10 g sample of propellant in small grains. Stopper the tubes and place them in the oven. Note the time of beginning change of color of paper to red. Cool the sample and save it until next day. b)*Test for Total Resistance*(Épreuve à la résistance totalisée). Replace the blue litmus paper with a new strip and continue heating in the oven until the change in color to red. Record the time and leave the sample at RT for at least 2 hrs(preferrably overnight). Change the paper and heat the sample as above, etc. During these tests, one will observe that the intervals betw beginning of heating and appearance of red color become shorter and shorter. As soon as this becomes one hour or less, stop the test and discard the sample. Count the total time of heating required to arrive at this point and this gives RT. Do not count the time between heatings. *Ref:* Book of Instructions issued by the Commission des Substances Explosives," Chapitre III, Articles 95–107 and Chapitre IV, Articles 230–1. This book may be obtained from the État Major de l'Armée, 2ème Bureau, Paris

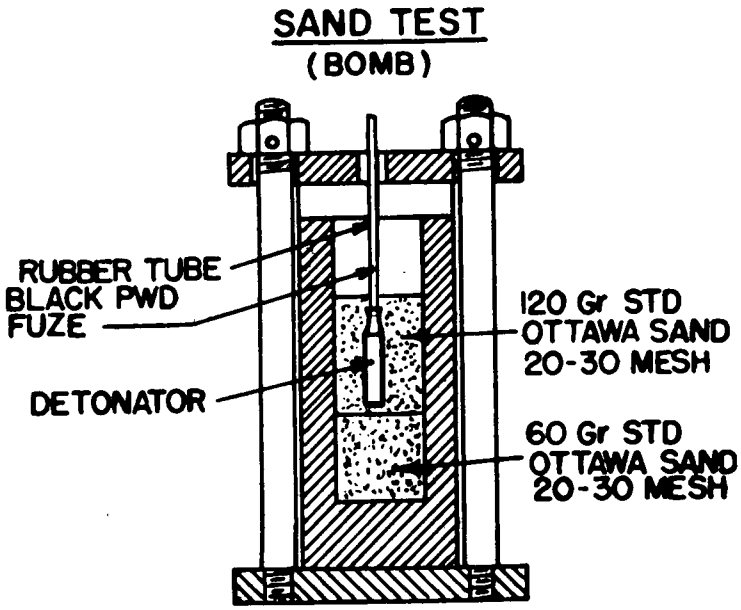
Rifle Bullet Test(Beschussprobe, in Ger). See Bullet Impact Sensitiveness Test

Rotter Impact Test. See FI(Figure of Insensitiveness) and also under Impact Tests

Sand Test or Sand Crushing Test(Sandprobe, in Ger) (Essai au sable, in Fr) (Prueba de la arena, in Span). This test, devised by W.O.Snelling in 1910 and studied extensively by C.G.Storm & W.C.Cope(Ref 1), is considered to measure the shattering(disruptive) power of an expl, called *brisance.** This characteristic is important because it determines the effectiveness with which an expl can fragment a shell, bomb casing, grenade or warhead of a rocket

The sand test consists essentially in detg the amt of standard sand(supplied by the Ottawa Silica Co, Ottawa, Ill), crushed by a std wt(usually 0.400 g) of expl. The original sand passes through No 20-mesh sieve(US Std) and contains no particles smaller than No 30-mesh. The test is conducted in a cylindrical steel bomb, 3½" in diam, 8½" with cylindrical cavity 1½" diam and 6" deep. *Procedure:* Transfer an accurately weighed 0.400 g portion of test expl, of such granulation as to pass through a No 100 sieve, to each of five empty No 6 commercial blasting cap shells(of Cu, Al or gilding metal) held during transfer in loading block. Insert in each shell a reinforcing cap provided with a small hole in the center and by means of a plunger subject the charge(previously placing it behind a barricade) to a pressure of 3000 psi for 3 mins. With a pin, prick the powder train in one end of a piece of miner's black powder fuse 8–9" long and crimp to the pricked end one of the above loaded No 6 caps, taking care that the fuse is held firmly against the

*According to W.R.Tomlinson, Jr, formerly of PicArsn, sand test det "energy" rather than brisance



charge in the cap. Pour into the bomb 80.0±0.1 g of std sand and level it by striking the bomb with a hammer. Lower the cap into the bomb so that the cap is centered at the axis of the bomb and just touches the sand. Pour 120±0.1 g of sand being careful not to disturb the position of the cap. Pass the upper end of the fuse through a tightly fitting rubber tubing which is then inserted in the hole of the bomb cover. Lower the cover into position and fasten it securely by means of two bolts with nuts. Ignite the fuse and after expln of chge, remove the cover. Transfer the contents of the bomb to a piece of glazed paper, cleaning the bomb and cover thoroughly. After removing pieces of cap and burnt fuse, transfer the sand to a No 30 sieve fitted with a bottom pan and a cover and shake for 3 mins on a mechanical shaker. Weigh to 0.1 g the sand which passed through the sieve and record the average of all five values. This wt is the *sand test value or brisance value*

If the expl chge cannot be initiated by flame, it is pressed without insertion of the reinforcing cap. Then a 0.300 g chge of LA is placed on top of the expl, followed by the reinforcing cap and another compression at 3000 psi. Then the fuse is inserted and the chge initiated as above. After detg the amt of sand crushed by these caps, subtract the wt of sand crushed by 0.300 g LA, when loaded alone in No 6 caps

A modification of this test applicable to liquid expls is described in Ref 9, pp 9 & 11. The sand test may also be applied to detg the amt of LA and/or tetryl, that must be used as initiator or booster to insure that the sample crushes the max net wt of sand. This is designated as *sensitivity to initiation test.* (See also Initiating Efficiency of Primary Explosives by Sand Test.) *Refs:* 1)US BurMinesTechPaper 125(1916) 2)US BurMines RI 2558(1923) 2a)H.Kast & A.Haid, SS 18, 166(1924) 3)US BurMines RI 3039(1930) 4)US BurMinesBull 346, (1931),109–13 5)L.V.Clark, IEC 25, 664 & 1387(1933) 6)OSRD Repts 803 & 804 (1942),24–7 7)Davis(1943),422–3 8)Pérez Ara(1945),124 9)PATR 1401,Rev 1(1950),7–12 9)TM 9–1910 (1955),60 10)PATR 1740,Rev 1(1958)

Sand Test for Detonators. See Initiating Efficiency of Detonators by Miniature Cartridge Test and also under Initiating Efficiency of Primary Explosives by Sand Test

Sensitivity to Detonation by Initiating Agents. See Sensitivity to Initiation by Detonators and Boosters

SENSITIVITY(Sensitiveness) of Explosives, Propellants and Pyrotechnic Compositions Tests may include the following: a)*Sensitivity to Detonation by Initiating Agents.* See Sensitivity to Initiation by Detonators and Boosters Tests described below b)*Sensitivity to Explosion from Glancing Blow.* See under Friction Sensitivity, Qualitative Tests, in this section c)*Sensitivity of Explosives to Glancing Blow Test.* Same as Item b d)*Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc.* See below e)*Sensitivity to Friction.* See Friction Sensitivity Tests f)*Sensitivity to Frictional Impact.* See under Friction Sensitivity Tests g)*Sensitivity to Heat.* See under Sensitivity to Flame, Heat, etc h)*Sensitivity to Impact.* See Impact Sensitivity Tests i)*Sensitivity to Inflammation*(Sensibilité à l'inflammation, in Fr). See Combustion Tests j)*Sensitivity to Initiation by Detonators and Boosters Tests.* See below k)*Sensitivity to Initiation by Electrostatic Discharge.* See under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc l)*Sensitivity to Initiation by Heat.* See under Sensitivity to Flame, Heat, Sparks, etc m)*Sensitivity to Initiation by Influence.* See Detonation by Influence Test n)*Sensitivity to Rifle Bullet Impact.* See Bullet Impact Sensitiveness Test o)*Sensitivity to Shock.* See Impact Sensitivity Test p)*Sensitivity to Sparks.* See under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc q)*Sensitivity to Sympathetic Detonation.* See Detonation by Influence Tests

SENSITIVITY TO FLAME, HEAT, SPARKS, ELECTROSTATIC DISCHARGES, ETC. These tests may be found listed in the literature under titles Burning Tests, Combustion, Fire Resistance Tests, Fire Tests, Index of Inflammability Test, Inflammability Tests and other names

US Bur of Mines(Ref 3) describes the following tests, under general title **Fire Resistance Tests:** a)*Fuse Test.* Insert an 8" long piece of squarely cut burning fuse into a test tube $\frac{7}{8}$" × 7" (clamped on a stand) with spit end against 3 g of the expl. Ignite the projecting end of the fuse and observe the behavior of the

expl behind a safety glass b)*Hemispherical Iron-Dish Test.* After heating an iron dish of hemispherical form; 4" in diam with a bottom 0.033±0.007" thick, to a red heat, drop(by means of a mechanical charging device operated behind a safety glass) on the bottom of the dish a charge of expl, not more than 0.5 g. If the 1st chge does not detonate, increase the quantity by ½ g increments up to 5 g. The point of "no explosion" is detd by trials in which no expln occurs with "a point of expln" occurring for a sample 0.5 g higher in weight c)*Red Hot Iron Test.* Heat to cherry-red(ca 900°) an iron bar 15 mm in diam over 10 cm of its length and bring it in contact with a small quantity expl placed on an asbestos board. If the expl burns without detonation, repeat the test using ca 100 g charge placed on an asbestos board. A permissible expl is considered to pass the test when it burns without deton and extinguishes itself when the source of heat is withdrawn. The iron bar should be brought in contact with the expl by a mechanical device while the operator is protected by a safety glass(Ref 3)

Similar tests are listed by Barnett(Ref 2) as Fire Tests

Marshall(Ref 1, p 435 describes under the Title **Sensitiveness to Heat** the following tests originated by H.Kast: a)*Behavior at Temperatures Near the Ignition Point* and b)*Behavior Towards Direct Heating.* Similar tests are described by Reilly(Ref 4) as *Inflammability Tests.* Médard(Ref 6) described the French Official tests. These tests are listed in this section under Combustion Tests. TM 9–1910(Ref 7) discusses the *Sensitivity to Heat and Spark,* and the *Sensitivity to Initiation by Electrostatic Discharge* is discussed in Refs 5 & 8. *Refs:* 1)Marshall 2, 435 2)Barnett(1919),216 3)US BurMinesBull 346, (1931),31–2 4)Reilly (1938),66 5)F.W.Brown et al, "Sensitivity of Explosives to Initiation by Electrostatic Discharges," US BM RI 3852(1946) 6)L.Médard, MP 33, 329–30(1951) 7)TM 9–1910(1955),49–50 8)PATR 1740, Rev 1(1958)

Sensitivity to Initiation by Primary(or Initiating) Explosives, Detonators and Boosters, Tests*(Sensitivity to Detonation by Initiating Agents)* (Sensibilité à l'amorce Essais, in Fr) [Zünd-(Initiier)-Vermögen Proben, in Ger] (Eficiencia como agente iniciador, Pruebas, in Span) (Sensibilità all' innescamento, Saggi, in Ital). Sensitivity to initiation of a HE can be expressed as the min weight of an initiating explosive required for complete detonation. It can also be expressed in the smallest No of a detonator required for complete detonation. If initiation of a HE cannot be achieved by a detonator alone and a booster is required, the min wt of booster expl and its name must be indicated

The tests are essentially the same as listed under Initiating Efficiency of Initiating Explosives, etc

In the US the sensitivity to initiation is conducted by the sand test using diminishing wts of an initiator, such as LA(lead azide), until there is obtained the min amt which will cause complete detonation of 0.40 g of powdered HE's when pressed in a blasting cap shell under a pressure of 3000 psi. When a HE(such as ammonium picrate) cannot be detonated by LA(or by other initiating expls) alone, the test is repeated by detonating 0.400 g of HE with a composite detonator consisting of 0.200 g LA and tetryl as a base charge. By repeating the tests with diminishing wts of tetryl, the min wt required to detonate the HE is detd(Refs 1 & 3)

In the Fr test, called *"sensibilité à l'amorce,"* described in detail by Médard (Ref 2) a 50 g sample of test HE contained in a Kraft paper cartridge, 30 mm diam and provided at one end with a mercury fulminate detonator and picric acid booster is laid horizontally on a lead plate 12×15 cm and 15 mm thick, resting on a steel plate at least 1 cm thick. After firing the cartridge, the appearance of the lead plate is observed. If the detonation is complete, the impression in the part of the plate farthest from the detonator would be somewhat deeper. In this case it is required to repeat the test using either a smaller detonator or a smaller booster. If the detonator is too small to achieve complete detonation, a larger size should be tried until detonation is complete. *Refs:* 1)PATR 1401, Rev 1(1950),7–11 2)L.Médard, MP 33, 339–42(1951) 3)TM 9–1910(1955),52–3 (See also Refs under Initiating Efficiency of Initiating Explosives, etc)

Setting Point Determination is described under Sodium Azide, Plant Analytical Procedures, p A613

Shaped Charge or Hollow Charge Efficiency(Cavity Charge Performance of Munroe-Neumann Effect). This term is applied to explosive charges with lined or unlined cavities formed in the charge opposite to the end of initiation. The lined or unlined hollow charge effect is sometimes referred to as simply *cavity effect.* A flat end explosive of high brisance produces a dent in a hard steel plate; the same explosive using the unlined cavity effect of Munroe-Neumann, erodes the target forming a smooth shallow crater; and a lined cavity in the same explosive and charge size produces a deep, narrow V-shaped hole in the steel plate. The application of this phenomenon represents one of the major advances in the use of explosives during WWII

The penetration action of a shaped charge is dependent upon a number of factors, such as a)the explosive used, b)type of liner material and its thickness, c)cone angle and other shape of cavity and d)*stand-off distance* or distance between the base of cavity and target. These factors must be determined experimentally for each explosive and for each type of shaped charge design

This test is conducted by placing the assembly vertically, at a known stand-off distance, above several layers of 0.5 inch thick armor-plate steel and detonating the charge. After detonation,the depth of hole, its average diameter at the top, and its volume are determined. *Refs:* 1)TM 9–1910(1955),78–85 2)PATR 1740, Rev 1(1958) 3)Cook(1958),226–64

Shell Impact Sensitivity Test See Armor Plate Impact Test, in this section

Silvered Vessel Test or Waltham Abbey Silvered Vessel Test(Silbergefässprobe, in Ger), designed for testing cordite, but suitable for testing other propellants and NC, is conducted as follows: A 50 g sample of cordite, cut into pieces $\frac{1}{8}$" long, is placed into a vacuum-jacketed round-bottom flask(''silvered vessel''), provided with a piece of tubing fused at the lower part of the neck and sealed at the other end. This tube serves as a means for observing color of gases evolved during heating of cordite. After closing the flask with a perforated cork and inserting a precision thermometer in the middle of the sample, the flask is placed in a thermostatically controlled air-bath at $80 \pm 0.1^{\circ}$. Reading of thermometer and the color of gases in the lateral tube are observed at regular intervals and the time of appearance of red fumes is recorded. A few hours after this, the temp of cordite begins to rise and as soon as the rise reaches 2°, the test is considered completed. A good ''service'' cordite will stand this test for 500–600 hrs. *Refs:* 1)F.L.Nathan, JSCI 28, 443–4(1909) 2)Marshall 2, (1917),663–4 3)Reilly(1938),81 4)Kast-Metz(1944),318

Small Lead Block Compression Test will be discussed under Compression Tests in Vol II

Small Lead Block Compression Test for Detonators. See under Esop's Test

Small Lead Block Expansion Test for Detonators. See under Trauzl Test

Sound Test for Detonators(Prueba acustica para detonadores, in Span). Martin(Ref 1) proposed to compare the brisance of detonators by observing the action of their sound wave on sensitive or vibrating flames. Other proposals have been made to measure the intensity of the sound by means of a microphone and sensitive galvanometer. In the opinion of Marshall (Ref 2) and of Pérez Ara(Ref 3), the weak point in these methods is that there is no necessary connection betw the intensity of sound and the efficiency of the detonator. *Refs:* 1)F.Martin, ChemZtg 37, 90(1913) 2)Marshall 2, (1917),532 3)Pérez Ara(1948),124

Specific or Normal Volume. See under Volume of Gases Evolved on Detonation(or Explosion)

Stability(Thermal) of Explosives and Propellants. See under Heat Tests

Steel Plate Denting Test or Steel Test. See Plate Denting Tests and under Plate Tests

Strength of Detonators, Determination. Same as Initiating Efficiency of Detonators, Tests

Strength of Dynamites. According to Davis(1943),338–9, the strength*(explosive force)* of a straight nitroglycerin dynamite is expressed by the percent of NG which it contains. Thus ''40% straight NG dynamite'' contains 40% NG but ''40% ammonia dynamite,'' ''40% gelatin dynamite,'' etc, whatever their compn may be, are supposed to have the same strength or explosive force as 40% straight dynamite. Strength of dynamites can be detd by Trauzl Test(qv)

Strength of Explosives, Test. According to Marshall 2, (1917),469 ''Trauzl's lead block test affords a ready means of ascertaining the approximate relative strength of explosives.'' As power of explosives is also detd by the same test, it seems that there is no difference between strength and power

65.5° and 80° Surveillance Tests are standard US stability tests for propellants. Transfer a 45 g sample to a dry 8 oz flint-glass bottle provided with an air-tight ground-glass stopper. Place the bottle in an oven or a special chamber(such as is represented by Fig 81, p 244, Ref 2) maintained at $65.5 \pm 1^{\circ}$. After heating for 24 hrs, reseat the stopper and continue heating. Observe the bottle every 24 hrs and note the number of days required to cause the evolution of red fumes(oxides of nitrogen). Test values of 20 days or less indicate a condition of hazardous instability calling for immediate disposal of the sample. Values of 90 days or less indicate insufficient stability

The test can be conducted at 80° when anticipatory data are required quicker than by the 65.5° test

Refs: 1)PATR 1401, Rev 1(1950),15–16 2)TM 9–1910(1955),243–4

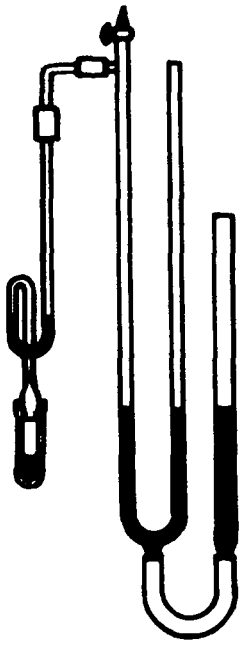
78° Surveillance Test, described by D.R.Wiggam & E.S.Goodyear, IEC, AnalEd 4, 73(1932), is similar to the above tests, except the temperature of heating is different

Sympathetic Detonation Test. See Detonation by Influence Test

Taliani Test for detg the thermal stability of NG, NC, and NG propellants was first described in 1921(Ref 1). The app consists of a glass tube, in which 1.3 g of sample is placed, closed with a ground-glass top and connected with a paraffin trap, the top of which is connected to a Hg manometer. The entire app, except the manometer, is heated in a specially constructed oven at 120° for NG or NG contg propellants, and at 135° for NC. After 30 min of heating the sample, the stopcocks on the sample tube and on the manometer are closed to keep the sample in contact with its decompn products. The pressure developed is measured at suitable intervals and the index of stability adopted is the time in minutes necessary to attain a press of 100 or 300 mm Hg

As first described the Taliani test was considered a good quantitative test of thermal stability but it was tedious and time consuming. Numerous investigators have subsequently modified the initial procedure. Goujon (Ref 2) heated NC, previously dried at 100° for 2 hrs, in a const vol at 135° in the presence of its decompn products and noted the time necessary to develop a press of 100 mm Hg. This time characterized the stability of the sample and the test was completed in 2 hrs. Wiggam & Goodyear(Ref 3) made modifications in the Taliani app and conducted the test on double-base powders at 120°. Haid et al(Ref 4) studied the time-press relationships of NC and HE's at 75°. Tonegutti(Ref 5) conducted this test at 120, 125 & 130° on NC, NG, NGu and other expls. Berl et al(Ref 6) used the ''glass-feather'' manometer,at 135° or higher

TALIANI APPARATUS



For research purposes a small scale micro-Taliani app has been constructed and applied to the study of HE's(Refs 8, 9 & 10). A description of this app and the procedure for its use will be prepd in a PA rept of std lab procedure. *Refs:* 1)M. Taliani, Gazz **51** I,184–93(1921) & CA **16**, 342(1922) 2)J.Goujon, MAF **8**, 837–902(1929); SS **26**, 217, 261, 289, 330, 361 & 400(1931) & CA **26**, 1444(1932) 3)D.R.Wiggam & E.S. Goodyear, IEC,AnalEd **4**, 73(1932) & CA **26**, 1444(1932) 4)A.Haid et al, SS **30**, 66–8 & 105–8(1935) & CA **29**, 4585(1935) 5)M.Tonegutti, IndustriaChimica **9**, 1334–42(1934) & CA **29**, 6061(1935); Chim e Ind **17**, 517–21(1935) & CA **30**, 1562(1936); and SS **33**, 185–6(1936) & CA **32**, 8145(1938) 6)E.Berl et al, IEC,AnalEd **10**, 220(1938) & CA **32**, 4338(1938) 7)Reilly(1938),88 8)NOL MemoRept 10288(1950) (Conf) 9)NAVORD Rept 2782(1953) (Conf) 10)PA MemoRept MR–55(1954) (Conf)

Temperature Developed on Detonation(or Explosion) can be detd experimentally with a fair degree of accuracy by optical methods and can also be estimated by calcn as discussed in the following: *Refs:* 1)É.Sarrau, "Théorie des Explosifs," Gauthier-Villars, Paris(1895),16–17 2)Marshall **2**, (1917),453 & 459–60 3)Barnett(1919),200 4)Vennin, Burlot & Lécorché(1932),36–50 5)Stettbacher(1933),85 6)H.Muraour, "Poudres et Explosifs," Paris(1947),71–3 7)Stettbacher(1948),14 8)Stettbacher(1952),17

Testing Galleries. See Galleries for Testing Permissible Explosives

Thermal Stability Tests. See Heat Tests

Time of Ignition(or Explosion) Test. See Ignition(or Explosion) Test

Transmission of Detonation Through Air. See Detonation by Influence(Sympathetic Detonation)

Transmission of Detonation Through Explosive Charge(Extent of Propagation of Explosion). See Ability to Propagate Detonation, in this section

Trauzl Test; Trauzl Lead Block Test or Lead Block Expansion Test(Cavité au bloc de Trauzl or Épreuve au bloc de plomb de Trauzl, in Fr) (Trauzlsche Probe or Bleiblockausbachung Methode, in Ger) (Prueba Trauzl or Prueba del bloque de plomo, in Span) (Metodo del Trauzl or Metodo del blocco di piombo, in Ital). According to Ref 5, this test measures the "comparative disruptive power" of an expl through enlargement of a cavity in a cylindrical lead block under carefully standardized conditions. Std conditions for conducting this test were defined by a Comm of the Fifth International Congress of Applied Chemistry(Ref 1). Although one of the oldest tests known for detg power, it is still widely used today but more common in Europe than in the USA

Procedure. A sample of the test expl(approx 10 g) is detonated in a cavity or borehole, 25 mm in diam and 125 mm deep, in a std lead block 200 mm in diam and 200 mm in height. The borehole is made centrally in the upper face of each block, previously cast in a mold from desilvered lead of the best quality. An electric blasting cap is placed centrally in the chge. After the chge and detonator are placed in the borehole, 40 cc of Ottawa sand are added and tamped lightly. An addnl 10 cc of sand are added and tamped more thoroughly. The volume of the hole made due to the press exerted by the exploding chge is then detd; and the distension(expansion) is calcd by subtracting from this value, the vol of the borehole before the chge is detonated. Three such tests are made and the results averaged. Expansions for equivalent wts of expls are calcd, and the test value is expressed in % of the expansion of an equivalent wt of TNT

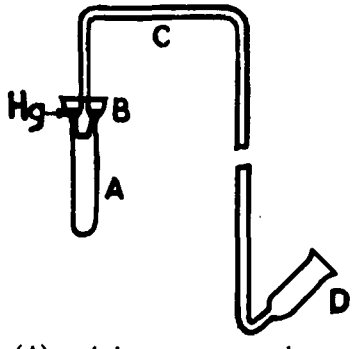
The Trauzl test in France is somewhat different in procedure although dimensions of the lead block are the same(See Coefficient d'utilisation practique,in this section)

Initiating efficiency(strength) of primary expls can be approx detd by firing a small chge(such as 1.0 g) in the cavity of a *small lead block*, such as 100 mm in height and 100 mm in diam. For testing detonators in such block, a hole is bored in the block of the exact diam of the detonator and of such a depth that the top of the detonator is flush with the top of the block(Ref 5, p 106 & Ref 5a, p 666)

Refs: 1)FifthIntCongAppldChem Vol 2 (1903),256 2)M.Neumann, ZAngChem 24, 2234(1911) 3)Marshall 2 (1917),469–72 4)Barnett(1919),179–81 5)US BurMinesBull 346 5a)L.V.Clark, IEC 25, 666(1933) 6)Stettbacher(1933),361–5 6a)Pepin Lehalleur(1935),64–6 7)OSRD 803 and 804(1942),18–21 8)Davis (1943),24–5 8a)Vivas, Feigenspan & Ladreda 4, (1944),111–14 8b)Pérez Ara(1945),113–17 8c)Belgrano (1952),23–8 9)TM 9–1910(1955),70–1 10)PATR 1740, Rev 1(1958)

Vacuum Stability Test was designed by Farmer(Ref 1) for detn of stability of explosives and propellants. In this test the thermal decompn of a sample is followed by observing the rise in pressure of the gases given off in vacuum. The test can be conducted at temps ranging from 80° to 180°(Ref 1), but in the US, the temps are 90° for propellants and 100° or 120° for HE

VACUUM STABILITY APPARATUS



Procedure: Transfer 5 g of thoroughly dried propellant or HE(use 1 g in case of initiating expls) to a glass heating tube(A) so designed that the ground neck(B) can be sealed with mercury after a calibrated capillary tube(C) with a ground stopper end has been connected. Place in the cup(D), attached to the lower end of capillary, ca 7 ml Hg and insert a rubber stopper with a tube connected to a vacuum pump. Tilt the app forward to free of Hg the capillary opening of the cup (D) and evacuate the apparatus until the press is reduced to ca 5 mm. This will force the Hg to rise in (C), nearly to the top. Disconnect the pump, add ca 1 ml of Hg to (D) and measure the total vertical height of the column in (C). Measure and subtract the vertical height of the column of Hg in (D). Note the RT and atm press. Insert the tube(A) in a bath maintained at desired temp ±0.5° and heat for 40 hrs unless an excessive amt of gas(11+ml) will be evolved in less time. Remove the app, cool RT and observe the atm press. Measure the total vertical height of the column of Hg in the capillary(C) and subtract the vertical height of the column in the cup(D). Calculate the vol of liberated gas from the difference betw the initial and final levels, as well as the vol of the capillary per unit of length, the vol of tube(A), and the atm press and temp conditions at the beginning and end of the test. The formula for this calcn is given in Ref 3, p 14. *Refs:* 1)R.C.Farmer, JCS 117, 1434–40(1920) 2)Reilly(1938),92 3)PATR 1401, Rev 1(1950),12 & 14–15 4)TM 9–1910(1955),56–7

Vapor Pressure of Explosives and Related Substances gives an idea as to their volatility and in some cases to their stability. Methods for measuring vapor pressure may be divided into static and dynamic. Their description can be found in the following : *Refs:* 1)J.Reilly & W.N.Rae, "Physico-Chemical Methods", VanNostrand,NY, v 1(1944),117 2)J.Strong, "Procedures in Experimental Physics," Prentice-Hall,NY (1945),Chap 3 3)A.Weissberger, "Physical Methods of Organic Chemistry," Interscience,NY, vol 1,part 2 (1949),141–251 4)J.H.Perry, Edit, "Chemical Engineers' Handbook," McGraw-Hill,NY(1950),98 & 149–73

Velocity of Detonation Tests. See Detonation Rate Tests

Vitesse de détonation. Fr for Detonation Rate

Vivacité, Determination de la. Fr for Quickness of Burning of Propellants, Determination

Volatility of Explosives and Related Substances may be expressed by the loss of wt per unit of its exposed surface at a given temp and in unit time. Two of the volatility tests used in the US are: *100° Heat Test*(qv) and *75° International Test*(qv)

R.Colson, MP 30, 55(1948) describes a French volatility test conducted by leaving a weighed sample in a container of a known surface in dry air at atm press and at a desired temp(such as 60°) for several hours or days and then reweighing. The loss of wt in milligrams per square decimeter and per one hour is known as *volatilité*

Volume of Gases Evolved on Explosion or Detonation may be either calcd or detd experimentally by exploding material in one of the bombs, such as Bichel Bomb(qv), and then collecting and measuring the volume. The volume(in liters) evolved by 1 kg of expl, measured at NTP, is called *specific volume* or *normal volume(Vo).* *Refs:* 1)Marshall 2 (1917),443 2)Pascal(1930),15 3)Vennin, Burlot & Lécorché(1932) 4)Stettbacher(1933),69 5)Stettbacher(1948),13 6)Stettbacher(1952),16

Waltham Abbey Silvered Vessel Test. See Silvered Vessel Test

Wax Gap Test is one of the Detonation by Influence tests(qv) and is similar to the Booster Sensitivity test(qv). The Wax Gap Test is described briefly under Ammonium Nitrate, p A354, Note c and in more detail in Spencer Chemical Company, "Safety Data," Kansas City,Mo(1960)

LIST OF ABBREVIATIONS, CODE NAMES AND SYMBOLS USED IN THIS WORK AND IN MILITARY ORGANIZATIONS OF THE USA AND OF VARIOUS OTHER COUNTRIES

(Items not listed here are given in the text. For German abbreviations see PATR 2510)

(See also Supplement, pages Abbr 59ff)

A

A or Abstr	abstract
A	acid (as MA, mixed acid)
A	aniline
A	argon
A	Army
A	atomic
A	after an Ordn term, indicates a standardized variation of a standard item
A(gamma)	Ital gelatin type expl contg NG
$\mathring{A}$	Angstrom
A°	absolute temperature
A1	first class; excellent
A1(Monobel); A1(Roundkol); A2(Monobel)	British explosives (see the text)
A/40	Rus 40/60 amatol
A/80	Rus 80/20 amatol
AA	antiaircraft
AA	Augusta Arsenal, Agusta, Ga
AAA	antiaircraft artillery
AAAAW	antiaircraft artillery automatic weapons
AAC	antiaircraft cannon
AADL	Artillery Ammunition Development Laboratory, PicArsn, Dover, N J (now AARDL)
AAE	American Association of Engineers
AAE	Aeroplane and Armament Establishment (Brit)
AAF	Army Air Forces
AAFCE	Allied Air Forces, Central Europe
AAG	antiaircraft gun
AAM	air-to-air missile
AAMG	antiaircraft machine gun
AARDL	Artillery Ammunition Rocket Development Laboratory, PicArsn, Dover, NJ (formerly AADL)
AASM	Association of American Steel Manufacturers
AATCC	American Association of Textile Chemists and Colorists
AAWC	Australian Advisory War Council
AB	Aktiebolaget (Swed)(same as Ger A-G)
AB	aviobamba(Rus)(aerial bomb)
ABBG	Aktiebolaget Bofors–Gullspång (Swed)
ABBN	Aktiebolaget Bofors–Nobelkrut (Swed)
abbrn	abbreviation
ABCC	Atomic Bomb Casualty Commission
ABCM	Association of British Chemical Manufacturers
Abel's Expl	see Picric Powder
ABL	Allegany Ballistics Laboratory Cumberland, Md
ABMA	Army Ballistic Missile Agency, Redstone Arsenal, Huntsville, Ala(see also OML)
ABNP	Aktiebolaget Norma Projektilfabrik(Swed)
ABNT	Aktiebolaget Nora Tandrofsfabrik(Swed)
ABSAV	Aktiebolaget Svensk Automat Vapen(Swed)
ABSKF	Aktiebolaget Svenska Krutfaktoriema, Landskrona(Swed)
absol or abs	absolute
abspn	absorption
abstr or A	abstract
abt	about
AC	Allied Chemicals, New York
AC	symbol for hydrogen cyanide (CWA)
Ac	acetyl(CH_3CO-, not CH_3COO-)
Ac	acid
AC	adjusted charge (Brit)

AC	Air Corps; aircraft
AC or ac	alternating current
A/C	anticoncrete
AC	armored car
AC	Army Corps
AcAn	symbol for 1,9-diacetoxy-pentamethylen–2,4,6,8-tetranitramine
Acad	Academy
ACC	American Cyanamide Co, New York
ACC	Army Chemical Center, Maryland (See also EA)
ACCCE	Association of Consulting Chemists and Chemical Engineers
accdg	according; according to
Acc of F	accuracy of fire
ACD	Armour Chemical Div, Chicago 9, Ill
acet	acetone
acet ac	acetic acid
ACNA	Aziende Chimiche Nazionali Associate (Cengio) (Ital)
AcH	acetaldehyde
ACHEMA	Ausstellung für Chemisches Apparatewesen
ACO	Army Corps Ordnance (Brit)
Ac_2O	acetic anhydride
AcOH	acetic acid
ACS	Allied Chiefs of Staff
ACS	American Chemical Society
ACSIRO	Australian Commonwealth Scientific and Industrial Research Organization
act	active
ACT-5	see the text
Actg	Acting
ad	advertisement
AD	Air Defense; ASTIA Document
AD	Ammunition Depot or Dump
AD	Anno Domini (after Christ)
A-day	Army Day
ADC	Air Development Center
Add	addenda
addn	addition
addnl	additional
ADE	Ital time & percussion fuzes used with aerial burst or impact projectiles (OP 1168, p63)
adj	adjective
Adj	Adjutant
ADL	Arthur D. Little, Inc, Cambridge, Mass
Adm	Admiral
Admy	Admiralty
adrm	airdrome
adv	adverb
AEA	Atomic Energy Act
AEC	Atomic Energy Commission
AECL	Atomic Energy of Canada, Ltd, Canada
AEDC	Arnold Engineering Development Center, Talahoma, Tenn
AEF	Allied Expeditionary Forces
AEG	Allgemeine Elektrizitäts Gesellschaft (General Electric Co of Germany)
AEL	Aeronautical Engineering Laboratory (US Naval Base, Phila, Pa)
AERE	Atomic Energy Research Establishment, Harwell, England
aerod	aerodynamics
aeron	aeronautics
AESC	American Engineering Standards Committee
AEU	Amalgamated Engineering Union
A°F	after firing(Brit)
AF	Air Force
AF	Armored Force
AF	Aviobomba, fugasnaya(Rus)(demolition bomb)
AFAC	Air Force Armament Center, Eglin, Fla
AFB	Air Force Base
AFBMD	Air Force Ballistic Missile Div, Inglewood, Calif
AFCA	Armed Forces Chemical Association
AFBDC	Air Force Base Development Center, Eglin, Fla
AFCRC	Air Force Cambridge Research Center, Bedford, Mass
AFFTC	Air Force Flight Test Center, Edwards AFB, Calif
AFMTC	Air Force Missile Test Center, Patrick AFB, Cocoa, Fla
AFNOR	Association Française de Normalisation(Fr Assocn for Standardization)
AFOSR	Air Force Office of Scientific Research, Washington, DC and Pasadena, Calif
AFOTC	Air Force Operational Test Center
AFPTRC	Air Force Personnel and Training Research Center
AFR	Admiralty Fuel Research(Brit)
AFS	Army Field Services, Fort Monroe, Va
AFSWC	Air Forces Special Weapons Center, Kirtland Air Force Base, NM
AFSWP	Armed Forces Special Weapons Project (changed to DASA)

AFT	adiabatic flame temperature
AFUS	Armed Forces of the United States
AFV	armored fighting vehicle (Brit)
AG	assault gun
A/G	antigas; air-to-ground
Ag	argentum (silver)
AgA	silver azide
AGARD	Advisory Group for Aeronautical Research and Development (NATO)
AGB	American Glycerin Bomb (see the text)
AGC	Aerojet-General Corp, Azusa, Calif
agcy	agency
AGDNW	Aktiengesellschaft Dynamit–Nobel, Wien (Austr)
AGE	Admiralty Gunnery Establishment (Brit)
Agfa	Aktiengesellschaft für Anilin-fabrikation (Ger chemical firm)
AGI	Ace Glass, Inc, Vineland, NJ
agitn	agitation
AGJ(Comp)	cast double-base propellants developed by ABL
Agr	Agriculture
agrl	agricultural
AHTCo	Arthur H. Thomas Company, Phila 5, Pa
AIC	American Institute of Chemists
AIC	Ammunition Identification Code
AIChE	American Institute of Chem Engineers
AIEE	American Institute of Electrical Engineers
AIHA	American Industrial Hygiene Association
AIIR	Air Intelligence Information Report
AIMME	American Institute of Mining and Metallurgical Engineers
AISC	American Institute of Steel Construction
AISI	American Iron and Steel Institute
A(ko)	Jap explosive (see text)
Al	aluminum
ALA	American Library Association
Alba	Alberta, Canada
Albanite	Brit propellant (see the text)
Albite	Ital expl (see the text)
alc	alcohol, alcoholic
ALCAN	Alaskan-Canadian Highway
ALCAN	Aluminum Company of Canada
ALCOA	Aluminum Company of America
ald	aldehyde
Aldorfit	Swiss expl (see the text)
alk	alkali, alkaline
Alk	alkyl
Alkalites	Belg safety expls (see the text)
alky	alkalinity
ALRL	Aluminum Research Laboratory (of ALCOA)
Alsilite	Belg expl (see the text)
Alsk	Alaska
alt	altitude
Alta	Alberta, Canada
Alumatols	see the text
a/m	above mentioned (Brit)
AM	Air Ministry (Brit)
Am or am	amyl
AM or am	when added to the designation of a Fr propellant, means that amyl alc was used as a stabilizer (eg BAm, BFAm, etc)
AM	ante meridiem (Latin for "before noon")
AM	Army Manual (Brit)
AMA	American Medical Association
AMA	American Military Attaché
amal	amalgam
am alc	amyl alcohol
Amatex	see the text
Amatol	see the text
A/MB	anti-motorboat
AMC	Army Medical Center
AMC	Army Medical Corps
AMES	Air Ministry Experimental Station (Brit)
AMILAT	American Military Attaché
AML	Admiralty Materials Laboratory (Brit)
AML	Aeronautical Materials Laboratory (US)
Amm	ammonium (NH_4)
ammo	ammunition
Ammonals	see the text
Ammonaru	Jap for Ammonal
Ammoniak krut	see the text
ammoniaku	Jap expl (see the text)
Ammonites	see the text
AmmP or AP	ammonium picrate
AmocoCC	Amoco Chemicals Corp, Chicago, Ill
amor	amorphous
amp	amperes
AMP	Applied Mathematical Panel
amph	amphibian

amp-hr	ampere-hour
AMPS	Army Mine Planting Service
AMRL	Army Medical Research Laboratories
AMSEF	anti-mine sweeping explosive float(Brit)
amt	amount
AMTB	anti-motor torpedo boat
AMTRAC(s)	amphibious tractor(s)
AMVETS	American Veterans of WW II
AN	ammonium nitrate
AN	ammonium nitrate based propellants
AN	Army-Navy
AN	after an Ordn term designates a standardized item for use by both the Army and Navy
AN-507	see the text
ANC	Argentine Naval Commission, New York 19, NY
AND	Army & Navy Design
ANG	Air National Guard
ANG	code name for nitroglycerin(NG) plus nitroglycol(NGc)
anhyd	anhydrous
Anilite	liq expl (see the text)
ANL	Argonne National Laboratory
Ann	Annals
anom	anomalous
anon	anonymous
Ans	anisole
ANS	Ital expl (Antisanzionite)(see the text
ANSB	Army-Navy Safety Board, Washington 25, D C
antifr	antifreezing
Antigel de sûreté Antigrisou(explosifs) Antigrisous Favier	Belgian safety expls (see the text)
Antigrisouteux (explosifs)	Fr permissible expls
antilog	antilogarithm
AO	aviobamba, oskolachnaya (Rus)(fragmentation bomb)
AOA	Army Ordnance Association, Washington, DC
AOAC	Association of Official Agriculture Chemists
AOD	Anniston Ordnance Depot, Anniston, Ala
AOD	Army Ordnance Depot
AOD	Arsenal Operations Division
AOR	Army Ordnance Regulations
AORG	Army Operational Research Group (Brit)
AOS	Army Ordnance Service
AOW	Alabama Ordnance Works,Childersburg, Ala
AP	American Patent(see USP)
AP	see AmmP(ammonium picrate)
A/P or AP	antipersonnel
AP	armor-piercing
APAT	armor-piercing, antitank
APC	armor-piercing, capped
APC	Atlas Powder Co, Wilmington 99, Del
APCHE	armor-piercing, capped,high explosiv
APCI	armor-piercing, capped,incendiary
APC-LC	armor-piercing capped, long case
APCI-T	armor-piercing capped,incendiary with tracer
APC-T	armor-piercing capped with tracer
AP-FS-DS	armor-piercing fin-stabilized, discarding sabot (arrow)
APG	Aberdeen Proving Ground, Md
APHA	American Public Health Association
APHE	armor-piercing, high explosive
APHEBC	armor-piercing, high explosive, ballistic cap
APHV	armor-piercing, hyper velocity
API	American Petroleum Institute
API	armor-piercing, incendiary
API-T	armor-piercing, incendiary with tracer
APL	Applied Physics Laboratory, Johns Hopkins University
APLN	armor-piercing, long nose
APMB	armor-piercing, monoblock
AP/NECL	Ardeer Plant of Nobel's Explosives Co, Ltd, Scotland
APO	Army Post Office
app or appar	apparatus
appd	approved
appld	applied
appln	application
approp	appropriate
approx or appr	approximate(ly)
apptd	appointed
appval	approval
Appx	appendix
APRN	armor-piercing, round nose
APT	appoint(see the text)
AP-T	armor-piercing with tracer
APWO	Association of Public Works Officials

APX-4A APX-5A	Amer exptl expls contg EDNA (see the text)
aq	aqueous
aq reg	aqua regia
aq soln(s)	aqueous solution(s)
AR	analytical grade reagent
Ar	aryl radical
AR	Army Regulations
AR	automatic rifle
ARC	Aeronautical Research Council (Brit)
ARC	Atlantic Research Corp, Alexandria, Va
Arcite	plastic propellant
ARD	Armament Research Dept (Brit)
ARDC	Air Research and Development Command, Baltimore, Md
ARDE	Armament Research and Development Establishment, changed to ARE (Brit)
ARDEC (changed to CARDE)	Armament Research and Development Establishment, Canada
ARE	Armament Research Establishment, Fort Halstead, Kent, England
ARE/RA	Armaments Research Establishment, Royal Arsenal, Woolwich, Engl
ARF	Armour Research Foundation, Chicago, Ill
Arg	Argentina
ARGMA	Army Rocket and Guided Missile Agency, Huntsville, Ala
ARL	Admiralty Research Laboratory (Brit)
ARL	Aeronautical Research Laboratory(Australia)
arith	arithmetic(al)
Arsn	Arsenal
Arsol	code name for trimethylenetrinitrosamine
Arty	Artillery
AS	Air Service (Brit)
A/S	anti-submarine
As	Arsenic
ASA	azide-styphnate-aluminum (Brit initiating mixt)
ASA	Ital expl(see the text)
ASCE	American Society of Civil Engineers
ASD	Ammunition Sub-Depot
ASDIC	Antisubmarine Detection Investigation Committee (Code name for ultrasonic underwater detector)
ASF	Army Service Forces
ASHVE	American Society of Heating and Ventilating Engineers
ASM	air-to-surface missile
ASM	American Society for Metals, Cleveland, Ohio
ASME	American Society of Mechanical Engineers
ASN	Ital expl and propellant
ASNE	American Society of Naval Engineers
ASP	Ammunition Supply Point
ASP	Army Supply Point
asph	asphalt
ASRE	Admiralty Signal Research Establishment(Brit)
ASSE	American Society of Safety Engineers
assoc	associate
assocd	associated
Assocn	Association
asstd	assorted
ASTIA	Armed Services Technical Information Agency, Arlington Hall Station, Arlington 12, Va
ASTM	American Society for Testing Materials
ASTM	American Standard Test Method
ASV	Active Service
ASV	aircraft searching apparatus
A/SW	antisubmarine warfare
asym or as	asymmetrical
AT	air temperature
AT	Rus for amatol
A/T or AT	antitank
AT	assay ton
at	atomic
ATA	air-to-air
ATC	Air Training Command
ATF	Rus for amatol,phlegmatized
ATG	air-to ground
A/TG	antitank gun
ATIC	Air Technical Intelligence Center(changed–see below)
ATIC	Aerospace Technical Intelligence Center

atm	atmosphere
at/no	atomic number
ATO	assisted take-off
Att	Attaché
ATT	attenuated ballistite(Fr)
attn	attention
at vol	atomic volume
at wt	atomic weight
ATX	symbol for 1,7-dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane
Au	aurum(Lat) (gold)
ÅU	Ångstrom unit
aut	automatic
aux	auxiliary
A/V	armored vehicle
av or avg	average
avdp	avoirdupois(1 lb = 453.59g)
Avigliana 3	Ital expl (see the text)
avn	aviation
AW	automatic weapon
AWC	Armstrong-Whitsworth Co, England
AWD	acoustic warning device
AWL	absent with leave
AWOL	absent without official leave
AWRE	Atomic Weapons Research Establishment (Brit)
az	azimuth
Az	azote (French for nitrogen)
azx	azoxy

B

B	base (of a bomb)
B	battery
B or benz	benzene
B	blind or dud (Brit)
B	bomb (er)
B	boron
B	brisance calcd by Kast formula(see the text)
B	broneprobivnoy(Rus)(armor-piercing)
B	used after an Ordn term, denotes a standardized item for use by both Army and Navy
B(gomma)	Ital gelatin type expd contg NG
B(salt)	one of Amer designations for ethylenediamine dinitrate
B_4	Jap incendiary expl (see text)
B(poudre)	Fr propellant named in honor of Gen Boulanger; formerly called V(poudre) because it was invented by Vieille
Ba	Barium
BA	Benicia Arsenal, Benicia, Calif
BA	benzaldehyde
BA	British Academy
BA	British Admiralty
BA	Bureau of Aeronautics, Washington, DC
BAC	Bell Aircraft Corp, Buffalo NY
BAC	Bristol Airplane Co (Brit)
BAc	benzoic acid
bacter	bacteriological
BAD	Base Ammunition Depot
B & I	base and increment
Bakufun	Jap primary expl (see the text)
Bakuhatsu-sei	Jap blasting cap
bal	ballistics
Balistita	Ital double-base propellant
Ballistites	double-base propellants
BalMort	ballistic mortar
Baln	Fr ballistite(see the text)
BalPend	ballistic pendulum
BAM or BAm	Fr propellants stabilized with amyl alcohol
BangT	bangalore torpedo
Baratol	Mil expl contg Ba nitrate & TNT
Barisutaito	Jap for ballistite
Baronal	Mil expl contg Ba nitrate, TNT & Al
BAS	Bessemer aerial steel
BAS	Ital projectile
BAS	Bulletin of Atomic Scientists
BA/T	battalion anti-tank
Battn	battalion
BB	barrage ballon
BB	buoyancy bomb(Brit)
BC	Before Christ
BC	British Columbia, Canada
BC	Fr propellant (see the text)
BCC	Baker Chemical Co, Phillipsburg, NJ
BCD	Becco Chemical Div, Buffalo, 7, NY
BCIRA	British Cast Iron Research Assocn
BCNL	Fr cannon propellant (see the text)
BCotIRA	British Cotton Industry Research Association
BCRA	British Coke Research Association
BCSO(NA)	British Commonwealth Scientific Office(North America)
BCURA	British Coal Utilization Research Association

BD	base detonating
BD	bomb disposal
BD	Fr propellant contg DPhA
B/D	bulldozer
BDS	Bomb Disposal Squad, now called EODT
BDSA	Business and Defense Service Administration (Commerce Dept, Washington 25, DC)
BDU	bomb disposal unit
BE	base ejection(chemical shell)
Bé	Baumé
Be	beryllium
BEAIRA	British Electrical and Allied Industries Research Association
Belg	Belgium, Belgian
Bellites	older Swed expls
benz	benzene
Berger(explosifs)	Fr expls(see the text)
BESA	British Engineering Standards Association
BESS	base ejection smoke shell
Bess	Bessemer
BethStCorp	Bethlehem Steel Corp
betw	between
BF	barrage fire
BF	before firing(Brit)
BF	poudre B fusil (Fr NC propellant for military rifle
BFF	Beretta Francese e Figli (Italfirm)
BFNL,	Fr military rifle propellants
BFP,BFP_1	(see the text)
BG	Birmingham gauge, for wire
BG, BG_4 & BGC	Fr propellants (see the text)
BG or BlG	blasting gelatin
BGOD	Blue Grass Ordnance Depot, Richmond, Ky
BGR	bombing and gunnery range
BH	Brinell hardness
BHC	benzene hexachloride
BHOD	Black Hills Ordnance Depot, Igloo, SDak
BHP	British horse-power
BHRA	British Hydromechanics Research Association
BI	base initiating; base ignition
Bi	bismuth
BI	Board of Investigation
BIB	baby incendiary bomb
bibl	bibliography
BICERA	British Internal Combustion Engine Research Association
bioch	biochemical
biol	biological
BIOS	British Intelligence Objectives Subcommittee
BIPM	Bureau International des Poids et Mesures(Fr)(International Bureau of Weights and Measures)
BISC	British Intelligence Sub-Committee
Bisoflex 102	Brit for triethyleneglycol dicaprylate
BISRA	British Iron and Steel Research Association
BJ	brass jacket
BJSM	British Joint Services Mission, Washington 6, DC
bk or blk	black
BkPdr	black powder
BKhV	boyevoye khimicheskoye veshchestvo(Rus)(CWA)
BL	base-loaded (shell)
BL	breech loading (separate loading ammo with bagged propelling charge)
BL	Burnside Laboratory, E.I. DuPont de Nemours & Co, Penns Grove, NJ
bl	blue
Blastin	Swed expl(see the text)
BLC	base-loaded capped (shell)
bldg	building
blk	see bk
BLMRA	British Leather Manufacturers Research Association
bln	balloon
BM	breech mechanism
BM or BurMines	Bureau of Mines, Pittsburg,Pa
BM(poudres)	Fr Navy(marine) propellant
BMG	Browning machine gun
BMI	Battelle Memorial Institute
bmr	bomber
BMRC	British Manufacture & Research Co
BMT	British Mean Time
BMTS	see BurMines TS
BN	poudre nouvelle(Fr modified propellant)
BNA	British Naval Attaché
BNF	bomb nose fuze
BNF	poudre nouvelle, fusil(Fr modified rifle propellant)
BNFMRA	British Non-Ferrous Metals Research Association
BNL	Brookhaven National Laboratory, Upton, NY
BNO	British Naval Officer

BNP	Bureau of Naval Personnel
BOAC	British Ordnance Ammunition Corps
BOAC	British Overseas Airways Company
BO	blown out(Brit)
BOD	Base Ordnance Depot
BOD	biochemical (biological)oxygen demand (capacity of water to absorb oxygen)
BOD	Birmingham Ordnance District, Birmingham, Ala
BOD	Boston Ordnance District, Boston 10, Mass
Bol	Bolivia
Bolovon O	Austr liq expl (HNO_3 + m-DNB)
Bonit	Swed for expl contg RDX & TNT
BOP	Burlington Ordnance Plant, Burlington, NJ
Boronites	expl mixts of amatols with boron salts (suitable for press-loading of ammo) (see PATR 1292 and the text)
BOV	boyevoye otravliayoushcheiye veshchestvo(Rus)(War poison substance) (poison gas)(CWA)
BOV	brown oil of vitriol (tech sulfuric acid)
BOW	Badger Ordnance Works, Baraboo, Wisc
BOW	Base Ordnance Workshop
BP	base point
bp	boiling point
BP	Bolta Products, Lawrence, Mass
BP	boyevyiye pripacy(Rus) (ammunition)
BP	British Patent (see BritP)
BP	broneprozhigayushchii(Rus) (burn through armor) (shaped charge)
B-P	bullet-proof
BP-152(polvere)	brown powder used by Italians in cannons prior to invention of smokeless propellant
BPB	black powder bag
BPCVMRA	British Paint, Colour & Varnish Manufacturers Association
BPD(polvere)	Ital sporting propellant
BPP	black powder pellet
BPZ	bronebrozhigayushchii-zazhigayushchii(Rus) (shaped charge, incendiary)
Br or Brit	British
Br	bromine
Br431(polvere)	brown powder used in Ital Navy prior to the invention of smokeless propellant
Braz	Brazil
Brit	British
brg	bearing
brghd	bridgehead
BRL	Ballistics Research Laboratory, Aberdeen, Md
bm	brown
Bros	brothers
BrP or BritP	British Patent
BRRA	British Rayon Research Association
BrS	Brown & Sharpe Manufacturing Co (wire gauge)
BRS	Buildings Research Station (Brit)
BrStd	British Standard
BS	bomb sight
BS	Bureau of Standards (see NBS)
BS(poudre)	Fr propellant (see the text)
BSA	Birmingham Small Arms Co (Engl)
BSI	British Standards Institution
BSIR	Bibliography of Scientific and Industrial Reports (US Dept of Commerce)
BSIRA	British Scientific Instruments Research Association
BSO	broadside on impact of projectile
BSP(poudre)	Fr propellant (see the text)
BSRA	British Shipbuilding Research Association
BSS	British Standard Screen
BSS	British Standard Specification
BSWG	British Standard Wire Gauge
BSX	symbol for 1,7-diacetoxy-tetramethylene-2,4,6-trinitramine or 1,7-diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane
BSX	symbol for 2,4,6-trinitro-2,4,6-triazaheptane-1,7-diol diacetate
B-t	boat-tailed (bullet)
BTEU	bis-(trinitroethyl)urea
BTL	Bell Telephone Laboratories
BTNES	bis(trinitroethyl)-succinate

Btry	battery (4 guns)
BTS	Bellini-Tosi System (of radio direction)
BTTN or BuTTN	butanetriol trinitrate
BTU	Board of Trade Unit (kilowat/hour)
BTU	British Thermal Unit(s)
Bu or Bur	Bureau
Bu or but	butyl(normal)
BuA	Bureau of Aeronautics, Washington 25, DC
Bucks	Buckinghamshire(Brit)
BuGDN	butyleneglycoldinitrate
Bulg	Bulgaria
Bull	bulletin
Buna	Butadiene-Natrium (synthetic rubber)
BuOrd or BUORD	Bureau of Ordnance
BurMines	Bureau of Mines, Pittsburg, Pa
BurMinesTS or BMTS	Bureau of Mines Test Station, Bruceton, Pa
BuShips	Bureau of Ships
but alc	butyl alcohol
BW	Biological Warfare
BW(poudre)	Fr propellant (see the text)
BWC	board wood cellulose (Brit)
BWG	Birmingham Wire Gauge
BWRA	British Welding Research Association
BWW	Biological Warfare Weapons
Bz	benzoyl, C_6H_5CO-
BzH	benzaldehyde, C_6H_5COH
Bzl	benzyl, C_6H_5CH-
BzOH	benzoic acid, C_6H_5COOH

C

°C	degree centigrade
C	capacitance
C	capped
C	carbamite(Brit for centralite)
C	carbon
C	cellulose
C	centigrade
C	Commanding
C or Conf	confidential
C	constant
C(explosif)	Fr explosive (see the text)
C-2(polvere)	Ital propellant similar to Brit cordite MD
C-7	Ital sporting propellant
Ca	calcium
ca	cathode
CA	Chemical Abstracts
ca	circa (about, approximately)
CA	Coast Artillery
CA	Contra-aereo (Ital)(antiaircraft)
C/A	counter-attack
CA_1; CA_2	coton azotique 1 et 2 (Fr)(NC contg ca 12%N)(see the text)
CAA	Civil Aeronautics Admin
CAC	Coast Artillery Corps
CAD	Central Ammunition Depot
CADO	Central Air Documents Office, now ASTIA
Cal	Caliber (inside diameter of a weapon)
cal	length of a cannon in calibers
Cal	see Calif
cal	gram-calorie
Cal	kg-calorie (see kcal)
CAL	Cornell Aeronautical Laboratory
calc	calculate
calcd	calculated
calcg	calculating
calcn	calculation
Calif	California
CalTech, Caltech, or CIT	California Institute of Technology, Pasadena, Calif (see also CIT)
CAM	Centro Armamento Marinha (Rio de Janeiro, Brazil)
Cambs	Cambridgeshire, Engl
camf	camouflage
Can	Canada, Canadian
can	canister
cap	capital letter
Carbamite	Brit for centralite
CARDE	Canadian Armament Research and Development Establishment (formerly ARDEC)
Carib	Caribbean
Carlsonites	older Swed expls
cart	cartridge
CAS	Canadian Army Staff, Washington 8, DC
CASEE	Canadian Army Signals Engineering Establishment
Cavy	cavalry
Cb	columbium
CB	Construction Battalion (its members, during WW II were called "Seabees")
CBR	chemical, biological and radiological (warfare)

CC	collodion cotton
cc	cubic centimetes
CCA	Celanese Corp of America, New York 6, NY
CCAE	(Joint) Congressional Committee of Atomic Energy
CCL	Chemists Club Library, 50E 41st St, New York 6, NY
CCLI	Charlotte Chemical Laboratories, Inc, Charlotte, NC
Cd	cadmium
CDB	Companhia Dinamitos do Brasil (Rio de Janeiro, Brazil)
CDEE	Chemical Defence Experimental Establishment(Brit)
CDRD	Chemical Defence Research Department
Ce	cerium
CE	Corps of Engineers
CE	"Composition Exploding" (Brit for tetryl)
CEC	Companhia Explosivos Cheddite (Brazil)
CEF	Canadian Expeditionary Force (in France)
CEG	Chemical Engineering Group, London
CEI	Commissariat à l'Énergie Atomique (Fr Atomic Energy Commission)
Cellamite	Fr expl contg AN, NG, CC and cellulose
cemf	counter electromotive force
CENCO	Central Scientific Co, Chicago 13, Ill
centf	centrifugal
Centr	Centralite (US); carbamite (Brit)
Centralite TA	Belg AN expl (see the text)
CEPE	Central Experimental & Proving Establishment (Canada)
CERN	European Council for Nuclear Research
CETME	Centro de Estudios Tecnicos de Materiales Especiales (Span)
cf	compare with; refer to
CFA	Canadian Field Artillery
cf ante	compare above
CFE	Central Fighter Establishment (Brit)
cfh	cubic feet per hour
cfm	cubic feet per minute
cf post	compare after
cfs	cubic feet per second
cg	centigram
CG	Commanding General
CG	code name for phosgene gas (CWA)
CG-13,CG-14	Ital double base propellants (see the text)
CGS	centimeter-gram-second
CGS	Central Gunnery School
CGS	Chief of the General Staff
CGS	Coast Guard Station
CGWI	Corning Glass Works, Inc, Corning, NY
CH	Case-hardened
Ch	chapter
char	character, characteristic
Ch D'Aff	Chargé d'Affaires
Chakatsuyaku	Jap for TNT (Sanshoki-teruoru)
Chanayaku	Jap expl (see the text)
Chaoyaku	Jap expl (see the text)
Chauyaku	Jap for cyclotol
Cheddites	Fr, Ital & Swiss chlorate expls
chem	chemical
Chemico	Chemical Construction Corp, New York 1, NY
ChemSoc	Chemical Society, London, Engl
ChemWarf	See CW
chemy	chemistry
chge	charge
Chikkaen	Jap for lead azide
Chin	Chinese
Chishoki-anin	Jap expl (see the text)
chlf	chloroform
Chujo-kayaku	Jap for cordite
CI	cast iron
CI	Chemical Inspectorate (Brit)
CI	Colour Index (Society of Dyers and Colourists (Brit)
CIA	Central Intelligence Agency
CIBA	Chemische Industries Basel (Swiss)
CIGM	Chief Inspector for Gun Mounting(Brit)
CIL	Canadian Industries Ltd, Montreal, Canada
CINCAF	Commander in Chief of Allied Forces
CINCEUR	Commander in Chief (of the US Forces) in Europe
CIOS	Combined Intelligence Objectives Subcommittee
CIOS	Comité International de l'Organisation Scientifique (Fr)(International Committee of Scientific Organization)

CIT	See CalTech
CIT	Carnegie Institute of Technology, Pittsburgh, Pa
CIT/GAL	California Institute of Technology/Guggenheim Aeronautical Laboratory
CIT/JPL	California Institute of Technology/Jet Propulsion Laboratory
civ	civil
CK	code name for cyanogen chloride gas (CWA)
cl	centiliter
Cl	chlorine
CLR	Chemical Laboratory Report (Pic Arsn)
cm	centimeter
CM	chemical mortar
CM	court martial
CMA	Canadian Manufacturers Association
CMLC	Chemical Corps
CNES	Companhia Nacional Explosivos Segurança(Brazil)
CNQB	Companhia Nitro Quimica Brasileira(São Miguel, Brazil)
CNR	Canadian National Railways
CNRS	Centre National de la Recherche Scientifique(Fr) (National Center for Scientific Research)
CNS	desgn for chloroacetophenone + chloropicrin in chlf (CWA)
CNTB	choking, nose, tear and blister gases(CWS)
c/o	care of
Co	cobalt
CO	Commanding Officer
Co	Company
Co-bomb	cobalt bomb
COC	Combat Operations Center
COD	cash on delivery, collect on delivery
COD	Charleston Ordnance Depot, N Charleston, SC
COD	Chicago Ordnance District, Chicago 6, Ill
COD	Cincinnati Ordnance District, Cincinnati 2, Ohio
COD	Cleveland Ordnance District, Cleveland 14, Ohio
coef	coefficient
COFORD or C of ORD	Chief of Ordnance

C of R	center of rotation
Col	colonel
col	colorless
Colinite	Fr expl (see the text)
coll	collective
collab	collaborator(s)
collecn	collection
Colo	Colorado
combd	combined
combn	combination
combstn	combustion
CombZ	combat zone
Comdg	commanding
Comdr	commander, commadore
Comdt	commandant
coml	commercial
Comm	commission, committee
comn	communication
comp	composite
Comp A-1	Amer explosive compositions based on RDX (see the text)
Comp A-2	
Comp A-3	
Comp B	
Comp B-2	
Comp C	
Comp C-2	
Comp C-3	
Comp C-4	
Comp D-2	binding agent and desensitizer contg paraffin, NC & lecithin
compar	comparative
compd	compound
compl	complete
compn	composition
compon	component
comprsn	compression
compt	compartment
CON	cash on delivery
con	connect
CONARC	Continental Army Command, Fort Monroe, Va
conc	concentrate(verb)
concd	concentrated
concg	concentrating
concln	conclusion
concn	concentration
cond	conductor
condy	conductivity
conf or C	confidential
cong	congress
conj	conjugate
conn	connect(verb)
Conn	Connecticut
cons	consult

consg	consulting
const	constant
constg	consisting
constrn	construction
cont	contain, continue, container
contd	contained, continued
contg	containing
contl	continental
contn	continuation
contr	contract, contractor
contrg	contracting
CONUS	Continental United States
conv	convenient
Convn	Convention
Co-op	cooperation
Coopalite	Belg expl contg AN, TNT, NG & wood flour (see the text)
COORDBD	Co-ordinating Board
coordn	coordination
Co-P	copilot
COP	Cornhusker Ordnance Plant, Grand Island, Neb
cor	corrected
Cordites	Brit propellants
CORG	Combat Operations Research Group, Fort Monroe, Va
Cornw	Cornwall, Engl
Coronit	Swed for PA
Corp	corporation
corr	correspond
corrn	corrosion
cos	cosine
cosc	cosecant
COSSAC	Chief of Staff to Supreme Allied Commander
cot	cotangent (see also ctn)
COW	Cactus Ordnance Works, Dumas, Tex
COW	Cherokee Ordnance Works Danville, Pa
COW	Coventry Ordnance Works (Brit)
Cox	coxswain
COXE	Combined Operations Experimental Establishment
CP	candle power
CP	chamber pressure, chemically pure
CP	common pointed (solid pointed shell having low armor penetration performance) (Brit)
C/P or CP	concrete-piercing
C_p	constant pressure
CP	coton poudre (Fr) (nitrocellulose)
CP_1	Fr NC (12.96 to 13.4% N)
CP_2	Fr NC (11.7 to 12.2% N)
CP_3	Fr NC (11.5% N) (old designation)
C_p100	carbon pourcent (Fr) (% of C)
CPC	common pointed capped (shell) (Brit)
CPC	Coors Porcelain Co, Golden, Colo
Cpl	corporal
CPO	Chief Petty Officer
CPPA	Canadian Pulp and Paper Association
CPR	Canadian Pacific Railway
CPRL	Chemical and Physical Research Laboratories (Australia)
CPS	Combined Planning Staff
CP_2/SD	CP_2 sans disolvant (Fr) (CP_2 gelatinized by NG using no solvent)
CPVA	Chemisch-physikalische Versuchsanstalt, Berlin (see also CTR)
CPVC	chlorinated polyvinyl chloride
Cr	chromium
CR	complete round
CRA	complete round of ammunition
C of R	center of resistance or drag
C of R	commencement of rifling
Cresylite	code name of 2,4,6-trinitro-m-cresol
CrFol	crown folio (size of a book 9.5" × 15")
crge	carriage
CRH	caliber-radius-head (radius of curvature of the ogival part of a shell expressed in calibers)
crit	critical
crkc	crankcase
CRL	Chemical Research Laboratory (Brit)
CRS	Canadian Rocket Society
Crs	cresol
CrsBl	Cresol Blue
CRST	cold-rolled steel
cruc	crucible
cryst(s)	crystal(s); crystalline
crystd	crystallized
crystg	crystallizing
crystn	crystallization
Cs	cesium
CS	Chemical Society (Brit)
C/S	Chief of Staff
CS	Civil Service
CSC	cartridge short case
CSC	Central Scientific Co, Chicago 13, Ill
CSC	Civil Service Commission
CSC	Commercial Solvents Corp, Terre Haute, Ind and New York 16, NY
CSE	coefficient de self-excitation (Fr) (transmission of detonation by influence)

CSE	Commission des Substances Explosives(Fr)
CSE(*exposifs*)	explosives developed or approved by the CSE(eg 55-CSE-1948)(see the text)
CSG	*Combat Service Group*
CSIR	Council for Scientific and Industrial Research
CSIRO	Commonwealth *Scientific and* Industrial Research Organization(Australia)
CSP	cast steel plate
CSS	cast semi-steel
CST	central standard time
CSUSA	Chief of Staff, US Army
CSUSAF	Chief of Staff, US Air Force
C/T	controlled target
CTA	cyanuric triazide
CTC	carbon tetrachloride
CTF	Commander Task Force
ctge	cartridge
CTMTN	cyclotrimethylenetrinitramine (RDX)
ctn	cotangent(see also cot)
CTR	Chemisch-technische Reichsanstalt, Berlin
CTRA	Coal Tar Research Association(Brit)
CU	*Chicago University*, Chicago 37, Ill
CU	Cornell University, Ithaca,NY
cu	cubic
Cu	cuprum(copper)
CUA	Catholic University of America, Washington 17, DC
CuCTez	copper chlorotetrazole
cu cm	cubic centimeter
cu ft	cubic foot
cu in	cubic inch
cu m	cubic meter
cum	cumulative
cuμ	cubic micron
Cumb	Cumberland, Engl
cu mm	cubic millimeter
CUP	coefficient d'utilisation pratique(Fr for modified Trauzl test value,relative to PA taken as 100%)
curr	current
cu yd	cubic yard
cv	calorific value
C_v	*constant volume*
c veh	combat vehicle
CW	chemical war(fare)
CWA	chemical warfare agent
CWC	Curtiss-Wright Corp, Woodridge, NJ
CWRE	Chemical Warfare Royal Engineers(Brit)
CWS	Chemical Warfare Service
CWSA	Chemical Warfare Service Army
cwt	hundredweight(used to designate different guns of the same caliber by indicating their weight)
Cy	cyan
cycl	cyclic
Cyclonite	same as cyclotrimethylene-trinitramine (RDX)
Cyclotol	cyclonite + TNT
cyl	cylinder
CyOx	*symbol for tetrahydro-3,5-dinitro-1,3,5,2H-oxdiazine*
CZ	Canal Zone(Panama)
CZ	Combat Zone
Cz-Sl	Czecho-Slovakia

D

d	density(g/cc)
d	dextrorotatory
d	differential
D	when added to the designation of a Fr propellant, means that DPhA is used as stabilizer (eg BD, BFD, etc)
D	Dunnite, Explosive D or ammonium picrate
D-2	"desensitizer 2" (see Comp D-2)
DA	*decontaminating agent*
DA	delay(ed) action
DA	Department of the Army(formerly part of War Dept)
DA	Detroit Arsenal, Centerline, Mich
DA	direction action(point detonating fuze) (Brit)
DA	Divisional Artillery
DAB	delayed action bomb
DAD	Divisional ammunition dump
DADNPh	diazodinitrophenol
DAER	Dept of Aeronautical Engineering Research (Brit)
DAF	delayed action fuze
DAF	Dept of the Air Force (formerly part of War Dept)
DAI	direct action impact(fuze)
Dak	Dakota

DAM	delayed action mine
Dan	Danish
DANC	decontaminating agent, non-corrosive
DanP	Danish Patent
DART	Code name of an Amer missile
DAS	Direction of Armament Supply(Brit)
DASA	Defence Atomic Support Agency (formerly AFSWP)
DATNB	1,3-diamino-2,4,6-trinitrobenzene
DAV	Disabled American Veterans
DB	depth bomb
DB	dive bomber
DB	double barreled
DB	driving band(rotating band)
DBP or DBuPh	dibutylphthalate
DBT	Rus expl contg DNB & TNT
DBX	depth bomb explosive(contains AN, RDX, TNT & Al)(see also Minex)
DC	depth charge
DC or dc	direct current
DC	District of Columbia
DCA	défense contre avion(Fr)(anti-aircraft defense)
DCC	Dow Chemical Co, Midland, Mich
DCDA	dicyandiamide
DCDRD	Director of Chemical Defence Research & Development(Brit)
DCT	depth charge thrower
DD	Design Dept(Brit)
DD	Fr expls contg PA & DNPh (see also MBT)
DD 60/40	Fr expl contg 60/40-PA/DNPh
DPBSA	Dupont do Brasil Sociedade Anônima Industrias Químicas (Duperiol)
D-day	Beginning of the action day
DDNP	see DADNPh(diazodinitrophenol)
DEA	diethanolamine
Dec	December
dec or decomp	decompose
Dechema or DECHEMA	Deutsche Gesellschaft für Chemisches Apparatewesen, Frankfurt a/Main, Germany
decomp or dec	decompose
decompd	decomposed
decompg	decomposing
decompn	decomposition
decontn	decontamination
def	defence
defgr	deflagrates
defgrg	deflagrating
defgrn	deflagration
deg	degree; °
DEG	diethyleneglycol
DEGDN or DEGN	diethyleneglycoldinitrate
DEGMN	diethyleneglycolmononitrate
dehyd	dehydrate(d)
dehydn	dehydration
Del	Delaware
deld	delayed
delq	deliquescent
delvd	delivered
demo	demolition
DEMS	defensively equipped merchant ship
Denb	Denbigshire, Wales
Densites	older Belg mining expls (see the text)
dep	departure
Dep	depot
Dept	department
deptml	departmental
Depy	deputy
der	see deriv
DER	Destroyer Escort Radar (vessel)
Derbs	Derbyshire, Engl
deriv	derivative
derivn	derivation
descrpn	description
desgn	designation
desic	desiccator
Designolle	Fr expls(see the text)
destn	destination
DETA	diethylenetriamine
det(d)	determine(d)
detg	determining
detn	determination
deton	detonation; detonates
detond	detonated
detong	detonating
deton vel	detonation velocity
Dets	detachments
DEUCE	digital electronic universal computing engine
dev	device
devel	develope(s)
develt	development
devn	deviation
Devon	Devonshire, Engl
dext	dextrinated
dextro	dextrorotatory
DF or df	direct fire
DF	direction finder
DFR	Director of Fuel Research(Brit)
dftg	drifting
dg	decigram

DGGM	Director General of Guided Missiles(Brit)
dgnl	diagonal
D/H	direct hit
DGOF	Director, General of Ordnance Factories(Brit)
DGWRD	Director of Guided Weapons Research & Development (Brit)
DI	degradation increase (of cellulose)
Di	Ger & Swiss desgn of DNT
diag	diagonal
diam	diameter
Diamin	Ger for ethylenediamine-dinitrate (EDD)
dibas	dibasic
dicta	dictaphone
dictn	dictation
dicty	dictionary
Didi	Ger & Swiss desgn of DEGDN
diffc	difficult(ly)
diffr	difference
dig	digest
dil	dilute
dild	diluted
dilg	diluting
diln	dilution
dimin	diminution
dimn	dimension
Dimple	Deuterium Moderated Pile, Low Energy, Harwell, Engl
DIN	Deutsche Industrie Normen (German Industry Standards)
Dina	Ger for dinitronaphthalene
DINA	diethanolnitramine dinitrate
Dinamaito	Jap for dynamite
Dinitryl	code name for glycero-*a*-2,4-dinitrophenylether dinitrate
Dinol	designation for diazodinitrophenol
dir(d)	direct(ed)
DPrGcDN	dipropyleneglycol dinitrate
Dir	Director
dirn	direction
dis	dissolve(s)
Di-salt	dimethyl ammonium nitrate (see PATR 2510, p Ger 37)
disc	discount
discon	disconnect
discond	disconnected
discont(d)	discontinue(d)
disd	dissolved
Disol	Ger & Swiss desgn of DNAns
disp	dispersed
displ	displacement
dissoc	dissociate(s)
dissocd	dissociated
dissocn	dissociation
dist	distance
distd	distilled
distg	distilling
distn	distillation
Distr	district
Ditetryl or Octyl	code names for N,N'-(hexanitrodiphenyl)-ethylenedinitramine
Dithekite 13	code name for liq expl contg NB & nitric acid
div	divided
Divn	Division
divn	division
dk or drk	dark
dkg	dekagram
dkl	dekaliter
dkm	dekameter
DL	dead load
dlvd	delivered
dm	decimeter
DMWD	Dept of Miscellaneous Weapons Development (Brit)
DMXRD	Director of Materials & Explosives Research & Development(Brit)
DN	dinitro-
DN or Dn	Fr for DNN (dinitronaphthalene)
DNA	dinitroaniline
DNAcet	dinitroacetone
DNAns	dinitroanisole
DNB	dinitrobenzene
DNBA	dinitrobenzaldehyde
DNBAc	dinitrobenzoic acid
DNC	Dept of Naval Construction
DNCPB or DNCB	dinitrochlorobenzene
DNCPH or DNCH	dinitrochlorohydrin
DNCrs or DNC	dinitrocresol
DND	Dept of National Defence (Canada)
DNDAPh or DNDAP	dinitrodiazophenol
DNDMOxm or DNDMeOxm	dinitrodimethyloxamide (see also MNO)

DNDMSA or DNDMeSA	dinitrodimethylsulfamide
DNDPhA	dinitrodiphenylamine
DNEU or DNEteU	dinitroethyleneurea
DNF	dinitrofurane
DNG	diglycerindinitrate
DNG	code name for NG, contg diglycerindinitrate serving as an antifreeze
DNGcU	dinitroglycoluril
DNM or DNMe	dinitromethane
DNMeA or DNMA	dinitromethylaniline
DNN	dinitronaphthalene
DNN	dinitronaphthol
DNO	Directorate of Naval Ordnance
DNPF	bis(dinitropropyl)-fumarate
DNPh	dinitrophenol
DNPN	bis(dinitropropyl)-nitramine
DNPS	dinitropropylsuccinate (see the text)
DNPT	see DPT
DNPTB	dinitropropyl-trinitrobutyrate (see the text)
DNR	dinitroresorcinol
DNT	dinitrotoluene
DNX	dinitroxylene
DO	Defence Order
doc	document
DOD	Detroit Ordnance District, Detroit 31, Mich
DOFL	Diamond Ordnance Fuze Laboratory, Washington 25, DC
dom	domestic
Dom	Dominion
Donarit	expl contg AN, TNT, NG, CC & vegetable meal (see the text)
Dors	Dorsetshire, Engl
DOS	Dept of State
DOS	Director of Ordnance Services(Brit)
DOT	direct oxidation test
DOV	distilled oil of vitriol (96% H_2SO_4)
DOVAP	Döppler Velocity and Position (see also EXRADOP; KOTAR and UDOP)
doz	dozen
DP	deck-piercing
DP	displaced person
DP	degree of polymerization
DP	distribution point (for supplies)
DPA	see DPhA(diphenylamine)
DPB	deep penetration bomb (Brit)
DPE	dipentaerythritol
DPEHN	dipentaerythritolhexanitrate
DPG	Dugway Proving Ground, Utah
DPhA	diphenylamine
DPT or DNPT	dinitropentamethylenetetramine; 2,6-dinitro(bicyclo)pentamethylene-2,4,6,8-tetramine or 3,7-dinitro-1,3,5,7-tetraza-bicyclo[3,3,1] nonane
Dr	Doctor
dr ap	dram apothecaries (0.0355 deciliter)
dr av	dram avoirdupois (1.7718 gram)
DRB	Defence Research Board (Canada)
DRBC	Defence Research Board of Canada
DRCL	Defence Research Chemical Laboratories (Canada)
DRD	Design Research Division
DRD	Directorate of Research and Development (US Air Force)
DRF	Deutsche Rezeptformeln (German Pharmacopeia)
DRI	Denver Research Institute, Univ of Denver, Denver 10, Colo
drk	dark
DRKL	Defence Research Kingston Laboratory(Canada)
DRML	Defence Research Medical Laboratories(Canada)
DRNL	Defence Research Northern Laboratory(Canada)
drtr	dram troy
DS	discarding sabot
DSI	duration of sustained injection (Rocketry)
DSIR	Dept of Scientific and Industrial Research(Brit)
DSIR/TIDU	DSIR Technical Information and Documents Unit(Brit)
DSIS	Defence Scientific Information Service(Canada)
DSP	Direction du Service des Poudres(Fr)
DSR	Director of Scientific Research & Experiments Dept (Naval)
DST	daylight saving time
DST	double set trigger
DSWV	Directorate of Special Weapons and Vehicles(Brit)
DTM	Directorate of Torpedoes and Mines (Brit)
Dualines	older Swed expls(see the text)
duct	ductile
DUKW	"Duck" (amphibian vehicle)
Dumb	Dumbarton, Scotland

Dumf	Dumfried, Scotland
Dunnite	Amm picrate or Expl D
dupl	duplicate
duPont	E.I.duPont de Nemours & Co, Wilmington, Del
DV	dymoobrazuyushcheyiye veshchestvo(Rus)(smoke agent)
DVA	Department of Veterans Affairs
DVV	drobiashcheye vzryvchatoye veshchestvo(Rus) (brisant explosive)
D-wave	detonation wave
DWE	Director of Weapons and Equipment(Brit)
DWR(D)	Director of Weapons Research (Defence) (Brit)
dwt	pennyweight(1.55 gram)
Dy	dysprosium
dyn	dynamite
Dynamit F	Swiss dynamite with 65%NG
Dz	diazole
DZ	dropping zone

E

E	East
E	electromotive force
E	(suffix) denotes an experimental variation of an ordnance item
e	electron or its charge
E	energy
e	erg
E	Jap & Swiss explosives (see the text and PATR 907)
E	Young's modulus
ea	each
EA	Edgewood Arsenal, Md (see also ACC)
EAON	except as otherwise noted
EB	Encyclopedia Britannica
E-boat	enemy boat (torpedo)
EC	"Explosive Company" (Brit propellant invented in 1880) (see the text)
EC(Blank Fire)	propellant contg GC, Ba & K nitrate, starch, DPhA and Aurine)
ECARL	expandable cluster aircraft rocket launcher
Echos or Escho	Fr & Ital expl(see the text)
ECNR	European Council for Nuclear Research
ECPI	Eastman Chemical Products, Inc, Kingsport, Tenn
Ecrasite	Amm trinitrocresylate
ECS	Electrochemical Society
ed	editor
ED	effective dose
ED	electron device
edd	edited
EDD	ethylenediaminedinitrate
EDF	European Defence Force
edn	edition
EDNA	ethylenedinitramine(same as Haleite)
EDNATOL	EDNA+TNT
EDTA	ethylenediaminetetracetic acid
EDVAC	electronic discrete variable automatic calculator
EEI	Edison Electric Institute
EES	Engineering Experiment Station, Annapolis, Md
EF	effective fire
EF	Expeditionary Forces
EF	Fr propellant for blank fire
EFC	equivalent full charges (number of firings with full charges; a Brit term used to assess the life of a gun)
effl	effloresccence
effy	efficiency
EFM	Engineering Field Manual
EGDN	ethyleneglycoldinitrate
EHP or ehp	effective horsepower
EI	end of injection (Rocketry)
eject	ejector
EKC	Eastman Kodak Co, Rochester, NY
Ekrasit	Ger for Amm trinitrocresylate
EL	Eastern Laboratory, Gibbstown, NJ (DuPont Co)
elec or electr	electric(al)
elem(s)	element(s)
elevn	elevation
EIP	electric primer
eman	emanation
emf	electromotive force
EMFWK	Eidgenössische Munitionsfabrik und Waffenkontrolle, Altdorf (Swiss Govt Munition Plant and Arms Inspection)

emgcy	emergency
EMMET	ethyltrimethylolmethane trinitrate or 1,1,1-trimethylolpropane trinitrate
Emp	Emperor, Empire
en	ethylenediamine(used in formulas only)
Encyc	encyclopedia
Energa	Belg A/T rifle grenade
eng	engine
Engl	England
Engr	engineer
engrg	engineering
Engrs	Engineers(troops)
ENIAC	electronic numerical integrator and computer
Enl	enlisted
Ennayaku	Jap expl(see the text)
Ens	ensign
EO	"explosion only"(Brit)(a very inferior explosion insufficient to be classed as a low order detonation)
EOC	Elswick Ordnance Co, Subsidiary of Armstrong, Elswick, Engl
EOCD	Eastman Organic Chemicals Dept, Rochester 3, NY
EOD	Erie Ordnance Depot, Port Clinton, Ohio
EODT	Explosives Ordnance Disposal Team, formerly BDS
EOR	Explosives Ordnance Reconnaissance
EP	English Patent(see BrP)
EP	Ital initiator for shaped chge projs (original type)
EPA	European Producing Agency
EPF	electrical percussion fuze
EPF	Emery Paper Figure(Brit)
EPFW	Eidgenössische Pulverfabrik in Wimmis(Bern)(Swiss Govt Powder Plant)
epm	explosions per minute
EPS	Ital initiator for shaped chge projs (improved type)
eq	equal
eqn(s)	equation(s)
EqS	equivalent to sheathed (explosives)
equil	equilibrium
equim	equimolecular
equiv	equivalent
Er	erbium
ERA	Engineering Research Association
ERAMA	Enfield Royal Arms Manufacturing Arsenal, England
ERDC	Evans Research & Development Corp, New York 17, NY
ERDE	Explosives Research & Development Establishment, Ministry of Supply, Waltham Abbey, Ess, Engl
ERDL	Engineering Research & Development Laboratories, Fort Belvoir, Va.
EREC	Esso Research & Engineering Co, Linden, NJ
ERETS	Experimental Rocket Engine Test Station
ERG	Explosives Research Group, Utah Univ
ERI	Engineering Research Institute, Univ of Michigan, Ann Arbor, Mich
ERL	Explosives Research Laboratory, Bruceton, Pa(existed during WW II)
ERRL	Eastern Regional Research Laboratory
ErTeN	erythritol tetranitrate
ESA	Explosivos Sociedad Anónima (Lurin, Peru)
Escho	same as Echo
ESP	end of sustained pressure (Rocketry)
esp	especially
Ess	Essex, Engl
EST	Eastern standard time
est	estimate(verb)
estb(d)	establish(ed)
estd	estimated
estg	estimating
estn	estimation
esu	electrostatic unit
Et or et	ethane
Et or et	ethyl(C_2H_5)
et al	*et alii*(Lat)(*and others*)
et acet	ethyl acetate
et alc	ethyl alcohol
ete or et	ethylene
eth	ether
Ethyltetryl	N,2,4,6-tetranitroethylaniline-
etl	ethanol
ETOUSA	European Theater of Operations, US Army

et passim	"and here and there"
Ets	Établissement(Fr)(Firm)
et seq	et sequentia (Lat) (and the following)
ETT	Explosion Temperature Test
EU	effect utile, also called travail pratique (see the text)(Fr)
Eu	europium
Eur	Europe
ev	electron volt(s)
evac(d)	evacuated(d)
evap(d)	evaporate(d)
evapg	evaporating
evapn	evaporation
evoln	evolution
EWD	Economic Warfare Division
EWT	Eastern winter time
ex(s)	example(s)
Ex	a Greek prefix meaning "out of"
exam(d)	examine(d)
examg	examining
examn	examination
Exc	Excellency
exc	excellent
exch	exchange
excl	exclusive
excld	excluded
Exogene	Ital for cyclonite
exp	exponent
expl	explode(s)
expl(s)	explosive(s)
expld	exploded
Expl D	ammonium picrate
explg	exploding
expln	explosion
expr	exploder
expt	experiment
exptl	experimental
ext	external
extr	extract
EXTRADOP	Extra Döppler (cf DOVAP)
extrd	extracted
extrg	extracting
extrn	extraction

F

°F	degree Fahrenheit
F	Farad
f	fathom(182.5cm)
F	February
F	fellow;member of an association
f	female
F	field
F	fleet
F	fluorine
f or ft	foot
F	force
f	force spécifique (Fr)(specific force)
f	frequency
f	fugacity
F	fugasnyi(Rus)(of great heaving effect)
F or Fz	fuze
FA	Field Artillery
F/A	fighter aircraft
FA	fixed ammunition
FA	Frankford Arsenal,Phila,Pa
F/a	fuel-air(ratio)
FAC	Federal Atomic Commission
FAD	Field Ammunition Depot
FADC	First Aid and Decontamination Center
FAdm	Fleet Admiral
FAFM	Field Artillery Field Manual
FAIF	Fabbrica Automobili Isotta-Fraschini (Ital)
FAM	fast air mine
FAN	first aid nurse
FAP	First Aid Post
FAP	forward ammunition point
FAS	forward area sight
FASP	final average sustained pressure(Rocketry)
fath	fathom(182.5cm)
Favier(explosifs)	Belg & Fr expls (see the text)
FB	fighter bomber
FB	flying boat
FB	fragmentation bomb
FB, FB-1	Ital solventless propellants
FBB	free balloon barrage(Brit)
FBI	Federal Bureau of Investigation
fbp	final boiling point
FC	Fort Custer, Mich
FC-4(polvere)	Ital solventless propellant
FCC	Federal Communications Commission
FCDA	Federal Civil Defence Administration
FCO	firing control order
FCS	Fellow of Chemical Society(Brit)
fcst	forecast
fcty	factory
FD	Field Depot
FD	Fire Department

FD	fuze delay
FDC	firing data computer
fdg	fading
f dr	fluid drachm(0.00355 l)
F-drive	front drive
Fdry	foundry
FE	Fábrica de Estrela(Vila Inhomerin, Brazil)
FE	Far East
Fe	ferrum(Lat)(iron)
FE	Fleet Engineer
Fe, Fe 2, Fe 3, Fe 4	Ital double base propellants (see the text)
Feb	February
Fed or Fedl	Federal
FEMW	Field Engineering and Mine Warfare(Brit)
FF	flying fortress
ff	and the following pages
FG	field gun
FG	fog gun
FGAN	fertilizer grade ammonium nitrate
FH	foghorn
FHA	Federal Housing Administration
Fi, fi	fighter
FI	Figure of Insensitiveness (Brit) (see the text)
fi	for instance
FI	Franklin Institute, Phila 3, Pa
FI	fuze, instantaneous
FIAT	Field Information Agency, Technical
FIAT	Fabbrica Italiana Automobili, Torino(Italy)
FID	fuze, instantaneous detonating
fig(s)	figure(s)
filt	filter
filte	filtrate
filtn	filtration
FIMC	Fabbrica Italiana Micce di Casale(Ital)
fin	finance
fisc	fiscal
FIU	Fighter Interception Unit
Fivolite	code name for tetramethylol-cyclopentanol pentanitrate or nitropentanol
Fivonite	code name for tetramethylol-cyclopentanone tetranitrate or nitropentanone
fl	fluid
Fl	fluorine
FLA	First Lord of the Admiralty(Brit)
Fla	Florida
Flak or FLAK	Flugabwehrkanone(Ger)(AA cannon)
flam	flammable
Flammivore	Belg safety expl (see the text)
flex	flexible
Flg	Flagship
flge	flange
floc	flocculent
fl oz	fluid ounce(0.02957 l) in USA and 0.02841 l in GtBrit)
FLP	Fábrica Lusitania de Pólvora (Portugal)
fl p	flash point
flshls	flashless
flwg	following
FM	Field Manual
FM	Field Marshall
FM	force-majeure(Fr) (disaster)
FM	frequency modulation
FM	fulminate of mercury(see MF)
FM	symbol for titanium tetrachloride(CWA)
FMP	full metal patched(bullet)
FN	flat-nosed
FN(fusil)	current Belg cal .30 rifle (see the text)
FNA	fuming nitric acid
FNAG	Fabrique Nationale d'Armes de Guerre, Herstal, Liége, Belgium
fnd	found
fndn	foundation
FNEA	Fábrica Naval de Explosivos, Azul (Argentina)
FNH	flashless, non hygroscopic (propellant)
FNMAL	Fábrica Nacional de Munições de Armas Ligeiras (Portugal)
fn p	fusion point
FNP	Ital expl contg AN, PETN & wax
FNP	Fábrica Nacional de Pólvora, near Mexico City
FNRL	Fixed Nitrogen Research Laboratory
FNV	Fábrica Nacional de Valladolid(Span)
FO	Foreign Office(Brit)
FOD	Field Ordnance Depot
folw	follow
folwg	following
Forcites	Belg & Swed expls(see the text)
forg(s)	forging(s)
Formit	mixt of MAN-salt, AN & Tri-salt (see PATR 2510, p Ger 52)
formn	formation
fort	fort, fortification

Fort	fortress
Fortex	older Fr AN expls (see the text)
FOSDIC	film optical sensing device for input to computers
FP	firing point
FP	fission product
FP or Fp	Füllpulver (Ger for filler)
FPEG	Fábrica de Pólvoras y Explosivos de Granada (Span)
FPL	Forest Products Laboratory
FPM	Fábrica de Pólvoras de Murcia (Span)
fpm	feet per minute
FPRL	Forest Products Research Laboratory (Brit)
fps	feet per second
FPV	Fábrica Presidente Vargas (Piquette, Brazil)
fr	franc (Fr)
Fr	France; French
Fractorites	Belg expls (see the text)
frag	fragile
fragm	fragment
fragn	fragmentation
FRB	Fire Research Board (Brit)
FREL	Feltman Research & Engineering Laboratories, Pic-Arsn, Dover, NJ
freqy	frequency
Fri	Friday
fricn	friction
FRITALUX	France, Italy and Benelux Countries
FrP	French Patent
fr p	freezing point
FRS	Federal Reserve System
FrT	Fragmentation Test
FrV	fragment velocity
fs	feet per second
FS	Faraday Society (Brit)
FS	Field Service (Brit)
FS	fin-stabilized
FS	fog siren
FS	US desgn for smoke-producing liq mixt of SO_3 & SO_3HCl (CWA)
FS	Foreign Service
fs	fuse
FSB	Federal Specification Board
FSB	Field Selection Board
FSC	Fisher Scientific Co, Pittsburgh 19, PA
FSD	Field Supply Depot
FSL	First Sea Lord (Brit)
FT	firing tables
FT	flame temperature
FT	flame thrower
ft	foot; feet
FTB	fleet torpedo bomber
FTC	Federal Trade Commission
ft-c	foot candle
FtD	Fort Dix, New Jersey
FTD	fuze time difference
FtKn	Fort Knox, Ky
ft-lb	feet pounds
ft-lb/min	feet pounds per minute
ft-lb/sec	feet pounds per second
ft/min	feet per minute
FtSam	Fort Sam Houston, Tex
ft/sec	feet per second
FtTh	Fort Thomas, Ky
fumg	fuming
Fus	fusilage
fusn	fusion
FUSAG	First United States Army Group
FVRDE	Fighting Vehicles Research & Development Establishment (Brit)
fut	future
FW	fog whistle
FYr	fiscal year
fz or F	fuze

G

G	gauge (pressure above atm)
G	gauss
G	Geiger
G	gheksoghen (Rus) (hexogen) (RDX)
G	ghil'za (Rus) (cartridge case)
G	glycerin, glycerol
g	grams(s)
G	granata (Rus) (grenade)
G	granata betonnoboynaya (Rus) (concrete-piercing projectile)
G	gun
G	Span single-base rifle propellant
Ga	gallium
Ga	Georgia
GAC	Goodyear Aircraft Corp, Akron 15, Ohio
GAFC	General Aniline & Film Corp, New York 14, NY
GALCIT	Guggenheim Aeronautical Laboratory of the California Institute of Technology, Pasadena, Calif

GAM	guided air missile
Gamsit	Swiss expl (see the text)
GAP	gun aiming point
GAP	gun aiming post
GAPA	ground-to-air-pilotless aircraft
GaR	Garand rifle
GAR	guided air rocket
Garr	garrison(Brit)
gas	gasoline
Gaub	gaubitsa(Rus)(howitzer)
Gaz	Gazette(Brit Govt publication
Gazz	Gazzetta (Ital)
GB	glider bomb
GB	See GtBrit(Great Britain)
GB	gunboat
GBC	green bag charge(used in Amer separate-loading ammunition)
Gc	glycol
GC	gun control
GC	guncotton
GCA	Geneva Convention Act
g-cal	gram-calorie
GCB	ground contamination bomb
GCC	Goodrich Chemical Co, Avon Lake, Ohio
g/cc	grams per cubic centimeter
GcDN	glycol dinitrate
GCL	Gibbs Chemical Lab, Harvard Univ, Cambridge, Mass
GCM	General Court Martial
GCRC	Goodrich Research Center, Brecksville, Ohio
GCT	General Classification Test
GcTNB	glycol trinitrobutyrate
Gd	gadolinium
GD	gelatin dynamite
GD	General Depot
GD	grenade discharger
GD	ground defence
GD1; GD2; GDII	Ital gelatin blasting expls contg NG
GDIM	same as above
GDN	see GcDN or NGc
GE	gas ejection
GE	General Electric Co
Ge	germanium
gelat	gelatinous
Gelatine-Aldorfit	Swiss expl (see the text)
Gelatin-Cheddite	Swiss chlorate expl(see the text)
gen	general
Gen	General
GEO	Ital gelatin blasting expl contg NG
geol	geological
GEOM	Ital same type of expl as GEO
Ger	German; Germany
g-g or GG	green-green(double star rocket)(AC sig
g gr	great gross(12 gross; 1728)
GH	gun howitzer
GHQ	General Headquarters
GI	Government issue(also nickname for an Amer soldier)
g/l	gram/liter
GL	grenade launcher
GL	gun limber
glac	glacial
GLEEP	Graphite Low Energy Experimental Pile
GLMC	Glen L. Martin Co, Baltimore 3, Md
Glos	Gloucestershire, Engl
GLR	General Laboratory Report (PicArsn)
GLTN	glycerinlactatetrinitrate
glyc	glycerol
GM	guided missile
GM	gun metal
G-Man	Govt man(FBI agent)
GMJ	gilding metal jacket (of a bullet)
GMLTeN	glycerin monolactate tetranitrate
g-mol	gram-molecule
gmv	gram molecular volume
gnd	ground
gnde	grenade
Gnr	gunner
GOC	General Officer, Commanding
Gomme(explosifs)	Fr gelatin dynamites
GOP	Gulf Ordnance Plant, Aberdeen, Miss
Gov	Governor
gov	governor(mechanical)
Govt	Government
GovtPrtgOff or GPO	Government Printing Office, Washington, DC
GP	general purpose
GP	Ital sporting propellant
GPA	Glycerine Producers Association, New York 17, NY
GPB	general purpose bomb
GPH	gallons per hour
GPO	General Post Office
GPO	See GovtPrtgOff
gr	grain(s)(0.0648gram)
gr	grey
gr	Gross(12 dozen=144)
GR	gunnery range
Grakrult	older Swed propellant (see the text)

gran	granular
GRE	ground radar equipment
GrF	grazing fire
Grf 88	Ger for PA
Grisou(dynamites)	Fr permissible dynamites
Grisonites; Grisoutines; Grisoutites	Fr & Belg permissible explosives (see the text)
grn	green
grnd	ground
Gs	gauss
GS	general service(Brit)
GS	General Staff
GS	gun sight
G-salt	nitroguanidine (see NGu)
GSC	green star, cluster
GSP	green star, parachute
G-suit	gravity suit
Gt	Great(as GtBrit)
GT	gun turret
GT'NB	symbol for ethyleneglycol-di-trinitrobutyrate(classified)
Gu	guanidine
guar	guarantee
Guate	Guatemala
Guer	Guernsey
GW	guided weapon
GWVA	Great War Veterans Association (of Canada)
Gy	gunnery
gyro	gyroscope

H

H	hardness
H	headquarters
H	henry
H	hexa
H	hotter variety propellants (Brit)
h	hour
H or How	howitzer
H	hydro-
H	hydrogen
H	code name for mustard gas
H_2	Jap expl known also as H_2-Kongo
H-6	Amer expl (classified)
H-8	double-base propellant
H-16	symbol for 2(or4)-acetyl -4(or 2),6,8-trinitro-2,4,6,8-tetrazanonane-1,9-diol diacetate
HA	Halstead Arsenal, Fort Halstead, Kent, Engl
HA	heavy artillery
ha	hectare
HA	high angle(for antiaircraft)
HAA	heavy antiaircraft artillery
HAB	high altitude bombing
HAC	Hague Arbitration Convention
HAC	Hughes Aircraft Co, Colver City, Calif
HACSIR	Honorary Advisory Council for Scientific and Industrial Research (Canada)
HADC	Holloman Air Development Center
HADN	hexamine dinitrate
Haensosan-bakuyaku	Jap expl(see the text)
Haishakuyaku	Jap expl(see the text)
Halelite	same as EDNA
Harb	harbor
Harrisite	same as Comp C-4
HB	hollow base (bullet)
H-bomb	hydrogen bomb
HBr	hardness, Brinell
HBRA	Howitzer Battery Royal Artillery (Brit)
HBX	high blast explosives(torpex type expls)
HC	hexachloroethane(smoke)
HC	high capacity(bomb)
HCC	Harshaw Chemical Co(see UCCC)
Hd	hogshead(238 1 in USA and 286.4 1 in GtBrit)
HD	Home Defence(Brit)
HD	horse-drawn
HD	symbol for Mustard Gas, distilled(blister gas) (CWA)
hdbk	handbook
He	helium
HE	high explosive
HEAP	high-explosive, armor-piercing
HEAT	high-explosive, antitank
HEBD	high-explosive, base detonating
HEC	Halstead Exploiting Centre(Brit)
HEDA	high-explosive delayed action
HEF	high-explosive, fragmentation (bomb)
HEH	high-explosive, heavy(projectile)
HEI	high-explosive, incendiary
HEIA	high-explosive immediate action
Heineiyaku	Jap for trinitrophenetole
HEI-T	high-explosive, incendiary with tracer

HELC	high-explosive, long case
Helv	Helvetica(Swiss)(adj)
HEP	high-explosive, plastic (corresponds to Brit HE/SH)
HEPL	High Energy Physics Laboratory
HEP-T	high-explosive, plastic with tracer
Heptryl	code name for 2,4,6-trinitrophenyl-trimethylol-methylnitramine trinitrate
Heref	Herefordshire, Engl
Herts	Hertfordshire, Engl
HES	Hercules Experiment Station, Woodale, Del
HES	high-explosive shell
HE/SH	high-explosive squashhead (Brit) (corresponds to Amer HEP)
HE-T	high-explosive with tracer
hex	hexagonal
HEX	high energy expls(US) (classified)
Hexa,Hexamine, Hexyl Urotropine	code names for hexanitro-diphenylamine(HNDPhA)
Hexa Hexamit or	Ger for hexanitrodiphenylamine Ger & Swiss expl contg TNT & HNDPhA(similar to Novit)
Hexanit	
Hexatonal	Swiss expl(see the text)
Hexogen	Ger & Swiss for cyclonite(RDX)
Hexógeno	Span for cyclonite (RDX)
Hexonite	Swiss expls contg RDX,NG & CC
Hf	hafnium
HF	harassing fire
HF	high frequency(3 to 30 megacycles/sec)
HFA	high frequency amplifier
HFC	high frequency current
HF/DF	high frequency direction finder
HFI	height finding instrument
HG	Hotchkiss gun(MG)
Hg	hydrargyrum(lat)(mercury)
HH	heavy hydrogen
H-hour	the time at which a planned operation is to begin(US); same as Brit Z-hour
HiC	high capacity
HiFi	high fidelity
H-ion	hydrogen ion
hist	historical
hi-volt	high voltage
H_2-Kongo	Jap expl(H_2 Mixture) (see the text)
HIHM	Hollandsche Industrie und Handel Maatschappij (Dutch)
hl	hectoliter
HM	His (or Her) Majesty's (Brit)
HM	hydrazine mononitrate
HMAC	House Military Affairs Committee
hmd	humid
HMF	His(or Her) Majesty's Force (Brit)
HMG	heavy machine-gun
HMG	His (or Her)Majesty's Government(Brit)
HMS	His(or Her) Majesty's Service (Brit)
HMS	His(or Her) Majesty's Ship (Brit)
HMSO	His(or Her) Majesty's Stationery Office (Brit)
HMT	see HMTeA
HMTD	see HMTPDA
HMTeA	hexamethylenetetramine
HMTPDA or HMTD	hexamethylenetriperoxide-diamine
HMX	His Majesty's Explosive (High Melting Explosive) (cyclo-tetramethylene-tetranitramine)
HN	"hotter than no flash" (Brit propellant)
HNAB	hexanitroazobenzene
HNG	hydrin-nitroglycerin(NG contg NSug as an antifreeze)
HNH	hexanitroheptane
HNCbl	hexanitrocarbanilide
HNDPh	hexanitrodiphenyl
HNDPhA	hexanitrodiphenylamine
HNDPhAEN	hexanitrodiphenylamino-ethylnitrate
HNDPhBzl	hexanitrodiphenylbenzyl
HNDPhGu	hexanitrodiphenylguanidine
HNDPhSfi	hexanitrodiphenylsulfide
HNDPhSfo	hexanitrodiphenylsulfone
HNDPhU	hexanitrodiphenylurea
HNEt	hexanitroethane
HNMnt	hexanitromannitol
HNO or HNOxn	hexanitrooxanilide
Ho	holmium
HO	Home Office(Brit)
HoC	hollow charge(same as SC)
HOCL	Hotchkiss Ordnance Co, Ltd, England
Holl	Holland
Holter	Span expl comparable to PBX
Holtex	Mil expl developed by Hispano-Suiza, Genève, Switz (its compn was not published)

Homocyclonite	same as HMX
homo-DPT	code name for 3,7-dinitro-1,5-endoethylene-1,3,5,7-tetrazacycloöctane
hon	honorary
horiz	horizontal
hosp	hospital
HOW	Holston Ordnance Works, Kingsport, Tenn
How or H	howitzer
h-p or h-press	high pressure
HP	hollow point(bullet)
HP or hp	horse power
HPAB	Hanssons Pyrotekniska Aktiebolaget(Swed)
HPC	Hercules Powder Co, Wilmington 99, Del
HPCC	Helenic Powder Cartridge Company at Daphni near Athens
hp-hr	horsepower-hour(s)
HQ	Headquarters
hr(s)	hour(s)
HR	humidity, relative
HRRL	Human Resources Research Laboratory
HS	US desgn for Mustard Gas and for chloroacetophenone & chloropicrin in chloroform (CWA's)
HS	high speed
HSC	hotter than "solventless carbamite propellant"(Brit)
HSL	high speed launch(Brit)
HSMS	high speed mine-sweeper (Brit)
HSS	high speed steel
HSSAB	Hispano-Suiza Sociedad Anónima, Barcelona(Span)
HSSAG	Hispano-Suiza Société Anonyme, Genève (Switz)
HT	half-tracked
HT	high tension
HTA	Amer expls(classified)
HTP	high test peroxide
HU	Harvard University, Cambridge, Mass
Hung	Hungary
Hunts	Huntingdonshire, Engl
hv	heavy
HV	high velocity or hyper-velocity
hv	high voltage
HV_2	Fr for polyvinyl acetate
HVAG	Holzverzuckerungs Aktiengesellschaft Domat-Ems(Swiss)
HVAP	hypervelocity, armor-piercing
HVAR	hypervelocity aircraft rocket
HVAT	hypervelocity,anti-tank
HVG	hypervelocity gun
HVTP	hypervelocity,target practice
HW	heavy weapon
hw	hectowatt
hwt	hundredweight (45.36kg in USA and 50.8kg in GtBrit)
Hwy	highway
Hx	hydroxy
hyd	hydrated, hydrolysis
hydr	hydraulics
hydrd	hydrodynamics
hydrs	hydrostatics
hydrx	hydroxide
hyg	hygiene
hygr	hygroscopic
hygry	hygroscopicity
Hyman	code name for N-nitro-N-methylglycolamide nitrate
hyp	hypothesis
HYPO	High Power Output Reactor
Hz	herz
Hz	hydrazo

I

I	impulse
I or Inc	incendiary
I	infantry
I or Inst	instantaneous
I	intensity of electric current flow
I	iodine
I or i	iso-(as applied to a type of organic compound)
I	Italy
IA	Indiana Arsenal, Charlestown, Ind
Ia	Iowa
IAAW&D	Inspector of Antiaircraft Weapons and Devices(Brit)
IAG	Industria ArmiGalesi(Ital)
IAR	Institute for Atomic Research
IASP	initial average sustained pressure(Rocketry)
IATM	International Association for Testing Materials
IAWR	Institute for Air Weapons Research

IB	incendiary bomb
IB	information bulletin
IBEN	incendiary bomb with explosive nose
IBHP	Institut Belge des Hautes Pressions, Bruxelles, Belg
ibid	ibidem(Lat) (in the same place)- used in repeating reference to the work last cited
ibp	initial boiling point
ICBM or IBM	intercontinental ballistic missile
ICC	Interstate Commerce Commission
ICI	Imperial Chemical Industries (Ltd) formerly Nobel Co(Brit)
ICIANZ	Imperial Chemical Industries of Australia & New Zealand
ICIL, Nobel Div	Imperial Chemical Industries, Ltd, Nobel Div, Stevenston, Ayrshire, Scotland
ICSU	International Council of Scientific Unions
ID or id	internal (inside) diameter
Ida	Idaho
IDR	Intelligence Division Report
ie	id est(Lat) (that is)
IE(balistita)	Span propellant
IEME	Inspectorate of Electrical and Mechanical Equipment(Brit)
IED	Industrial Engineering Division
ign	ignition
ignt	ignite(s)
igntg	igniting
IIT	Index of Inflammability Test(Brit)
Ill	Illinois
illumg	*illuminating*
IM	interceptor missile
IMk	identification mark
immed(y)	immediate(ly)
immisc	immiscible
imp(s)	impurity(ies)
imp	impulse
IMP	Industrial Mobilization Plan
IMP	initial maximum pressure (Rocketry)
imp gal	imperial gallon(4.54 l)
impr	improved
imprg(d)	impregnate(d)
imprt	important
IMR	*improved military rifle(Pdr)(Brit)*
in or "	inch
In	indium
inact	inactive
inc	incendiary
Inc	Incorporated
INCEP	interceptor
incl	inclusive
incld	included
inclg	including
Ind	India, Indiana
ind	indirect
ind	industrial
indef	indefinite
indvdl	individual
Inf or Infy	infantry
infl	inflammable
info or infn	information
infra	Lat prefix meaning "below" as in infrared of the invisible spectrum
in Hg	inches of mercury
inj	*inject*
injn	injection
in-lb	inch-pound(s)
inorg	inorganic
INS	Iodine number and saponification number
in/sec	inches per second
insol	insoluble
insoly	insolubility
Insp	*Inspector*
inspn	inspection
inst	instantaneous
Inst	Institute
InstFz	instantaneous fuze(Brit)(a percussion fuze with no delay action)
Instn	Institution
instrn	instruction
instru	instrument
int	internal
interch	interchangeable
intl	international
IN/TN	insoluble nitrogen to total nitrogen
intro or intrn	introduction
Io	Iowa
IOP	Iowa Ordnance Plant, Burlington, Iowa
IP	initial point
IPD	Ingersoll Products Div of Borg-Warner Corp, Kalamazoo, Mich
ipm	inches per minute
ips	inches per second
IR	infrared
Ir	iridium
IRBM	intermediate range ballistic missile
IRC	initial rate of climb

IRE	Institute of Radio Engineers
irreg	irregular
IS	impact sensitiveness
ISA	Ignifera Societa Anonima in Locarno-Minusio(Swiss) (see also ZSF)
ISC	Iowa State College
iso-Bu	iso-butyl
isom	isomeric
iso-Pr	iso-propyl
isoth	isothermal
Isp	specific impulse
ISTM	International Society for Testing Materials
ISWG	Imperial Standard Wire Gauge (Brit)
IT	infantry tank
ITB	infantry training bomb
Ital	Italian
Ital P	Italian Patent
IUPAC	International Union of Pure and Applied Chemistry

J

J	Ger symbol for iodine
J	joule
J or j	journal
J	Fr sporting propellant
JA	Joliet Arsenal, Joliet, Ill
JAAF	Joint Army-Air Force
Jan	January
JAN	Joint Army-Navy
JANAF	Joint Army-Navy-Air Force
Jap	Japanese
JapP	Japanese Patent
JATO	jet assisted take off, also called RATO or "booster rocket"
JF	jet fighter
JHU	Johns Hopkins University, Baltimore, Md
JHU/APL	Johns Hopkins Univ, Applied Physics Laboratory, Silver Springs, Md
JHU/ORO	Johns Hopkins Univ, Operations Research Office
JOP	Joint Operations Center
JP	jet propelled
JP-1, JP-2, etc	jet fuels(see the text)
JPC	Jet Propulsion Center
JPG	Jefferson Proving Ground Madison, Ind
JPL	Jet Propulsion Laboratory Pasadena 3, Calif
JPRS	Joint Publications Research Service
Jr	junior
JRDB	Joint Research and Development Board(US Army & Navy)
JS&TIC	Joint Scientific and Technical Intelligence Committee (Brit)
Jugo	See Yugo
Juinite	Fr code name for ethylene-bisurethane
juncn	junction
JUSMAPG	Joint United States Military Advisory & Planning Group
JUSMG	Joint United States Military Group
just	justice
juv	juvenile

K

°K	degree Kelvin
K	kalium(Lat) (potassium)
K	constant
K	Jap expl(see the text)
k	kilo = 10^3
K	knot
K-1 & K-2	Rus expls contg TNT & DNB
Kaipinites	Fr expls contg Amm perchlorate, Na nitrate and TNT or TNN
Kan	Kansas
KAPL	Knolls Atomic Power Laboratory
Karitto	Jap black powder(see the text)
Kasshokuyaku	Jap brown powder(see the text)
KC	Kellog Company, Jersey City, NJ
kc	kilocycle(s)
kcal or Cal	kilocalorie(s)
KDNBF	potassium dinitrobenzofuroxan
Keyneyaku	Jap for trinitrophenetole
kg	kilogram(s)
kg/cu m	kilograms per cubic meter
Kg-m	kilogram-meter
kg-m/s	kilogram-meter per second
kg/sq m	kilograms per square meter
KGC	Kontes Glass Co, Vineland, NJ
KhF	khimicheskii fugass(Rus) (chem land mine)
Kibakuyaku	Jap initiating expl(see the text)
Kibakuzai	Jap primer or percussion charge
kj	kilojoule
kl	kiloliter

KLG	Kimble Laboratory Glassware, Toledo 1, Ohio
km	kilometer
kMc/s	kilomegacycles per second (=1000 megaherz)
km/hr	kilometers per hour
KMOP	Kings Mills Ordnance Plant, Kings Mills, Ohio
km/s	kilometers per second
KNSFNV	Koninklijke Nederlandsche Springstoffenfabrieken Naamlose Venoodschap
Ko	Jap Amm perchlorate type expl contg ferrosilicon
Kokoshokuyaku	Jap black powder type expl
KOP	Kansas Ordnance Plant, Parsons, Kan
KOP	Kingsbury Ordnance Plant, LaPorte, Ind
Koshitsu	Jap expls (see also Shouyaku-koshitsu)
KOTAR	Correlated Tracking & Ranging (cf DOVAP & UDOP)
KOW	Kankakee Ordnance Works, Joliet, Ill
KOW	Keystone Ordnance Works, Meadville, Pa
KR	Kings Regulations(Brit)
Kr	krypton
KV	kapciul'-vosplamenitel' (Rus) (igniter cap)
kv	kilovolt(s)
kva	kilovolt-ampere
kw	kilowatt(s)
kw-hr	kilowatt-hour
Ky	Kentucky
KZ	Kraftzahl(see the text)

L

l	laevorotatory
l	liter(s)
L	elevated railroad
L	latun'(Rus)(brass)
/L or |L|	for Land Service(Brit)
L_1, L_2, L_3	Belg propellants(see the text)
La	lanthanum
LA	lead azide
LA	Light Artillery
LA	Los Alamos(N Mex)
LA	Los Angles(Calif)
LAA	light antiaircraft artillery
lab	laboratory
LaC	Lance-Corporal(Brit)
lachr	lachrymator
Lact ON	lactose octanitrate
LAG	light automatic gun
Landskrona	older Swed propellant
lang	language
LAOD	Los Angeles Ordnance District Pasadena, Calif
LASL	Los Alamos Scientific Laboratory, Los Alamos, NM
Lat	Latin
lat ht	latent heat
LB	light bomb
lb	pound(s)
lb ap	pound apothecary, see lb tr
lb av	pound avoirdupois(453.59g)
lb/cu ft	pounds per cubic foot
lb-ft	pound-foot(feet)
lb/HP	pound(s) per horsepower
lb-in	pound-inch(es)
LBM	lever of breech mechanism
lb/mol	pound molecule
lb/sq in	pound per square inch
lb tr	pound troy(373.2418g)
lb/yd	pound(s) per yard
LC	landing craft
LC	Library of Congress, Wash 25,
LC	light case (chemical)
LC	long case
LCA	Lake City Arsenal, Independe Mo
LCA	landing craft assault(ship)
LCB	light case (chemical) bomb(Br
LCG	landing craft, gun
LCG(M)	landing craft gun (medium)
LCR	landing craft, rocket
LCT	landing craft, tank
LCT(R)	landing craft, tank(rocket)
LCSE	Laboratorie de la Commission des substances Explosives(Fr
LCV	landing craft, vehicle
ld	laevo-and dextrorotatory
LD	lethal dose
LD	long delay
LD	long distance
LD50	killing 50% of subjects under
LD50/30	killing 50% of subjects under in 30 days
LD50 time	killing 50% of personnel after indicated time
LDNR	lead dinitroresorcinate
LDWTI	Lucidol Division, Wallace & Tiernan Inc, Buffalo 5, NY
LDT	long distance telephone

LE	Lee-Enfield(rifle) (Brit)
LE	low explosive
LEC	Laboratory Equipment Corp, St Joseph, Mich
Leics	Leichestershire, Engl
LEWCF	Lanza Elektrizitätswerke und Chemische Fabriken Aktiengesellschaft (Basel)
LF	land forces
LF	low frequency(30 to 3000 kilocycles per second)
lft(s)	leaflet(s)
LG	Lewis gun(Brit)
lg	logarithm
lgt	light
lg t	long ton (Brit) (1016.05 kg or 2240 lb)
lgth	length
l/hr	liter(s) per hour
l-hr	lumen-hour(s)
LI	Lefax Inc, Phila 7, Pa
LI	light infantry
Li	lithium
LIAB	Lindesberg Industries Aktiebolaget(Swed)
Lieut or Lt	Lieutenant
ligr	ligroin
liq	liquid
Lithofracteurs; Lithotrite	older Belg expls(see the text)
LLA	Lend-Lease Administration
LLA	low level attack
LM	land mine
lm	lumen
LMG	light machine-gun
lmt	limit
LN	liquid nitrogen
LN	long nose
ln	logarithm, natural(also $\log_e$)
loc cit	loco citato(Lat) (in the place cited)-used when several footnotes intervene betw two citations not only to the same work, but also to the same place in that work
LOD	Letterkenny Ordnance Depot, Chambersburg, Pa
LOD	Lima Ordnance Depot, Lima, Ohio
LOD	low order detonation
LOF	line of fire
log	logarithm
$\log_e$	see ln
Logs	logistics
LOMC	Lenape Ordnance Modification Center, Newark, Del
Lond	London
LOP	Louisiana Ordnance Plant, Shreveport, La
LORAC	long range accuracy(system of radio navigation)
LORAN	long range navigation
LOW	Longhorn Ordnance Works, Marshall, Tex
LOX	liquid oxygen explosives (oxyliquits)
lp	liquid propellant(Rocketry)
	Lyddite
LP	Livens projector
LP(poudre)	Belg propellant(see the text)
LPC	Liberty Powder Co, Mt Braddock, Pa(Olin Industries)
LPIA	Liquid Propellants Information Agency
lpw	lumens per watt
LR	long range
LRB	long range bomb
LRG	long range gun
LRL	Livermore Research Lab
LRPG	Long Range Proving Ground
LRWE	Long Range Weapons Establishment(Brit)
LSOP	Lone Star Ordnance Plant, Texarkana, Tex
LST	landing ship tanks
LSt	lead styphnate
Lt	See Lieut
Lt,lt	light
LtAA	light antiaircraft artillery
Ltd	Limited
LTk	light tank
LTRS	Low Temperature Research Station(Brit)
Lu	lutetium
lubr	lubricant, lubrication
Lux	Luxemburg
LVD	low velocity dynamite
LVT	landing vehicle tracked
l/w	lumens per watt
Lyddite	Brit for cast PA
LZ	landing zone

M

M	Mach number
M	Manual
M	Mark(model)

M	followed by a number(as M2) signifies a standardized Ordn item (Roman numerals as MII are used by the Brit)
m-	meta(position)
M	symbol for a metal(M^{II} means a divalent metal, etc)
m	meter(s)
m	mile
m	milli(1/1000)
M	mine
M	minomët(Rus) (mine thrower)
M	molar (as applied to concentration)(not molal)
M	Mono-
M	mortar
M	mortira(Rus)(mortar)
μ	mu(Greek letter)-meso-position, micro(1 millionth of a unit); micron
M	multitubular propellant(Brit)
M, M_4, M_6, M_8, & M_{10}	Ital propellants contg metriol trinitrate
Ma	masurium
MA	medium artillery
MA	Milan Arsenal, Milan, Tenn
ma	milliampere(s)
mÅ	milliångström
MA	Military Attaché
MA	Ministry of Aviation (Brit)
MA	mountain artillery
MABT	Fr & Ital expl contg PA, TNT & DNPh
macarite	Belg expl(see the text)
mach	machine
machy	machinery
Macmillan	The Macmillan Co, NY
MADAEC	Military Application Division of the Atomic Energy Commission
MAEE	Marine Aircraft Experimental Establishment(Brit)
mag	magazine
mag	magnet
magn	magnitude
Maj	Major
Maj-Gen	Major-General
Man	Manitoba, Canada
Man-Salt	methylamine nitrate (see PATR 2510, p Ger 108)
manuf	manufacture
manufd or mfd	manufactured
manufg or mfg	manufacturing
MAP	Ministry of Aircraft Production (Brit)
Mar	March
Mar	marine
mart	martial
MAS	Military Agency for Standardization
MASB	motor anti-submarine boat
masc	masculine
Mass	Massachusetts
MAT	Fr, Ital & Jap expl contg PA & TNT
mat	material
Matagnites	Belg expls (see the text)
math	mathematical
maths	mathematics
Matsu	Jap for blasting gelatin
max	maximum
max	metal and explosive (mixture giving maximum performance for each metal and explosive system)
max cap	maximum capacity
MB	medium Besa(Brit machine gun)
MB	medium bomber
mb	millibar
MB	monoblock
MB	motor boat
MB	mountain battery
MB	Fr & Ital sporting propellant
mbl	mobile
MBT	Ital expl contg PA & DNPh(see also DD)
MC	machine carbine
MC	medium capacity
mc	megacycle
mc	millicurie
MC	motor car
MC(Cordite)	modified cordite contg cracked mineral jelly(Brit)
MCA	Manufacturing Chemists' Association, Washington 5, DC
MCB	Matheson, Coleman & Bell, Norwood, Ohio
MCB	medium capacity bomb
MCC	Monsanto Chemical Co, St Louis, Mo
McGraw-Hill	McGraw-Hill Book Co, Inc, NY
MCI	Merck & Co, Inc, Rahway, NJ
Md	Maryland
MD	mean deviation
MD	Medical Doctor
MD	military district
MD	mine depot, mine detector
MD(cordite)	modified cordite(Brit)
MDN or MDn	mélinite-dinitronaphthaline (Fr expl contg PA & DNN)
MDPC	mélinite-dinitrophénol-crésylite (Fr expl contg PA,DNPh & TNCrs)

MDW	Military District,Washington,DC
Me or me	methyl
ME	military engineer(ing)
me	milliequivalent
ME	muzzle energy
MeAN	methylamine nitrate
MEC	Metalab Equipment Co, Hicksville, LI, NY
mech	mechanical
Med	medicine, medical
Med	medium
MedArty	Medium Artillery
MEDINA	methylenedinitramine
MeEDNA	N-methylethylenedinitramine
Mégadyne	Belg expl(see the text)
Meiayaku	Jap for tetryl
Mel	melamine
Mélanite	Belg expl(see the text)
Melinit	Ger & Rus for PA
Mélinite	Fr for PA
Mem	Memorandum
Mém	Mémorial
Mems	Memoirs
MeN	methyl nitrate
MeNENA	l-nitroxytrimethylene-3-nitramine
Menkayaku	Jap for NC(shokamen)
Menyaku	Jap for guncotton
MeOr	methyl orange
mep	mean effective pressure
meq	milliequivalent
MeR	methyl-red
Meri	Merionetshire,Wales
MERL	Mechanical Engineering Research Laboratory(Brit)
Mes	mesitylene
Messrs	Messieurs(Fr)
met	methane
mete	methylene
meth	method
methanol	methyl alcohol
Metilites	liquid expls used in mine clearing
metlrg	metallurgical
M et R	Fr sporting propellant
Metr	metriol
MetrTN or MTN	metriol trinitrate
mev	million electron volts
MEXE	Military Engineering and Experimental Establishment(Brit)
MF	medium frequency(300 to 3000 kilocycles per second)
MF	mercuric fulminate
mf	millifarad
μf	microfarad
MFA	Manual of Field Artillery
mfd or manufd	manufactured
MFDN	Fr expl contg PA & DNN
mfg or manufg	manufacturing
MFU	mobile floating unit
MG	machine gun
Mg	magnesium
mg	milligram(s)
M-gas	motor gasoline
MGB	machine gun belt
MGB	motor gunboat
mhy	millihenry
mi	mile
MI	Military Intelligence
Mich	Michigan
microsc	microscopic
Midx	Middlesex, Engl
mi/hr	miles per hour
MIIR	Mellon Institute of Industrial Research
MIL or Mil	military
min or minim	minimum
min(s)	minute(s)
Minérite	Belg expl(see the text)
Minex	expl contg Amm carbonate RDX, TNT & Al
Minite	Belg expl(see the text)
Minn	Minnesota
Minol	expl contg AN, TNT & Al
Minolex	expl contg AN, RDX, TNT & Al
Miny	Ministry
misc	miscellaneous
misci	miscible
Miss	Mississippi
MIT	Massachusetts Institute of Technology
mixt	mixture
MJ	metal jacketed(bullet)
MJ	mineral jelly(Brit for vaseline)
Mk	Mark(used by the Brit with a Roman numeral to designate a model as MkI) Amer practice is to use an Arabic numeral
Mk1 (cordite)	original Brit cordite(see the text under Cordite)
Mk2	Jap expl (same as Nigotanyaku Mk2)
m-kg	meter-kilogram(s)
ml	milliliter(s)
ML	motor launch

ML	muzzle loader, muzzle loading (in mortars)
Mlt	maltose
MltON	maltose octanitrate
mm	millimeter(s)
mμ	millimicron
mM	millimole
m/min	meters per minute
MMMC	Minnesota Mining & Manufacturing Co, St Paul, Minn
MMN or MMn	mélinite-mononitronaphthaline (Fr expl contg PA & MNN)
Mn	manganese
MN	mononitro
MN or Mn	Fr for MNN
MNA	mononitroaniline
MNAns	mononitroanisole
MNB	mononitrobenzene
MNBA	mononitrobenzaldehyde
MNBAc	mononitrobenzoic acid
MNCrs	mononitrocresol
MNDT	Fr & Ital expl contg AN, DNN & TNT (same as Siperite)
MNM	mononitromethane
MMeA	mononitromethylaniline
MN or Mn	Fr for MNN(mononitronaphthalene)
Mnn	mannose
MNN	mononitronaphthalene
MnnHN	mannose hexanitrate
MNO	symbol for N,N'-dinitrodimethyloxamide (DNDMOxm)
Mnt	mannitol
MNT	mononitrotoluene
MntHN	mannitol hexanitrate
MNX	mononitroxylene
MO	Medical Officer
MO	Military Observant
MO	Mining Officer
Mo	molybdenum
MO	money order
mob	mobile
mobn	mobilization
Mod	Model
modifd	modified
Modifn	Modification
M of F	method of filling(Brit)
moist	moisture
mol	molecule, molecular, molar
mol-%	molar percentage
mol compd	molecular compound
mol ht	molecular heat
mol wt	molecular weight
Mon	Monday
Mon	Montana
MONAB	Mobile Operational Naval Air Base(Brit)
monocl	monoclinic
monogr	monograph
Mons	Monmouthshire, Engl
Montgom	Montgomeryshire, Wales
MOP	Michoud Ordnance Plant, New Orleans, La
MOP	Muskegon Ordnance Plant, Muskegon, Mich
Mor or Mort	mortar
MOS	Ministry of Supply(Brit)
MOUSE	Minimum Orbital Unmanned Satellite of the Earth
movt	movement
MOW	Maumelle Ordnance Works, Little Rock, Ark
MOW	Morgantown Ordnance Works, Morgantown, WVa
MOX	expl mixts of metal,oxydizer & an expl(US)
MP	machine pistol
MP	Marine Police
MP	mélinite paraffiné (Fr)(paraffined PA)
mp	melting point
MP	Member of Parliament
MP	Metropolitan Police (London)
MP	Military Police
MP	Mounted Police
MP	multiperforated(propellant)
mpb	mean point of burst
mpg	miles per gallon
mph	miles per hour
MPI	mean point of impact(Brit)
mps	meters per second
MR	machine-rifle
mr	milliroentgen
MRBM	medium range ballistic missile
MRI	Midwest Research Institute, Kansas City, Mo
MRL	multiple rocket launcher
MROD	Mt Rainier Ordnance Depot, Tacoma, Wash
M/S	mine-sweeper
MS	Ministry of Supply(Brit)(now MA)
MSA	Military Service Act
MS/ERDE	Ministry of Supply, Expls Res & Development Establishment Waltham Abbey, Essex, Engl
MS or MSc	Master of Science
msec	millisecond

Msl	missile
MST	Fr & Ital expls contg AN, DNN & TNT (Same as Nougat)
MT	mean time(or proof of time fuzes) (Brit)
MT	mechanical time
MT	metric ton
MTA	metriol triacetate
MTB	motor torpedo boat
MTk	medium tank
MTN	metriol trinitrate
MTR	materials testing reactor
MTrk	motor truck
MTS	Malta Test Station, Schenectady, NY
MTSQ	mechanical time, superquick
MTTC	mélinite-tolite-trinitro-crésole (Fr & Ital expl contg PA, TNT & TNCrs)
MTX	mélinite-tolite-xylite (Fr & Ital expl contg PA, TNT & TNX)
mtzd	motorized
mu(μ)	micron(μ)
MU	University of Michigan, Ann Arbor, Mich
Muenkayaku or Muenyaku	Jap smokeless propellants
mun(s)	munition(s)
MunBd	Munition Board
mut	mutual
MUV	modernizirovannyi uproshchennyi vzryvatel' (Rus) (modern simplified pull fuze)
MV	mechanical vehicle
MV	methyl-violet
mv	millivolt(s)
MV	muzzle velocity
MVD	medium velocity dynamite
mw	milliwatt
mw	molecular weight
Myrol	Ger for methyl nitrate
MZD	meena zamedlennago deystviya(Rus) (delayed action mine)

N

N	National
N	nautical
N	Naval, Navy
N	in design of Fr expls or propellants indicates the presence of a nitrate such as AN, etc
N	nitro(see MN-mononitro)
N	nitrogen
N	"no-flash" propellant(Brit)
N	normal(as applied to concn)
n	normal (as applied to type of org compd)
N	North
n	noun
n	index of refraction (n_D at 20° represents n at 20° and Na light, line D)
/N; |N| or NS	for Naval Service(Brit)
N(explosif)	Fr AN expls(see the text)
N(poudre)	poudre noire(Fr)(black powder)
Na	natrium(Lat) (sodium)
NA	Naval Aviation
NAAI	North American Aviation, Inc, Downey, Calif
NAC	nitroacetycellulose
NAC	Ital propellants based on NAC
NACA	National Advisory Committee for Aeronautics(changed to NASA)
NACO	Navy cool propellants
NAD	Navy Ammunition Depot, Crane, Indiana
NADC	Naval Air Development Center
NAE(CAN)	National Aeronautical Establishment(Canada)
NAES	Naval Air Experimental Station
NAMC	Naval Air Material Center
NAML	Naval Aircraft Materials Laboratory(Brit)
NAMTC	Naval Air Missile Test Center, Point Mugu, Calif
NARTS	Naval Air Rocket Test Station,Lake Denmark, Dover, NJ
NASA	National Aeronautics and Space Administration, Washington, DC (formerly NACA)
NATC	Naval Air Test Center, Patuxent River, Md
NATO	North Atlantic Treaty Organization
Nauckhoff	Swed expls(see the text)
naut	nautical
Nav	Naval
navig	navigation
NavOrd; NAVORD	Naval Ordnance

Nb	niobium
NB	nitrobenzene
NB	Fr propellant contg NÉO
NBFU	National Board of Fire Underwriters
NBJK	nitrobenzène-jaune de potasse (Fr) (mixt NB + $K_4FeCy_6 \cdot 3H_2O$)
NBruns	New Brunswick, Can
NBS	National Bureau of Standards
NBSX	same as ATX
NBYA	symbol for bis(trinitroethyl) urea
NC	Naugatuck Chemicals, Naugatuck, Conn
NC	Nitrocellulose
NC or NCar	North Carolina
NC-82	symbol for bis(trinitroethyl) urea
NCB	National Coal Board(Brit)
NCh	normal charge
NCO	non-commissioned officer
NCRE	Naval Construction Research Establishment(Brit)
NCT	nitrocellulose, tubular
Nd	neodymium
ND	New Deal
ND	non-delay
ND or NDak	North Dakota
$n_D^{20°}$	index of refraction for line D of Na at 20°C
NDA	National Defence Act
NDAC	National Defence Advisory Committee
ND/ACC	Nitrogen Division, Allied Chemical Corp, NY 6, NY
ndls	needles
NDNT	Fr expl contg AN, DNN & TNT
NDRC	National Defence Research Council
Ne	neon
NE or NEngl	New England
NE	North-East
Neb	Nebraska
NEB	nuclear, electronic, biological
NEC	Nitrogen Engineering Corporation
NECL	Nobel's Explosives Co, Ltd, Stevenston, Scotland
neg	negative
NEL	Naval Electronics Laboratory
Nellite	Brit expl contg PA & DNPh
NENA	N-(2-nitroxy)-nitraminoethane
NENO	dinitrodi(β-nitroxyethyl)-oxamide
NÉO	Fr for coml DEGDN
NEPA	Nuclear Energy for Propulsion of Aircraft
neut	neutral
neutn	neutralization
Nev	Nevada
Newfld	Newfoundland
NF	National Formulary
NF	normal factor(concentration)
NF	night fighter
NF	nose fuze(bomb)
NFMC	National Filter Media Corp, Salt Lake City 10, Utah
NFOC	National Fireworks Ordnance Corp, West Hanover, Mass
NG	National Guard
NG	nitroglycerin
NGAB	Nitroglycerin Aktiebolaget, Gyttorp, Swed
NGc	nitroglycol
NGF	Naval gunfire
Ngl	Fr & Ger for nitroglycerin (NG)
NGTE	National Gas Turbine Establishment
NGU	See DNGcU
NGu	nitroguanidine
NH	New Hampshire
NH	non-hygroscopic
Ni	nickel
NIBTN	nitroisobutylglycerol trinitrate
NID	Naval Intelligence Department
Nigotanyaku Mk2	Jap for cyclotol
Nigu	Ger for nitroguanidine
Niperit (Nyperite)	same as PETN
NIP	nitroindene polymer
NiSt	nickel steel
Nitorogurisen	Jap for nitroglycerin
Nitramidon	Fr for nitrostarch
Nitramit	expl contg AN, TNT & Al
Nitramite	Ital expl contg AN, pitch & Al
Nitrocooppalite	Belg expl
Nitroferrite	Belg expl
Nitrogomme	Belg expl
	(see the text)
Nitrolit	60/40 TNAns/AN
Nitropenta	same as PETN
Nizol	Swiss Mil castable expl contg TNT & DNB
NJ	New Jersey
NJIC	National Joint Industrial Council(Brit
NK	Nobelkrut (see Bofors CoPropellants in the text)
NK1	Nobelkrut 1 (same as Ballistite) (see Bofors Co Propellants)

NK7	Nobelkrut 7 (see the text)
NL	Navy List
NlM	nautical mile
NLRB	National Labor Relations Board
NM	New Mexico
NMD	Naval Mine Depot, Yorktown, VA
NME	National Military Establishment
NMe	nitromethane
NNTP	symbol for 2-nitrimino-5-nitroxy-1,2-diazacyclohexane
NO	Naval Officer
NO	Naval Ordnance
no	not observed on firing reports (Brit)
No; no or #	number
Nobelit	Swed & Ger expls(see the text)
Nobélite	Fr plastic expl (see the text)
Nobel's 704	Brit expl contg AN, TNT & Al
Nobel-SGEM	Nobel-Società Generale di Esplosivi e Munizioni(Ital)
NOD	Navajo Ordnance Depot, Flagstaff, Ariz
NOD	Naval Ordnance Dept(Brit)
NOG or NsoGu	nitrosoguanidine
NOL	Naval Ordnance Laboratory, White Oak, Silver Spring, Md
NOMTF	Naval Ordnance Missile Test Facility, White Sands Proving Ground, Las Cruces, NM
Nom	nomenclature
nom	nominal
NOMP	Niskayuna Ordnance Modification Plant, Schenectady, NY
NOP	Nebraska Ordnance Plant, Wahoo, Neb
NOR	Naval Ordnance Research, Univ of Minnesota, Minneapolis, Minn
Normelit	Swed expl
Norw	Norway; Norwegian
NorwP	Norwegian Patent
Nos	numbers
NOTS	Naval Ordnance Test Station, Inyokern, China Lake, Calif
Notts	Nottinghamshire, Engl
notwg	notwithstanding
Nougat	same as MST
Nov	November
Novit	Swed underwater expl contg TNT & HNDPhA(similar to Hexamit)
NP	nitropenta(same as PETN)
NP	new pattern
NP	New Providence, Engl
NP	nickel-plated
NP	nitropenta(Fr) (PETN)
N/P	"no-flash/contg potassium sulfate (Brit propellant)
NP	non-persistent
NPA	National Planning Association
NPF	Naval Powder Factory, Indian Head, Md(changed to NPP)
NPG	Naval Proving Ground, Dahlgren, Va
NPI	National Petroleum Institute
NPL	National Physical Laboratory
NP.Mn 95/5	nitropenta(PETN) 95, MNN 5% (Fr explosive)
NPP	Naval Propellant Plant (formerly NPF)
NR	natural rubber
NR	Navy Regulations
NR	Fr AN expls resistant to water (see the text)
NRC	National Research Council (USA)
NRC(Can)	National Research Council (Canada)
NRC Compd	soln of NC, China wood oil & rosins in methyl acetate used in loading of ammo
NRL	Naval Research Laboratory, Belleview, Md
NROP	New River Ordnance Plant, Radford, Va
NRTS	National Reactor Testing Station, Arico, Idaho
NRX	National Research Experimental (Reactor)(Canada)
NS	new series; new system
NS	nitrate of sodium(Brit)
NS	nitrostarch
NS	North-South
NS	Nova Scotia, Can
NSA	National Service(Armed Forces)Act
NSA	naval small arms
NSC	National Security Council
NSF	National Science Foundation
Nso	nitroso
NsoGu	nitrosoguanidine
NSug	nitrosugar
NSX	Amer demolition expl contg NS, Ba nitrate, MNN, p-MNA and oil
Nt	nitron, now termed Rn
NT	nitrate-trotyl(Fr expl contg AN & TNT such as amatol)

NT	normal temperature
NTD	Naval Torpedo Depot
NTN	Fr expl contg AN & TNN
N2TN	Fr expl contg AN, Na nitrate & TNN
NTP	normal temp & pressure (0°C & 760 mm Hg)
NV	Naamlose Venoodschap (Dutch for Inc)
NV	nozzle velocity
NW	north-west
NWG	National Wire Gauge
n wt	net weight
NX	Fr expl contg AN & TNX
NY	New York
NYAS	New York Academy of Science
NYC	New York City
NYOD	New York Ordnance District, New York 14, NY (includes ROD)
Nyperite	same as PETN
NYU	New York University, New York 53, NY
NZ	New Zealand
NZAOC	New Zealand Army Ordnance Corps

O

O	Octa-
O	office
O	Officer
O	Ohio
O	order
O	Ordnance
o-	ortho
O	orúdiiye(Rus) (gun or cannon)
O	oskolochnyi (Rus) (fragmentation) (adj)
O	oxygen
O	Fr explosive(see the text)
OA	Ordnance artificer
OAC	Ordnance Ammunition Command Joliet, Ill
OAPC	Office of Alien Property Custodian
OB	observation balloon
OB	see OBd
OB	oxygen balance(OB to CO_2 or OB to CO expressed in %)
OBA	oxygen breathing apparatus
OBD	Ordnance Base Depot
OBd	Ordnance Board
Obr	Obrazets(Rus) (Model)
obsn	observation
obsol	obsolete
Obsy	observatory
obt	obtain
obtd	obtained
obtg	obtaining
O/C	Officer-in-Charge
OC	Ordnance Committee
OC	Fr chlorate type expls
occpn	occupation
occsly	occasionally
OCCWS	Office, Chief of the Chemical Warfare Service
OCD	Office of Civilian Defence
OCE	Office of Chief of Engineers Washington 25, DC
OCFGC	Owens-Corning Fiberglass Corp
OCO	Office, Chief of Ordnance Dept of the Army Washington 25, DC
OCO-ORDTA	OCO-Ammunition Branch
Oct No	octane number
Octol	Amer expl(classified)
Octyl	Brit for Bitetryl
OD	Officer of the Day
OD	olive drab
OD	Ordnance Department or Depot
OD or od	outside diameter
OEEC	Organization for European Economic Cooperation
OF	oskolochno-fugasnyi(Rus) (fragmentation with heaving action)
OF	oxidizing flame
OfA	offensive arms
OFD	Ordnance Field Depot
OFF	Office
Off	Officer
OFM	Ordnance Field Manual
OFS	Ordnance Field Service
OFSO	Ordnance Field Safety Officer, Jeffersonville, Ind
og	ogival
OGM	Office of Guided Missiles
OGMS	Ordnance Guided Missiles School, Redstone Arsenal
OHMS	On His(Her) Majesty's Service
OIS	Office of Information Services
OJCS	Office of Joint Chiefs of Staff
OK	all right
OK'd	approved
OKh	oskolochno-khimicheskii (Rus) (fragmentation-chemical)
Okla	Oklahoma
OL	Ordnance Lieutenant (Navy)

OMCC	Olin-Mathieson Chem Corp, East Alton, Ill
OLCr	Ordnance Lieutenant-Commander (Navy)
OME	Ordnance Mechanical Engineer
OMGUS	Office of Military Government of the United States
OML	Ordnance Missile Laboratory, Redstone Arsenal, Huntsville, Ala
Onayaku	Jap for trimonite
ONR	Office of Naval Research, Washington 25, DC, Chicago 11, Ill & Pasadena, Calif
Ont	Ontario, Canada
OO	Ordnance Officer
OOR	Office of Ordnance Research, Duke Univ, Durham, NC
OOW	Oklahoma Ordnance Works, Pryor, Okla
OP	observation post
OP	open point(SA Ammo)
OPA	Office of Price Administration
OPC	Ordnance Procurement Center, New York
op cit	opere citato(Lat) (in the work cited)-used when several footnotes intervene between two citations to the same work in the same chapter
OPD	Operations Department
OPDEVFOR	Operations Development Forces(Navy)
OPM	Ordnance Proof Manual
opn	operation
OPNAV or OpNav	Office of the Chief of Naval Operations
opp	opposite
opt	optical, optics
Opts	optical sight
OQMG	Office of the Quartermaster General
or or orn	orange
ORD	Office of the Chief of Ordnance
Ord; ORD or Ordn	Ordnance
ORDAmm Dept	Ordnance Ammunition Department
ORDB	Ordnance Board

Ordnance Field Service (Code names)

ORDFA	Ammunition Supply Branch
ORDFI	Requirements Branch
ORDFM	Maintenance Branch
ORDFQ	General Supply Branch
ORDFT	Operations Branch
ORDFX	Executive Branch
ORDFX-PM	Planning & Management Office
ORDGA	Office Service Branch
ORDGB	Ordnance Board
ORDGC	Ordnance Comptroller
ORDGL	Legal Branch
ORDGL-AD	Administrative Office
ORDGL-CS	Special Council for Contracts
ORDGL-ID	Special Council for Industrial Division
ORDGL-RD	Special Council for Research and Development Division
ORDGM	Management Office
ORDGN-SA	Intelligence, Security and Safety Office
ORDGX	Executive Office
ORDGX-H	Historical
ORDGX-OTL	Technical Liaison
ORDHO	Military Training and Organization
ORDI	Ordnance Industrial Division

Ordnance Industrial Division (Code names)

ORDIF	Facilities Branch
ORDIM	Ammunition Branch
ORDIP	Production Service Branch
ORDIR	Artillery Branch
ORDIS	Small Arms Branch
ORDIT	Automotive Branch

Ordnance Research and Development Branch (Code names)

ORDTA	Artillery Ammo Branch
ORDTB	Research & Materials Branch
ORDTQ	Ammo Development Branch
ORDTR	Artillery Development Branch
ORDTS	Small Arms Development Branch
ORDTT	Tank and Automotive Branch
ORDTU	Guided Missiles Branch
ORDTX	Executive Branch

Ore	Oregon
org	organic
org chem	organic chemistry
orgn	organization
orig	origin
ORINS	Oak Ridge Institute of Nuclear Studies
ORNL	Oak Ridge National Laboratory, Tenn
ORO	Operations Research Office

ORS	Operational Research Section (Brit)
ORS(India)	Operational Research Section, India
ORSORT	Oak Ridge School of Reactor Technology
Os	osmium
OSA	Office of the Secretary of the Army
OSA	Official Secret Act(Brit)
OSAF	Office of the Secretary of the Air Force
Oshitsuyaku	Jap desensitized RDX
Oshiyaku	Jap desensitized PA
Oshokuyaku	Jap for pressed PA
OSM	Ordnance Safety Manual
OSR	Office of Scientific Research
OSRD	Office of Scientific Research & Development
OSS	Office of Strategic Services
OSU	Ohio State University, Columbus 10, Ohio
OSWAC	Ordnance Special Weapons Ammunition Command, Pic Arsn, Dover, NJ
OT&AC	Oerlicon Tool & Arms Corp
OTAC	Ordnance Tank-Automotive Command, Detroit, Mich
OTAN	l'Organisation du Traité de l'Atlantique du Nord (French for NATO)
OTC	Ordnance Training Command, APG, Md
OTIA	Ordnance Technical Intelligence Agency, Arlington, Va
Otsu-B or A(ko)	Jap for Hexamit
OTS/USDC	Office of Technical Services, United States Dept of Commerce
OV	oil of vitriol (sulfuric acid)
OV	otravliayushcheiye veshchestvo (Rus) (toxic substance)(war gas)
OV(Propellant)	see the text
OWC	Ordnance Weapons Command, Rock Island, Ill
OWI	Office of War Information
ox	oxalate, oxalic
Oxam	oxamide
Oxan	oxanilide
oxid	oxidize
oxidn	oxidation
Oxonite	Belg expl (see the text)
Oxyliquit	liquid oxygen explosive (LOX)
oz	ounce(s) (28.35g)
Oz	Ozone
oz ap	apothecary's ounce see oz tr
oz av	ounce avoirdupois(28.31 g)
oz fl	ounce fluid(29.5737cc) (US)
oz fl	ounce fluid(28.4130cc) (Brit)
oz tr	ounce troy(31.1035g)

P

p	page
p-	para
p(s)	part (s)
P	partial detonation
P	Patent
P	penta
P	percussion
P	phosphorus
P	pistolet(Rus) (pistol)
P	plastic
P	podkalibernyi(Rus)(subcaliber)
P	potassium sulfate contg propellant(Brit)
p	pressure(absol)
P	publication
P	pushka(Rus)(gun or cannon)
P	Fr & Ital explosive (see the text)
P(salt)	symbol for piperazine dinitrate
P^1	symbol for methyleneglycol dinitrate
P^2	symbol for (methylenedioxy)-dimethanoldinitrate
PA	Pack Artillery
PA	Pan American
Pa or Penna	Pennyslvania
PA or Pic Arsn	Picatinny Arsenal
PA	Picric Acid
Pa	protactinium
PAA or PANAIR	Pan American (World) Airways
PAA	picramic acid
Pac	Pacific
PAC(R)	parachute and cable(rocket) (Brit)
PACLR	Picatinny Arsenal Chemical Laboratory Report
PAK or Pak	Panzerabwehrkanone (Ger) (antitank gun)
pam	pamphlet
PAM	Ital expl contg PETN
PAMETRADA	Parsons and Marine Engineering Turbine Research & Development Association(Brit)

PANA	Ital expl contg PETN
PANAIR or PAA	Pan-American(World)Airways
Panclastites	Fr & Brit liq expls contg liq N_2O_4 and liq fuels
par	paragraph
PAS	Philadelphia Astronautical Society
PAT	platoon anti-tank
pathol	pathological
PatOff	Patent Office
PAU	Pan-American Union
PB	pilotless bomber
Pb	plumbum(lead)
PB	Publication Board of Office of Technical Services(US)
PB_1; PB_2; PB_3(poudres)	prismatic brown powders used in Fr Naval guns prior to the invention of smokeless propellant
PBL	Publication Board L

Note: The L following PB means that the rept was housed and reproduced at the Library of Congress. The L no longer has any significance since all the PB repts are now available from the Photoreproduction Service, Library of Congress

PBM	patrol bomber Martin(flying boat)
PBOW	Plum Brook Ordnance Works Sandusky, Ohio
PB-RDX	Amer expl(classified)
PBRept	Publication Board Report of OTS
PBU or PhBU	phenylbenzylurethane(Brit gelatinizer for NC)
PBX	plastic bonded explosive (US)(composition is classified)
PC	Panama Canal
pc	per centum, %
PC	Pflaudler Co, Rochester 3, NY
PCC	Polverificio Comocini di Como (Ital)
pcf	pounds per cubic foot
pcpn	precipitation
PCX	symbol for 3,5-dinitro-3,5-diazapiperidinium nitrate
PCZ	Panama Canal Zone
Pd	palladium
PD	partial detonation(Brit)
PD	point detonating(fuze)
PDF	point detonating fuze
PDNA	propylene-1,2-dinitramine
Pdr	pounder(eg 18 Pdr; used to designate a gun firing a projectile weighing 18 lb)(Brit)
pdr	see powd
PDT	Plate Dent Test
PE	pentaerythritol
PE	plastic explosive
PE	point d'ébullition(Fr)(boiling point)
PE	Post Exchange
PEAP	Brit for pentaerythritol diacetate dipropionate
PEFL	Polverificio Esercito di Fontana Liri(Ital)
PEI	Prince Edward Island, Can
Pemb	Pembrokeshire, Wales
pend	pendulum
Penna or Pa	Pennsylvania
Pent	Pentagon Building, Washington, DC
pent	pentagonal
Pent	pentolite
Pentastit	Swiss expl contg PETN & pentaerythritol tetrastearate(PETS)
Penthrite	Brit for PETN
Pentolite	mixts of PETN & TNT
Pentoriru	Jap for pentolite
Pentralita	Span expl (see the text)
Pentrinit	Swiss expl contg PETN & NG
Pentro	see Pentryl (Swiss)
Pentrol	Ital expl contg PETN & TNT
Pentryl	β(2,4,6-trinitrophenyl-nitramino) ethyl nitrate
Pentryl(Swiss) or Pentro	expl mixt of PETN, TNT & Al manufd by a special process
Pentyl	Same as PETN
PEP	expls contg PETN & Gulf Crown E oil
per	period; periodical
PERA	Production Engineering Research Association
Perammon	Fr Amm perchlorate expl
percn	percussion
perfn	perforation
perm	permanent
permn	permission
Permonite	Belg expl (see the text)
pers	personnel
PERSPEX	acrylic resin;may be used as a binding or coating agent, in expls & propellants
pertg	pertaining
Pertite	Ital for PA

PETA	pentaerythritol tetracetate
PETN	pentaerythritol tetranitrate
petr	petrol; petroleum
petr eth	petroleum ether
Petrin	pentaerythritol trinitrate
Petrin Acr	Petrin acrylate (classified)
PETS	pentaerythritol tetrastearate (component of Pentastit)
PETX	symbol for tetra-(nitraminomethyl)-methane
PF	percussion fuze
PF	picrylfluoride
Pfc	private first class
PG or PhGer	Pharmacopeia Germanica
PG	pivot gun
PG	Proving Ground
pge	page
pgh	paragraph
Pgh	Pittsburgh
PGS	Polverificio Giovanni Stacchini (Ital)
PGTN	pentaglycerin trinitrate (same as TMMMT)
Ph	phenyl, phenol
pH	symbol for the logarithm of the reciprocal of the hydrogen ion concentration
ph	telephone; phone
pharmacol	*pharmacological*
PhBr	Pharmacopeia Britannica
PhBr	phenylbromide
PhCl	phenylchloride
PhD	Philosophiae Doctor(Lat)(Doctor of Philosophy)
PHE	plastic high explosive(Brit)
PhEI	Philips Electronics, Inc, Mount Vernon, NY
PhF	phenylfluoride
PhI	phenyliodide
PHIB	amphibian
Phil	Philippines
Phila	Philadelphia, Pa
PHLW	pressurized heavy and light water (Reactor)
photom	photometry
phpht	phenolphthalein
PHS	Public Health Service
Ph-salt	ethylenediamine dinitrate
Pht	phenetole
PhUS	Pharmacopeia of the US
phys	physical
phys chem	*physical chemistry*
physiol	physiological
PI	Philippine Islands
PI	point initiating
PIAT	projector infantry anti-tank
PIB	Polytechnic Institute of Brooklyn, N
PIC	Parr Instrument Company, Moline, Ill
Pic Arsn	Picatinny Arsenal, Dover, NJ
Picramide	same as TNA
Picratol	mixt of Amm P & TNT (US)
Picric Powder or Abel's Explosive	Brit expl contg Amm P, Al, sawdust & crude petroleum
Picrinita	Span for picric acid
Picrite	Brit for nitroguanidine
Picurinsan	Jap for PA(Oshokuyaku)
Picurinsan-ammonia	Jap for ammonium picrate
Pierrit	Swiss blasting expl(see the text)
PIG	Percentage Initiation by Grit (Brit)
Piombite	Ital mil expl of WWI(see the text)
PIPE	Amer expl contg PETN & Gulf Crown E oil
PkArty	pack artillery
pl	plural
Plancastita	Span expl(see the text)
plast	plastic
Plastit	Swiss expl
Plastita	Span expl
Plastolit	Swiss expl
Plat	platoon
Plomoplastrita	Span expl
Plumbatol	Amer expl contg Pb nitrate & TNT
Pluto	reconnaissance & rescue plane, *ground* based
PLW	Pressurized Light Water (Reactor)
PLX	Picatinny liquid explosives (nitromethane ethylene-diamine)
PM	parachute mine
PM	Post meridiem(Lat) afternoon
PM	powder metallurgy
PM	Prime Minister(Brit)
PM	Provost Marshall
PN	performance number
PNA	pentanitroaniline
PNDPhEth	pentanitrodiphenylether
PNDPhEtl	pentanitrodiphenylethanol
PNDPhSfo	pentanitrodiphenylsulfone
PNG	persona non grata(Lat) undesirable person
PNP	Ital expl contg PETN, AN & Wax
Po	polonium
POD	Philadelphia Ordnance District, Phila 2, Pa
POD	Pittsburgh Ordnance District, Ptgh 22, Pa

POD	Pueblo Ordnance Depot, Pueblo, Colo
polym	polymer
POP	Pantex Ordnance Plant, Amarillo, Tex
pos	positive(as an adjective)
poss	possible
pot	potential
Potentite	Belg expl, same as tonite
Potentites	mixts of NC & K nitrate used in Fr as propellants and as demolition expls
Poudre B	see B(poudre)
Poudre NB	see NB(poudre)
Poudre NB JK	see NB JK(poudre)
POW	prisoner of war
powd	powder(ed)
pp	pages
PP	percussion primer
PP	picric powder
PP	pistolet-pulemët(Rus) (machine pistol)
PP	pilotless plane
PP	Polverificio Piemontese(Ital)
PP	power plant
PP	proof paper(firing report)(Brit)
PPB't	pin point bombardment
PPC(B)	Phillips Petroleum Co, Bartlesville, Okla
PPC(McG)	Phillips Petroleum Co, McGregor, Tex
PPD	pistolet-pulemët Degtiarëva (Rus)
ppm	parts per million
PPP	plastic protective plate(Brit)
PPRI(Can)	Pulp & Paper Research Institute of Canada
PPSh	pistolet-pulemët Shpagina (Rus)
ppt	precipitate
pptd	precipitated
pptg	precipitating
pptn	precipitation
PQ	Province of Quebec, Canada
pr	pair
Pr	praseodymium
Pr	Proceedings
Pr or pr	propyl(normal)
PR	public relations
PR	Puerto Rico
Pr 4/5 & Pr 20/24	Ital propellants(see the text)
prac	practice
pract	practical
pr alc	propyl alcohol
PrBr	propylbromide
prcht	parachute
PrCl	propylchloride
Pref	preface
prel	preliminary
Prem	Premier
prep(d)	prepare(d)
prepg	preparing
prepn	preparation
pres	presence
Pres	President
press	pressure
Pri	private
prim	primary
prin	principal
proc	procedure, proceedings
prod	produce, product
prodn	production
prof	professor
prog	progression
proj(s)	projectile(s)
pron	pronoun
prop(s)	property(ies)
proplnt(s)	propellant(s)
propn	propulsion
PRP	petrolatum, rosin & paraffin wax (used in the USA for coating AN crysts)
PRS	Pacific Rocket Society, Los Angeles, Calif
prtg	printing
Pruss	Prussian
pry	priority
PS	percussion shrapnel
ps	per second
PS	point de solidification(Fr) (setting point)
PS	Police Sergeant
PS	Post Scriptum(Lat)
PS	private secretary
PSA	Pacific Science Association (Hawaii)
pseud	pseudonym
psf	pounds per square foot
PSG	Percentage Sensitization by Grit (Brit) (see the text)
psi	pounds per square inch
psia	" " " " (absolute)
psig	" " " " (gauge)
PSR	Polverificio Stacchini di Roma (Ital)
PST	Pacific Standard Time
pt	pint(s) (0.568 l)
Pt	platinum

Pt	point(s)
Pt	port
PT	primary target(Arty)
PT-boat	patrol torpedo-boat
PTC	patrol torpedo-craft
PT-Div	patrol torpedo-boat division
Pte	private(soldier)
pte	private
PTRD	protivotankovoye ruzhiö Degtiarëva(Rus) (A/T rifle of Degtiarëv)
PTRS	A/T rifle of Semënov (Rus)
PTX1 & PTX2	Picatinny ternary expls (RDX, tetryl & TNT or RDX, PETN & TNT)
pty	party
Pu	plutonium
PU	Princeton University, Princeton, NJ
PU	Purdue University, Lafayette, Ind
Publ	Publication
publ(d)	publish(ed)
publg	publishing
publn(s)	publication(s)
pulv(d)	pulverize(d)
pulvn	pulverization
PUNS	permanently unfit for Naval service
PV	patrol vessel
PVA	polyvinyl acetate
PVA-4	Amer expl contg polyvinyl acetate, RDX & DBuPh
PVAlc	polyvinyl alcohol
PVC	polyvinylchloride
Pvt	private
PW	powder weight(in a charge)
PW	prisoner of war
PWA	Public Works Administration
PWC	Brit for "paper wood cellulose" (contg 85% of α-cellulose)
PWD	Petroleum Warfare Development (Brit)
PWP	plasticized white phosporus
PWT	propulsion wind tunnel
PX	Post Exchange; Army Exchange
Py or py	pyridine or pyridyl
pyc	pycnometer
pyr	pyrometer
Pyro	pyrocellulose, pyrocotton
pyro or pyrot	pyrotechnics
Pyrolithes; Pyronomes	Belg black powder type expls
pz	piezo

Q

Q	gunner's quadrant(Arty)
Q	quantity of electricity, Coulombs
Q	quantity of heat
Q	quebrachitol
Q or Qm	quartermaster
Q-boat or Q-ship	vessel for trapping boats
Q_c	heat of combustion
Q_c^p	heat of combustion at constant pressure
Q_c^v	heat of combustion at constant volume
Q-clearance	clearance for classified atomic information; issued by AEC
QE	quadrant elevation (angle of elevn of a gun above horizon)(Brit)
Q_e	heat of explosion
Q_f	heat of formation
Q_f^p	heat of formation at constant pressure
Q_f^v	heat of formation at constant volume
QF	quick firing(of guns using fixed or semi-fixed cartridge cases) (Brit); rapid firing(US)
QFA	quick firing ammunition
QFG	quick-firing gun(Brit)
qlfd	qualified
QM or Q	quartermaster
QMC	Quartermaster Corps
QMG	Quartermaster General(Can)
QOC	Quaker Oats Co, Chicago 54, Ill
QPN	quebrachitol pentanitrate
QR	Queen's Regulations(Brit)
Q-ship	same as Q-boat
qt(s)	quart(s) (for liquids 0.946 l in the US and 1.136 l in GtBrit)
qual	qualitative; quality
qualy	qualitatively
quant	quantitative; quantity
quanty	quantitatively
Que	Quebec, Canada
quotn	quotation
qv	quot vide(Lat) (which see)
qy	quarry

R

°R	degrees Rankine
R	radical(organic)
R	radius
R	range
R	gas constant(1.9885 cal/degree)

°R	degrees Réaumur (80°R=100°C)
R	recoilless
R	relay
R or Rept	report
R	Republic
R	resistance
R	revolver
R	Reynolds number (Physics)
r	rheostat
R	rifle
R or Rus	Russia(n)
R or V	ruzhiö or vintovka(Rus) (rifle)
/R	ribbon propellant (Brit)
R(poudre)	Fr sporting propellant
ρ^6	symbol for Fivonite
RA	Radford Arsenal, Radford, Va
RA	Raritan Arsenal, Metuchen, NJ
RA	Ravenna Arsenal, Apco, Ohio
RA or RSA	Redstone Arsenal, Huntsville, Alabama
RA	Royal Arsenal (Brit)
RA	Royal Artillery
RABRM	Research Association of British Rubber Manufacturers
rac	racemic
RAC	Royal Armoured Corps (Brit)
rad	radio
RADAR	Radio Detection and Ranging
RADC	Rome Air Development Center (US)
RAE	Royal Aircraft Establishment (Brit)
RAF	Royal Air Force (Brit)
Raibun	Jap primary expl (see the text)
Raiko	Jap for MF
RAOC	Royal Army Ordnance Corps (Brit)
RAOD	Royal Army Ordnance Depot (Brit)
RArty	Royal Artillery (Brit)
Ra-Th	radium-thorium
RATO	rocket assisted take off, called also "booster rocket"; more common name is JATO
RB	rifle bomber
Rb	rubidium
RBT	Rifle Bullet Test
RC	Rand Corp, Santa Monica, Calif
RCA	Radio Corp of America
RCAF	Royal Canadian Air Force
RCAT	radio controlled airplane target
RCh	reduced charge
RCL	Radiation Counter Laboratories, Chicago, Ill
RCOC	Royal Canadian Ordnance Corps
RD	Research Department
R&D	Research & Development
Rd	road
rd(s)	round(s) (of Ammo)
RD	rural delivery
RDB(cordite)	Research Department "B" (Brit propellant used during WW I)
RDB	rifle double-base (propellant)
rds/m	rounds per minute
RDX	Research Department Explosive (cyclonite, hexogen or cyclo-trimethylenetrinitramine)
Ré	Réaumur
Re	rhenium
RE	Royal Engineers (Brit)
rec(d)	receive(d)
recogn	recognition
recryt(d)	recrystallize(d)
recrystn	recrystallization
redn	reduction
redox	reduction and oxidation
Ref(s)	Reference(s)
refg	refrigerating
refgn	refrigeration
refl	reflux
Reg	regiment
Regl	regimental
reinfd	reinforced
Reinhold	Reinhold Publishing Corp, New York 22, NY
Reintri	Ger & Swiss for purified TNT
rel	relative
reln	relation
Rep	Republic
req(d)	require(d)
res	reserve
RES	Reynolds Experimental Station, of Atlas Powder Co, Tamaqua, Pa
RESB	Royal Engineers Standards Board (Brit)
resp	respectively
restr	restricted
retd	returned
RETMA	Radio-Electronics-Television Manufacturers Association
Rev(s)	Review(s)
revn	revolution

RF	rapid firing; rimfire
rf	rifle
RFA	radio frequency amplification
RFA	Royal Field Artillery(Brit)
RFAmmo	*rimfire ammunition*
RFC	Reconstruction Finance Corp
RFF	Royal Firearms Factory, England
RFG	rifle fine grain(propellant)(Brit)
RFN	red fuming nitric
RFNA	red fuming nitric acid
Rg	*range*
RG	rifle grenade
RG	ruchnaya granata(Rus)(hand grenade)
RGb	river gunboat
RGF	Royal Gun Factory(Brit)
RGM	rounds per gun per minute
RGn	recoilless gun
RH	relative humidity
Rh	rhodium
RHB	radar homing beacon
RHC(H)	Rohm & Haas Co, Huntsville, Ala
RHC(Ph)	Rohm & Haas Co, Phila 5, Pa
rheo	rheostat
rhmb	rhombic
RHN	Rockwell hardness number
RI	refractive index
RIA	Rock Island Arsenal, Rock Island, Ill
RIC	Royal Institute of Chemistry (Brit)
Rio	Rio de Janeiro, Brazil
RIPE	Amer expl contg RDX & Gulf Crown E oil
riv	river
riv	rivet
RJ	ramjet
Rkt	rocket
RL	rocket launcher
RLG	rifle, large grain (propellant)
RLT	rocket, light tube
Rlwy	railway
RMArty	Royal Marine Artillery
RMC	Royal Marine Corps(Brit)
RMC	Royal Military College(Brit)
RMD/TCC	Reaction Motors Division, Thiokol Chemical Corp
RMI	Reaction Motors, Inc, Rockaway,NJ
Rn	radon
Rn	range(Brit)
RN	round nose
RN	Royal Navy(Brit)
RNPRC	Royal Naval Personnel Research Committee(Brit)
RNZA	Royal New Zealand Army
RNZArty	Royal New Zealand Artillery
RNZN	Royal New Zealand Navy
RO	radar operator
RO	radio operator
ROD	Rochester Ordnance District, Rochester, NY(See NYOD)
ROD	Royal Ordnance Depot(Brit)
ROF	Royal Ordnance Factory(Brit)
Rohtri	Ger & Swiss for crude TNT
Rom	Roman
Romite	Ital cheddite type expl
ROP	Ridgewood Ordnance Plant, Cincinnati, Ohio
ROP	Riverbank Ordnance Plant, Riverbank, Calif
ROP	Rockford Ordnance Plant, Rockford, Ill
ROR	rocket on rotor
Rossite	code name for guanylnitrourea (nitrodicyandiamidine)
ROTC	Reserve Officers Training Corps
ROW	Radford Ordnance Works, Radford, Va
Roy	Royal
RP	rocket projectile
RP	rocket propellant
R/P	rocket projector
RPD	Rocket Propulsion Department
rpg	rounds per gun
rpgpd	rounds per gun per diem
rpgpm	rounds per gun per minute
rpm	*revolutions per minute*
rpm	rounds per minute
rprt	reprint
rps	revolutions per second
RP's	rocket projectiles
RR	railroad
RRA	Red River Arsenal, Texarkana, Tex
RRC	Rolls-Royce Co, England
RRE	Radar Research Establishment (Brit)
RRI	Rocket Research Institute
RRL	Reynolds Research Laboratory, Tamaqua, Penna(Atlas PC)
RRL	Road Research Laboratory(Brit)
RRS	Reaction Research Society, Glendale, Calif
RS	rolled steel

R-salt	cyclotrimethylenetrinitrosamine
RSigG	rocket, signal, green
RSigR	rocket, signal, red
RSL	Royal Society, London
RSM	rocket sea-marker
RSOP	reconnaissance, selection and organization of artillery positions
RSVP	répondez s'il vous plaît(Fr) (please answer)
R/T	radio telegraphy or radio telephony(Brit)
rt	rate
RT	room temperature
RTCC	Reilly Tar & Chemical Corp, Indianapolis 4, Ind
Ru	ruthenium
Rum	Rumania; Rumanian
Rupturita	Braz expl manufd by SAER
Rus	Russia; Russian
RV	remaining velocity
Ry(s)	railway(s)
RyBn	Railway Battalion
RyJn	railway junction

S

s	second
s-	secondary(as applied to a type of organic compound)
S	series
S	service
s	single
S	slotted tubular propellant (Brit)
S	solventless propellant(Brit)
S	south; southern
S	staff
S	sulfur
s- or sym-	symmetrical
S	Fr & Ital expls (see the text)
S	Fr sporting propellant
S1; S2 etc	Ger & Ital expls used in underwater ammo(see PATR 2510, pp 170 & 212)
Sa	samarium
SA	samokhodnaya artilleriya(Rus) (self-propelled artillery)
Sa	Saturday
SA	Secretary of the Army
SA	semi-automatic
SA	small arms
SA	Sociedad Anónima(Span) Società Anonima(Ital) Société Anonyme(Fr) Sociedade Anônima(Port) (Joint Stock Company)
SA	South America
SA	Space Agency(US)
SA	Springfield Armory, Springfield 1, Mass
SA152	Ital propellant(obsolete)
SAA	small arms ammunition
SAA	Société Anonyme d'Arendonck (Belg)
SAA	Standards Association of Australia
Sabulites	Belg & Ital expls of various compns
SAAC	Scientific Advisor to the Army Council(Brit)
SAAD	small arms ammunition depot
SAB	svetiashshayacia aviobomba (Rus)(illumination aerial bomb)
SABPD	Società Anonima Bombrini-Parodi-Delfino(Ital)
Sabulite	Belg safety expl(see the text)
SAC	Scientific Advisory Council(Brit)
SAC	Strategic Air Command
SAC	Supreme Allied Commander
SACNA	Società ACNA(Ital)
SAD	Sociedade Anônyma de la Dinamita(Portug)
SADN	Società Anonima Dinamite Nobel Avigliana(Ital)
SAEF	Société Anonyme des Explosifs Favier (Belg)
SAEH	Société Anonyme des Explosifs d'Havré(Fr)
SAEPC	Società Anonima di Esplodenti e Prodotti Chimiche(Villafranca) (Ital)
SAEPC	Société Anonyme d'Explosifs et de Produits Chimiques
SAEPCM	Società Anonima di Esplodenti e Prodotti Chimiche, Montecatini(Ital)
SAER	Sociedade Anonima Explosivos Rupturita(Brazil)
SAFA	Società Anonima Fabbrica Armi(Ital)
SAFAT	Società Anonima Fabbrica Armi Torino(Ital)
SAICE	Società Anonima Consomatori Esplosivi(Orbetello)(Ital)

SAIM	Società Anonima Italina Micce (Ponte Stazzemese)(Ital)
Sakuma-dainamaito	Jap for gelignite
Sakura-dai-namaito	Jap dynamite(see the text)
salv	salvage
SAM	surface-to-air missile
SAMCM	Società Anonima Munizione e Cartuccheria Martignoni (Ital)
SAME	Society of American Military Engineers
SANACC	State Army-Navy-Air Co-ordinating Committee
Sanshōki-mechiru nitoruamin	Jap for tetryl (same as Meiayaku)
Sanshōki-toruoru	Jap for TNT
SAP	semi-armor-piercing
sapon	saponification, saponify
sapond	saponified
sapong	saponifying
sap val	saponification value
SAR	semi-automatic rifle
Sask	Saskachewan, Can
sat	saturate
satd	saturated
satg	saturating
satisf	satisfactory
satn	saturation
SB	Selection Board
SB	shell bullet(exploding bullet)
SB	Siege Battery
SB	smooth-bore
Sb	stibium(Lat)(antimony)
SB	submarine boat
SBAS	standard beam approach system
SBCP	slow burning cocoa powder (see the text)
Sc	scandium
S&C	Schaar & Co, Chicago 7, Ill
Sc or Sci	science
SC	shaped charge
Sc or Sch	schneiderite (Fr, Ital & Rus expl)
SC	short case
SC	solventless, carbamite (Brit propellant contg centralite)
SC	South Carolina
SCA	Seacoast Artillery
ScD	Scientiae Doctor(Lat); Doctor of Science
SCC	Stauffer Chemical Co, New York 17,
sch	schedule; school
Sch or Sc	schneiderite(Fr, Ital & Rus expl)
Schiesswolle 18 or TSMV 1-101	expl contg HNDPhA, TNT & Al (see PATR 2510, p 172)
Sci or Sc	science
SCI	Society of Chemical Industry
sclt	scarlet
ScM	Master of Science
SC/RDL	Signal Corps, Research and Development Laboratories
scrng	screening
Scy	Secretary
SD	Salvage Depot
SD	sans dissolvant(Fr)(solventless)
SD	sawdust
SD	self-destroying
SD	Service Depot
SD	shell dressing
SD	Signal Department(Brit Navy)
SD or SDak	South Dakota
SD	straight dynamite
SD	Submarine Department
SDC	Shell Development Company
SDC	Special Devices Center
SD NÉO	Fr solventless propellant based on DEGDN
SD Ngl	Fr solventless propellant based on NG
SDO	synthetic drying oils (used in some incendiaries)
SDVM	Società Dinamite Villafranca-Mulazzo(Ital)
Se	selenium
SE	Society of Engineers
SE	south-east
Sébomites	Fr chlorate expls of pre WW I
sec(s)	second(s)
Sec or Secry	Secretary
SECI	Société d'Études Chimiques pour l'Industrie(Fr)
secn	section
Sécurites	Belg expls of various compns (see the text)
seg	segment
Segurança	Braz safety expls manufd by CNES
Seigata	Jap expl(see the test)
SEM	Società Esplodenti e Munizione (Roma, Italy)
Sen	Senator
Sen	Senior
Sengite	Belg expl(see the text)
sens	sensitive
sensy	sensibility, sensitivity

SEPC	Società Esplodenti et Prodotti Chimiche(Torino)(Ital)
sep(d)	separate(d)
sepg	separating
sepn	separation
SEPR	Société d'Étude de la Propulsion par Réaction(Fr) (Society for the Study of Propulsion by Reaction Technique)
Sept	September
seq	sequence
ser	series
Seranin	older Swed AN dynamite
Serg	Sergeant
Serj Maj	Sergeant-Major(Brit)
SERL	Signals Engineering Research Laboratory
Serv	Service
SES	Suffield Experimental Station (Can)
SET	Société d'Explosifs Titanite
Sévranites	Fr plastic expls contg PETN and Amm perchlorate
SEX	symbol for 1-acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine
SF	safety fuze
SF	San Francisco
SF	sans flammes(Fr)(flashless)
SF	sans fumée (Fr)(smokeless)
SF	Santa Fe
sf	self-feeding
SF	semi-fixed
SF	signal flare
SF	supersonic frequency
SFE	Société Française des Explosifs
SFHEPC	Société Franco-Hellénique d'Explosifs et Produits Chimiques(Ktipito, Greece)
Sfi	sulfide
SFIEC	Société Franco-Italienne d'Explosifs Cheddite
SFMCTG	Société Française des Munitions de Chasse, de Tir et de Guerre(Fr)
Sfo	sulfone
SFOD	San Francisco Ordnance District, Oakland, Calif
SG	smoke generator
SG	specialist in gunnery
SGACI	Scientific Glass Apparatus Co, Inc, Bloomfield, NJ
SGD	straight gelatin dynamite
SGEC	Société Générale d'Explosifs Cheddite
SGEM	Società Generale di Esplosivi e Munizione(Ital)
SGIMC	Società Generale per l'Industria Mineraria e Chimica(Ital)(see also SAPCM)
SGM	ship-to-ground missile
SGP	Sécurite-Grisou-Poussière (Belg expls safe to use in gaseous and dusty coal mines)
SGR	sodium graphite reactor
Sh	shrapnel' (Rus)(shrapnel)
SH	Squashhead(Brit) (see also HE/SH and HEP)
ShCh or SC	shaped charge
SHAEF	Supreme Headquarters, Allied Expeditionary Forces
Shakunetsuzai	Jap for thermite
SHAPE	Supreme Headquarters of Atlantic Powers in Europe
shell	hollow projectile filled with expl or other material
Shellite	Brit expl contg PA 70 & DNPh 30% (See also Tridite)
SHF	super high frequency(3000 to 30000 megacycles per second)
Shimose; Shimose-bakuyaku or Shimosite	Jap cast PA prepd by a special method
Shoanbakuyaku	Jap AN expl
Shoanyaku	Jap coal mining expls
Shōbenyaku	Jap expls(see the text)
Shoeiyaku	Jap for PETN
Shokamen	Jap for NC(Menkayaku)
Shokayaku	Jap propellant
Shonayaku	Jap expl
SHORAN	short range navigation
shot	solid projectile or slug
Shotoyaku	Jap 50/50 amatol
Shouyaku	Jap for cyclonite and some of its expls(see also Tanayaku)
SHQEA	Supreme Headquarters of the European Army
sh t	short ton(907.18 kg or 2000 lb) (US)
shth	sheath
shthg	sheathing
sh/w	short wave
Si	silicon
SI	specific impulse
SI	start of ignition(of liq propellants in rockets)

SIA	Società Italiana Ansaldi (Ital)
Sib	Siberia
SIB	Special Intelligence Bureau
SIB	Special Investigation Board
SIDB	Società Italiana Davy Bickford at Taino(Ital)
SIEB	Società Italiana Ernesto Breda (Ital)
Siem	Ital sporting propellant
SIEP	Società Italiana dell'Esplosivo Prometheus
SIG	Schweizerische Industrie-gesellschaft(Neuhausen)(Swiss)
sig	signal
SigC	Signal Corps
sign	signature; signify
Silites	older types Fr & Ital cannon propellants
Simplonit	Swiss expl(see the text)
sin	sine
sing	singular
SIPE	Società Italiana Prodotti Esplodenti(Milano)
Sipe	Ital sporting propellant
Siperite	same as MNDT
SIPRE	Snow, Ice and Permafrost Research Establishment
sit	situate
SIT	spontaneous ignition temperature
sitd	situated
sitn	situation
Sixolite	code name for tetramethylol-cyclohexanolpentanitrate
Sixonite	code name for tetramethylol-cyclohexanonetetranitrate
SJ	steel jacket
SJOD	San Jacinto Ordnance Depot, Channelview, Tex
SL	separate-loading
sl	slight(ly)
S/L	streamlined
SLEEP	Swedish Low-Energy Experimental Pile
SLOD	San Louis Ordnance District, St Louis 1, Mo
S/Lt	Second Lieutenant
sl sol	slightly soluble
sly	slowly
Sm	samarium
SM	strategic missile
S/M	submarine
SMAC	Senate Military Affairs Committee
SMC	Sten machine carbine(Brit)
SMG	sub-machine gun
Smith Inst	Smithsonian Institute, Washington, DC
smk sh	smoke shell
smk sig	smoke signal
SMLE	short magazine(or model) Lee-Enfield(rifle) (Brit)
smls	seamless
smpl	sample
SMRE	Safety in Mines Research Establishment(Brit)
SN	saponification number
SN	sodium nitrate
Sn	stannum(Lat), tin
snafu	situation normal, all fouled up (Amer slang)
SNECMA	Société Nationale d'Étude et de Construction de Moteurs d'Aviation(Fr)
SNF	Société Nobel Française
SNL	Standard Nomenclature List
SNM	Society of Nuclear Medicine
SNSE	Society of Nuclear Scientists and Engineers
SO	Stationery Office(Brit)(see HMSO)
SOA	School of Artillery
SOC	Shell Oil Co, San Francisco, Calif
SOC	Standard Oil Co, Whiting, Ind
SOCONY	Standard Oil Co of New York
Socy	Society
SOD	Savanna Ordnance Depot, Savanna, Ill
SOD	Seneca Ordnance Depot, Romulus, NY
SOD	Sierra Ordnance Depot, Herlong, Calif
SOD	Sioux Ordnance Depot, Sidney, Neb
SOD	Springfield Ordnance District, Springfield 1, Mass
SODC	Standard Oil Development Company
SOFAR	sound fixing and ranging
Sofranex A	Fr plastic expl contg NG, CC, AN, Al & liq DNT
sol	soluble
Solenita	Ital rifle propellant
soln	solution
solv	solvent(s)
soly	solubility
solys	solubilities
Soms	Somersetshire, Engl
Son	Sonora, Mexico
SONAR	Submarine Sound Operation, Navigation and Ranging
SOP	Scranton Ordnance Plant, Scranton,Pa

SOS	smoke obscuring screen
SOS	sniping, observation and scouting distress
SOS	international distress signal
SOW	Sunflower Ordnance Works, Lawrence, Kan
SP	self-propelled
SP	Service des Poudres(Fr)
SP	single-perforated(propellant)
SP	smokeless propellant
sp	solidification or setting point
SP	solid propellants(Rocketry)
sp	specific
SP	Supply Post
SP_1; SP_2; SP_3	black powders used by the French in cannons prior to invention of smokeless propellants
SPAR	Super Precision Approach Radar
SPAT	self-propelled antitank
SPCEBC	Société des Produits Chimiques et d'Explosifs Bergès, Corbin et Companie(Grenoble)(Fr)
SPCGM	self-propelled caterpillar gun-mount
SPE	Society of Plastics Engineers
Spec(s)	Specification(s)
SPG	self-propelled gun
sp gr	specific gravity
sp ht	specific heat
SPI	Society of the Plastics Industry
SPIA	Solid Propellants Information Agency, Johns Hopkins University, Silver Spring, Md
splty	specialty
SPM	self-propelled mount
spont	spontaneous
spr	sapper
sp vol	specific volume
SpW	Special Weapon
SPXEC	Special Executive
spzd	specialized
Sq	Squadron
sq	square
SQ	superquick(Brit)
SQC	Soldier's Qualification
sq cm or cm^2	square centimeter(s)
Sq(D)	dive squadron
sq ft	square foot(feet)
sq in or in^2	square inch(es)
sq km	square kilometer(s)
sq m or m^2	square meter(s)
sq mi	square mile(s)
sq mm	square millimeter(s)
sq yd	square yd(s)
Sr	Senior; Señor(Span)
SR	short rifle
SR	sound ranging
SR	Special Regulation(s)
SR	Special Report(s)
Sr	strontium
SR	synthetic rubber
SRDE	Signals Research and Development Establishment
SRED	Scientific Research and Experiments Department(Naval)
SRI	Southwest Research Institute, San Antonio, Tex
SRI	Stanford Research Institute, Stanford, Calif
SRMLE	short rifle military, Lee-Enfield(Brit)
SS	single shot(firearm)
SS	star shell
SS	steamship
SS	supersensitive
SSA	Selective Service Act
SSA	Social Security Administration
SSAGCD	Schweizerisch Sprengstoff Aktiengesellschaft Cheddit & Dynamit (Liestal & Isleton, Switzerland)
SSEGB	Société Suisse des Explosifs, Gamsen-Brigue
SSF	Schweizerische Sprengstoff Fabrik, AG, Dottikon, Switzerland
SSG	submarine guided missile
SSLI	Sargent Scientific Laboratory Instruments, Chicago 30, Ill
SSM	ship-to-ship missile
SSM	surface-to-surface missile
SSME	Società Sarda Materie Esplodenti (Caglieri, Sardinia) (Ital)
SSN	symbol for nuclear powdered submarine
SSN	Specification Serial Number
SSP	start of sustained pressure (Rocketry)
SSPF	Staatliche Schwarzpulverfabriken (Aubonne & Chur) (Switz)
SSR	symbol for radar picket submarine
ST	starting time
Sta	Station
Stab	stabilizer

STAF	Strategic Air Force Staffs, Staffordshire, Engl
(star)*	after Mark as MkV* denotes a minor design modification in Ordn items(Brit)
std	standard
stdz	standardize
stdzn	standardization
Sté	Société (Fr)(Society)
Sté Ayme	Société Anonyme(Fr) (Joint Stock Company)
stl	stilbene
STP	standard temperature and pressure
strg	strength, strong
STR	Submarine Thermal Reactor (as in the Nautilus)
SU	"solventless, urethane" (Brit propellant)
Sub	Subaltern(Brit)
sub or subm	submarine
sub	subway
sub-cal	sub-caliber
subd	subdivision
subj	subject
subl	sublime(s)
subln	sublimation
subm or sub	submarine
subs	subsidiary
subseq	subsequent
subs	substance, substitute
Suc	sucrose
succr	successor
SucON	sucrose octanitrate
SUE	Société Universelle des Explosifs
suff	sufficient
Suff	Suffolk, Engl
Sug	sugar
SUM	surface-to-underwater(guided) missile
Sund	Sunday
Superforcite	Belg gelatin dynamite
supers	supersaturated
SupHqs	Supreme Headquarters
SUPO	Super Power Water Boiler (Reactor)
suppl	supplement
supra	Lat for "above"-used to refer to earlier parts of the book
Supt	Superintendent
SuptDoc	Superintendent of Documents
surf	surface
Surr	Surrey, Engl
suspd	suspended
suspn	suspension
Suss	Sussex, Engl
SUV	Saybolt Universal Viscosity
SV	saponification value
SV	striking velocity(Brit)
SVB	Società Vulcania di Brescia (Ital)
SW	short waves
SW	South Wales
SW	South-west
SWC	Special Weapons Center
Swed	Sweden, Swedish
SwedP	Swedish Patent
SWG	Standard Wire Gauge(Brit)
SwissP	Swiss Patent
Switz	Switzerland
sym- or s-	symmetrical
symb	symbol
syn	synonym
Synd	syndicate
synth	synthetic
syr	syrup
syry	syrupy
syst	system

T

T or Tk	tank
T	target angle
T	technical
T	temperature
t- or tert-	tertiary(as applied to type of organic compound
T	time
T	toluene(Fr & Ital)
t	ton, short(US) = 2000 lb = 907.18 kg = 0.8929 lg t
t(lg)	ton, long(Brit) = 2240 lb = 1016.05 kg 1.1200 t (short)
T	metric ton = 1000 kg = 2204.62 lb = 1.1023 t = 0.9842 lg t
T	torpedo
T	tracer
/T	with tracer(Brit)
T	tri
T	tritium
T	trotil or tol(Rus)(TNT)
T	Troy (weight) (1 lb = 373.2418g)
T	tubular propellant(Brit)
T	followed by a number (eg T28) signifies an experimental unstandardized item
T(poudre)	Fr sporting propellant
/T	with tracer(Brit)

T_1	Fr & Ital for MNT
T_2	Fr & Ital for DNT
T_3	Fr & Ital for TNT
T_4	Fr & Ital for RDX
Ta	tantalum
TA	triacetin
TAC	Tables Annuelles Internationales de Constantes et Données Numériques (See in Abbreviations for Books and Journals)
TAC	Technical Assistance Committee
TACAN	Tactical Air Navigation (System)
TACC	Tactical Air Control Center
TAM/DNLC	Titanium Alloy Mfd, Div of National Lead Co
tan	tangent
Tanayaku	Jap RDX expls(see also Shouyaku)
Tanoyaku	Jap expl contg RDX, TNT & tetryl
TAP	time and percussion (fuze)
TAPPI	Technical Association of the Pulp and Paper Industry
TATNB	triaminotrinitrobenzene (classified)
TAX	symbol for 1-acetylhexahydro -3,5-dinitro-s-triazine
TB	Technical Bulletin
Tb	terbium
TB	tracer bullet
TBD	torpedo-boat destroyer
Tbis	Fr pistol propellant
TC	Tennessee Corp, Atlanta, Ga
TC	tracer composition
TCA	Twin Cities Arsenal, Minneapolis, Minn
TCC	Thiokol Chemical Corp, Trenton, NJ and Elkton, Md (see also RMD/TCC)
TD	tank destroyer
TD	torpedo detonating
T-Day	Termination of War Day
TDE	Technical Development Establishment(India)
Te	tellurium
Te	tetra
TeAA	tetraazylazide
tech	technical
Tech	Technician(a specialist rating)
TEE	Torpedo Experimental Establishment
TEG	triethyleneglycol
TEGDN or TEGN	triethyleneglycol dinitrate
TEGMN	triethyleneglycol mononitrate
TEL	tetraethyllead
teleg	telegram
teleph	telephone
Telsit	Swiss dynamite
TeMeAN	tetramethylammonium nitrate
temp	temperature
TEN	Rus designation for PETN
TeNA	tetranitroaniline
TeNAns	tetranitroanisole
TeNAzxB	tetranitroazoxybenzene
TeNB	tetranitrobenzene
TeNBPh	tetranitrobiphenyl(same as TeNDPh)
TeNCbl	tetranitrocarbanilide
TeNCbz	tetranitrocarbazole
TeNDG	tetranitrodiglycerin
TeNDMBDNA	tetranitrodimethylbenzidine-dinitramine
TeNDPhA	tetranitrodiphenylamine
TeNDPhEta	tetranitrodiphenylethane
TeNDPhEth	tetranitrodiphenylether
TeNDPhEtla	tetranitrodiphenylethanolamine
TeNHzB	tetranitrohydrazobenzene
TeNMe	tetranitromethane
TeNMA or TeNMeA	tetranitromethylaniline
Tenn	Tennessee
TeNN	tetranitronaphthalene
TeNOx or TNO	tetranitrooxanilide
TeNPhMNA or TeNPhMeNA	tetranitrophenylmethylnitramine
tent	tentative
TeNT	tetranitrotoluene
TeNTMB or TeNTMeB	3,5,3',5'-tetranitro-4,4'-tetramethyldiaminobiphenyl
TePhUr	tetraphenylurea
TERI	torpedo effective range
Territ	Swed plastic perchlorate type expl(see the text)
tert	tertiary
TETeA	triethylenetetramine
Tetracene	guanylnitrosaminoguanyltetrazene
Tetra-Di-Salt	tetramethylammonium dinitrate
Tetralita	Span for tetryl
Tetra-Salt	tetramethylammonium nitrate (see PATR 2510, p Ger 197)
Tetra-tetryl	tetra(2,4,6-trinitro)-phenyl-nitraminomethyl) methane

Tétréthyl	Fr for ethyltetryl
tetrg	tetragonal
tetrh	tetrahedral
Tetroxyl	2,4,6-trinitrophenyl-methoxynitramine
Tetryl	N,2,4,6-tetranitromethylaniline
Tetrytol	mixt of tetryl & TNT
Tex	Texas
Tez	tetrazole
TF or TiF	time fuze
Tg	target
TG	trotil-gheksoghen(Rus for TNT-RDX mixts)
TGB	torpedo gunboat
TGSC	Texas Gulf Sulfur Co, New York 17, NY
Th	thorium
theor	theoretical
therm	thermal
therm	thermometer
thermo	thermostat
thermoch	thermochemical
thermod	thermodynamics
Thional	code name for pentanitrodiphenyl-sulfone
THOD	Terre Haute Ordnance Depot
Thur	Thursday
TI	technical information
Ti	titanium
TIB	target identification bomb
TIB	Technical Information Bureau(Brit)
TIC	temperature of initial combustion
TID	Technical Information Division
TIDU	Technical Intelligence Documents Unit
TiFz	time fuze
TILS	Technical Information and Library Services(Brit)
TIS	Technical Information Service (Canada)
Titanites	Fr expls contg AN,TNT & charcoal from curcuma
titr	titrate
titrn	titration
Tk, tk or T	tank
TkV	tracked vehicle
Tl	thalium
TLP	torpedo land plane (Navy; Coast Guard) (US)
TLV	troop landing vessel
TM	tactical missile
TM	technical manual
TM	technical memorandum
Tm	thulium
TM	time, mechanical(fuze)
TM	trade mark
TM	training manual
TM	trench mortar
T-Man	treasury department man
TMB	trench mortar bomb
TMC	Thompson machine gun
TMD	Torpedo and Mine Department
TMEMT	trimethylolethylmethane trinitrate
TMG	turret machine gun
TMMMT	trimethylolmethylmethane trinitrate
T-Mor	trench mortar
T-Mun	trench munition
TN	total nitrogen
TN or Tn	Fr for TNN (trinitronaphthalene)
TNA	trinitroaniline
TNAmPh	trinitroaminophenol
TNAns	trinitroanisole
TNB	trinitrobenzene
TNBA	trinitrobenzaldehyde
TNBA	trinitrobenzoic acid
TNBzN	trinitrobenzyl nitrate
TNC	see TeNCbz
TNClB or TNCB	trinitrochlorobenzene
TNCrs	trinitrocresol
TNDMA or TNDMeA	trinitrodimethylaniline
TNDPhA	trinitrodiphenylamine
TNEB or TNEtB	trinitroethylbenzene
TNEDV or TNEtDNV	trinitroethyldinitrovalerate
TNETB or TNEtTNBu	trinitroethyltrinitrobutyrate (classified)
TNG	trinitroglycerin
TNM or TNMe	trinitromethane
TNMA or TNMeA	trinitromethylaniline
TNMel	trinitromelamine
TNMes	trinitromesitylene
TNN	trinitronaphthalene
TNO	see TeNOx
TNPE	tetranitro de pentaeritrita (Span) (PETN)
TNPh	trinitrophenol (PA)
TNPht	trinitrophenetole
TNPhBuNA	trinitrophenylbutylnitramine
TNPhDA	trinitrophenylenediamine
TNPhENA or TNPhEtNA	trinitrophenylethylnitramine
TNPHMNA or TNPhMeNA	trinitrophenylmethylnitramine
TNPhMNNAPh or TNPhMeNAPh	trinitrophenylmethylnitramino-phenol

TNR	trinitroresorcinol
TNRS	trinitroresortsinat svintsa (Rus) (lead styphnate)
TNStl	trinitrostilbene
TNT	trinitrotoluene (TNT)
TNTAB	trinitrotriazidobenzene
TNTClB	trinitrotrichlorobenzene
TNTMNA	trinitrotolylmethylnitramine
TNX	trinitroxylene
TO	Technical Order (US Air Forces) (corresponds to TM of US Army)
To	see lg t
TOD	Tooele Ordnance Depot, Tooele, Utah
Tol or Tolit	Rus for TNT
Tolamite	Fr plastic expl contg NG, CC, AN, liq DNT & Wood meal
Tolite	Fr for TNT
Ton(Brit)	long ton of 2240 lb
Tonite	Belg & Brit expl contg GC and Ba & Na nitrates
TopSec	top secret
Tor	torpedo
TORPCM	torpedo counter measures and deception
Torpex	expl contg RDX, TNT & Al
TorT	torpedo tube
tot	total
Totalit	Swiss expl contg AN & paraffin
toxy	toxicity
T/P	tank piercing
TP	target practice
Tp	troop
TPC	Trojan Powder Co, Allentown, Pa
TPEON	tripentaerythritol octanitrate (classified)
Tpk	turnpike
TP-T	target practice, tracer
tr or T	trace, tracer
Tr	transactions
TR	true range
trac	tractor
Tri	Ger & Swiss for TNT
Trialen	Ger & Ital expls (see PATR 2510, p 203)
tric	triclinic
Tridites	mil expls contg PA & DNPh in various propns (see also Shellite & Nellite)
trig	trigonal
Trilita	Span for TNT
Trilite	same as TNT
trim	trimetric
Trimonite	mil expl contg PA & MNN
Trinal	same as TNN
Trinitril	code name for glycerol-α,2,4,6-tri-nitrophenolether dinitrate
Triogen	a term proposed by Dr H.J. Matsuguma of PicArsn for cyclotrimethylenetrinitrosamine (R-salt)
Tri-Salt	trimethylammoniumnitrate
Trisol	Ger & Swiss name for TNAns
trit	triturate
Tritolo	Ital for TNT
Triton	same as TNT
Tritonal	expl contg TNT, AN & Al
Tri-Trinol	Ger expl (see PATR 2510, p 209)
trnsln	translation
Trojan(explosive)	nitrostarch expl contg Na nitrate, Ba nitrate, oil & a stabilizer (see the text)
Trotil	Rus for TNT
Trotyl	Brit, Ger & Swiss for TNT
TS	top secret
TS	torpedo shell
TSG	Technical Service Group
T/Sgt	Technical Sergeant
TSMG	Thompson sub-machine gun
TSMV 1-101	see Schiesswolle 18
TSP	torpedo seaplane
TSQ	time and superquick
TSQ	torpedo seaplane (Navy)
TSR	torpedo-spotter-reconnaissance (airplane)
T-Stoff	Ger designation for 80-85% H_2O_2
TSVP	tournez s'il vous plaît (Fr) (over)
TT	teletype
TT	torpedo tube
TT	towed target
TTE	Tropical Testing Establishment (Brit)
TU	University of Texas, Austin 12, Tex
TU	toxic unit
Tu	Tuesday
Tube (of ammo)	primer used with separate-loading ammo and firing through the breech mechanism of the gun (Brit)
turp	turpentine
TV	television
TV	terminal velocity
TVA	Tennessee Valley Authority
°Tw	degree Twadell (concentration)
TWA	Trans-World Air Lines

Ty	Fr designation for Tetryl
typ	typical
Type 1, Type 2, etc	Jap expls(see the text)
typog	typographical
typw	typewriter
Tz	triazole

U

U	unclassified
U	underground
U	unitarnyi(Rus)(fixed round)
U or Univ	University
U	uranium
U	Utah
UAL	United Air Lines
UAM	underwater-to-air missile
U-boat	submarine(from the Ger Unter-seeboot)
u/c	undercharge
UC	University College(London)
UC	University of California, Berkeley, Calif
UCC	Union Carbide Corp, New York
UCCC	Union Carbide Chemicals Co, New York 17, NY
UCDWR	University of California, Dept of War Research
UCLA	University of California, Los Angeles
UCRL	University of California Radiation Laboratory
UDMH	unsymmetrical dimethyl hydrazine
UDOP	Universal (ultra hi-frequency) Döppler(supersedes DOVAP)
UDU	Underwater Demolition Unit
UEESA	Union Española de Explosivos, Sociedad Anónima(Span)
UERL	Underwater Explosives Research Laboratory, Woods Hole, Mass
UFla	University of Florida, Gainesville, Fla
UHF	ultra-high frequency(300 to 3000 megacycles per second)
UI	University of Illinois, Urbana, Ill
UK	United Kingdom(GtBritain)
UKAEA	United Kingdom Atomic Energy Authority
UKSM	United Kingdom Scientific Mission(in USA)
ult	ultimate
u/m	undermentioned(on Brit firing records)
UM	University of Michigan, Ann Arbor, Mich
Umbrite	Ital amatol-type expl
UMC	Universal Match Corp, Maynard, Mass and Ferguson, Miss
UMT	Universal Military Training
UMWA	United Mine Workers of America
UN	United Nations
unabr	unabridged
unacc	unaccompanied
UNAEC	United Nations Atomic Energy Commission
unexpl	unexploded
unif	uniform
Univ	University
unpub	unpublished
uns	unsymmetrical
unsat	unsatisfactory
unsatur	unsaturated
unst	unstable
unsym- or u-	unsymmetrical
UOD	Umatilla Ordnance Depot, Ordnance, Oregon
UP	Union Pacific(Railroad)
UP	United Press
UP	University of Pennsylvania, Philadelphia, Pa
UP	unrotating projectiles (former Brit name for rockets)
UPRB	Usines des Poudreries Réunies de Belgique
UrN	urea nitrate
UrP	urea picrate
Uru	Uruguay
US	ultrasonic(or supersonic)
US	Under-Secretary
US	United Services(Armed Forces)
US	United States
USA	Union of South Africa
USA	United States Army
USA	United States of America
USAAC	United States Army Air Corps
USAEC	United States Atomic Energy Commission
USAF	United States Air Force
USAF	United States Army Force
USAFIT	United States Air Forces Institute of Technology
USBM	United States Bureau of Mines
USBS	United States Bureau of Standards (see NBS)
USCG	United States Coast Guard
USCSC	United States Civil Service Commission
USDA	United States Department of Agriculture, Washington 25, DC

USDC	United States Department of Commerce, Washington 25, DC
USE	United States Engineers
USEF	United States Expeditionary Forces
USG	United States Gage(wire caliber)
USG or USGovt	United States Government
USGPO or USGovtPtg-Off	United States Government Printing Office, Washington 25, DC
USI	United Services Institution (Brit)
USI	United States Industrial Chemical Co, New York 16, NY
USIA	United States Information Agency
USIS	United States Information Service
USL	United States Lines (Steamship Co)
USM	underwater-to-surface missile
USM	United States Marines
USMA	United States Military Academy, West Point, NY
USMC	United States Marine Corps
USMCA	United States Marine Corps Aviation
USMCEB	United States Marine Corps Equipment Board, Quantico, Va
USMP	United States Military Police
USN	United States Navy
USNA	United States Naval Academy, Annapolis, Md
USNAD	United States Naval Ammunition Depot, Crane, Ind
USNAOTS	United States Naval Aviation Ordnance Test Station, Chincoteague, Va
USNAS	United States Naval Air Service
USNATC	United States Naval Air Test Center, Patuxant River, Md
USNEL	United States Naval Electronics Laboratory
USNG	United States National Guard
USNRDL	United States Naval Radiological Defence Laboratory
USO	United Services Organizations
USO	Unit Security Officer(Canada)
USP	United States Patent
USP	United States Pharmacopeia
USPO	United States Patent Office
USPO	United States Post Office
USQMC	United States Quartermaster Corps
USRC	United States Rubber Co, Passaic, NJ
USS	United States Senate
USS	United States Ship
USS	United States Standard
USS	United States Steel Corp, Pittsburgh 30, Pa
USSAF	United States Strategic Air Force
USSG	United States Standard Gage
USStd Sieve	United States Standard Sieve
USVA	United States Veterans Administration
USW	ultra short waves
USWB	United States Weather Bureau
UU	University of Utah, Salt Lake City, Utah
UV	ultraviolet
UV	uproshchennyi vzryvatel' (Rus) (simplified pull fuze)
U/W	underwater
UW	University of Wisconsin, Madison, Wisc
UWE	underwater explosive
UX	unexploded
UXAA	unexploded antiaircraft(shell)
UXB	unexploded(HE) bomb
UXIB	unexploded incendiary bomb
UXPM	unexploded parachute mine
UXTGM	unexploded gas-type mine

V

V	value
V	vanadium
V or veh	vehicle
V or vel	velocity
v	very
v	volt(s)
V	vintovka(Rus) (rifle)
V or vol	volume
V	vystrel razdel'nago zariazheniya (Rus) (separate-loaded round)
V(poudre)	"poudre V," original name of smokeless propellant invented by Vieille; the name was changed to "poudre B" in honor of Gen Boulanger, then Minister of War
VA	Veterans Administration
VA	Vickers-Armstrong(Brit concern manufg arms, ordnance and ships)

Va or Vir	Virginia
va	volt-ampere
vac	vacuum
VAL	Vickers-Armstrong, Ltd,England
Van Nostrand	D.VanNostrand Co Inc, Princeton,NJ(publishers)
vap	vapor
VAP	vinylacetylene polymer
vapzn	vaporization
VAR	Volunteer Air Reserve
vas	vaseline(See also PG)
VB	variable bomb(guided bomb)
VB	code name for a bomber of the Naval Air Service
VBF	code name for a fighting bomber plane of the Naval Air Service
VC	valeur calorimétrique(Fr)
vc	volt-coulomb
vd	vapor density
VD	velocity of detonation
V-Day	Victory Day
VDI	Verein Deutscher Ingenieure (Association of German Engineers)
VDT	variable density tunnel(aero-dynamics)
VE	volume in cc occupied by 1kg of an expl at a given density(Fr)
VEB	Volkseigener Betrieb(E Ger) (People's Own Concern)
VE-Day	Victory in Europe Day
veh	vehicle
vel	velocity
Veltérine	see Wélterine
Veltex	Amer expl(classified)
VEPE	Vehicle Experimental & Proving Establishment(Can)
Vergé	older Swiss dynamites
ves	vessel(s)
vet(s)	veteran(s)
veter	veterinary
VF	code name for a fighter plane of the Naval Air Service
VF	Fr ballistite(see the text)
VG	Vickers gun(automatic)(Brit)
VG	vintovochnaya granata(Rus) (rifle grenade)
VH	code name for a helicopter of the Naval Air Service
VHF	very high frequency(130 to 300 megacycles per second)
VHN	Vickers hardness number
VI	Vancouver Island, Can
VI	Virgin Islands
VI	viscosity index
Vibrite	Ital expl(see the text)
vic- or v-	vicinal
Vic	Victoria
Victoria	Ital sporting propellant
Victorite	Ital cheddite type expl
Vic	Victoria
vide ante	Lat for "see above"
Vigorine	older Swed expl
Vincennite	poisonous mixt used during WW I in Fr chem shells
viol	violet
VIP	very important person (eg, Vice President)
Vir or Va	Virginia
Virite	Ital black powder type expl
visc	viscosity
Vixorite	Span expl(Resina explosiva)
viz	videlicet(Lat) (namely)
VLF	very low frequency(10 to 30 kilocycles per second)
v/m	volts per meter
VM	code name for the Marine Corps Aviation
VMG	Vickers machine gun
VNP	vinylnitrate polymer
Vo	initial velocity
Vo	initial volume
VOA	Voice of America
VOC	Vickers Ordnance Co, England
Voc	vocabulary
vol	volume
volat	volatile, volatilizes
volaty	volatility
VOLSCAN	volume scanning(Radio system)
VOM	volt-ohmmeter
VOW	Volunteer Ordnance Works, Chattanooga, Tenn
VP	Vice President
VPA	Véry pistol ammunition
VPB	code name for the Navy Patrol bomber
VPH	Vickers plate hardness
VPI	Virginia Polytechnic Institute, Blacksburg, Va
VPT	code name for the patrol torpedo plane of the Naval Air Service
vs	versus
VS & ML	Vickers Sons & Maxim Ltd, England
VSW	very short waves
VT	vacuum tube(radio)
Vt	Vermont

VTF	variable time fuze(proximity fuze)
vv	vice versa
VV	vzryvchatoiye veshchestvo(Rus) (expl subst)
VX	code name of an experimental plane of the Naval Air Service

W

W	Wales
W	Waltham Abbey Arsenal, Ess, Engl
W	War
w	water
w	watt(s)
W	West
W	Westinghouse
w	with
W	wolfram(tungsten)
/W	propellant mfd at Waltham Abbey(Brit)
W(poudre)	an old Belg black powder manufd at Wetteren
W(Pulver)	an old Austrian black powder
WA	Watertown Arsenal, Watertown 72, Mass
WA	Watervliet Arsenal, Watervliet, NY
WA	Woolwich Arsenal, England
WAA	War Assets Administration
WAAC	Women's Army Auxiliary Corps (Brit)
WAAF	Women's Auxiliary Air Force (Brit)
WAASC	Women's Army Auxiliary Service Corps(Brit)
WAC or Wac	Women's Army Corps(USA)
WADC	Wright(Patterson)Air Development Center, Patterson Air Base, Ohio
WADF	Western Air Defence Force
WAL	Watertown Arsenal Laboratory
Wallonites	older Belg mining expls
WAPD	Westinghouse Atomic Power Division
W/armt	with armament
Warws	Warwickshire, Engl
WAS	Washington Academy of Sciences
Wash	Washington
Wash, DC	Washington, District of Columbia
WAVES	Women Accepted for Volunteer Emergency Service
WBNS	Water Boiler Neutron Source (Reactor)
WC	War Cabinet(Brit)
WC	War College
w/c	watt per candle
WC	weapon carrier
WCC	War Claims Commission
WCC	War Crimes Commission
WCC	World Council of Churches
WCC	Wyandotte Chemical Corp, Wyandotte, Mich
WCSAC	War Cabinet Scientific Advisory Committee(Brit)
WD	War Department(now DA & DAF)
WDC	Western Defence Command
WE	Western Electric
WE	Westinghouse Electric
Wed	Wednesday
Wélterines or Vélterines	Belg expls based on Amm trinitrocresylate
W/F	wave frequency
WFAGS	Waffenfabrik Aktiengesellschaft Salothurn(Switz)
WFN or WFNA	white fuming nitric acid
wg	weighing
WG	Western Germany
WG	wire gauge
wh	white
WhC	"White Compound" (1,9-dicarboxy-2,4,6,8-tetranitrophenazine-N-oxide)
WhH	White House, Washington,DC
WHOI	Woods Hole Oceanographic Institution,Mass
WhP or WP	white phosphorus
whr	watt-hour(s)
WI	West India
WI	West Indies
WI	wrought iron
Wiley	J.Wiley & Sons, New York 17
Wisc	Wisconsin
WIT	Washington Institute of Technology
Witol	Ger for synthetic toluene
wk	work
wkg	working
wkly	weekly
WM	wood meal
/WM	modified propellant mfd at Waltham
WMFBC	Werkzeugmaschinenfabrik Bührle & Co, Oerlikon-Zürich (Switz)
WO	War Office(Brit)

WO	Warrant Officer
wo	without
WOAS	when on active service
WOD	Wingate Ordnance Depot,Gallup,NM
Worcs	Worcestershire, Engl
w or wo	with or without
WO/W	without weapon
WP	West Point (US Military Academy)
WP or WhP	white phosphorus
WP	wood pulp
WP	Würfelpulvèr(Ger cube or die shape propellant)
WPAFB	Wright-Patterson Air ForceBase, Ohio
WPB	War Production Board
WPC	War Problems Committee
wpc	watt per candle
wpfg	waterproofing
wpn(s)	weapon(s)
WRAC	Women's Royal Army Corps(Brit)
WRAMA	Warner-Robins Air Matériel Area
WRC	War Resources Council
WRD/ES	Woolwich Research Dept/ Explosives Section, England
WRE	Weapons Research Establishment(Australia)
WRNS	Women's Royal Naval Service (Brit)
WROW	Wabash River Ordnance Works, Newport, Ind
WS	Wireless Station
WSEG	Weapons System Evaluation Group
WSMR	White Sands Missile Range, Las Cruces, NM (formerly WSPG)
WSOW	Weldon Springs Ordnance Works, Mo
WSPG	White Sands Proving Ground, Las Cruces, NM (now WSMR)
WSS	War Savings Stamp
WT	War tax
WT	weapon training
wt	weight
WT Inc	Wallace & Tiernan Inc, Buffalo 5, NY(See also LDWTI)
WU	Western Union
WUDO	Western Union Defence Organization
WUTC	Western Union Telegraph Co
WV	Women's Volunteers
WVa or WV	West Virginia
WVOW	West Virginia Ordnance Works, Pt Pleasant, WVa
W/W	with weapons
WWI	World War I
WWII	World War II
Wyo	Wyoming
WyoUniv	University of Wyoming, Laramie, Wyo

X

X	experimental
X	explosive(such as RDX)
X	xenon
X(hour)	code name for the beginning of action(see also H-hour and Z-hour)
X-1, etc	Jap unknown name expls(see the text)
XB	experimental bomber
Xe	Xenon
Xilit	Rus for TNX
Xmas	Christmas
Xylite	Fr for TNX

Y

Y	year
Y	yttrium
Yb	ytterbium
yd	yard(91.44cm)
yel	yellow
Y-gun	code name for a depth-charge launcher
yld	yield
YMCA	Young Men's Christian Association
Yonckites	Belg coal mining expls(see the text)
Yorks	Yorkshire, Engl
yr(s)	year(s)
Yu	Yukon, Canada
Yuc	Yucatan, Mex
Yugo	Yugoslavia
YTS	Yuma Test Station, Yuma, Ariz
YU	Yale University,New Haven, Conn
YWCA	Young Women's Christian Association
y-y or YY	yellow-yellow (double star rocket) (AC signal)

Z

Z	zazhigatel'nyi(Rus)(incendiary)
Z	zenith
z	zero
z	zone
ZA	zenitnaya artilleriya(Rus)(AA artillery)
ZAB	zazhigatel'naya aviobomba(Rus) (incendiary aerial bomb)
Zac	Zacatecas, Mex
Zar	zariad(porokhovoy)(Rus)(charge propellant)

ZEEP	zero energy experiment pile
ZF or Z/F	zone of fire
Z-hour	Zero hour(Brit) same as H-hour(US)
Zn	zinc
ZP	code name for a small dirigible
Zr	zirconium
ZSF	Zündschnurfabriken in Schindellegi (Switz) (see also ISA)
ZV	zazhigatel'noye veshchestvo (Rus) (incendiary substance)

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SUPPLEMENT TO ABBREVIATIONS

ABC	atomic, bacteriological, chemical (warfare)
ack-ack	antiaircraft (originated among Brit signalmen)
Acrolein	Fr CWA of WWI, desgnd Papite
Adamsite	Brit design for diphenylamino-chloroarsine (CWA)
ADCC	Air Defence Control Center
ADF	auxiliary detonating fuze
ADGB	Air Defence of Great Britain
ADRDE	Air Defence Research and Development Establishment (Brit)
AFF	Army Field Forces
AFR	after flame ratio
AFSC	Armed Forces Staff College, Norfolk, Va.
AIR	air-arming impact rocket
Ala	Alabama
Alas	Alaska
ALBM	air-launched ballistic missile
AMSCE	Army Map Service, Corps of Engineers, Washington, DC
ANPD/GE	Aircraft Nuclear Propulsion Department, General Electric
AOMC	Army Ordnance Missile Command
AOTS	Aviation Ordnance Test Station, Chincoteague, Va
APC-BC	armor-piercing capped, ballistic cap
APCC	Americal Potash & Chemical Corporation
APD	Atomic Products Division
APDS	armor-piercing, discarding sabot
Aquinite	Fr desgn for chloropicrin (CWA)
AR	aircraft rocket
ARDC/BMD	Air Research and Development Command, Ballistic Missile Division, Palo Alto, Calif
ARL	aircraft rocket launcher
ARPA	Advanced Research Projects Agency
ART	Army Research Task
ART	automatic range tracking
ARTS	Army Research Task Summary
ASAES	Army Small Arms Experimental Station
A-Stoff	Ger desgn for chloroacetone (CWA)
ATAR	antitank aircraft rocket
ATRAN	automatic terrain recognition and navigation (system)
AUW	air-to-underwater
AW	above water (Brit)

B

BA	US desgn for bromoacetone (CWA)
BAR	Browning automatic rifle
BASF	Badische Anilin & Sodafabriken, Germany
BDF	base-detonating fuze
BENELUX or Benelux	Belgium-Netherlands – Luxemburg
BER	British experimental rocket
Berthollite	Fr for chlorine (CWA of WWI)
BFP	Bureau of Fire Prevention
Bibi	Fr desgn for dibromomethylether (CWA)
Blaukreuz	Ger desgn for some CWA's (See Blue Cross Ammunition in the text)
BLC	base-loaded, capped (shell)
BLC	breech-loading, converted (Brit)
BL&P	blind loaded and plugged (inert loaded proj with plugged tracer cavity)
BL&T	blind loaded with tracer (inert loaded proj with tracer)
Bn	Ger desgn for bromomethylethylacetone (CWA)
BNW or BuWeps	Bureau of Naval Weapons
Bretonite	Fr desgn for iodoacetone (CWA)
BRLI	Bjorksten Research Laboratories, Incorporated
B-Stoff	Ger desgn for bromoacetone (lachrymator)
BuAer	Bureau of Aeronatuics (US Navy) (now part of BuWeps)
Buntkreugz-schiessen	See PATR 2510 (1958), p 271
BuOrd	Bureau of Ordnance (US Navy) (now part of BuWeps)
Burrowite	Mil expl contg AN, TNT & Al (see the text)
BuWeps	Bureau of Naval Weapons (which assumed the responsibility of the BuAer & BuOrd)

C

c	centi = 10^{-2}
|c|	common to both Land and Naval Service (Brit)
CA	US desgn for α-bromobenzylcyanide (CWA)
Camite	Fr desgn for CA (CWA)
Campiellite	Ital desgn for cyanogen bromide (CWA)
CB	Brit abbrn for cyanogen bromide (CWA)
CCIA	Chemical Corps Intelligence Agency
CDA	US desgn for Clark II
CERCHAR	Centre d'Études et de Recherches des Charbonnages, Verneuil, Franc
CF	center fire (Brit)
CGM	Committee on Guided Missiles(US)
CIC	Combat Information Center
Cici	Fr desgn for dichloromethylether (CWA)
CK	symbol for cyanogen chloride(CWA
Clark I	Ger desgn for biphenylchloroarsine (CWA)
Clark II	Ger desgn for biphenylcyanoarsine (CWA)
CN	US desgn for chloroacetophenone (tear gas) (CWA)
CNC	symbol for chloroacetophenone in chloroform
Collongite	Fr desgn for phosgene and diphosgene(CWA)
combstn	combustion
COMP	Charlotte Ordnance Missile Plant, Charlotte, NC
condn	condition
crcb	crucible
CS	cast steel (Brit)
CS	common shell (Brit)
C-Stoff	Ger CWA of WWI, dimethylsulfate 75 & methylchlorosulfonate 25%
CWLR	Chemical Warfare Laboratories Reports
Cyclite	Fr desgn for benzylbromide (CWA)
Cyclon	Ger desgn for mixt of tech methyl- & ethylcyanoformates with ca 10% of esters of chloroformic acid (CW

D

d	deci = 10^{-1}
D	Di-, such as Dinitrobenzene
D	in jato unit nomenclature designates a cast double-base propellant
DA	US desgn for biphenylchloroarsine (CWA)
DAG	Dynamit Aktiengesellschaft, Germany
DC	symbol for biphenylcyanoarsine (C
DEFA	Direction et Études des Fabricati d'Armement (Fr)
DI	dark ignition
Dick	Ger desgn for ethyldichloroarsine (CWA)
Diphosgene	symbol for perchloromethylformate (CWA)

dk	deka = 10
DM	US desgn for Adamsite (diphenylaminochloroarsine) (CWA)
DME/RD	Directorate of Materials and Explosives, Research and Development (Brit)
DMIC	Defense Metals Information Center, Batelle Memorial Institute, Columbus, Ohio
DN	Department of the Navy
DO	dissolved oxygen
DOD	Department of Defense (US)
DP	symbol for diphosgene (trichloromethylchloroformate) (CWA)
DR	direction ranging
DR	distant range
DRE	Defense Research and Engineering
DRL	Defense Research Laboratory
DSFS	discarding sabot fin stabilized (projectile)
D-Stoff	Gern desgn for phosgene and diphosgene (CWA)
DT	day tracer (Brit)
DWM	Deutche Waffen- und Munitionsfabriken, Germany

E

ED	US desgn for Ger CWA "Dick" (ethyldichloroarsine)
EDS	Explosives Development Section, PicArsn, Dover, NJ
EI	Experiments Incorporated (US)
elecy	electricity
EOD	Explosive Ordnance Disposal
EODS-NPP	Explosive Ordnance Disposal Service – Naval Propellant Plant
Erlen fl	Erlenmeyer flask
E-Stoff	Ger desgn for cyanogen bromide (CWA)
EW	Electronic Warfare

F

FFAR	folding fin aircraft rocket
FG	fine grain (Brit)
FM	fulminate of mercury (see MF)
FOCOL	field of fire, observation cover & concealment, obstacles, lines of communication
Forestite or Vincennite	Fr desgns for hydrocyanic acid (CWA)
FOURA	forward observation unit, Royal Artillery (Brit)
FP	flashless propellant
FPC	Fire Prevention Code
FQ	quick fuze
FR	flash ranging
Fraissite	Fr desgn for benzyliodide (CWA)
FS	flash spotting
FS	forged steel (Brit)
F-Stoff	Ger desgn for $TiCl_4$, smoke-producing agent of WWI

G

G	giga = 10^9
GA	symbol for ethylphosphorodimethylamidocyanadate or **Tabun** (see PATR 2510, p Ger 204, under Trilons) (CWA)
Gargoyle	See KUD-1
GASM	guided air-to-surface missile
GB	green star, blinker, parachute (US)
GB	symbol for isopropylmethylphosphonofluoridate or **Sarin** (see PATR 2510, p Ger 204, under Trilons) (CWA)
GB-4	glide bomb (see the text)
GD	symbol for pinacolylmethylphosphonofluoridate or **Soman** (see PATR 2510, p Ger 204, under **Trilons**) (CWA)
Gelbkreuz	Ger desgn for Mustard Gas and for some other CWA's (see Yellow Cross Ammunition in the text)
GF	gunfire
GG	Gardner-Gatling (Brit)
GGS	gyro gunsight
giga	10^9
GMCM	guided missile countermeasure
GOCO	Government owned, contractor operated
GOGO	Government owned, government operated
GOOSE	code name for air- to-air missile with turbojet engine
GOW	Gopher Ordnance Works, Rosemont, Minnesota
Grünkreuz	Ger desgn for some CWA's (See Green Cross Ammunition in the text)
GY	green-yellow, double star (US)

H

h	hecto = 10^2

HA	high angle (Brit)
HAPO	Hanford Atomic Products Operation
HC	US desgn for smoke-producing agent of WWI: Zn + $ZnCl_2$ + ZnO
HEF	high energy fuel
HEL	high-explosive, light (shell)
HEL	Human Engineering Laboratory, Aberdeen PG, Md
HES	high-explosive, smoke (shell)
HL	desgn for Mustard Gas-Lewisite mixt (CWA)
HN	desgn for nitrogen Mustard Gas (CWA)
Homomartonite	Fr desgn for bromomethylethylacetone (CWA)
HOP	Hoosier Ordnance Plant, Indiana Arsenal, Charleston, Ind
HT	symbol for "Mustard Gas-Agent T" (CWA)
HumRRO	Human Resources Research Office
HVAPDSFS	hypervelocity armor-piercing, discarding sabot, fin stabilized (projectile)
HVTP-T	hypervelocity, target practice, tracer
H-Warhead	warhead contg a nuclear fusion device

I

IED	Industrial Engineering Division (changed to IMED), PicArsn, Dover, NJ
IGFarbenind	Interessengemeinschaft Farben-industries, Germany
IM	insoluble matter
IMED	Industrial Maintenance and Engineering Division, PicArsn, Dover, NJ
INS	International Notational System
invest	to investigate
investd	investigated
investg	investigating
investn	investigation
IRFNA	inhibited red fuming nitric acid
IV	initial velocity

J

JANMB	Joint Army-Navy Munition Board
JB-2	an Amer version of the Ger V-1 (see PATR 2510, p Ger 213) (called also Loon)
JCS	Joint Chiefs of Staff

K

kilo	10^3
Klop	Ger desgn for chloropicrin (CWA)
K-Stoff	Ger desgn for CWA of WWI: monochloromethylcarbonate 91.4 & dichloromethylcarbonate 8.6%
K2-Stoff or KII-Stoff	Ger desgn for phenyl-iso-cyanochloride (CWA)
KT	Brit desgn for $SnCl_4$ used as smoke-producing agent
KUD-1	a remote controlled glide bomb, also called **Gargoyle**
KW	symbol for signal pistol

L

L	Lewisite (CWA)
LA	light artillery
Lacrimite	Fr desgn for thiophosgene (CWA)
Lchr	launcher
LEDC	low energy detonating cord
LEL	lower explosion limit
Lewisite	US desgn for vinylchloroarsine
LG	large grain (Brit)
LM	Lee-Metford (rifle) (Brit)
LMNR	lead mononitroresorcinate
LMR	light machine rifle
Loon	see JB-2
Lost or Gelbkreuz	Ger desgns for Mustard Gas (CWA)
LOZ	liquid ozone (oxidizer for some liq rocket fuels
LS or |L|	Land Service (Brit)
LVP	Launch Vehicles Programs, Washington, DC

M

M	mega = 10^{12}
MA	medium artillery
MAA	Mathematical Association of America, Univ of Buffalo, NY
MAA	medium antiaircraft (artillery)
MAC	maximum allowable concentration (in determination of toxicity)
Manganite or Vincennite	Fr desgns for hydrocyanic acid (CW
Martonite	Fr desgn for CWA of WWI: bromoacetone 80 & chloroacetone 20%
MATS	Military Air Transport
Mauginite or Vitrite	Fr desgns for cyanogen chloride (CWA)
MB	missile battalion
MC	muzzle cap
MD	US desgn for methyldichloroarsine (CWA)

M-day	the day on which mobilization shall begin
MDF	mild detonating fuze
MDS	modified strip cordite
MDT	modified tubular cordite
ME	Martini-Enfield (rifle) (Brit)
MED	Materials Explosives Division (Brit)
mega	10^6
mesrt	measurement
MethylDick	Ger desgn for methyldichloroarsine (CWA)
MH	Martini-Henry (rifle) (Brit)
micro	10^{-6}
milli	10^{-3}
MISUM	Monthyl Intelligence Summary
ML	metal lined (Brit)
MLD	minimum lethal dose (toxicity)
MLE	magazine Lee-Enfield (rifle) (Brit)
M^{le}	Modèle (Fr for Model)
MLM	magazine Lee-Metford (rifle) (Brit)
MLRG	muzzle-loading rifled gun
MRC	Mathematics Research Center, US Army, Univ of Wisconsin
MTF	mechanical time fuze
Mustard Gas	Brit desgn for dichlorodiethylsulfide (CWA) (See also Yperite)
MWDD	Miscellaneous Weapons Development Department (Brit)

N

n	nano = 10^{-9}
NAADC	North American Air Defense Command
nanon	10^{-9} meter, millimicron
NARL	Naval Aeronautical Rocket Laboratory
NAS	National Academy of Science
NASC	National Aeronautics and Space Council
NDCA	Nuclear Development Corporation of America
NE	nose ejection
NFPA	National Fire Prevention Association
NFSAIS	National Federation of Science Abstracting and Indexing Services
n^o	Fr abbreviation for numéro (number)
NOLC	Naval Ordnance Laboratory, Corona, Calif
NP	Napalm
NPGS	Naval Post Graduate School, Monterey, Calif
NQ	nitroguanidine flashless (propellant) (Brit)
Nr	Ger abbreviation for Nummer (number)
NS	Naval Service (Brit) (see also /N)
NSF	National Science Foundation
NT	night tracer (Brit)
NTS	Naval Torpedo Station, Newport, RI
NWL	Naval Weapons Laboratory, Dahlgren, Va

O

OARP	Office of Advanced Research Programs, Washington, DC (superseding OASR)
OASD	Office of Assistant Secretary of Defense
OASR	Office of Aeronautical and Space Research (now OARP)
OC	Ordnance Corps
OEHO	Ordnance Engineering Handbook Office, Duke Univ, Durham, NC
OMRO	Ordnance Material Research Office
OMTF	Ordnance Missile Test Facility, White Sands, NM
ONI	Office of Naval Intelligency
Opacite	Fr desgn for $SnCl_4$, smoke-producing agent of WWI
ORDP	Ordnance Pamphlet
OSFD	Office of Space Flight Development (now OSFP)
OSFP	Office of Space Flight Programs, Washington, DC (superseding OSFD)
OT	ordinary temperature
OTCM	Ordnance Technical Committee Minutes
OTIO	Ordnance Technical Intelligence Office
OTIS	Ordnance Technical Intelligence Service (Aberdeen Proving Ground, Md)
OvF	Overhead fire
OZTC(HA)	Organization and Training Center (Heavy Artillery) (US)

P

p	pico = 10^{-12}
PAC	pilotless aircraft
PANAGRA	Pan-American-Grace Airways

Papite	Fr desgn for acrolein used in WWI as a lachrymator
PBAA	polybutadiene acrylic acid (used in some Thiokol propellants)
PD	symbol for phenyldichloroarsine (CWA)
Per	Ger desgn for trichloromethyl-chloroformate (CWA)
Phosgene	desgn for carbonylchloride, $COCl_2$ (CWA)
pico	10^{-12}
picon	10^{-12} meter = micromicron
PIR	pressure-arming impact rocket
Pk How	pack howitzer
pltlts	platelets
plts	plates
P(Mixture)	a mixture of pebble and fine grain propellants (Brit)
PN	percussion nose
posn	position
PPCo	Pacific Powder Company, Tenino, Wash
prpnt or proplnt	propellant
PS	US desgn for chloropicrin (CWA)
PT	percussion tube (Brit)

Q

Q	chemical agent of specialized application
QB or qb	quick burning (propellant)
QFC	quick firing converted (Brit)

R

RAD	radiation absorbed dose (a unit of absorbed dose of ionizing radiation)
RAT	rocket-assisted torpedo
Rationite	Fr desgn for smoke-producing mixt of WWI: chlorosulfonic acid & dimethylsulfate
RBLG	rifled breech-loading gun
RC	reduced charge (ammunition)
RD 38	a system of interior ballistics used by the Brit
RDF	radio direction finder
reqt	requirement
RF	rye flour
RGF	Royal Gun Factory (Brit)
RGPF	Royal Gunpowder Factory (Brit)
RL	Royal Laboratory (Brit)
RML	rifle medium light (Brit)
RNTF	Royal Navy Torpedo Factory (Brit)
ROD	Rochester Ordnance District is now absorbed into NYOD
ROD	Rossford Ordnance Depot, Toledo, Ohio
RPE	Rocket Propulsion Establishm (Brit)
RR	recoilless rifle
RSAF	Royal Small Arms Factory

S

S(mixture)	Brit smoke-producing agent of WWI: K nitrate 45, pitch 30, sulfur 12, borax 9 & glue 4%
S20	an Ital expl (see the text)
SAE	Society of Automotive Enginee
S&W	Smith & Wesson (revolver)
SC	solid case (Brit)
SC	solventless cordite (Brit)
SCAR	subcaliber aircraft rocket
SCEL	Signal Corps Engineering Laboratory
SD	short delay
SDT	self-destroying tracer
SEM	Station d'Essais de Montluçon (Fr)
SFG	sulfurless fine grain (powder) (Brit)
SID	Scientific Intelligence Digest
SK	Brit & US desgn for ethyliodo-acetate (CWA)
SLOP	St Louis Ordnance Plant, St Louis, Mo
smkls	smokeless
SMLE	short model Lee-Enfield (rifle) (Brit)
SMW	School of Mine Warfare, Yorktown, Va
SPCGM	self-propelled caterpillar gunmount
SSR	spin-stabilized rocket
STRAC	Strategic Army Corps
Sulvanite	Fr desgn for ethylchlorosulfo-mate and for bromine (CWA)
Surpalite	Fr desgn for trichloromethyl-chloroformate (CWA)
SWAC	Special Weapons Ammunition Command
SWC	Special Weapons Center

T

T	tera = 10^{12}
T	symbol for CWA of specialized application
TA	tractor-drawn artillery
TCBM	transcontinental ballistic missile
tera	10^{12}
TH	symbol for incendiary CWA "thermate"
TH1	symbol for specific composition of CWA "thermite"
TH2	symbol for specific compn of CWA "thermate"
TH3	specific compn of chemical agent "thermate"
TJ	turbojet
TML	tetramethyl lead
TNTBP	mixt of TNT with black powder
Tonite	Fr desgn for chloroacetone (CWA)
TOP	total obscuring power (area in sq ft covered by the smoke produced by 1 lb of material)
T-Stoff	Ger desgn for xylylbromide (CWA)

U

UERD	Underwater Explosions Research Division, Norfolk Naval Shipyard
UOS	Underwater Ordnance Station, Newport, RI
USAOMC	United States Army Ordnance Missile Command, Redstone Arsenal, Alabama

Note: Subordinate units included ABMA, ARGMA and WSMR

USL	Underwater Sound Laboratory, New London, Conn
USPHS	US Public Health Service
UUM	underwater-to-underwater missile

V

VAR	vertical aircraft rocket
VGFAG	Vereinigte Glanzstoff-Fabriken Aktiengesellschaft
Vicastrong	Vickers-Armstrong (Brit)
Villantite	Fr desgn for ethylchlorosulfonate (CWA)
Vincennite or Manganite	Fr desgns for hydrocyanic acid (CWA)
VINITI	Vsesoyuznyi Institoot Naoochnoi i Tekhnicheskoi Informatsiyi (Rus) (All Union Institute of Scientific & Technical Information)
Vitrite	Fr desgn for cyanogen chloride (CWA)
VKRPF	Vereinigte Köln-Rottweiler Pulverfabriken, Germany
Vomiting Gas	Brit desgn for chloropicrin
VS	vent sealing (Brit)
VTOL	vertical take-off and landing

W

W	chemical agent
WAAC	Woolwich Arsenal
WASAG	Westfälisch-Anhaltische Sprengstoff Aktiengesellschaft, Germany
WB	white star, blinker, parachute
WF	wood flour
WhF	wheat flour
WSC	white star cluster
WSP	white star-parachute

Y

Yperite	Fr desgn for dichlorodiethylsulfide (CWA); Brit desgn Mustard Gas; US desgn HS and Ger desgns Lost or Gelbkreuz

LIST
OF
ABBREVIATIONS FOR BOOKS AND PERIODICALS USED AS REFERENCES IN THIS WORK

(Abbreviations not included in this list are the same as used by the American Chemical Society in Chemical Abstracts)(See also supplement to this list, pp 75-6)

Note: When the name of a journal was changed, the words, "changed to," "formerly," "now," etc are followed here not by the full name of the journal but by the abbreviation used in this dictionary. This is done because the journals are arranged alphabetically according to their abbreviations and not according to the full names of the journals

ADL Punch Cards	Arthur D. Little Inc, "Punch Card Recording of Data on Explosives," Cambridge, Mass (1954)
AFChJ	Armed Forces Chemical Journal (Washington, DC)
AHThCatalog	Arthur H. Thomas' Catalog of Apparatus and Reagents, Philadelphia (1950)
AIChE	American Institute of Chemical Engineers (Journal published beginning 1955)
All&EnExpls(1946)	APG, "Allied and Enemy Explosives," Aberdeen, Md (1946)
AmChemJ	American Chemical Journal (discontinued in 1913)
AmJPhys	American Journal of Physics
Anal	Analyst(Cambridge, England)
AnalChem	Analytical Chemistry (formerly IEC,Anal Ed)
AnalChimActa	Analytica Chimica Acta(Amsterdam)
AngChem	Angewandte Chemie, formerly Zeitschrift für Angewandte Chemie (Berlin)
Ann	Annalen der Chemie (Justus Liebeg's)
AnnActas	Annales de l'Association Canadienne–Française pour l'Avancement des Sciences (Montreal)
AnnChim(Paris)	Annales de Chimie (Paris), formerly AnnChimPhys
AnnChim(Rome)	Annali di Chimica(Rome), formerly AnnChimAppl
AnnChimAnal	Annales de Chimie Analytique, Paris
AnnChimAppl	Annali di Chimica Applicata(Rome), now AnnChim(Rome)
AnnChimPhys	Annales de Chimie et de Physique(Paris), now AnnChim
AnnPhys	Annales de Physique, Paris
AnnPhysik	Annalen der Physik, Leipzig
APG	Aberdeen Proving Ground, Maryland
ArchParm	Archiv der Pharmazie(Berlin)(suspended in 1944 and resumed in 1950)

ArchToxicol	Archiv für Toxicologie (Berlin)
ArmamentEngrg(1954)	Anon, "Elements of Armament Engineering," US Military Academy, West Point, NY (1954)
Arms & Expls	Arms and Explosives (Brit) (discontinued Dec 1920)
ArOrdn	Army Ordnance, changed in 1947 to Ordn
ARSJ	American Rocket Society Journal, formerly JetPropn and before that JARS
ASTMBull	American Society for Testing Materials Bulletin
ASTMProc	American Society for Testing Materials Proceedings
ASTMStds	American Society for Testing Materials Standards
AttiAccadLinceiMem	Atti della Accademia Nazionale dei Lincei, Memorie
AttiAccadLinceiRend	Rendiconti (formerly Atti della Reale Accademia dei Lincei)
AttiAccadTor	Atti della Accademia delle Scienze di Torino
Barnett(1919)	E. deBarry Barnett,"Explosives,"Van Nostrand, NY (1919)
Bebie(1943)	J. Bebie,"Manual of Explosives, Military Pyrotechnics and Chemical Warfare Agents,"Macmillan, NY, 1943
Beil	Beilstein's Organische Chemie, 4th ed
Beil 1,350,(170), [200] & {550}	Beilstein vol 1, p350 of the Hauptwerk; p(170) of the Erstes Ergänzung; p [200] of the Zweites Ergänzung and p {550} of the Drittes Ergänzung
Belgrano(1952)	C. Belgrano, "Gli Esplosivi," Hoepli, Milano (1952)
Ber	Berichte der Deutschen Chemischen Gesellschaft, now called ChemBer
Beyling&Drekopf(1936)	C. Beyling & K. Drekopf,"Sprengstoffe und Zündmittel,"Springer, Berlin(1936)
Bichowsky & Rossini (1936)	F.R.Bichowsky & F.D. Rossini, "Thermochemistry of Chemical Substances," Reinhold, NY (1936)
BIOS Rept	British Intelligence Objectives Sub-Committee Report
Blaster's Hdb(1952)	Blaster's Handbook, E.I. duPont de Nemours & Co, Wilmington, Del
Blinov(1948 - 1952)	A.D.Blinov, "Artillery Courses," Voyenizdat, Moscow ((1948 - 1952)
BrA	British Abstracts (discontinued Dec 1953)
BrJApplPhys	British Journal of Applied Physics (London)
BrP or BritP	British Patent
Brunswig, Expls (1912)	H.Brunswig,"Explosives,"Wiley,NY(1912) (Translated by Munroe & Kibler)
Brunswig,Props(1926)	H.Brunswig,"Das Rauchlose Pulver,"W. De Gruyter, Berlin(1926)
Budnikov et al(1955)	M.A.Budnikov et al, "Explosives and Propellants,"Oborongiz, Moscow (1955)
BullBelg	Bulletin de la Société Chimique de Belgique(Brussels)
BullBurStds	Bulletin of the Bureau of Standards
BullFr	Bulletin de la Société Chimique de France
BurMines	US Bureau of Mines, Pittsburgh, Pennsylvania
BurStds	National Bureau of Standards, Washington, DC
C	See Conf
CA	Chemical Abstracts
CanChemProcessing	Canadian Chemical Processing (Toronto)
CanJChem	Canadian Journal of Chemistry(Ottawa), formerly CanJRes,Sect B
CanJRes	Canadian Journal of Research(Ottawa). Its Sect B is now the CanJChem and its Sect F, CanJTechn
CanJTech	Canadian Journal of Technology(Ottawa), formerly CanadJRes,Sect F
CanNatlResCouncilRepts	Canada National Research Council Reports
CEARapp	Commission Énergie Atomique Rapport (France)
Cellulosechem	Cellulosechemie (Berlin)(discontinued in 1936)

C&EN	Chemical and Engineering News
Chem	Chemist(New York)
ChemAge	Chemical Age (London)
ChemAnal	Chemist Analyst(Phillipsburg,New Jersey)
ChemBer	Chemische Berichte (supersedes Ber)
ChemEngrg	Chemical Engineering, formerly Chem & MetEngrg
ChemEngrgProgr	Chemical Engineering Progress(New York)
Chem&Ind	Chemistry and Industry(London), published together with JSCI, but is now separated
ChemInd	Chemical Industries, changed to ChemIndWeek, on Jan 20, 1951
ChemIndWeek	Chemical Industries Week, changed to ChemWeek on June 2, 1951
ChemMetEngrg	Chemical and Metallurgical Engineering, now ChemEngrg
ChemN	Chemical News and Journal of Industrial Science
ChemObzor	Chemický Obzor(Chemical Review, Prague)(now ChemPrûmysl)
ChemPrûmysl	Chemický Prûmysl(Prague)
ChemRevs	Chemical Reviews(Baltimore, Md)
ChemRubHdb	Chemical Rubber Publishing Co, Handbook of Chemistry & Physics, Cleveland, Ohio, 38th ed (1956-7)
ChemWbl	Chemisch Weekblad(Amsterdam)
ChemWeek	Chemical Week(formerly ChemIndWeek)
ChemZtg	Chemiker Zeitung (Köthen,Anhalt). In Jan 1951 the name was changed to "Chemiker Zeitung ehemalsKöthen"(Stuttgart) and in Jan 1954 to "Chemiker Zeitung mit Chemische Apparatur und Chemie-Börse"(Heidelberg)
ChemZtr	Chemisches Zentralblatt(Berlin)
Chim&Ind(Paris)	Chimie et Industrie(Paris)
Chim e Ind(Milan)	Chimica e l'Industria(Milan)
CIOS Rept	Combined Intelligence Objectives Sub-Committee Report
Clark & Hawley(1957)	G.L.Clark & G.G. Hawley, Edits,"The Chemical Encyclopedia,"Reinhold, NY (1957)
Colver(1918)	E.deW.S.Colver, "High Explosives," Van Nostrand, NY (1918)
CondChemDict(1956)	A. & E.Rose, ed,"The Condensed Chemical Dictionary,"Reinhold, NY(1956),
CondChemDict(1950)	former edition(1950) was directed by F.M.Turner
Conf or C	Confidential
CR	Comptes Rendues de l'Académie des Sciences(Paris)
Cranz(Vol & year)	K.J.Cranz,"Lehrbuch der Ballistik",Springer,Berlin(1925-1927)
Cundill(1889)	J.P.Cundill,"A Dictionary of Explosives,"London(1889). The French translation was published in MP 5,235-354(1892) and 6,7-132(1893)
Daniel(1902)	J.Daniel,"Dictionnaire des Matières Explosives,"Dunod, Paris (1902)
Davis(1943)	T.L.Davis,"The Chemistry of Powder and Explosives,"Wiley, NY(1943)
Degering(1950)	E.F.Degering,"An Outline of Organic Nitrogen Compounds,"Univ Lithoprinters, Ypsilanti,Mich(1950)
DoklAkadN	Doklady Akademii Nauk(Proceedings of Academy of Science,Russia)
Dorée(1947)	C.Dorée,"The Methods of Cellulose Chemistry,"Chapman & Hall, London (1947)
DRP	Deutsches Reichs Patent(German State Patent)
Durrans(1957)	T.H.Durrans,"Solvents,"Van Nostrand, NY(1957)
Elkins(1950)	H.B.Elkins, "The Chemistry of Industrial Toxicology," Wiley(1950)
Ephraim(1949)	F.Ephraim, "Inorganic Chemistry," Interscience, NY(1949)

Erikson, Wiley & Wystrach(1956)	J.G.Erikson, P.F.Wiley & V.P.Wystrach, "The 1,2,3- and 1,2,4-Triazines, Tetrazines and Pentazines," Interscience, NY(1956)
Escales,NG & D(1908)	R.Escales, "Nitroglyzerin und Dynamit," Veit & Co, Leipzig(1908)
Escales,Ammonspr(1909)	R.Escales, "Ammonsalpetersprengstoffe," Veit,Leipzig(1909)
Escales,Chloratspr(1910)	R.Escales, "Chloratsprengstoffe," Veit, Leipzig(1910)
Escales, Schwarzpulver (1914)	R.Escales, "Schwarzpulver und Sprengsalpeter," Veit, Leipzig (1914)
Escales, Nitrospr(1915)	R.Escales, "Nitrosprengstoffe," Veit, Leipzig(1915)
Escales, Initialspr(1917)	R.Escales, "Initialsprengstoffe," Veit, Leipzig(1917)
Explosivst	Explosivstoffe, formerly SprTech(Mannheim)
ExplsEngr	The Explosives Engineer, Hercules Powder Co, Wilmington, Del
Faith, Keyes & Clark(1957)	W.L.Faith, D.B.Keyes & R.L.Clark, "Industrial Chemicals," Wiley,NY(1957)
Feodos'ev & Siniarev (1956)	V.J.Feodos'ev & G.B.Siniarev, "Introduction to Rocket Techniques, Oborongiz, Moscow(1956)
FIAT	Field Information Agency, Technical
Fieser & Fieser(1950)	L.F.Fieser & Mary Fieser, "Organic Chemistry," Heath, Boston(1950)
FM	Field Manual
Franklin(1935)	E.C.Franklin, "The Nitrogen System of Compounds," Reinhold,NY(1935)
FrP	French Patent
Gazz	Gazzetta Chimica Italiana(Rome)
GerP	German Patent
Gilman(Vol & year)	H.Gilman, Edit, "Organic Chemistry," Wiley, NY v1(1949), v2(1950), v3(1953), v4(1953)
Giua, Dizionario(Vol & year)	M.Giua & C.Giua-Lollini, "Dizionario di Chimica Generale e Industriale," UTETT, Turin, vols 1-3(1948-1950)
Gmelin(Syst Nr & year)	Gmelin-Krauts Handbuch der Anorganischen Chemie, Verlag Chemie, Berlin,8th ed(1928-1958)
Gody(1907)	L.Gody, "Traité des Matières Explosives," Wesmael-Charlier,Namur(1907)
GornyiZh	Gornyi Zhurnal(Mining Journal, Russia)
Gorst(1957)	A.G.Gorst, "Propellants and Explosives," GIOP, Moscow(1957)
Groggins(1958)	Ph.H.Groggins,ed, "Unit Processes in Organic Synthesis," McGraw-Hill, NY(1958)
Hackh's(1944)	Hackh's Chemical Dictionary, "Blackiston, Philadelphia(1944)
HACSIR	Honorary Advisory Council for Scientific and Industrial Research(Canada)
Hayes(1938)	T.J.Hayes, "Elements of Ordnance," Wiley, NY(1938)
Helv	Helvetica Chimica Acta(Basel,Switzerland)
Heuser(1944)	E.Heuser, "The Chemistry of Cellulose," Wiley,NY(1944)
Hickinbottom(1948)	W.J.Hickinbottom, "Reaction of Organic Compounds," Longmans-Green, London(1948)
Houben 4(1941)	J.Houben, "Die Methoden der Organischen Chemie," G.Thieme, Leipzig, v4(1941)
IA	Iron Age
ICT	International Critical Tables,McGraw-Hill,NY, v 1(1926), v 2(1927), v 3 (1928), v 4(1928, v 5(1929), v 6(1929) & v 7(1930)
IEC	Industrial and Engineering Chemistry
IEC, AnalEd	Industrial and Engineering Chemistry Analytical Edition(changed to AnalChem)
IGFarbenind	Interessengemeinschaft Farbenindustrie
IndChem	Industrial Chemist(London)

InorgSynth(Vol & year)	Collective,"Inorganic Syntheses,"McGraw-Hill,NY, vols 1-4(1939-1953)
Instr	Instruments(Pittsburgh)
Instrn	Instrumentation(Philadelphia)
IzvestAkadN	Izvestiya Akademii Nauk(Bulletin of Academy of Science, Russia)
Izzo,Pirotechnia(1950)	A.Izzo, "Pirotecnia e Fuochi Artificiali," Hoepli, Milano(1950)
Izzo,Minatore(1953)	A.Izzo, "Manuale del Minatore Esplosivista," Hoepli,Milano(1953)
Jacobs(1949)	M.B.Jacobs, "The Analytical Chemistry of Industrial Poisons, Hazards and Solvents," Interscience, NY,(1949)
JACS	Journal of the American Chemical Society
JAgrFChem	Journal of Agricultural and Food Chemistry
JapP	Japanese Patent
JApplChem(London)	Journal of Applied Chemistry(London), called J SCI prior to 1951
JApplPhys	Journal of Applied Physics, formerly called Physics
JahresberCTR	Jahresbericht der Chemisch-Technischen Reichsanstalt
JAOAC	Journal of the Association of Official Agricultural Chemists
JARS	Journal of the American Rocket Society(changed to JetPropn and then to ARSJ)
JChemEduc	Journal of Chemical Education
JChemPhys	Journal of Chemical Physics
JChimPhys	Journal de Chimie Physique(Paris)
JCS	Journal of the Chemical Society(London)
JetPropn	Jet Propulsion(formerly JARS, now ARSJ)
JFrankInst	Journal of the Franklin Institute
JIEC	Journal of Industrial Engineering Chemistry,changed in 1923 to IEC
JISI(London)	Journal of the Iron and Steel Institute(London)
JMakrChem	Journal für Makromolekuläre Chemie, formerly JPrChem. Called MakrChem since 1947
JOC	Journal of Organic Chemistry
JOil & Col	Journal of the Oil and Color Chemists Association(London)
JOptSocAm	Journal of the Optical Society of America
Jordan(1954)	T.E.Jordan, "Vapor Pressure of Organic Compounds," Interscience, NY(1954)
JPhChem	Journal of Physical Chemistry, except the years 1947-1951 when it was called JPhCollChem
JPhCollChem	Journal of Physical and Colloid Chemistry (See JPhChem)
JPolymerRes	Journal of Polymer Research, now JPolymerSci
JPolymerSci	Journal of Polymer Science, formerly JPolymerRes
JPraktChem	Journal für Praktische Chemie, discontinued in May 1943. Continued as JMakrChem, then as MakrChem. Resumed as a separate journal since March 1956
JRNBS	Journal of Research of the National Bureau of Standards
JRussPhChemSoc	See ZhRusFiz-KhimObshch
JSCI	Journal of the Society of Chemical Industry, called JApplChem since 1951
JScInst	Journal of Scientific Instruments
Karrer(1950)	P.Karrer,"Organic Chemistry," Elsevier,NY(1950)
Kast(1921)	H.Kast,"Spreng- und Zündstoffe," Braunschweig(1921)
Kast-Metz(1944)	H.Kast & L.Metz,"Chemische Untersuchung der Spreng- und Zündstoffe," Vieweg,Braunschweig(1944)
KhimReferatZh	Khimicheskii Referativnyi Zhurnal, now called ReferatZhKhim
Khimstroi	Journal for Projecting and Construction of the Chemical Industry (discontinued in 1935)

KhimTekhnTopliva	Khimia i Teknologiya Topliva (Chemistry and Technology of Fuels, Russia)
Kirk & Othmer (vol and year)	R.E.Kirk & D.F.Othmer, eds, "Encyclopedia of Chemical Technology," Interscience, NY, vols 1 to 15(1947-1956) and Supplements
KollBeih	Kolloid-Beihefte, merged in 1943 with KollZts
KollZh	Kolloidny Zhurnal(Colloid Journal,Russia)
KollZts	Kolloid-Zeitschrift, formerly Kolloid-Beih
Kunstst	Kunstoffe (Munich)
Land-Börnst (Vol & year)	Landolt-Börnstein, Physikalisch-Chemische Tabellen, J.Springer, Berlin, 5th ed, v 1(1923), v 2(1923) and Supplements; 6th ed, v 1(1950-2), v 2(1956) and v 4(1956-7) (vol 3 was not available)
Lange(1956)	N.A.Lange,"Handbook of Chemistry,"Handbook Publishers, Sandusky,Ohio, 9th ed(1956)
MAF	Mémorial de l'Artillerie Française(Paris)
MakrChem	Die Makromolekuläre Chemie(Munich), formerly JMakrChem
Marshall 1(1917)	A.Marshall,"Explosives,"Chruchill, London,vol 1 (1917)
Marshall 2(1917)	Ditto, vol 2 (1917)
Marshall 3 (1932)	Ditto, vol 3(1932)
Mellor, (Vol & year)	J.W.Mellor,"A Comprehensive Treatise on Inorganic and Theoretical Chemistry,"Longmans Green & Co, London and NY(1922-1947)
Mellor(1946)	G.D.Parkes & G.W.Mellor, "Mellor's Modern Inorganic Chemistry,"Longmans-Green, London (1946)
MemRept or MR	Memorandum Report
Merck(1952)	Anon, The Merck Index, Merck & Co, Inc,Rahway, NJ(1952)
Merriam-Webster's(1951)	"Merriam-Webster's Unabridged Dictionary," Merriam Co Springfield, Mass(1951)
Meyer(1943)	M.Meyer,"Explosives,"Crowell, NY(1943)
MikrChem	Mikrochemie(combined with Mikrochimica Acta)
Molina(1930)	R.Molina, "Esplodenti e Modo di Fabricarli," Hoepli,Milano(1930)
Monatsh	Monatshefte für Chemie(Vienna)
MP	Mémorial des Poudres(Paris)
MR or MemRept	Memorandum Report
MSCE	Mémorial des Services Chimiques de l'État(Paris)
Naoúm, Expls(1927)	P.Naoúm,"Schiess- und Sprengstoffe,"Steinkopf, Dresden & Leipzig(1927)
Naoúm, NG(1928)	P.Naoúm,"Nitroglycerin and Nitroglycerin Explosives,"Williams & Wilkins, Baltimore(1928)(Translated by Symmes)
Nature	Nature(London)
NavOrd Rept	Naval Ordnance Report
NBSJR	National Bureau of Standards, Journal of Research (see JRNBS)
NC	Nitrocellulose (combined with SS in 1943)
NDRC Rept	National Defense Research Council Report
NOrd or NORD Rept	Naval Ordnance Report
OffGaz	OfficialGazette, US Patent Office, Dept of Commerce, Washington 25,DC
OffJ	Official Journal(British Patents)
Ohart(1946)	T.C.Ohart,"Elements of Ammunition,"Wiley, NY (1946)
ONRRR	Office of Naval Research, Research Reviews
OpNav(Publications)	Office of the Chief of Naval Operations(Publications), Washington,DC
Ordn	Ordnance, formerly ArOrdn
OrgSynth(Vol & year)	"Organic Syntheses",Wiley,NY, Coll Vols 1(1941), 2(1943), 3(1955) and individual vols 30(1950), 31(1951), 32(1952), 33(1953), 34(1954), 35(1955) & 36(1956)
OSRD Rept	Office of Scientific Research and Development Report

Ott (1954-1955)	E.Ott,ed,"Cellulose and Cellulose Derivatives,"Interscience,NY, vol 5; Part 1(1954), Part 2(1954), Part 3(1955)
P or Pat	Patent
PA or PicArsn	Picatinny Arsenal, Dover, NJ
PACLR	Picatinny Arsenal Chemical Laboratory Report
PAGLR	Picatinny Arsenal General Laboratory Report
PACT	Prévention des Accidents-Contrôles Techniques(Bruxelles)
PAMR or PicArsnMemRept	Picatinny Arsenal Memorandum Report
Partington(1950)	J.R.Partington, "A Textbook of Inorganic Chemistry," Macmillan, London(1950)
Pascal(1930)	P.Pascal,"Explosifs, Poudres, Gaz de Combat,"Hermann, Paris(1930)
PATR or PicArsnTechRept	Picatinny Arsenal Technical Report, Dover, New Jersey
PATR 1401(Rev 1)(1950)	Wm. H.Rinkenbach & A.J.Clear, Picatinny Arsenal Technical Report No 1401, (1950), "Standard Laboratory Procedure for Sensitivity, Brisance and Stability of Explosives"
PATR 1740(Rev 1)(1958)	W.R.Tomlinson, Jr & O.E.Sheffield, Picatinny Arsenal Technical Report No 1740, Revision 1 (1958), "Properties of Explosives of Military Interest"
PATR 2510(1958)	B.T.Fedoroff et al, Picatinny Arsenal Technical Report No 2510(1958) "Dictionary of Explosives, Ammunition and Weapons" (German Section)
PB Rept	Publication Board's Report (of the US Office of Technical Services)
PBL Rept	Ditto, Library of Congress
Pepin Lehalleur(1935)	J.Pepin Lehalleur, "Traité des Poudres, Explosifs et Artifices," Ballière et Fils,Paris(1935)
Pérez Ara(1945)	A.Pérez Ara,"Tratado de Explosivos,"Cultural, La Habana(1945)
Perry(1950)	J.H.Perry,"Chemical Engineers' Handbook",McGraw-Hill, NY(1950)
PhilMag	Philosophical Magazine(London)
PhilTr	Philosophical Transactions of the Royal Society of London
PHS Rept	Public Health Service Report(USA)
PhysRevs	Physical Reviews
PhysZSow	Physikalische Zeitschrift der Sowjetunion(Leipzig)(discontinued in 1938)
PicArsn	See PA
PrChSoc	Proceedings of the Chemical Society(London)
PromOrgKhim	Promyshlennost' Organicheskoy Khimii(Organic Chemical Industry, Russia)
Protar	Protar (Solothurn, Switzerland)
PrRoySoc	Proceedings of the Royal Society(London)
PrzChem	Przemysl Chemiczny(Warsaw)
QuartRevs	Quarterly Reviews(London)
Quím y Ind	Química y Industria(Barcelona)(discontinued in 1941)
R or Rept	Report
Reagent Chemicals(1950)	"Reagent Chemicals," ACS Specifications, Washington, DC(1950)
Rec	Recueil de Travaux Chimiques des Pays Bas(Amsterdam)
ReferatZhKhim	Referativnyi Zhurnal, Khimiya (Abstract Journal, Chemistry)(Russia)
Reilly(1938)	J.Reilly,"Explosives, Matches and Fireworks,"Van Nostrand,NY(1938)
Rept or R	Report
Rept Invn or RI	Report of Investigation
Res(London)	Research(London)
RevChimInd	Review of Scientific Instruments
RI or ReptInv	Report of Investigation
Riegel,ChemMach(1953)	E.R.Riegel,"Chemical Machinery,"Reinhold,NY(1953)
Riegel,IndChem(1949)	E.R.Riegel,"Industrial Chemistry",Reinhold,NY(1949)
RoczChem	Roczniki Chemii (Chemical Annual, Poland)(formerly ChemikPolski and Chemji)

Sancho(1941)	E.E.Sancho, "Química de Explosivos," A.Aguado,Madrid(1941)
Sanford(1896)	P.G.Sanford, "Nitro-Explosives," Crosby-Lockwood, London(1896)
Sax(1957)	I.Sax, "Dangerous Properties of Hazardous Materials," Reinhold, NY(1957)
Sci	Science(New York)
SciAbs	Science Abstracts
SciAm	Scientific American
Seidell 1(1940)	A.Seidell, "Solubilities of Organic Compounds," Van Nostrand, NY, v 1(1940)
Seidell 2(1941)	v 2(1941) and Suppl (1952)
Seidell Suppl(1952)	
Shidlovskii(1954)	A.A.Shidlovskii, "Pyrotechnics," Oborongiz, Moscow(1954)
Shilling(1946)	A.D.Shilling, "Explosives and Loading of Ammunition" Oborongiz, Moscow, (1946) (in Rus)
Shreve(1956)	R.N.Shreve "The Chemical Process Industries," McGraw-Hill, NY(1956)
Sidgwick,OrgChem of N (1937)	N.V.Sidgwick,"Organic Chemistry of Nitrogen,"Oxford Univ Press, London (1937)
Sidgwick, ChemElems 1 (1950)	N.V.Sidgwick, "The Chemical Elements and Their Compounds," Oxford Univ Press London, v 1(1950)
Siniarev & Dobrovol'skii (1957)	G.B.Siniarev & M.B.Dobrovol'skii "Liquid Propellant Rocket Motors," Oborongiz, Moscow(1957)
Sprgtech	Sprengtechnik (Manheim)(See SS and Explosivst)
SS	Zeitschrift für das gesamte Schiess- und Sprengstoffwesen-Nitrocellulose, suspended in 1944 and followed after WW II by Sprengtechnik(1952) and since 1952 by Explosivstoffe(München)
Stettbacher(1933)	A.Stettbacher,"Die Schiess- und Sprengstoffe,"Barth,Leipzig(1933)
Stettbacher(1948)	A.Stettbacher,"Spreng- und Schiesstoffe,"Rascher Verlag, Zürich(1948)
Sukharevskii & Pershakov (1932)	M.Sukharevskii & F.Pershakov, "Explosives," Moscow(1932)
Sutton(1956)	G.P.Sutton, "Rocket Propulsion Elements,"Wiley,NY(1956)
SvenskKemTidskr	Svensk Kemisk Tidskrift(Stockholm)
Tables Annuelles 5(1926)	Tables Annuelles de Constantes, Gauthier-Villars, Paris v 5(1926)
TAPPI	Technical Association of the Pulp and Paper Industry
Taylor & Gay(1958)	G.Taylor & P.F.Gay, "British Coal Mining Explosives," G.Newnes, London(1958)
TechMan or TM	Technical Manual(US Army)
TechOrd or TO	Technical Order(US Air Force)
TechRept or TR	Technical Report
Tharaldsen(1950)	Th.Tharaldsen, "Ekplosivstoffer," Dreyes Vorlag, Oslo, Norway(1950)
Thorpe(Vol & year)	"Thorpe's Dictionary of Applied Chemistry,"Longmans-Green, London vols 1-12(1937-1956)
Thorpe 4(1949)	Ditto, vol 4(1949)
Thorpe(1917)	Ditto, old edition(it contains some information not given in the new edition)
TM	See TechMan
TO	See TechOrd
TR	See TechRept
Trety'akov(1946)	G.M.Tret'yakov, "Artillery Ammunition," Voyennizdat,Moscow(1946)
TrFaradSoc	Transactions of the Faraday Society(London)
TrRoySoc	Transactions of the Royal Society(London)
U	Unclassified
Ugol'	Coal(Moscow)

Ullmann(Vol & year)	F.Ullmann,"Enzyklopädie der Technischen Chemie,"Urban & Schwarzenberg, Berlin,2nd ed, vol 4(1926) and 3rd ed, vol 1(1951) and following volumes
USNIP	United States Naval Institute Proceedings
UspKhim	Uspekhi Khimii (Progress in Chemistry,Russia)
Van Gelder & Schlatter (1927)	A.P.Van Gelder & H.Schlatter, "History of the Explosives Industry in America," Columbia Univ Press, NY(1927)
Vennin,Burlot & Lécorché(1932)	L.Vennin,E.Burlot et H.Lécorché, "Les Poudres et Explosifs," Ch. Béranger, Paris(1932)
VestnikMoskovUniv	Vestnik Moskovskago Universiteta(Bulletin of University of Moscow)
Vivas,Feigenspan & Ladreda	M.Vivas, R.Feignespan & F.Ladreda, "Polvoras y Explosivos Modernos," Morata, Madrid, v 1(1945), v 2(1946), v 3(1948), v 4(1944) and v 5(1947)
Walker(1953)	J.F.Walker, "Formaldehyde," Reinhold, NY(1953)
Warren(1958)	F.A.Warren, "Rocket Propellants," Reinhold, NY(1958)
Weaver(1917)	E.M.Weaver, "Military Explosives," Wiley NY(1917)
Webster's Collegiate(1953)	"Webster's New Collegiate Dictionary," Merriam & Co, Springfield, Mass(1953)
Weichelt(1953)	F.Weichelt, "Handbuch der Gewerblichen Sprengtechnik," Marhold, Halle-Saale (1953)
Wheland(1949)	G.H.Wheland, "Advanced Organic Chemistry," Wiley, NY(1949)
Zakoshchikov(1950)	A.P.Zakoshchikov, "Nitrocellulose," Oborongiz, Moscow(1950)
ZAnalChem	Zeitschrift für Analytische Chemie, Fresenius(München)
ZAngChem	Zeitschrift für Angewandte Chemie, called AngChem since 1932
ZAnorgChem	Zeitschrift für Anorganische und Allgemeine Chemie (Leipzig)
ZavodLab	Zavodskaya Laboratoriya(Factory Laboratory Journal, Russia)
ZElektrochem	Zeitschrift für Elektrochemie(Berlin)
ZhAnalKhim	Zhurnal Analiticheskoy Khimii(Journal of Analytical Chemistry, Russia)
ZhFizKhim	Zhurnal Fizicheskoy Khimii(Journal of Physical Chemistry,Russia)
ZhKhimProm	Zhurnal Khimicheskoy Promyshlennosti(Journal of Chemical Industry,Russia)
ZhNeorgKhim	Zhurnal Neorganicheskoy Khimii(Journal of Inorganic Chemistry, Russia)
ZhObshchKhim	Zhurnal ObshcheKhimii(Journal of General Chemistry, Russia)
ZhPriklKhim	Zhurnal Prikladnoy Khimii(Journal of Applied Chemistry, Russia)
ZhRusFiz-KhimObshch	Zhurnal Russkago Fiziko-Khimicheskago Obshchestva(Journal of the Russian Physico-Chemical Society)(discontinued in 1930)
ZhTekhnFiz	Zhurnal Teknicheskoy Fiziki(Journal of Technical Physics, Russia)
ZKrist	Zeitschrift für Kristallographie (Frankfurt a/M)
ZPhysChem(Frankfurt)	Zeitschrift für Physikalische Chemie (Frankfurt a/M)
ZPhysChem(Leipzig)	Zeitschrift für Physikalische Chemie(Leipzig)
ZPhisiolChem	Zeitschrift für Physiologische Chemie(Berlin)

[See also "List of Periodicals Abstracted by Chemical Abstracts", The Chemical Abstracts Service, The Ohio State University, Columbus 10, Ohio (1956)]

SUPPLEMENT TO THE LIST OF ABBREVIATIONS FOR BOOKS AND PERIODICALS

Blatt OSRD 2014 (1944)	A.H.Blatt, "Compilation of Data on Organic Explosives," OSRD Report 2014(1944)
Charlot & Bézier (1957)	G.Charlot & D.Bézier, "Quantitative Inorganic Analysis, Methuen, London (1957)
Clift & Fedoroff (Vol & year)	G.D.Clift & B.T.Fedoroff, "A Manual for Explosives Laboratories," Lefax, Phila, Pa, vols 1-4(1942-1946)
CLR	Chemical Laboratory Report (Picatinny Arsenal)
Cole, Underwater (1948)	R.H.Cole, "Underwater Explosions," Princeton Univ Press, Princeton, NJ (1948)
Collier's Encycl (Vol & year)	Coll, "Collier's Encyclopedia," P.F.Collier, NY, vols 1-20(1957)
Compt Rend Acad Sci (USSR)	See DoklAkadN
Cond Chem Dict (1942) (Editions 1950 & 1956 are listed on p Abbr 63)	F.M.Turner, Edit, "The Condensed Chemical Dictionary," Reinhold,NY (1942) (Contains table of expls, pp 287-92 not given in newer editions)
Cook(1958)	M.A.Cook, "The Science of High Explosives,"Reinhold, NY(1958)
DoklAkadN	See p Abbr 63 [listed sometimes in CA and in some papers as Compt Rend Acad Sci (USSR)]
EncyclBritannica (Vol & year)	Coll,"Encyclopaedia Britannica," London, vols 1-23(1952)
GLR	General Laboratory Report (Picatinny Arsenal)
Gorst(1957)	A.G.Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Propellants and Explosives), Gosizdatobororprom, Moscow (1957)
Inorg Synth (Vol & year)	Coll, "Inorganic Syntheses," McGraw-Hill, NY, vol 1 (1939); v 2 (1946); v 3 (1950); v 4 (1953) & v 5 (1957)
Mangini, Esplosivi (1947)	A.Mangini, "Quaderni di Chimica Industriale No 14, Explosivi," Pàtron, Bologna (1947)
Org Analysis (Vol & year)	Coll, "Organic Analysis," Interscience, NY, vol 1 (1953); v 2 (1954) & v 3 (1956)
Olsen & Greene (1943)	L.A.Olsen & W.G.Greene, "Laboratory Manual of Explosive Chemistry," Wiley, NY (1943)
Scott & Furman (1939)	W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis," VanNostrand, NY (1939)
Shidlovskii (1954)	A.A.Shidlovskii, "Osnovy Pirotekhniki" (Fundamentals of Pyrotechnics), Gosizdatoboronprom, Moscow (1954)
Shriner, Fuson & Curtin (1956)	R.L.Shriner, R.C.Fuson & D.Y.Curtin, "The Systematic Identification of Organic Compounds," Wiley, NY (1956)

Siggia (1949)	S.Siggia, "Quantitative Organic Analysis via Functional Groups," Wiley, NY (1949)
Snell & Biffen (1944)	F.D.Snell & F.M.Biffen, "Commercial Methods of Analysis," McGraw-Hill, NY (1944)
Stettbacher (1952)	A.Stettbacher, "Pólvoras y Explosivos," G.Gili, Buenos Aires (1952)
Sutton & Grant (1955)	F.Sutton & J.Grant, "A Systematic Handbook of Volumetric Analysis," Butterworth, London (1955)
TM 9-1910(1955)	Anon, "Military Explosives," Department of the Army Technical Manual TM 9-1910 and Department of the Air Force Technical Order TO 11A-I-34, Washington, DC(1955)
Tobolsky & Mesrobian (1954)	A.V.Tobolsky & R.B.Mesrobian, "Organic Peroxides," Interscience, NY (1954)
Treadwell & Hall 2 (1949)	F.P.Treadwell & W.T.Hall, "Analytical Chemistry," Wiley, NY, vol 2 (1949)
Webster's Unabridged Dictionary (1951)	See Merriam-Webster's (1951)
Yaremenko & Svetlov (1957)	N.E.Yaremenko & B.Ya.Svetlov, "Teoriya i Tekhnologuiya Promyshlennykh Vzryvchatykh Veshchestv" (Theory & Technology of Industrial Explosives), Promstroyizdat, Moscow (1957)
ZhEkspTeoretFiz	Zhurnal Ekperimental'noi i Teoreticheskoi Fiziki (Journal of Experimental & Theoretical Physics) (Russia)
ZPhysChem or ZPhysChem Leipzig) (See also p Abbr 74)	Zeitschrift für physikalische Chemie, published in Leipzig(must not be confused with the journal published in Frankfurt since about 1953)
ZPhysChem (Frankfurt)	Zeitschrift für physikalische Chemie (Frankfurt a/M)

ENCYCLOPEDIA OF EXPLOSIVES AND RELATED ITEMS

"106". Code designation for 1,9–Dinitroxy –2,4,6,8–tetranitro–2,4,6,8–tetrazanonane described under Dihydroxytetrazanonane

"121". See Firing or Igniter Composition in PATR 2510(1958), p Ger 49

A(explosif). A Fr expl prepd by mixing Amm perchlorate 94 & CC(collodion cotton), in the form of jelly 6%

Ref: Commission des Substances Explosives, MP **12**, 18(1903–4)

A(series). Ger rockets, **A**(Raketen) (see PATR 2510, p Ger 1)

A1(Monobel)Brit permitted expl: AN 60, NG 10, WM 9, K chloride 20 & moisture 1%; max charge 28 oz and Bal Pend Swing 2.78"
Ref: Barnett(1919), 134

A1(Roundkol). A current, granular Brit permitted coal mining expl: AN 53.6–56.5, Na nitrate 9–11, NG & NGc 9–11, vegetable fibers 11–13, Na chloride 11–13, Amm phosphate(dibasic) 0–0.5, resin 0–0.5 & moisture 2%. Power 61% of BG and d 0.70
Refs: 1) Thorpe 4(1940), 556 2)J.Taylor, Detonation in Condensed Explosives, Clarendon Press, Oxford(1952), 20

A2(Monobel). An earlier type of Brit mining expl: AN 59, NG 10, WM(wood meal) 9, K chloride 20, Mg carbonate 1 & moisture 1%; max charge 22 oz and Bal Pend swing 2.44" (See also Monobel No2)
Ref: Barnett(1919), 134

A–4(Rocket). Same as V–2 described in PATR 2510(1958), p Ger 213

A6(Fuseheads). See PATR 2510(1958), p Ger 1

A–9/A–10(Guided Missile). See PATR 2510 (1958), p Ger 1

Abbcites. Brit permitted expls: a)AN78–82, NG 9–11, WM(dried at 100°) 8–10 & moisture 0.5–2.5%(Ref 1) b)AN 58, NG 8, DNT 2, WM 9 & Na chloride 23%

Ref: Escales, "Ammonsprengstoffe"(1909), 187

Abbreviations of Ordnance and other terms are given at the beginning of the volume. Abbreviations of German Ordnance terms are given in PATR 2510(1958), pp Ger 309-45

Abel, Sir Frederick Augustus(1827–1902), was a leading Brit scientist in the fields of propellants and explosives. Introduced the practice of beating nitrocellulose to effect stabilization, devised a stability test for explosives which is named after him, and was the author of numerous patents and publications on explosives
Refs: 1)J.Spiller, JCS **87**, 565–70(1905) 2)T.Urbański, MAF **13**, 837–41(1934) 3)Pérez Ara(1945), 362 4)Giua, Dizionario, v1(1948), p 1

Abel & Dewar Smokeless Propellant, invented in 1889, consisted of high nitrogen NC gelatinized by acetone or ethyl acetate & NG
Ref: Cundill's Dictionary, MP **5**, 279(1892)

Abel Powder or Picric Powder. A mixt for priming PA(picric acid) invented in 1869 by Sir F.A.Abel: Amm picrate 40 & K nitrate 60% (Ref 1). French used a similar compn called Brugère(poudre). In Ref 2, the compn of **picric powder** is given as Amm picrate 43 & K nitrate 57%; yel solid,dec without melting; brisance – less than TNT; ballistic strength ca 75% TNT; deton rate ca 3500 m/s vs 6900 for TNT; sensitivity to impact, rifle bullet and initiation – comparable to tetryl; stability and compatibility with metals – comparable to Amm picrate. Was used during WWII by the British as a booster in AP projectiles filled with *Shellite*(qv)
Refs: 1)Thorpe 4(1940), 483 2)All&En Expls(1946), 104

Abel's Researches on Guncotton are described in JCS **20**, 310–357 & 505–576(1867)

Abel Smokeless Propellant, invented in 1886, consisted of a mixt of AN & NC coated with petroleum w or wo camphor

Ref: Cundill's Dictionary, MP **5**, 279(1892)

Abel's Test or KI–Starch Test was designed by Sir F.A.Abel to determine the stability of propellants and explosives. It involved heating a small sample of an expl in a test tube closed with a stopper provided with a hook on which is suspended a strip of KI–Starch paper, moistened at the upper half with 50% glycerin in water. The tube is heated in a constant temp bath and the time necessary to produce a slight brownish (or other) coloration at the border between the moistened and dry areas of the indicator paper is observed. The longer the time required the greater the stability of substance under test. The test is usually conducted either at 65.5° or 82.2°, but other temps may also be used. More detailed descriptions of the test are given under propellants and under some expls, such as TNT

Notes: a)Although this test is one of the oldest in existence, it is still used very extensively b)In this test, nitrogen dioxide, which starts to evolve at the moment of decompn of a propellant or an expl, forms, on contact with wetted portion of the test paper, a mixture of nitrous and nitric acid. The acids attack KI and the liberated iodine colors the starch paper c)Koehler & Marquerol(Ref 2) do not recommend the use of Abel's test for NC propellants contg Ca carbonate – Bergmann–Junk test(qv) gives more reliable results

Refs: 1)Marshall **2**(1917), 644 & 657 2)A. Koehler & M.Marqueyrol, MP **23**, 11–18(1928) 3)Marshall **3**(1932), 213 4)Reilly(1938), 71–7, 5)A.L.Olsen & J.W.Greene, Wiley, NY(1943), 28–30 6)Kast–Metz(1944), 227–32,307–9 & 458–60 7)PATR 1401, Revision 1(1950) 13 & 17–18

Abelite. A type of Brit dynamite patented by Sir F.A.Abel: NG 65.5, GC(gun cotton) (finely divided) 30.0, Na nitrate 3.5 & Na carbonate 1.0%

Refs: 1)Daniel, Dictionnaire (1902), p 1 2)Pérez Ara(1945), 330

Abelite No. 1. A type of Brit "permitted" expl: AN 68, TNT 6.7, DNB(dinitrobenzene) 7.0, Na chloride 17.5, moisture 0.5 & unac 0.3%

Ref: Barnett(1919), 132

Abelli, Modesto(1859-1911). Ital scientist who specialized in expls. Was director for a numberofyears of the Nobel Dynamite Plant at Avigliana

Ref: L.Cesaris, SS **6**, 381–2(1911)

Abelli Propellant: NC 30–45, NG 45–30 & NGu 20–25%

Ref: M.Abelli, USP 899,855(1908) & CA **3**, 377(1909)

Note: It seems that incorporation of NGu in propellants as a cooling agent was not originally a German idea but that of Abelli [See PATR 2510(1958), p Ger 81(Gudolpulver) and Ger 121(Nitroguanidin or Nigu)]

Aberdeen Chronograph. See under Chronographs

Aberdeen Proving Ground is the US Ordnance Proving Ground located in Maryland, near Baltimore. Its mission is outlined in Ordnance Corps Order 4-57, 11 Feb 1957 and in Change 1, 25 July 1958, Dept of the Army, Chief of Ordnance, Washington 25, D C

Abietates. See under Abietic Acid and Derivatives

ABIETIC ACID AND DERIVATIVES

Abietic Acid or Sylvic Acid (1,2,3,4,4a,4b,5,6,10,10a-Decahydro-7-isopropyl-1,4a-dimethyl-1-phenanthrene-carboxylic Acid), $C_{19}H_{29}COOH$,

MW 302.44. Leaflets, mp 174–5°, bp 200° at 1mm, d 1.132 at 25°, n_D^{25} 1.514, $[\alpha]_D^{22}$ 115.6°. Insol in w, very sol in alc & eth. May be obtained from the resin of pine species(colophony) or by other methods (Refs 1, 2 & 5). A lab method of prepn is described in Ref 6. Its toxicology, fire hazard, storage and handling are discussed in Ref 7. It was claimed (Ref 3) that the ignition sensitivity life of igniter compds contg cuprous acetylide(or other metallic acetylides) is improved by the addition of small amounts of abietic acid

Refs: 1)Beil **9**,[424] 2)L.F.Fieser & W.P. Campbell,JACS **60**,159 & 166(1938) 3)G.F. Rolland, USP 2,388,368(1945) & CA **40**,1036 (1946) 4)Kirk & Othmer **1**(1947),148 5)H.H. Zeiss, ChemRevs **42**,163–4(1948) 6)Org Synth **32**(1952),1–4 7)Sax(1957),227

Abietic, Azido Derivative, $N_3 \cdot C_{19}H_{28} \cdot COOH$ – not found in Beil or CA through 1956

Abietic Acid, Diazido Derivative, $(N_3)_2 C_{19}H_{27} \cdot COOH$ – not found in Beil or CA through 1956

Mononitroabietic Acid, $(O_2N)C_{19}H_{28} \cdot COOH$ – not found in Beil

Dinitroabietic Acids. The dinitro compd of the formula $(O_2N)_2 C_{19}H_{27} \cdot COOH$, mp 178–184°, reported to be obtained by Johansson on nitrating abietic acid (Refs 1 & 2),could not be identified as dinitroabietic acid by later investigators. Goldblatt et al(Refs 3 & 4) reported that by nitrating abietic acid with nitric acid(d 1.42) in AcOH or in boiling alc, they obtained white crysts, mp 171.2–171.4°, corresponding to the formula, $(O_2N)_2 C_{18}H_{28} \cdot COOH$. Fieser & Campbell (Refs 5 & 6) prepd the compd $(O_2N)_2 C_{19}H_{26} \cdot COOH$, ndls decomposng at 178–185°, by nitrating dehydroabietic acid, $C_{19}H_{27} \cdot COOH$, with fuming nitric or mixed nitric-sulfuric acid. This compd was identified as 6,8-*dinitrodehydroabietic or pyroabietic acid* and had the same props as comp reported by Jo-hansson as dinitroabietic acid

Refs: 1)Beil **9**,[428] 2)D.Johansson, ArkivKemi Min Geol(Stockholm) **6**, No 19, 20pp(1917) & CA **12**,583(1918) 3)Beil **9**, [430] 4)A.Goldblatt et al,JACS **52**,2133(1930) 5)Beil **9**,[448] 6.L.F.Fieser & W.P.Campbell, JACS **60**, 165(1938)

Trinitroabietic Acid, $(O_2N)_3C_{19}H_{26} \cdot COOH$. This compd, crysts, mp 177–8°, claimed to be prepd by Dubourg(Refs 1 & 2) proved to be identical with 6,8-dinitrodehydroabietic acid, $(O_2N)_2 C_{19}H_{28} \cdot COOH$ prepd by Fieser & Campbell(Ref 3)

Refs: 1)Beil **9**,[428] 2)J.Dubourg,BullInst-Pin(Fr) No **41**,241–6(Oct 1927) & CA **22**, 593(1928) 3)L.F.Fieser & W.P.Campbell, JACS **60**,165(1938)

Polynitro Derivative of Abietic Acid(no formula given), yel amorphous solid which exploded on heating and gave bright red Na, K & Amm salts, sol in w. It was prepd by dissolving abietic acid in fuming nitric acid (heated, if necessary) and pouring the soln into a large amt of ice cold water

Refs: 1)Beil **9**,[428] 2)J.Dubourg,BullInst-Pin(Fr) No **41**,241–6(Oct 1927) & CA **22**, 594(1928)

Abietic Acid, Organic Derivatives(Abietates): It has been claimed that the incorporation of 5–10% of an aliphatic or aromatic abietate (eg ethyl, methyl, phenyl or benzyl abietates) in single- or double-base propellants reduced the temp of burning and eliminated the muzzle flash

Ref: S.G.Norton,USP 1,788,438(1931) & CA **25**,1086(1931)

Following are some organic abietates:

Benzyl Abietate, $C_{19}H_{29}CO_2 \cdot CH_2C_6H_5$. Semi-liq, bp 294–297° at 4mm, d 1.036 at 15/4°and n_D 1.551. Used as a plasticizer and was recommended as a flash reducer in smokeless propellants

Ref: 1)Beil **9**,[431] 2)C.C.Kesler, JACS **49**, 2902–3(1927) 3)S.G.Norton, USP 1,788,438(1931)

Ethyl Abietate, $C_{19}H_{29} \cdot CO_2 \cdot C_2H_5$. Yel oil

freezing at –45°, bp 204–207°at 4mm, d 1.032 at 15/4°, n_D 1.5265. Used in lacquers and recommended as a flash reducer in smokeless propellants

Refs: 1)Beil **9**,[431] 2)C.C.Kesler et al, JACS **49**,2901(1927) 3)A.C.Johnston, IEC **21**, 688(1929)

Ethyl Abietate, Nitration with HNO_3 (d 1.42) in alcoholic soln produced a solid, mp 157.5–157.8°, corresponding to the formula $C_{21}H_{30}N_2O_6$ with N=6.90%. The same compd was obtained by refluxing an alcoholic suspension of the Na salt of $C_{19}H_{26}N_2O_6$ with diethyl sulfate

Refs: 1)Beil **9** not found 2)L.A.Goldblatt et al, JACS **52**,2135(1930)

Methyl Abietate, $C_{19}H_{29}\cdot CO_2\cdot CH_3$. Liq, bp 225–6° at 16mm, d 1.050 at 15/4° and n_D 1.3344. Recommended as a plasticizer for NC

Refs: 1)Beil **9**,[430] 2)C.C.Kesler et al, JACS **49**,2902(1927)

Phenyl Abietate, $C_{19}H_{29}\cdot CO_2\cdot C_6H_5$. Semisolid, distilled at 330–333° at 4mm giving a dark-colored gum which did not become lighter in color on redistilling: d 1.056 at 15/4° and n_D 1.5354. Recommended as a flash reducer in propellants

Refs: 1)Beil **9**[431] 2)C.C.Kesler et al, JACS **49**,2901(1927)

Ablation. According to Nicholls et al(Ref 1), there is considerable contemporary interest in the phenomenon of ablation, or mass loss from solids as a result of their immersion in an environment from which there is a large rate of energy transfer

The study of ablation in shock tubes conducted in Canada is briefly described in open literature (Ref 1), whereas the studies of ablation conducted in the USA are classified(Ref 2)

Refs: 1)R.W.Nicholls et al, JApplPhys **30**, 797–8(1959)(16 refs) 2)N.Beecher,National Research Corp, "Ablation Mechanism Study", Contract DA–19–020–ORD–4689, Progress Report No 7(1959) and previous repts(C)

Abnormal Temperature Testing of Propellants is discussed in the US Ordnance Proof Manual No 40–32(1949). The purpose of these tests is to determine the effect of extreme temps (as low as –70°F and as high as 160°F) on ballistic uniformity of a propellant and the adequacy of the ignition system. In these tests,the projectiles are fired at various temps to determine the relationship of velocity/temp and pressure/temp

Abonachit 2. A Ger expl used during WWII for filling grenades. See Filler No 57 in PATR 2510(1958),p Ger 47

Abrasive. Any grinding or polishing material, such as emery, ground glass, carborundum, infusorial earth, pumice etc. Some of these materials are used in priming compositions in order to increase the sensitivity of other components (such as MF, $KClO_3$ etc) to friction or impact. Crystalline Sb_2S_3 used in priming compns functions not only as a fuel but also as an abrasive(See under Primers)

Refs: 1)Kirk & Othmer **1**(1947),1–12(12 refs) 2)Riegel,Industrial Chemistry(1949),334–41

Absolute Method of Measurement of Power of Explosives. See under Power of Explosives, Measurements

Absolute Rate Theory(also known as Transition State or Activated Complex Theory). A theory of reaction rates based on the postulate that molecules form, before undergoing reaction, an activated complex which is in equilibrium with the reactants. The rate of reaction is controlled by the concn of the complex present at any instant. In general, the complex is unstable and has a very brief existance(See also Collision Theory of Reaction)

Refs: 1)H.Eyring, JChemPhys **3**,107–15(1935) (The activated complex in chemical reactions) 2)W.Wynne-Jones &H.Eyring, JChemPhys **3**,

492–502(1935)(The absolute rate of reactions in condensed phases) 3)C.F.Prutton & S.H.Maron, Fundamental Principals of Physical Chemistry, MacMillan,NY(1951), pp642–3 4)A.A.Frost & R.G.Pearson, Kinetics and Mechanism, Wiley,NY(1953), pp85–90 5)E.S.Freeman & S.Gordon,J Phys Chem **60**,867–71(1956)(The application of the absolute rate theory to the ignition of propagatively reacting systems)(10 refs)

Absorbent. Any body or substance which imbibes or takes up another either by penetration into the pores, crevices, or capillary spaces of the absorbent, or by dissolving it. Energy of various kinds may also be taken up by the absorbent. The absorbent may be a liquid or a solid and the absorption (qv) may occur with or without chemical action. Illustrative examples are: wood which absorbs water, water which absorbs gases such as ammonia, solid anhydrous Ca chloride which absorbs water, liquids which absorb light rays (see Absorption Spectroscopy) colored solutions which absorb lights of different wave lengths than the color of the absorbent
(See also **Absorption** and **Adsorption**)

Absorbent Materials to Control Exudation were discussed by R.W.Heinemann, FREL, EDS, PicArsn, Dover, NJ, May 1959(See also under Exudation)

Absorbent(Adsorbent) Materials in Dynamites. See Dopes

Absorbent(Adsorbent) Materials for Liquid Explosives. See under Liquid Explosives (Oxyliquits)

Absorbent Materials for Nitrogen Oxides. A satd soln of K bichromate or 0.02M Kpermanganate in concd sulfuric acid can be advantageously used to replace PbO_2 in analytical organic combustions for the determination of C, H and N

Ref: P.J.Elving & W.H.McElroy,IEC,Anal Ed **13**,660(1941) & CA**35**,6896(1941)

Absorbent Materials from Potatoes. A fibrous material, obtained by washing potatoes until the remaining fibers contain 15% or less starch and then drying and comminuting the fibers, was proposed as an absorbent for liquid explosives (such as NG),etc(Ref 1). In another patent by the same firm, the absorbent is prepd by evaporating the liquid used in washing potato starch, followed by drying and pulverizing the residual fibrous mass, which contains but little starch(Ref 2)

Refs: 1)N.V.M.A.Scholten's Aardappelmeelfabrieken, BritP 506,929(1939) & CA **34**,550 (1940) 2)Ibid, GerP 726,576(1942) & CA **37**, 6462(1943)

Absortiometer, an apparatus for analysis of metals, and probably suitable for analysis of metal-contg expl & pyro compns, is described in the book by F.W.Haywood& A.A.R.Wood, "Metallurgical Analysis by Means of the Spekker Photoelectric Absorptiometer," Hilger & Watts,London(1957)

Absorption(see also Adsorption) is an act or process of taking up (incorporating) gases, liquids or solids inside a liquid or solid substance which may be called the "absorbent" Absorptions may be classed principally as physical, chemical, thermal (radiation), electrical and physiological. Only the first two are treated here

In physical absorption no chemical reactions take place and the absorbed material (absorbate) is held by the absorbing material (absorbent) only by the forces of cohesion or capillary action in the pores of the solid. Physical absorption is a reversible process. As examples may be cited the absorption of gases such as nitrogen or oxygen in water, and absorption by soda lime or KOH of carbon dioxide

In chemical absorption definite chemical bonds are produced between the atoms and molecules inside the "absorbents" and the atoms and molecules of the "absorbates"

This is usually accompanied by considerable evolution of heat and the reaction is very difficult to reverse. For instance, when cold platinum sponge is held in the vapors of alcohol absorption proceeds with enormous evolution of heat – the sponge becomes red hot and ignites the alcohol. This property has been used in some "lighters"

From the point of view of industry, the book of Brown on Unit Operations(Ref 4,p32) defines **absorption** as "an operation in which significant or desired transfer of material is from the vapor phase to the liquid phase". Absorption usually, but not always, designates an operation in which the liquid is supplied as a separate stream independent of the vapor being treated

Refs: 1)H.C.Carlson et al, IEC **38**, Jan 1948 and following years under Unit Operations: "Absorption and Humidification" 2)Kirk & Othmer **1**(1947)14–32(25 references) 3)Perry (1950),667–711 4)G.G.Brown, Edit,"Unit Operations,"Wiley,NY(1950) 5)T.K.Sherwood & R.L.Pigford, "Absorption and Extraction," McGraw-Hill,NY(1952),pp 115–390

Additional References on Absorption: a)M. Voogd,CanP 362,060(1936) & CA **31**,1563 (1937)(Absorption of nitrogen oxides from gases leaving the acid absorption system of a nitric acid plant by contact with sufficient gaseous NH_3 to neutralize the N oxides in the gas stream and render it slightly alkaline) b)S.N.Ganz & L.I.Mamon, ZhurPriklKhim **26**, 1005–13(1953) & CA **48**,7982(1954)(Absorption of NO by $FeSO_4$) c)G.Cherubin, BullFr **1954**,192–5 & CA **48**,9684(1954)(The nitration of cellulose with mixed HNO_3–H_2SO_4 depends on the absorption of acids into the fibers)

Absorption Coefficient. See Coefficient of Absorption

Absorption, Electronic. See under Absorption Spectroscopy

Absorption Spectroscopy is the technique devoted to the study of radiations absorbed on passing through matter of various forms. Essentially, the method consists in placing a transparent solid or liq material or soln in quartz containers, called cells, between the source of light (visible, ultraviolet etc) and a spectrometer, and observing which lengths of radiation are absorbed. Absorption spectroscopy is used in anaylsis of expls and propellants

The technique which determines the relationship between the wave length(frequency) of radiation and its attentuation by absorption upon passage through a particular medium, is called **absorption spectrophotometry**

Following are selected refs on absorption spectroscopy, absorption spectrophotometry, electronic spectroscopy, etc: 1)F.Twyman & C.B.Allsop, "The Practice of Absorption Spectrophotometry, Adam Hilger,London(1934) 2)H.Mohler, "Absorptionsspektrum der chemischen Bindung,"reproduced by Edwards Bros, Ann Arbor, Mich(1943) 3)L.Pauling,OSRD Rept5953(1945) "Absorption Spectra of Explosives and Other Compounds of Interest in the Study of Smokeless Powder"(Conf)(not used for this dictionary) 4)G.R.Harrison, R.C.Lord & J.R.Loofbourow, "Practical Spectroscopy,"Prentice-Hall,NY(1949) 5)W. West in Weissberger's "Physical Methods of Organic Chemistry,"Interscience,NY,v1,part2 (1949,pp 1295–1312 6)G.F.Lothian, "Absorption Spectrophotometry," Adam Hilger, London(1949) 7)M.G.Mellon, et al "Analytical Absorption Spectroscopy", Wiley,NY(1950) 8)K.Dobriner, "Infrared Absorption Spectra", Interscience,NY(1953) 9)J.Deschamps, MSCE **38**,335(1953)(Ultraviolet absorption spectra of nitronium and nitrosonium ions) 10)N.Norrish et al, PrRS **A227**,423–33(1955) & CA **49**,7391(1955)(Explosive combustion of hydrocarbons – comparative investigation and study of continuous spectra) 11)H.M.Hershenson, "Ultraviolet and Visible Absorption Spectra", Index for 25 years – 1930 to 1954, Academic Press, NY(1956) 12)A.Gillam & E.S.Stern, "An Introduction to Electronic Spectroscopy in Organic Chemistry", St Martins,London(1958)(See also Infrared Spectroscopy, Ultraviolet Spectroscopy and Visible Spectroscopy)

Absorption Towers or Columns are tall cylindrical structures designed for absorption(qv) of gases by liquids. There are several types of towers, such as:

b)Plate tower. The simplest type consists of a closed vertical cylinder the inside of which is partitioned by a set of horizontal trays installed one above the other. Each tray has two openings, one in the center for a bubble cap,the other on the side for an overflow tube. The liquid moves from the top to the bottom of the tower while the gas passes counter current. The liquid flows across the first tray and then falls to the second tray. It flows in this tray in the opposite direction and falls to the third tray, thus following a twisting path down the column

b)Packed tower consists of a closed cylinder filled with different solids such as pieces of stone, brick, glass, coke, Raschig rings, Berl saddles, Lessing rings, Nielson propeller packing, Hechenbleikner blocks, Stedmann packing, Bregeat multiple spirals etc. Here, as in the case of plate towers, the liquid flows from the top of the tower and the gas enters at the bottom

c)Spray towers contain fine-spray nozzles through which the liquid is forced under pressure against the incoming gas to be absorbed. Another method of "atomizing" the liquid consists of impinging the liquid against a disk rotated at very high speed. In the so-called "cyclone-spray scrubber" the gas enters tangentially and is forced into a spiral path by a system of deflector plates, while the absorbing liquid (such as water) enters a perforated tube located in the center of the column and is sprayed against the particles of gas. This method is very suitable for removing dust, fumes, etc from the air. Plate and packed towers are also used in the fractional distillation of liquids

Refs: 1)Kirk & Othmer 1(1947),15–18 2)Perry (1950) 3)Riegel, ChemMach(1953),254–8 & 501 4)G.A.Morris & J.Jackson, Absorption Towers, Butterworth, London(1953)

Acacia. See Gum Arabic

AcAn. Code name for 1,9-Diacetoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane, described under Diacetoxytetrazanonane. It is also called 1,9-Diacetoxypentamethylene-2,4,6,8-tetranitramine and 2,4,6,8-Tetranitro-2,4,6,8-tetrazanonane-1,9-diol-diacetate

Acapnia. An Ital sporting propellant (polvere da caccia), similar to Schultze Propellants(qv) formerly manufd by the Societe Italienne pour la Fabrication de l' Acapnia

Refs: 1)Daniel, Dictionnaire(1902),2 2)Belgrano(1952),292

ACARDITE OR AKARDIT

Acardites are compds developed in Germany as stabilizers-gelatinizers for NC in smokeless propellants. There are three acardites of which Acardite I was developed first

Acardite I (asym-Diphenylurea or N,N-Diphenylurea)(Akardit I or Stabilit in Ger), $(C_6H_5)_2N{\cdot}CO{\cdot}NH_2$, mw 212.24, N 13.20%, OB to CO_2 – 233.7%, OB to CO – 135.7%. Col ndls, mp 189°, d 1.276.Can be prepd by one of the methods mentioned in Refs 1 or 2 by the method used in Germany during WWII and communicated to us by Dr Hans Walter (Ref 9). The manuf in Germany was conducted in two stages: a) Treatment in the cold of diphenylamine with phosgene in the presence of soda ash in an autoclave under atmospheric pressure: $(C_6H_5)_2NH + Cl{\cdot}CO{\cdot}Cl + 1/2\ Na_2CO_3 \rightarrow (C_6H_5)_2N{\cdot}CO{\cdot}Cl + NaCl + CO_2$ b)Treatment of the resulting diphenylcarbamylchloride with ammonia gas, in the presence of soda ash, conducted in the same autoclave but under pressure and at a temp of about 100°: $(C_6H_5)_2N{\cdot}CO{\cdot}Cl + NH_3 + 1/2\ Na_2CO_3 \rightarrow (C_6H_5)_2N{\cdot}CO{\cdot}NH_2 + NaCl + CO_2$

Following are some props of Acardite I: **solubilities**(appr) at RT, g/100 ml of solvent (Ref 6a & other sources): acet 1.50, benz 0.306, carbon disulfide 0.24, chlf 5.80, alc (95%) 0.94, ethylene chloride 2.31, eth 0.209, methanol 2.93 toluene 0.105, & petr eth 0.17;

nearly insol in w; **heats of combstn** 1605.4 kcal/mol at C_v, 18°, H_2O liq(Refs 7 & 8) or 1606.2 at C_p(Ref 7); **heat of formn** 28.3 kcal/mol at C_v or 32.6 at C_p(Ref 7)

Acardite I was used in Germany as a stabilizer -gelatinizer and as a muzzle flash reducer in NC smokeless propellants. When used in small quantities(say 0.8%), acardites served as stabilizers, while in larger quantities they acted as moderators of burning rate and as flash reducers. Acardite I was considered inferior in all respects to acardites II and III(Ref 6). According to Ref 5, Acardite I does not exercize any gelatinizing action on NC, especially if NC is of high nitrogen content

Reudler(Ref 3), studied the nitration of Acardite I and obtained asym-dinitrodiphenylureas and asym-tetranitrodiphenylurea

Analytical procedures for acardites are briefly described following Acardite III

Refs: 1)Beil **12**,429,(255)&[241] 2)H.Kast, Spreng- und Zündstoffe, Braunschweig(1921), 181 3)J.F.L.Reudler,Rec **33**,49–55(1914) 4)Stettbacher(1933),197 5)Anon PB Rept 11544(1944) 6)O.W.Stickland et al, PB Rept 925(1945) 6a)R.Dalbert & J.Tranchant, MP **30**,338(1948) 7)L.Médard & M.Thomas, MP **34**,422,430 & 439–40(1952) 8)P.Tavernier, MP **38**,307–8 & 329(1956) 9)Dr Hans Walter, PicArsn,Dover,NJ; privage communication(1958)

Acardite II or Methylacardite(N'-Methyl-N,N-diphenylurea)(Akardit II in Ger), $CH_3{\cdot}NH{\cdot}CO{\cdot}N(C_6H_5)_2$, mw 226.27, N 12.38%, OB to CO_2 -240.4%, OB to CO -141.4%. White crysts, mp 170.5° for a tech sample and 171.2° for samples recrystallized from chloroform or ethanol. Soly at RT in chlf 15.2g per 100g of solvent and in methylene chloride 14.9g(Ref 3a). It was prepd in Germany by treating equimolecular quantities of DPhA and methylamine, dissolved in CCl_4, with phosgene in the presence of limestone. This was followed by fractional distillation. The reaction proceeded as follows:

$$CH_3NH_2 + COCl_2 + NH(C_6H_5)_2 + CaCO_3 \longrightarrow CH_3{\cdot}NH{\cdot}CO{\cdot}N(C_6H_5)_2 + CaCl_2 + CO_2 + H_2O$$ (Ref 2)

Heat of combustn 1771.5 kcal/mol at C_v, 18° & H_2O liq or 1772.6 at C_p(Refs 4 & 5); heat of formn 24.1 kcal/mol at C_v or 29.1 at C_p(Refs 4 & 5)

Acardite II was proposed in Germany as a stabilizer-gelatinizer in NC smokeless propellants. As a stabilizer, it was considered superior to Acardites III & I and as a gelatinizer inferior to Acardite III but superior to Acardite I(Ref 3)

Refs: 1)Beil – not found 2)Dr Hans Walter, PicArsn; private communication 3)O.W. Stickland et al, PB Rept 925(1945),18 3a) R.Dalbert & J.Tranchant, MP **30**,340(1948) 4)L.Médard & M.Thomas, MP **34**,423 & 430 (1952) 5)P.Tavernier, MP **38**, 307 & 329(1956)

Acardite III or Ethylacardite (N'-Ethyl-N,N-diphenylurea)(Akardit III, in Ger), $C_2H_5{\cdot}NH{\cdot}CO{\cdot}N(C_6H_5)_2$, mw 240.29, N 11.66%, OB to CO_2 -246.4%, OB to CO -146.5%. White cryst, mp 72.3° for a tech sample and 73.1° for samples recrytd from ethanol or chlf(Ref 4). It was prepd in Germany by treating with phosgene a soln in carbon tetrachloride of equimolar quantities of ethylamine and diphenylamine in the presence of limestone. This was followed by fractional distillation. The following reaction took place:

$$C_2H_5{\cdot}NH_2 + COCl_2 + NH(C_6H_5)_2 + CaCO_3 \longrightarrow C_2H_5{\cdot}NH{\cdot}CO{\cdot}N(C_6H_5)_2 + CaCl_2 + CO_2 + H_2O$$ (Ref 2)

Heat of combustn 1922.7 kcal/mol at C_v, 18° & H_2O liq(Refs 4 & 5) or 1924.1 at C_p (Ref 4); heat of formn 35.0 kcal/mol at C_v or 40.5 at C_p(Ref 4)

Acardite III was proposed in Germany as a stabilizer-galatinizer in NC smokeless propellants. As a stabilizer it was considered superior to Acardite I and inferior to Acardite II and as a galatinizer superior to Acardite II

Refs: 1)Beil – not found 2)Dr Hans Walter, PicArsn; private communication 3)O.W. Stickland et al, PB Rept 925(1945),18 4)L. Médard & M. Thomas, MP **34**,423 & 431(1952) 5)P.Tavernier, MP **38**,307–8 & 329(1956)

Acardites, Analytical Procedures. Following methods are based on Refs 1,2,3 & 5:

Method 1 (when only acardite I is present and no urethanes, DPhA or centralites): a)Extract with chloroform or methylene chloride(5–10g) a finely divided propellant using a Soxhlet or other extractor(ca 15 hrs for complete extraction). Evaporate chlf under reduced pressure and weigh the dry residue (wt 1) b)Check the mp and if it is close to 189°, no further analysis is necessary c)If mp is not 189°, shake the residue with 50cc CCl_4(in which acardite I is only sl sol), add 50cc of N/5 K bromide-bromate soln, and 10cc concd HCl: $H_2N{\cdot}CO{\cdot}N(C_6H_5)_2 + 2Br_2 \longrightarrow H_2N{\cdot}CO{\cdot}N(C_6H_4Br)_2 + 2HBr$. This bromination is complete if conducted in the dark for 6 hrs. d) Determine the amt of unreacted bromine by adding K iodide soln: $Br_2 + 2KI \longrightarrow 2KBr + I_2$, and titrating the liberated iodine with N/5 Na thiosulfate soln in presence of starch. Calculate the amt of acardite I, knowing that 1cc of N/5 thiosulfate = 0.0106 g of akardite I(wt 2). If the wt 2 is smaller than wt 1, some impurity is present(Refs 2 & 5)

Note: According to Dalbert & Tranchant(Ref 3), the above direct bromination of acardite I is not as convenient as their method, which consists essentially of: a)saponification of acardite I(extracted from propellant) by boiling with 3N NaOH soln for 2 hrs: $H_2N{\cdot}CO{\cdot}N(C_6H_5)_2 + H_2O \longrightarrow C_6H_5{\cdot}NH{\cdot}C_6H_5 + CO_2 + NH_3$ b)Bromination of the resulting diphenylamine with bromide-bromate(½ hr): $C_6H_5{\cdot}NH{\cdot}C_6H_5 + 2Br_2 \longrightarrow BrC_6H_4{\cdot}NH{\cdot}C_6H_4Br + 2HBr$ c)Determination of unreacted bromine by adding to the soln KI and titrating the liberated iodine with N/10 thiosulfate in presence of starch. Calc the amt of akardite I knowing that 1cc of N/10 thiosulfate corresponds to 0.0021g DPhA or 0.0053g acardite I

Method 2(when acardite I and a centralite are present): a)Extract the finely ground sample of propellant, as in Method 1, evaporate the solvent and weigh the dry residue (wt 1), which is equal to centralite & acardite b) Treat the residue with aq AcOH at pH ca 4. This will hydrolize the centralite, leaving acardite I intact c)Wash the residue with w, dry, and weigh(wt 2). Wt 2 is equal to acardite I and wt 1 – wt 2 is equal to centralite d) Det mp of acardite I and if ca 189°, no further analysis is necessary e)If mp is not 189°, detn the amt of acardite either by direct bromination, proc (d) of Method 1 or by the method of Dalbert & Tranchant(Ref 3)

Method 3(when acardite I and substituted urethanes are present in propellants not contg NG or DEGDN) : a)Extract with chlf or methylene chloride) a large sample(ca 10g) of finely ground propellant, evaporate the solvent and weigh it(wt 1). This is equal to acardite I & urethanes b)Stir the residue with 50cc of toluene, previously saturated with acardite I, and filter through sintered glass crucible, under vacuo. Rinse the residue with few cc of w, dry, and weigh (wt 2). Wt 2 is equal to acardite I and wt 1 – wt 2 is equal to urethanes such as diphenylurethane, ethylphenylurethane, etc (Ref 3, pp 338–9)

Method 4(when acardite II alone is present as stabilizer and gelatinizer): a)Extract a finely divided sample of propellant(ca 10g) with methylene chloride or chlf and evap the solvent b) Dry at 110° and weigh(wt 1) (acardite II is not volatile at 110°) c)Det the mp and if it is ca 170.5°, the identity of acardite II is established. If mp differs from 170.5°, boil the residue with ethanol and aq sulfuric acid to split the acardite II:

$CH_3{\cdot}HN{\cdot}CO{\cdot}N(C_6H_5)_2 \xrightarrow{HOH} CO_2 + C_6H_5{\cdot}NH{\cdot}C_6H_5 + CH_3{\cdot}NH_2$ d)Distil off quantitatively methylamine into a flask contg AcOH and save the residue contg DPhA e)Treat

the soln of methylamine with aq Na nitrite : $CH_3 \cdot NH_2 + NaNO_2 + CH_3COOH \longrightarrow N_2 + CH_3COONa + CH_3OH + H_2O$, and collect the liberated nitrogen into a gas burette. Calc from the amt of N, the amt of acardite II (wt 2). If wt 2 is smaller than wt 1, then something else than acardite II is present f)In order to establish that the sample contains acardite II and not acardite III, treat the soln(after removal of N gas) with chromic acid mixt($K_2Cr_2O_7 + H_2SO_4$) to oxidize the methanol to formaldehyde. The pungent odor of formaldehyde indicates the presence of acardite II in propellants. More definite results are obtained by treating the oxidized soln with fuchsin, previously discolored by treatment with SO_2 gas. Bluish-red coloration indicates the presence of formaldehyde(and of acardite II) and the intensity of coloration can be detnd in a colorimeter g)Another way to check the results of analysis is to det the amt of DPhA in residue of proc (d). For this wash the residue and dry & weigh it(wt 2 = DPhA) h)Verify the identity of DPhA by dissolving the residue in concd sulfuric acid and det the amt of DPhA colorimetrically without delay using as reagent aq KNO_3 soln(Refs 3 & 5)

Method 5(when acardite II & centralites are present): a)Extract with chlf or methylene chloride ca 10g of finely divided propellant, evap the solvent and weigh the dried extract (wt 1 = centralite + acardite II) b)Treat the extracted material with aq AcOH at pH ca 4 and filter the mixt through tared sintered glass crucible. Rinse the residue, dry it to const wt and weigh(wt 2 = acardite II and wt 1 – wt 2 = centralites) c)Continue the analysis as described in procedures (c) to (g) of Method 4

Method 6(when acardite III alone is present as stabilizer and gelatinizer): Procs a) to e)–same as in Method 4 f)In order to establish that the sample contains acardite III and not acardite II, treat the soln(after removal of N gas) with chromic mixt($K_2Cr_2O_7 + H_2SO_4$) in order to oxidize ethanol to acetaldehyde. Treat the oxidized soln with fuchsin, previously discolored by treatment with SO_2 gas. No change in coloration indicates the absence of formaldehyde, which would form in the presence of acardite II. Procs g) & h)–same as in Method 4

Method 7(when acardite III and centralites are present): Procs a) & b)–same as in Method 5; c), d) & e) – same as in Method 4; f), g) & h) – same as Method 6

Method 8(when acardite II and diphenylamine are present): a)Extract ca 10g of finely divided propellant with methylene chloride or with chlf, evap the solvent and dry and weigh the extracted residue (P = x + y, where x is the wt of acardite II and y is the wt of DPhA in propellant) b)Dissolve the extracted material in 50cc chlf, add 300cc water and an excess of K bromide-bromate soln of known concn c)After brominating for 4 hrs at RT, add aq soln of KI and titrate the liberated iodine with N/10 Na thiosulfate (1cc of thiosulfate is required for 0.0021 g of DPhA) d)If the calcd wt of DPhA is equal to P', mol wt of DPhA = 169 and wt of 2 mols of acardite II is equal to 452, the P' = 169/452 x + y = 0.374 x + y

Eg: If P = 2.5000 g and P' = 1.8000 g, then x + y = 2,500 and 0.374 x + y = 1.800, then x = 1.119 g and y = 1.381 g (Ref 3, p 342)

Method 9(when acardite III and DPhA are present)–same as in Method 8 except the formula in proced(d) shall be $P_1 = 169x/480 + y = 0.352x + y$

Note: In Ref 4 are given colorimetric reactions with aq K nitrate–sulfuric acid for *acardite I*(color of ring brn-red & violet and color after mixing grn-brn turning into yel-brn) and for *acardite III*(color of ring blue-grn, and color after mixing blue-grn turning into violet)

Refs: 1)F.Becker & G.A.Hunold, SS **28**,285(1933) 2)Kast-Metz(1944),166 3)R.Dalbert & J.Tranchant, MP **30**,335-42(1942) 3a)T.C.J.Ovenston, Analyst **74**,344-51(1949)(Chromatographic detn of acardites in propellants) 4)F.vonGizyki & L.Reppel,ZAnalChem **144**,110-11(1955)(Color reactions of acardites I & III) 5)Dr Hans Walter,

PicArsn; private communication(1959)

Accelerographs, Accelerometers and other devices for experimental study of movement of projectiles in guns are described by P. Libessart, MAF **11**,1077–1117(1932)

Accenzione(Ital). Ignition

Acceptable Explosives belong to the group of "Dangerous Chemicals"(qv), as defined by the ICC (Interstate Commerce Commission), which may be safely transported by railroads, motor vehicles and steamships subject to certain regulations

Acceptable explosives may be divided into three classes:

Class A: dangerous explosives(detonating or otherwise) of maximum hazard. Their distinguishing characteristic is the susceptibility to deton by a blasting cap. Typical examples are: dynamite, PA, TNT, NC, NG and AN and chlorate expls. Black powder is also included in this group although it cannot be detonated by a commercial blasting cap(ICC Sec 73.53)

Class B: less dangerous explosives than A. In general they function by rapid combustion rather than by deton. Typical examples: some smokeless propellants, some pyrotechnic powders(flash powders) and signal devices (ICC Sec 73.88)

Class C: relatively safe expls(minimum hazard). They are defined as certain types of manufd articles which contain class A or class B expls, or both, as components but in restricted quantities. Eg: small arms ammunition and certain types of fireworks (ICC Sec 53.100)(See also Forbidden Explosives)

Refs: 1)US War Dept Tech Manual TM 3–250 (1940),pp 4–5 2)Agent H.A.Campbell's Tariff No 10, publishing the "Interstate Commerce Commission Regulations for Transportation of Explosives and other Dangerous Articles by Land or Water", 30 Vesey St, New York 7, NY(1957)

Accessibility is the ratio between the portion of a cellulose sample which is accessible to a given reagent (such as Ac_2O, $Ac_2O + HNO_3$, $AcONO_2$ etc) and the portion which is not accessible

It has been claimed that the cryst(or ordered) regions of cellulose resist the penetration of reagents while the amorphous regions are more reactive. This definition is only approximate

Refs: 1)J.Chédin & A.Tribot, MSCE **36**,42 (1951)(7 refs) 2)Ott, **5**, part 1(1954),pp 7 & 266 3)E.Dyer & H.Williams, TAPPI **40**, No 1,14–20(1957)(16 refs)

Acciaio(Ital). Steel

Acciaio fuso(Ital). Cast steel

Accidental Explosions in Process Industry Plants. Causes of such explosions may sometimes be determined by a study of resulting missiles as well as of any corpses

Ref: C.Field, ChEng **54**,102–4(Jan 1947); 126–8(Feb 1947); 118–20(March 1947)

Accidental Scientific Discoveries. Title of a booklet by B.E.Schaar, published in 1955 by Schaar & Co, 754 W.Lexington St, Chicago 7,Ill. Among many interesting items in the booklet, mention is made of the accidental discoveries of dynamite, acetylene, the benzene ring, iodine, oxygen, petroleum jelly, plastics, radioactivity, and X-rays. All these substances and phenomena are of importance in the expls industry

Accidents in Industry(Laws, Prevention, Statistics, etc). See the following publications:

1)G.C.Whalen, ChemInds **54**,852–3(1944) "Accident Analysis in Wartime Chemical Plants" 2)H.H.Judson & J.M.Brown, "Occupational Accident Prevention",Wiley,NY(1944) 3)C.G.Daubney,Metallurgia **33**,41–4(1945) "Accident Investigations" 4)US Army, Corps of Engineers Safety and Accident Prevention Div, Safety Requirements,Pamphlet,US Govt

Printing Office(1946) 5)H.W.Heinrich,"Industrial Accident Prevention; A Scientific Approach," 3rd ed, McGraw-Hill,NY(1950) 6)National Safety Council,"Accident Prevention Manual for Industrial Operations,"2nd ed, Chicago,Ill(1951) 7)Underwriters Laboratories,Inc,"Lists Relating to Accident Equipment",NY(1951) 8)National Fire Protection Association,"National Fire Codes for the Prevention of Dust Explosions," Boston(1952) 9)W.M.Kunstler,"The Law of Accidents," Oceana Publications,NY(1954) 10)US Bureau of Mines,"Accidents from Explosives at Metal and Non-metallic Mines," July(1956)(See also Safety Measures in Industry)

Accumulators(Storage Batteries) are frequently used in Ordnance plants and laboratories. Since they represent certain explosive hazards some knowledge of their handling is desirable. The following references contain such information:

Refs: 1)G.W.Vinal,"Storage Batteries," Wiley, NY(1940) 2)G.W.Jones et al, USBurMines Tech Paper **612**,pp 1–10(1940) & CA **34**, 8284(1940)(Danger of explosion in storage battery rooms due to evolution of hydrogen and formation of explosive mixtures with air) 3)J.Reilly & W.N.Rae,"Physico-Chemical Methods,"Van Nostrand,NY(1943),pp 228–36 4)Kirk & Othmer **2**(1948),pp 340–60 under Batteries (17 refs) 5)Perry(1950),pp 1792–3

Accuracy Life. The number of rounds a particular weapon can be fired before wearing of the barrel (tube) would cause inaccuracy of firing to exceed the permitted tolerance

Accuracy Tests of Small Arms Ammunition are described in Ordnance Proof Manual 7–14(1945)(13 pages)

ACENAPHTHENE AND DERIVATIVES

Acenaphthene(Ethylenenaphthalene or 7,8-Dihydroacenaphthylene), $C_{10}H_6{<}^{CH_2}_{CH_2}$, mw 154.1, col ndls, mp 95°, bp 227°, d 1.0678. It is one of the products of coal tar distillation; insol in w and sol in hot alc; used in org synth in the manuf of dyes(Ref 3). Its prepn & props are discussed in Beil(Ref 1). A qualitative test for acenaphthene(by nitrating it to 5-nitroacenaphthene) is given in Ref 3

Refs: 1)Beil **5**,586(274) & [494] 2)G.T.Morgan & H.A.Harrison, JSCI **49**,413T to 421T (1930) 3)Hackh(1944),5 4)C.Y.Vanag & E.A.Zalukaeva, ZhurAnalKhim **5**,315–18 (1950) & CA **44**,10605(1950) 5)E.D.Bergmann & J.Czmuszkovicz, JACS **75**,2760(1953) & CA **49**,5408(1955)(A new synthesis of acenaphthene)

Note: M.Berthelot, CR **65**,508(1867), tried to obtain an explosive by nitration of acenaphthene but the highest product of nitration was a non-explosive dinitro compd which melted with decompn at 206°. The same product was later prepd by F.Sachs and G.Mosenbach, Ber **44**,2860(1911) and identified as 5,6-dinitronaphthene(see below)

Acenaphthene, Azido Derivative, $N_3 \cdot C_{12}H_9$, mw 195.22, N 21.53% – not found in Beil, but one isomer, **4-Triazoacenaphthene**, col crysts, mp 66–8°, is described by G.T.Morgan & H.A. Harrison, JSCI **49**,415T(1930). Its expl props were not examined

Acenaphthene, Diazido Derivative, $(N_3)_2C_{12}H_8$ not found in Beil or CA through 1956

Acenaphthene-4-diazonium Chloroaurate, $C_{12}H_9N_2Cl_4Au$, pale yel ppt decomp violently on heating

Refs: 1)Beil – not found 2)G.T.Morgan & H.A.Harrison JSCI **49**,415T & 419T(1930)

Mononitroacenaphthene, $O_2N \cdot C_{12}H_9$ – not found in Beil, but two isomers **2- and 4-nitroacenaphthenes** are described by G.T. Morgan & H.A.Harrison, JSCI **49**,415T & 419T(1930). Another isomer **5-nitroacenaphthene** is described by T.Ishii & Y.Yamazaki, Memor Faculty Technol, Tokyo MetropolUniv, No 1,21–9(1951) & CA **47**,2159(1953)

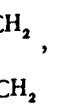
Dinitroacenaphthene, $(O_2N)_2C_{12}H_8$, mw 244.20, N 11.47%. Following isomers are described in the literature:

2,5-Dinitroacenaphthene, yel ndls(from AcOH), mp dec 205–6° (Ref 2,p 419T)

2,7-Dinitrocenaphthene, brn-yel ndls (from AcOH), mp 155–6° (Ref 2,p 419T)

5,6-Dinitroacenaphthene, crysts, sinter ca 210° and melt at 220–4°(Refs 1 & 3)

Refs: 1)Beil **5**,588,(277) &[498] 2)G.T.Morgan & H.A.Harrison, JSCI **49**,419T(1930) 3)I.Honda & M.Okazaki, JSocOrgSynthChem (Japan) **7**,25–9(1950) & CA **47**,6922(1953)

Trinitroacenaphthene, $(O_2N)_3C_{10}H_3$  ,

mw 289.20, N 14.53% – not found in Beil or CA through 1956

Acenaphthene Picrate, $C_{12}H_{10} + C_6H_3N_3O_7$, mw 383.31, N 10.96%. Orange-red prisms, mp 161–162.5°(Refs 1,2,3 & 5), expl at 418° (Ref 4). Can be prepd by mixing equimolecular quantities of acenaphthene and picric acid in hot alc, followed by cooling

Refs: 1)Beil **5**,(276) 2)A.Behr & W.A.van Dorp,Ann **172**,265(1874) 3)R.Meyer & A. Tanzen,Ber **46**,3193(1913) 4)R.L.Datta & N.R.Chatterjee, JCS **115**,1008(1919) 5)E.D. Bergmann & J.Szmuszkovicz, JACS **75**,2760 (1953) & CA **49**,5408(1955)

Acerit. Same as Polygalit(1,5-Anhydro-d-sorbitol)

ACETAL AND DERIVATIVES

Acetal or Acetaldehyde-diethylacetal(Acetol, Ethylidene Diethyl Ether or 1,1-Diethoxyethane), $CH_3CH(OC_2H_5)_2$, mw 118.17, OB to CO_2 -230.2%, OB to CO -148.9%. Col liq n_D^{20} 1.3819, d 0.825 at 20°/4; sl sol in w, sol in eth, miscible with alc(See also Ref 8). Heat of combstn at C_v ca 929 kcal/mol or ca 930.5 at C_p; spec heat 0.51 cal/g/°C . Can be prepd by treating acetaldehyde with ethanol in the presence of anhyd Ca chloride (Ref 2) or by other methods(Ref 1)

Acetal is stable under neutral or sl alkaline conditions, but hydrolyzes in the presence of acids to form acetaldehyde

It has been used as a solvent and as an intermediate in the manuf of chemicals used in the expl industry and of synthetic rubber (Ref 4). During WWII, acetal(as well as acetaldehyde) was used in Germany as hypergollic fuel in liquid rocket propellants in conjunction with red or white fuming nitric acid which served as an oxidizer. Acetal was later replaced by catechol(Brenzcatechin or Brenzol in Ger)(Ref 10)

Acetal is dangerous when exposed to heat or flame and it can react vigorously with oxidizing materials. Its toxicity, toxicology and fire hazards are discussed in Ref 7

Acetal is a poor solvent for NC but its admixture with anhydrous alcohol(see Acetal Solvent) greatly increases its solvent power (Ref 8)

Refs: 1)Beil **1**,603,(326) & [671–2] 2)Org-Synth,CollVol **1**(1941),1–2 3)W.J.Huff, US BurMinesReptInvest **3669**(1942) & CA **37**, 1869–70(1943)(The lower limit of inflammability of acetal at atm pressure and at 25° is 1.65% by vol and the ignition temp at 0° is 230° in air and 174° in oxygen) 4)Ullmann **3**(1953),13–17 5)P.Dugleux & P.Laffitte, CR **221**,661–3(1945) & CA **40**,3951(1946) (Studies of spontaneous inflammation of mixtures of acetal with air) 6)Kirk & Othmer **1** (1947),40–5 7)Sax(1957),228 8)Durrans(1957), 116 9)Carbide and Carbon Corp, Bulletin "The Physical Properties of Synthetic Organic Chemicals" 10)Dr Hans Walter,PicArsn, Dover,NJ; private communication

Acetal, Analytical Procedures are discussed in: 1)Kirk & Othmer **9**(1952),607 2)Ullmann **3** (1953),17 3)Organic Analysis, Interscience, NY, **1**(1953),309–328

Acetal Solvent consists of a mixt of acetal and abs alc. The 90% mixt has d 0.82, boiling range 75–85° and fl p 38°. Acetal solvent gelatinizes NC much better than straight acetal and also dissolves many resins

Ref: Durrans(1957),116

Acetal Compounds of Pentaerythritol are described by A.Scrabal & S.Kalpasanow,Ber **61B**,55–78(1928) & CA **22**,1328(1928)(See also under Pentaerythritol)

ACETALDEHYDE AND DERIVATIVES

Acetaldehyde or Aldehyde(Ethylaldehyde, Ethylidene Oxide or Ethanal), CH_3CHO, mw 44.05, OB to CO_2 – 181.6%. Col liq, freezing ca –123.5°, bp 20.8°, d 0.7833 at 18/4°, $n_D^{20°}$ 1.3316, Q_c^P 281.9 and Q_f^P 47.9 kcal/mol (Ref 1a). It is miscible with w, alc and eth (See also Ref 7). Various methods of prepn are given in Refs 1,3 & 9. In Ref 2 is described the catalytic production of acetaldehyde from acetylene and steam over activated carbon and promoted by phosphoric acid. Yields of 85–90% of theor were reported. The reaction of acetaldehyde with sulfuric acid is exothermic and when uncontrolled proceeds with almost expl violence. Mixts of acetaldehyde vapor with air(4 to 57% acetaldehyde by vol) are highly inflammable and expl. Uses and applications of acetaldehyde are listed in Ref 3,pp 32 & 39. It was used extensively during WWI as an intermediate for making acetic acid, which was transformed to acetone

Acetaldehyde(as well as acetal) was used during WWII in Germany as a hypergolic(qv) fuel in liquid rocket propellants in conjunction with oxidizers, red or white concd nitric acids. These fuels were later replaced by catechol(Brenzcatechol or Brenzöl, in Ger) (Ref 5). Acetaldehyde,(together with formaldehyde and hydrated lime) has been used for the prepn of pentaerythritol(Ref 4)

Toxicity, toxicology and fire and expl hazards are discussed in Refs 7 & 8. Its expl hazard is severe when exposed to flame. Expl range in air is 4.0 to 57% of acetaldehyde(Ref 8)

Typical US specifications for a technical grade acetaldehyde are: color–water white; acetaldehyde(minim) 99%; acidity as AcOH 0.5%(max), sp gr 0.770 to 0.790 at 20°

Acetaldehyde shows a great tendency to polymerize. A few drops of concd H_2SO_4 added to anhydrous acetaldehyde causes it to polymerize to:

Paraldehyde or 2,4,6-Trimethyl-1,3,5-trioxane $(CH_3CHO)_3$, mw 132.16, OB to CO_2 -181.6%. Col liq with pleasant odor, fr p ca.12.5°, bp 124°, d 0.9943 at 20°, $n_D^{20°}$ 1.4049, fl p 42°, abs visc at 15° 0.1359, sp heat 0.459 cal/g/°C and heat of fusion 25.2 gcal/g. Sol in w (13.3% at 8.5° and 5.8% at 75°); sol in eth, alc & chlf(Ref 3,p 42). It is used as a solvent for cellulose derivatives, fats, oils, waxes, gums, etc,as well as for many other purposes (Refs 6 & 7)

At a lower temp and with a smaller quant of sulfuric acid a solid polymer *Metaldehyde* is formed. It is used as the solid fuel "*Meta*" (See also Ref 4a)

Refs: 1)Beil **1**,594,(321) & [654] 1a)J.Thomas, ZPhCh **52**,347(1905) 2)A.Yakubovich et al, ZhurPriklKhim(Russia) **19**,973–88(1946)(A complete English translation,No RJ–64, is available from Associated Technical Services, PO Box 271, East Orange,NJ 3)Kirk & Othmer **1**(1947),32–39 & 42 4)Walker(1953),222 4a) Ullmann **3**(1953),12–13 5)Dr Hans Walter, PicArsn,Dover,NJ; private communication 6)CondChemDict(1956),822–3 7)Durrans(1957), 116–17 8)Sax(1957),228 9)Faith,Keyes & Clark(1957),1–7

Additional References on Acetaldehyde: a)W.J. Huff, USBurMinesReptInvest **3669**(1942) & CA **37**,1871(1943)(Ignition temp of acetaldehyde at 0° in air is 165° and in oxygen 159°) b)Saburo Yagi, RevPhysChemJapan **19**,106–30 (1945) & CA **44**,2346–7(1950)(Oxidation reactions of acetaldehyde and explosion of AcH mixtures with oxygen c)P.Gray & A.D.Yoffe,

JCS **1950**,3184(Inflammation of acetaldehyde-nitrogen dioxide mixtures) d)A.G.White & E.Jones, JSCI **69**,206–12(1950)(Limits for the propagation of flame in acetaldehyde-oxygen-nitrogen mixtures) e)J.d'Ans et al, AngChem **66**,633–5(1954) & CA **49**,10832 (1955)(Peroxide derivatives of acetaldehyde, prepd by treating AcH with peroxy acids. All the compds are highly explosive)(16 references)

Acetaldehyde, Analytical Procedures are discussed in the following references: 1) Beil **1**,601,(326) & [668–70] 2)H.A.Iddles & C.E.Jackson, AnalChem **6**,454–6(1936) (Precipitation of acetaldehyde as hydrazone using 2,4-dinitrophenylhydrazine as a reagent) 3)Kirk & Othmer **1**(1947),38(Qualitative and quantitative methods of determining acetaldehyde) 4)Jacobs(1949),476–8(Qualitative and quantitative methods of detn) 5)Ullmann **3** (1953),11

Acetaldehyde-Nitrogen Dioxide Mixtures, with a large propn of dioxide, expl vigorously when heated to 350–400°. The expln may be considered to be the result of a chain-thermal process

Ref: P.Gray & A.Yoffe, JCS **1950**,3184

Acetaldehyde, Azido Derivative(Azidoacetaldehyde or Triazoacetaldehyde), $N_3 \cdot CH_2 \cdot CHO$, very unstable oil, expl mildly on heating. Was prepd in impure state from chloroacetaldehyde hydrate and Na azide. Dec by KOH with violent evoln of ammonia & nitrogen

Refs: 1)Beil **1**,627 2)M.O.Forster & H.E. Fierz, JCS **93**,1870–1(1908)

Mononitroacetaldehyde, $(O_2N)\,CH_2 \cdot CHO$, was prepd according to Beil **1**, [684], in aq soln but not isolated

Dinitroacetaldehyde, $(O_2N)_2\,CH \cdot CHO$ – not found in Beil or CA through 1956

Acetaldehydepicrylhydrazone or Ethylidene-[2,4,6-trinitrophenylhydrazine], $(O_2N)_3C_6H_2 \cdot NH \cdot N{:}\,CH \cdot CH_3$, mw 269.18, N 26.02%. Brn lflts, mp 119–20°; v sl sol in w, sl sol in eth, fairly sol in alc & AcOH. Was prepd by heating 2,4,6-trinitrophenylhydrazine with acetaldehyde

Refs: 1)Beil **15**,495 2)A.Purgotti, Gazz **24** I, 575(1894) & JCS **68** I,28(1895)

Acetaldehyde Superoxide. See Diethylidene Diperoxide

ACETALDOL AND DERIVATIVES

Acetaldol or β-Hydroxybutyraldehyde also called **Aldol**, $CH_3CH(OH)CH_2CHO$, mw 88.10. Col liq when freshly distilled at reduced press, bp 72° at 12mm, d 1.103 and sp heat 0.737 cal/g/°C. May be prepd by the aldol condensation reaction(qv) of two acetaldehyde molecules in the presence of a small amt of an alkali. Other methods of prepn and props are given in Refs 1,2,3 & 4. Aldol is used in solvent mixts

During WWII aldol was used in Germany for the prepn of 1,3-butyleneglycol (by hydrogenation) which was either dehydrated to form butadiene or nitrated to the expl 1,3-butyleneglycol dinitrate

On standing aldol changes to a viscous dimer from which *paraldol*, $[CH_3CH(OH)CH_2CHO]_2$ separates. Wh triclinic cryst, d 1.345 at 15.6/4°, mp 95–97°. It boils in vacuo, under which condition part of it is reconverted to aldol. Sol in w or alc, sl sol in eth. Unlike paraldehyde it shows some props of the aldehydes. Used as a raw material for making resins for plastics and synth coatings(Ref 4). Acetaldol may be hydrogenated to form 1,3-butyleneglycol from which the expl 1,3-butyleneglycol dinitrate may be prepd.(See also Aldol and Aldol Condensation)

Refs: 1)Beil **1**,824,(419) & [868] 2)Kirk & Othmer **1**(1947),p 41 3)Ullmann **3**(1953),169–73(under Aldol) 4)CondChemDict(1956),823

Acetaldol, Analytical Procedures are briefly discussed under Aldol in Ullmann **3**(1953),172

ACETALDOXIME AND DERIVATIVES

Acetaldoxime or Aldoxime(Acetaldehydeoxime

or Ethanaloxime), $CH_3CH(:NOH)$, mw 59.07, N 23.71%, Ndls, mp 47°, bp 114–5°, d 0.965 at 20/4°, $n_D^{20°}$, 1.4278, Q_c^P, 340.6 kcal/mol, Q_f 12.8; sol in H_2O, misc with alc, sol in eth, acet and sl sol in gasoline. Was discovered in 1882 by V.Meyer, described by Petraczek(Ref 2) and then by Franchimont (Ref 3). Can be prepd from acetaldehyde, hydroxylaminehydrochloride and Na_2CO_3 in aq soln or by other methods. Some of its props were detnd by Landrieu(Ref 4)

Refs: 1)Beil 1,608,(327)&[675] 2)J.Petraczek, Ber **15**,2783(1882) 3)A.Franchimont,Rec **10**, 236(1891) 4)P.Landrieu,CR **140**,867(1905) 5)Kirk & Othmer **5**(1953),692(under Oximes) 6)Merck(1952),p 5

Acetaldoxime, Analytical Porcedures are not discussed in Organic Analysis,Interscience, vols 1–3(1953,1954 & 1956), but identification of oximes in general are discussed by R.L.Shriner & R.C.Fuson, "Systematic Identification of Organic Compounds", Wiley,NY (1940),167–8

Acetaldoxime, Azido Derivative, $N_3 \cdot CH_2 \cdot CH(:NOH)$ – not found in Beil or in CA through 1956

Mononitroacetaldoxime or Methazonic Acid, $(O_2N)\,CH_2 \cdot CH(:NOH)$, mw 104.07, N 26.92%, crysts, mp 79–80°; sol in w, alc, eth, acet and warm benz or chlf. Can be prepd by treating nitromethane in aq NaOH or by other methods(Refs 1 & 4). Its ammonium salt, $C_2H_3N_2O_3 \cdot NH_4$ obtained by the action of ammonia on nitromethane, dec on heating with evoln of poisonous hydrogen cyanide(Ref 3). Its K salt $C_2H_3N_2O_3K$, yel ppt expl on heating with evoln of lt blue flame(Ref 2) and its Ag salt, $C_2H_3N_2O_3Ag$, pale yel ppt, expl ca 100°(Ref 3)
Refs: 1) Beil 1,627 2)O.Schultze,Ber **29**, 2289(1896) 3)W.R.Dunstan & E.Goulding, Ber **42**, 2030–1(1909)

Nitroacetaldoxime, Anhydride, $C_4H_4N_4O_4$, mw 172.10, N 32.56%. Two isomers, α-, mp168-72° (dec)& β-, mp 121-2° are described in the literature. Ag & Na salts of α-isomer were reported to be mild expls

Refs: 1)Beil **2**,(332) & [684] 2)W.Steinkopf, JPraktChem **81**, 228(1910) 3)H.Wieland, Ann **444**, 15(1925)

ACETAMIDE AND DERIVATIVES

Acetamide, Amide or Ethanamide (Acetic Acid Amide), $CH_3CO\,NH_2$ (abbreviated to $AcNH_2$) mw 59.0, OB to CO_2 -149.2%, OB to CO -94.91%. Hygr cryst mp 81°, bp 221.2°, d 1.159 20/20°, $n_D^{78.3°}$ 1.4274 and vapor pres at 105° 10.0 mm Hg. Sol in w, alc and other solvents. Solys of several subst in acetamide were investigated by Stafford (Ref 2). Various methods of prepn are listed in Refs 1 & 4. The method from Amm acetate is described in Ref 2. When heated to decompn it emits highly toxic fumes of cyanides (Ref 6)

Numerous uses of acetamide are discussed in Refs 4 & 5. According to Ref 4, p 47, the neutral and amphoteric characteristics of acetanilide make it valuable as an anti-acid in expl compns

Refs: 1)Beil **2**, 175,(80) & [177] 2)O.F. Stafford, JACS **55**,3987(1933) 3)OrgSynth, CollVol 1(1941), 3-4 4)Kirk & Othmer **1** (1947),45-8 4a)Giua, Dizionario **1**(1948), 464 4b)Ullmann **6**(1955), 802 5)Cond ChemDict(1956),5 6)Sax(1957), 229

Acetamide, Analytical Procedures are discussed in Kirk & Othmer **1**(1947), 47 and in Organic Analysis, Interscience, **3**(1956), 188 & 192

Acetamide, Azido Derivative (Azidoacetamide or Triazoacetamide) $N_3 \cdot CH_2 \cdot CO \cdot NH_2$ mw 100.08 N55.99%. Col ndls(from benz), mp 58° expl on further heating; easily sol in alc & w, diff sol in benz & petr eth. Can be prepd by shaking azidoacetic ester with aq ammonia

Refs: 1)Beil **2**,229 2)M.O.Forster & H.E. Fierz, JCS **93**,80-1(1908)

Mononitroacetamide $O_2N \cdot CH_2 \cdot CO\,NH_2$, mw 104.07, N26.92% crysts, mp 102-3°. Was prepd by introducing ammonia into an ethereal soln

of nitroacetyl chloride (Ref 4). It forms salts, some of which are expl

Distillation of Amm nitroacetamide with concd KOH gives a tribasic acid, $C_4H_5N_3O_6$ and the Amm salt of this acid gives with Ag nitrate a yel solid which is expl. Another nitroacetamide deriv, $C_4H_5N_3O_5$, gives with Ag nitrate a wh cryst compd, $AgC_4H_4N_3O_5$, which expl violently on heating

Refs: 1)Beil **2,** 226 & (100) 2)F.Ratz, Monatsh **25,** 716 & 739(1894) 3)W.Steinkopf, JPraktChem **81,**207,Anm 210(1910) 4)W. Steinkopf & M.Kühnel, Ber **75,**1328(1942) & CA **37,**4687(1943)

Dinitroacetamide, $(O_2N)_2CH.CO.NH_2$, mw 149.07, N28.19%–not found in Beil or CA through 1956

Note: However, this compd is listed in ADL Punch Cards and Reports as Compound No 351 and also in the following confidential reports: 1)J. Farago NOrd 9951(1950) 2)N.D.Mason, NavOrd 1589, NOL(1950) 3) J.Farago et al, NavOrd 483,BuOrd(1952)

Trinitroacetamide, $(O_2N)_3.C.CO.NH_2$ –not found in Beil or CA through 1956

Acetamidine or Ethaneamidine. Same as *α*-Amino-*α*-imino-ethane

Acetamido-6-amino-uracil or 5-Acetamino-6-amino-2,4-dioxy-pyrimidine, $C_6H_8N_4O_3$, treated with 70% perchloric acid gives **diperchlorate,** $C_6H_8N_4O_3 + 2HClO_4$, crysts, decompg at 205-7°

Refs: 1)Beil **25,**[387] 2)H.Bredereck et al, Ber **86,** 853(1953)

ACETAMIDOANISOLE AND DERIVATIVES

Acetamidoanisole, Methoxyacetanilide, Acetaminoanisole or Acetaniside, (Acetaminomethyläther or Essigsäureanisidid, in Ger), $CH_3.CO.NH.C_6H_4.O.CH_3$. Three isomers are listed in Beil **13,**371,416,461,(113,133, 160) & [172,243]

Acetamidoanisole, Azido Derivative, $C_9H_{10}N_4O_2$ –not found in Beil or in CA through 1956

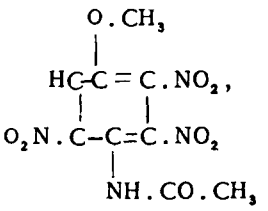
Acetamidoanisole, Diazido Derivative, $C_9H_9N_7O_2$ –not found in Beil or in CA through 1956

Mononitroacetamidoanisole, $CH_3.CO.NH:C_6H_3(NO_2).O.CH_3$. Several isomers are listed in Beil **13,** 388, 389, 390, 422, 521, 522, (136,137,186)& [192,193,194,195,216, 287]

Dinitroacetamidoanisole, $CH_3.CO.NH.C_6H_2(NO_2)_2.O.CH_3$. Several isomers are listed in Beil **13,**393,394,425,526,527,528, 530,(123,137,138,139)& [290,292]

Trinitroacetamidoanisole or Trinitroacetaminoanisole, $C_9H_8N_4O_8$, mw 300.19, N 18.67%. Following isomers are listed in Beil:

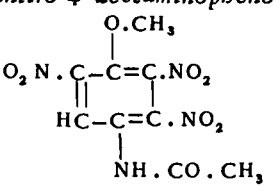
2,3,5-Trinitro-4-acetamidoanisole or 2,3,5-Trinitro-4-acetaminophenolmethylether,



(called by Lorang 4-Methoxy-*2,3,6-trinitro-1-acetylamino-benzene),* mp 242°(from dil AcOH or aq alc((Ref 2), mp 246°(from alc) (Ref 3). Was first prepd by Reverdin (Ref 2) from 2,3,5-trinitro-4-amidoanisole and acetic anhydride in the presence of a little concd sulfuric acid. Lorang (Ref 3) prepd it by nitrating 2,3-dinitro-4-acetamidoanisole with mixed nitric-sulfuric acids. Its expl props were not examined

Refs: 1)Beil **13,**(195) & [294] 2)F. Reverdin,Ber **43,**1851(1910) 3)H.Lorang, Rec **46,**638(1927) & CA **22,**230(1928)

2,3,6-Trinitro-4-acetamidoanisole or 2,3,6-trinitro-4-acetaminophenol-methylether



(called by Meldola & Kuntzen *Methyl Ether of 2,3,5-Trinitro-4-acetylaminoanisole),* wh ndls(from alc), mp 194°. Was prepd by treating Ag salt of 2,3,6-trinitro-4-acetaminophenol with methyliodide(Refs 1 & 2). Its expl props were not examined

Refs: 1)Beil **13,**(197) 2)R.Meldola & H.Kuntzen, JCS **97,**455(1910)

Tetranitroacetamidoanisole, $C_9H_7N_5O_{10}$ – not found in Beil or in CA through 1956

Acetamidobenzene. See Acetanilide

ACETAMIDOBENZENEDIAZONIUM HYDROXIDE AND DERIVATIVES

1-Acetamidobenzene–3–diazoniumhydroxide or 1-Acetamino-3-diazonium hydroxide-benzene (N-Acetyl-3-diazoniumhydroxide-aniline; Acetanilide-3-diazoniumhydroxide),

$$C_6H_4 \begin{cases} NH.CO.CH_3 \\ N_2.OH \end{cases}$$

Known only in the form of salts, some of which are expl. For instance, its *chromate* $CH_3.CO.NH.C_6H_4.N_2O(CrO_3H)$ violently explodes when pressed by a spatula or by the action of cold ammonia(Ref 1). The -ombination of its chloride with antimony chloride, $2CH_3CONH.C_6H_4N_2Cl + SbCl_3$, melts ca 94° with slight decompn (Ref 2)

Refs: 1)Beil **16,**[306] 2)W.H.Gray, JCS **1926,** 3180-1

1-Acetamidobenzene-4-diazoniumhydroxide or 1-Acetamino-4-diazoniumhydroxide-benzene (N-Acetyl-4-anilinediazonium-hydroxide or Acetanilide-4-diazoniumhydroxide),

$$C_6H_4 \begin{cases} NH.CO.CH_3 \\ N_2.OH \end{cases}$$

Known only in the form of salts, some of which are expl. For instance, its *bromide,* mp ca 116°, explodes when heated on a platinum plate; its *chromate,* $CH_3CONH.C_6H_4.N_2O(CrO_3H)$, has mp 136° with mild expln; its *picrate,* $CH_3CONH.C_6H_4.N_2.[C_6H_2(OH)(NO_2)_3]$, has mp 146.5° with mild explon (Ref 2). Its *perchlorate* is also known but its expl props have not been investigated [Ref 1, p (372)]. Its acetate and chloride are unstable and slowly evolve N_2 at 100°

Refs: 1)Beil **16,**603-4,(372) & [307] 2) W.H.Gray, JCS **1926,**3180-1

ACETAMIDODIPHENYLETHER AND DERIVATIVES

Acetamidodiphenylether, $CH_3.CO.NH.C_6H_4.O.C_6H_5$ is described in Beil **13,**414,(161) & [172,245)

Acetamidodiphenylether, Azido Derivative, $N_3.C_{14}H_{12}NO_2$ –not found in Beil or CA through 1956

Acetamidodiphenylether, Diazido Derivative, $(N_3)_2C_{14}H_{11}NO_2$ –not found in Beil or CA through 1956

Mononitroacetamidodiphenylether, $C_{14}H_{12}N_2O_4$. Two isomers are described in Beil **13,**[285,287]

Dinitroacetamidodiphenylether, $C_{14}H_{11}N_3O_6$. One isomer *2',4'-Dinitro-4-acetamido-diphenylether,* $CH_3.CO.NH.C_6H_4.O.C_6H_3(NO_2)_2$ is described in Beil **13,** 463

Trinitroacetamidodiphenylether, $C_{14}H_{10}N_4O_8$ -not found in Beil or CA through 1956

Tetranitroacetamidodiphenylether, $C_{14}H_9N_5O_{10}$, mw 407.25, N 17.20%, OB to CO_2 -88.4%, Following isomer is listed in Beil:

3,5,2',4'–Tetranitro-4-acetamidodiphenyl-ether or 3,5-Dinitro-4-acetamidophenol-[2',4'-dinitrophenyl]-ether, $CH_3.CO.NH.C_6H_2(NO_2)_2.O.C_6H_3(NO_2)_2$, wh ndls(from glac AcOH). Can be prepd by warming 3,5,2',4'-tetranitroamidodiphenylether with acetic anhydride and some concd sulfuric acid. Its expl props were not examined

Refs: 1)Beil **13,**530 2)F. Reverdin & A Dresel, Ber **38,**1595(1905)

Pentanitroacetamidodiphenylether,

$C_{14}H_8N_6O_{12}$ –not found in Beil or CA through 1956

Hexanitroacetamidodiphenylether, $C_{14}H_7N_7O_{14}$– not found in Beil or CA through 1956

ACETAMIDOFURAN AND DERIVATIVES

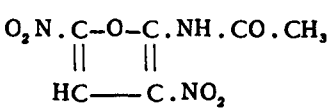
Acetamidofuran or Furylacetamide, $C_6H_7NO_2$ is described in Beil **17,**248

Acetamidofuran, Azido Derivative, $N_3 . C_6H_6NO_2$ –not found in Beil or in CA through 1956

Acetamidofuran, Diazido Derivative, $(N_3)_2 . C_6H_5NO_2$ –not found in Beil or in CA through 1956

Mononitroacetamidofuran, $(O_2N)C_6H_6NO_2$ – not found in Beil

3,5-Dinitro-2-acetamidofuran,



mw 215.12, N 19.54%, pale yel crysts, mp 155°, dec 160°. Was prepd by gradually adding acetamidofurancarboxylic acid to a stirred mixt of AC_2O and nitric acid cooled to −7°. Its expl props are not discussed in CA

Refs: 1)Beil–not found 2)T.Sasaki, Bull-InstChemResearch,Kyoto Univ **33,** 39-48(1955) (in English) & CA **50,**14705(1956)

ACETAMIDOGUANIDINE AND DERIVATIVES

Acetamidoguanidine, $CH_3.CO.NH.NH.C(:NH).NH_2$ reported in Beil **3,** 120 & [95] in the forms of its nitrate and picrate

Acetamidoguanidine, Azido Derivative, $N_3 . C_3H_7N_4O$–not found in Beil or in CA through 1956

Acetamidoguanidine, Diazido Derivative, $(N_3)_2 . C_3H_6N_4O$–not found in Beil or in CA through 1956

Acetamidoguanidine Nitrate, $CH_3 . CO . NH . NH . C(:NH) . NH_2 + HNO_3$, mw 179.14, N 39.09%. Cryst s(from abs alc), mp 145.5-146.5°(Ref 2), 142-3°(Ref 1). Sol in w and alc. Can be prepd by heating aminoguanidine, AcOH and a trace of nitric acid on a water bath (Ref 1). Q_c^v 471.55 kcal/mol and Q_f 118.04(Ref 2)

Refs: 1)Beil **3,**120 & [95] 2)M.M.Williams et al, JPhChem **61,**264 & 266(1957)

N-Acetamido-N'-nitroguanidine or 1-Acetamido-3-nitroguanidine(called in Ref 3, p 264, 1-Acetamido-2-nitroguanidine),$CH_3 . CO . NH . NH . C(:NH) . NHNO_2$, mw 161.13, N 43.46%, OB to CO −34.8%. Crysts(from w), mp 194-5° (dec). Can be prepd either from nitroaminoguanidine, AcOH and Ac_2O or from acethydrazide and methylnitrosonitroguanidine(Ref 2). Q_c^v ca 475 kcal/mol Q_f (46.3(Ref3). May be suitable as an ingredient of propellants

Refs: 1)Beil–not found 2)R.A.Henry, JACS **72,**5343-4(1950) 3)M.M.Williams et al, JPhChem **61,**264 & 266(1957)

1-Acetamidomethylhexamine Nitrate, $C_9H_{18}N_6O_4$, mw 274.28, N30.64%, crysts, mp 183-4°. One of the compds obtained by Bachmann et al in the course of investigation of the action of acetic anhydride on hexamine mononitrate, under a contract recommended by NDRC

Ref: W.E.Bachmann, E.L.Jenner & L.B. Scott JACS **73,** 2775-7(1951)

ACETAMIDONAPHTHALENE AND DERIVATIVES

Acetamidonaphthalene, Acetylnaphthylamine, Acetonaphthalide, or Naphthylacetamide, $C_{12}H_{11}NO$. Several isomers are described in Beil **12,** 1230, 1284,(524,538) & [684,719]

Acetamidonaphthalene, AzidoDerivative, $N_3 . C_{12}H_{10}NO$,–not found in Beil or CA through 1956

Acetamidonaphthalene, Diazido Derivative $(N_3)_2 C_{12}H_9NO$–not found in Beil or in CA through 1956

Mononitroacetamidonaphthalene, $(O_2N)C_{12}H_{10}NO$. Several isomers are described in Beil **12,**1258,1260,1261,1313, 1315,(530,544) & [704,705,731,732,733]

Dinitroacetamidonaphthalenes, $(O_2N)_2C_{12}H_9NO$. Several isomers are described in Beil **12,**1263,1264,1316,(532) & [709, 735]

Trinitroacetamidonaphthalenes, $(O_2N)_3C_{10}H_4.NH.O.CH_3$, mw 308.21, N18.18%, OB to CO_2 -98.6%. Following isomers are described in the literature, but their expl props were not discussed:

2,4,5-Trinitro-1-acetamidonaphthalene, cryst, dec ca 275°. Was prepd by treating 2,4,5-trinitro-1-aminonaphthalene with acetic anhydride & concd sulfuric acid

Refs: 1)Beil **12,** [709] 2)W.H.Talen, Rec **47,**356(1928)

2,4,8-Trinitro-1-acetamidonaphthalene, creamy plates, mp 207°. Was prepd by treating 2,4,6-trinitro-1-aminonaphthalene with acetic anhydride & concd sulfuric acid

Refs: 1)Beil–not found 2)E.R.Ward & L. A.Day, JCS **1951,** 785 & CA **45,**9014(1951)

1,6,8-Trinitro-2-acetamidonaphthalene, yel crysts, mp 239-40°. Was prepd by treating 1,6,8-trinitro-2-aminonaphthalene with acetic anhydride & concd sulfuric acid

Refs: 1)Beil **12,**[739] 2)E.J.van der Kam, Rec **45,**729(1926)

Tetranitro-, Pentanitro- and Hexanitroacetamidonaphthalenes–are not found in Beil or in CA through 1956

ACETAMIDOPHENETOLE AND DERIVATIVES

Acetamidophenetole, Ethoxyacetanilide or Acetaminophenolethylether, $CH_3.CO.NH.C_6H_4.O.C_2H_5$. All isomers are listed in Beil **13,**371,416,461,(113,133, 160) & [172,244]

Acetamidophenetole, Azido Derivative, $N_3.C_{10}H_{12}NO_2$–not found in Beil or in CA through 1956

Acetamidophenetole, Diazido Derivative, $(N_3)_2C_{10}H_{11}NO_2$–not found in Beil or in CA through 1956

Mononitroacetamidophenetole, $CH_3.CO.NH.C_6H_3(NO_2).O.C_2H_5$. All isomers are listed in Beil **13,**388,389,391,522,(136,137) & [193,194,285,287]

Dinitroacetamidophenetole, $CH_3.CO.NH.C_6H_2(NO_2)_2.O.C_2H_5$. Several isomers are listed in Beil **13,**394,526,(139,193) & [290, 292]

Trinitroacetamidophenetole, $CH_3.CO.NH.C_6H(NO_2)_3.O.C_2H_5$ mw 314.21, N 17.83%. Following isomer is listed in Beil:

2,3,5-Trinitro-4-acetamidophenetole or 2,3,5-Trinitro-4-acetaminophenolethylether (called by Lorang *4-Ethoxy-2,3,6-trinitroacetanilide),* crysts (from alc), mp ca 260° (dec); sol in acet & AcOH, diff sol in hot alc or benz. Can be prepd by nitrating 2,3-dinitro-4-acetaminophenetole with mixed nitric-sulfuric acid at RT. Its expl props were not examined

Refs: 1)Beil **13,**(196) & [295] 2)H.Lorang, Rec **46,** 644(1927)

Tetranitroacetamidophenetole, $C_{10}H_9N_5O_{10}$ – not found in Beil or in CA through 1956

ACETAMIDOPHENOL AND DERIVATIVES

Acetamidophenol or Acetaminophenol $CH_3.CO.HN.C_6H_4.OH$, mw 151.16, N9.27%. All existing isomers are described in Beil **13,** 370,415,460,(113,132,159) & [171,213, 243]

Acetamidophenol, Azido Derivative, $N_3.C_8H_8NO_2$–not found in Beil or CA through 1956

Acetamidophenol, Diazido Derivative,

$(N_3)_2C_8H_7NO_2$—not found in Beil or CA through 1956

3-Azido-2,6-dinitro-4-acetamidophenol (called in Ref 2 2,5-Dinitro-4-acetylamino-3-triazophenol) $CH_3 . CO . NH . C_6H(N_3)(NO_2)_2 . OH$, mw 282.18, N29.79%. Ocher-colored ndls or golden yel scales (from alc), mp 167-8°. Can be prepd by treating 2,3,6-tritro-acetamidophenol with Na azide in warm w. Its expl props were not examined

Refs: 1)Beil **13,**(198) 2)R.Meldola & H.Kuntzen, JCS **99,**43(1911)

Mononitroacetamidophenol, $C_8H_8N_2O_4$, mw 196.16,N 14.28%. All possible isomers are described in Beil **13,** 422-3, 520-1, (136-7) & [191,193-4,285,287]

Dinitroacetamidophenol, $C_8H_7N_3O_6$, mw 241.16, N17.43%. All possible isomers are listed in Beil **13,** 396,425,528,530,(193-4) & [197,216, 290,292]

Trinitroacetamidophenol, $C_8H_6N_4O_8$, mw 286.16, N19.58%. The following isomers are described in the literature:

2,3,5-Trinitro-4-acetamidophenol, CH_3CO:NH . $C_6H(NO_2)_3$. OH, lt brn scales (from AcOH), mp 191-2°(dec), easily sol in alc & AcOH. Can be prepd by treating 3,5-dinitro-4-acetamidophenol with fuming nitric acid at 0° or by other methods. Its explosive props were not examined *Refs:* 1)Beil **13,**(195) 2)F. Reverdin & R. Meldola, ,JPrChem **88,** 798(1913) & JCS **102,** 1493(1913)

2,3,6-Trinitro-4-acetamidophenol, CH_3CO:HN . $C_6H(NO_2)_3$. OH, yel ndls (from AcOH), mp 178-9°(dec); sol in AcOH and hot alc; diff sol in hot w. Can be prepd by treating 3-nitro-4-acetamidophenol with fuming nitric acid (Ref 2) or by other methods (Ref 1,4 & 5). Forms numerous salts of which the **cobalt, nickel, and silver** salts are mild explosives (Ref 3)

Refs: 1) Beil **13,** 533,(197) & [296] 2) R. Meldola & J.G.Hay, JCS **95** 1380(1909) 3)R. Meldola & H.Kuntzen, JCS **97,**449-51 (1910) 4)F.Reverdin & R.Meldola, JPr Chem **88,**797(1913) & JCS **103,**1492(1913) 5)A.Girard,BullFr [4] **35,**776(1924) & JCS **126i,** 959(1924)

3.5.6-Trinitro-2-acetamidophenol, 2-Acetamido-3,5,6-trinitrophenol or 2-Hydroxy-3,4,6-trinitroacetanilide, lt yel leaflets mp 151°. Was prepd by adding 2-acetamido-5-nitrophenol to nitric acid

Refs: 1)Beil—not found 2)G. Heller et al, JPraktChem **129,**242(1931) & CA **25,**2129 (1931)

Tetranitroacetamidophenol, $C_8H_5N_5O_{10}$, mw 331.16, N 21.15%—not found in Beil or CA through 1956

ACETAMIDOTETRAZOLE AND DERIVATIVES

5-Acetamido-1H-tetrazole, called by Thiele & Ingle Acetyl-[5-amino-tetrazol] and in Beil

Tetrazolon-(5)-anil, $CH_3 . CO . NH . C(=N-NH-N)$ or $CH_3 . CO . N{:}C(-NH-N)$ (HN—N), mw 127.11, N55.10%.

Wh leaflets (from w or alc) or prisms(from acetanhydride), mp 269°(dec)(Refs 1,2&3), mp 271°(Ref 4); easily sol in alc, acetanhydride & hot w, nearly insol in ether. Can be prepd by heating 5-aminotetrazole with an excess of acetanhydride or acetylchloride. Its X-ray diffraction pattern is given in Ref 5

Refs: 1)Beil **26,** 405 & [243] 2)J.Thiele & H.Ingle, Ann **287,** 234(1895) 3)R.Stollé, Ber **62,**1121(1929) 4)L.Birkofer, Ber **76,** 773(1943) & CA **38,**970(1944) 5)L.A.Burkhardt & D. W. Moore, AnalChem **24,**1583(1952) & CA **47,**2010(1953)

Acetamidotetrazole, Azido Derivatives—not found in Beil or CA through 1956

Acetamidotetrazole, Nitro- and Dinitro-Derivatives—not found in Beil or CA through 1956

ACETAMIDOTOLUENE AND DERIVATIVES

Acetamidotoluene, Methylacetanilide or Acetoluidide, $C_9H_{11}NO$. The three isomers are described in Beil **12**, 792,860,920,(379, 400,420), [439,468,501]

Acetamidotoluene, Azido Derivative, $N_3 . C_9H_{10}NO$—not found in Beil or in CA through 1956

Acetamidotoluene, Diazido Derivative, $(N_3)_2C_9H_9NO$—not found in Beil or in CA through 1956

4-Acetamidotoluene-4-diazoniumhydroxide or 4-Acetamido-2-diazoniumhydroxide-toluene (N-Acetyl-4-methyl-3-diazonium-hydroxide-aniline or 2-Methyl-5-acetamino-benzenediazoniumhydroxide),

$CH_3C_6H_3$ (NHCOCH$_3$ / N_2OH). Known only in the form of its salts some of which are stable and a few expl. For instance, the *bromide*, $CH_3 . CO . NH . C_6H_3(CH_3) . N_2 . Br$, prepd by Wallach from 2-amino-4-acetaminotoluene (Ref 3) and designated by him as "acetparatoluidine-o-diazobromide" (Refs 1 & 2), is a yel solid which explodes on rapid heating. The *chloride*, designated as "acetparatoluidine-o-diazochloride" and prepd from 4-acetaminotoluene-2-diazopiperidide (Ref 4), also explodes on heating

Refs: 1)Beil **16**,608 2)O.Wallach,Ann **235**,249(1886) 3)Beil **13**, 133,(41) & [62] 4)Beil **20**,91

Mononitroacetamidotoluene, $C_9H_{10}N_2O_3$. Several isomers are described in Beil **12**, 843, 845,847,849,876,877,998,1002,(392,393,394, 408,440) & [458,459,460,476,477,534,536]

Dinitroacetamidotoluene, $C_9H_9N_3O_5$. Several isomers are described in Beil **12**, 851,1010, (396,409,442) & [462,463,479,480,481,482, 538]

Trinitroacetamidotoluene, $C_9H_8N_4O_7$, mw 284.19, N19.72%, OB to CO_2 -80.9%. One isomer is listed in Beil

2,4,6-Trinitro-3-acetamidotoluene, $(O_2N)_3 . C_6H$ (CH$_3$ / NH . CO . CH$_3$), microscopic ndls(from alc), mp 249° (dec). Was prepd by warming 2,4,6-trinitro-3-amidotoluene with acetic anhydride and some concd sulfuric acid. Its expl props were not discussed

Refs: 1)Beil **12**, [482] 2)J.W.Cook & O.L.Brady, JCS **117**, 752(1920)

Tetranitroacetamidotoluene, $C_9H_7N_5O_9$—was not found in Beil or in CA through 1956

ACETAMIDOTRIAZOLE AND DERIVATIVES

4-Acetamido-asym-triazole, called by H & S "Acetyl-hydrotetrazin," HC=N—N (| ||), mw

$CH_3 . CO . NH . N$—— CH

126.12, N 44.43%, apparently cryst compd, very sol in w & alc and insol in eth, ligroin & chlf. Was prepd by heating 4-amino-asym-triazole with acetyl chloride

Refs: 1)Beil **26**,19 2)A.Hantzsch & O. Silberrad, Ber **33**,84(1900)

1-Acetamido-asym-triazole, called in CA **1,2,4-Triazole-1-acetamide**, HC·N(CH$_2$·CO·NH$_2$)·N, shiny plates, mp 185-

N —— N

6°. Was obtained from ethyl-1,2,4-triazole-1-acetate in methanol satd with ammonia at RT

Refs: 1)Beil-not found 2)C.Ainsworth & R.Jones, JACS **77**, 622(1955) & CA **50**,1785 (1956)

Acetamidotriazole, Azido-Derivative—not found in Beil or CA through 1956

Acetamidotriazole, Nitro- and Dinitro-Derivatives—not found in Beil or CA through 1956

ACETANILIDE AND DERIVATIVES

Acetanilide, Acetamidobenzene or Acetylaniline (N-Phenylacetamide, Antifebrin or Acetanil), $C_6H_5 . NH . CO . CH_3$, mw 135.16, OB to CO_2—231%, OB to CO -136.1%. Col crysts, mp 114.2°, bp 303.8°, d 1.21 at 4/4°, Q_c^v 1014.4 kcal/mol, heat of

vaporization at 154° 136 cal/g, mean heat capacity (from 0° to 99.6°) 0.339 cal/g°C, sol in w, alc & eth. Can be prepd by heating aniline with AcOH or by other methods. A lab prepn from aniline and $(CH_3CO)_2O$ is described in Ref 2. Its fire hazard is small when exposed to flame(Ref 6)

Various uses of acetanilide, among them as a stabilizer for hydrogen peroxide and for cellulose, are listed in Refs 4 & 5. According to Ref 4, p 52, a considerable quantity of acetanilide was used during WWII for the manuf of acetylsulfonyl chloride

Note: According to one of the Hercules Co Laboratory manuals, acetanilide was used as one of the ingredients of smokeless propellants. Some nitrated acetanilide derivatives are explosive

Refs: 1)Beil **12**,237,(190) & [137] 2) OrdSynth, CollVol 1(1941),332 3)H.E.Fierz-David & L.Blangey,"Grundlegende Operationen der Farbenchemie,"Wien(1943),125 Kirk & Othmer **1**(1947),48-52 4a)Ullmann **3**, (1953), 653-4(under Aniline) 5)Cond Chem Dict (1956), 6 6)Sax(1957), 229 7)Faith,Keyes & Clark(1957),8-10

Acetanilide, Analytical Procedures are discussed in Refs: 1)Kirk & Othmer **1**(1947), 52 and 2)Organic Analysis, Interscience, NY, **2**(1954), 44,133,142,162, & **2**(1956),186, 188,190

Acetanilide, Azido Derivatives, $C_8H_8N_4O$, mw 176.18, N 31.80%. Following isomers are described in the literature:

Azidoacetanilide or Triazoacetanilide, $C_6H_5 . NH . CO . CH_2 . N_3$; ndls, mp 83-83.5°. Was prepd by treating aniline with azidoacetamide

Refs: 1)Beil **12**, 245 2)M.O.Forster & R.Müller, JCS **95**,201(1909)

4-Azidoacetanilide, Acet-[4-azido]-anilide or Acetyl-p-aminotriazobenzene, $N_3 . C_6H_4 . NH . CO . CH_3$ crysts, mp 122.5-124°. Was prepd by treating p-acetamido-benzenediazoniumperbromide with ammonia

Refs: 1)Beil **12**,772 2)H.Rupe & K.von Majewski, Ber **33**,3406(1900) 3)O.Silberrad & B.J.Smart, JCS **89**,170(1906)

Acetanilide, Diazido Derivative, $N_3 . C_6H_4 . NH . CO . CH_2 . N_3$—not found in Beil or CA through 1956

Mononitroacetanilides (MNAA), $C_8H_8N_2O_3$. All isomers are described in Beil **12**,245,691, 703,719,(193,342,347,351) & [371,380,389]. A new method of prepn of p-MNAA is given in USP 2,406,578(1948), by E.H.Bart,CA **41**, 153(1947)

Note: nitration of acetanilide has been discussed in the following refs:

1)A.P.Terent'ev & B.M.Kedrov, SciRepts-MoscowUniv **1936**, No 6,213-14 & CA **32**, 2519(1938) 2)H.McCormack, IEC **29**, 1333-5 (1937) 3)P.P.Shorygin et al, ZhObshKhim **8**, 981-5(1938) & CA **33**,3781(1939)(Nitration of acetanilide with an excess of liq nitrogen dioxide)

Nitronitrosoacetanilides, $C_8H_7N_3O_4$. Several isomers were prepd by H.France et al, JCS **1940**,370 & CA **34**,3700(1940)

Dinitroacetanilides (DNAC), $C_8H_7N_3O_5$, mw 225.16, N 18.66%. Several isomers are listed in Beil **12**,447,754,758,759,(362) & [405,410, 414]

Note: B.B.Dey et al, JSciIndianResearch **10B**,140-4(1951) & CA **47**,3257(1953) discuss the prepn of 3.4-DNAA, mp 145-6° and of 2,3-DNAA, mp 186-7°

2,4-Dinitro-N-nitrosoacetanilide, $(O_2N)_2-C_6H_3 . N(NO) . CO . CH_3$, mw 254.16, N 22.05%, oil, prepd by treating 2,4-dinitroacetanilide with nitrosyl chloride in the presence of K acetate. Its expl props were not investigated

Refs: 1)Beil—not found 2)H.France et al, JCS **1940**,370-1 & CA **34**,3700(1940)

Dinitro-N-nitroacetanilide, $(O_2N)_2C_6H_3 . N(NO_2)-CO.CH_3$, mw 270.16 N 20.74% —not found

in Beil or in CA through 1956

Trinitroacetanilides, $C_8H_6N_4O_7$, mw 270.16, N20.74, OB to CO_2 -71.1%, OB to CO -23.7%. Following isomers are described in the literature:

2,3,5-Trinitroacetanilide or N-Acetyl-2,3,5-trinitroaniline and 3,4,5-Trinitroacetanilide or N-Acetyl-3,4,5-Trinitroaniline, $(O_2N)_3C_6H_2.NH.CO.CH_3$, were prepd by acetylation of corresponding trinitroanilines(TNA's)

Refs: 1)Beil–not found 2)E.Maciotta, AnnChimAppl(Rome) **36,**212(1946) & CA**41,** 4115(1947)

2,4,6-Trinitroacetanilide or N-Acetyl-2,4,6-Trinitroaniline, solid, mp 235° (with decompn). Was prepd by treating 2,4,6-trinitroaniline with acetic acid and a little concd sulfuric acid. Its expl props are not mentioned in Refs 1,2 & 3. For absorption spectra see Ref 4

Refs: 1)Beil **12,**767 & [423] 2)O.N.Witt & E.Witte, Ber **41,**3092(1908) 3)W.Borsche, Ber **56,** 1940(1923) 4)K.Masaki, BullChem-Soc Japan **7,** 352(1932) & CA **27,** 904(1933)

Trinitro-N-nitrosoacetanilide, $(O_2N)_3C_6H_2.N(NO).CO.CH_3$–not found in Beil or in CA through 1956

Trinitro-N-nitroacetanilide, $(O_2N)_3C_6H_2.N(NO_2).CO.CH_3$–not found in Beil or CA through 1956

2,3,4,6-Tetranitroacetanilide or N-Acetyl-2,3,4,6-tetranitro-aniline, $(O_2N)_4C_6H.NH.CO.CH_3$, mw 315.16, N 22.22%, OB to CO_2 -48.2%, OB to CO -7.6%. Yel ndls(from benz +AcOH), mp 169-70° (with decompn). Was prepd by heating 2,3,4,6-tetranitroaniline with acetic anhydride and a little concd sulfuric acid. TeNAA is an expl and was proposed by Flürscheim(Ref 2) as an ingredient of expl compns. It is rather unstable, even at low temp and sensitive to shock (Ref 3)

Refs: 1)Beil **12,**(372) & [428] 2)B.J. Flürscheim ChemZtr **1912,** I, 184 3)C.F. vanDuin & B.C.R.vanLennep,Rec **39,**149 (1920) & CA **14,**2708(1920) 4)B.J.Flürscheim & E.L.Holmes, JCS **1928,**3046

Tetranitro-N-nitrosoacetanilide, $(O_2N)_4C_6H.N(NO).CO.CH_3$, mw 344.16, N24.42%–not found in Beil or in CA through 1956

2,3,4,6-Tetranitro-N-nitroacetanilide or N,2,3,4,6-Pentanitroacetanilide, $(O_2N)_4C_6H.N(NO_2).CO.CH_3$, mw 360.16, N23.34%. Yel solid, unstable; decomp slowly in air and in boiling w. Was prepd by treating 2,3,4,6-tetranitrophenylnitramine with acetyl chloride

Refs: 1)Beil–not found 2)E.Macciotta & Z.Orani, Gazz **60,** 408(1930) & CA **24,** 4280 (1930)

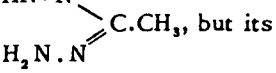
Acetaniside. Same as Acetamidoanisole

Acetates. See under Acetic Acid and Derivatives

Acetatopentamminecobalt(III)nitrate, $[CO(NH_3)_5C_2H_3O_2](NO_3)_2$, crysts. Its lab method of prepn from carbonatopentamminecobalt(III)nitrate is described in Inorg Synth **4**(1953),175-6

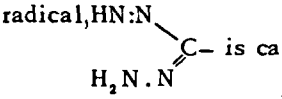
Acetato-plumbo Complexes. See Diacetatoplumbo Complexes

Acetazidine, Azidine or Acethydrazidine. Beil **2,** p 4 gives its formula as:

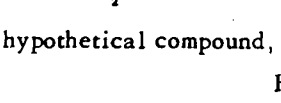


but its prepn is not given

According to Beil **16,** pp 4-5, the hypothetical radical,



is called *formazyl* and the hypothetical compound,



is called *formazan* or *formazylhydride* (Formazylwasserstoff). This means that *acetazidine* may be called *C-methylformazan.*

Acethydrazidine or Azetazidine. See Formazans and Formazyls

ACETIC ACID AND DERIVATIVES

Acetic Acid (Ethanoic or Acetonecarboxylic Acid)(AcOH)(Essigsäure,, in Ger), CH_3COOH, mw 60.05, OB to CO_2 -106.6%. Col liq, mp 16.7°, bp 118.1°, d 1.049 at 20°/4°, fl p 104°E, Q_c^p 208.5 kcal/mol(Ref 4). Miscible with w, alc & eth; insol in CS_2. It is found in the products of distn of wood and for this reason is called "pyroligneous acid ". Can be prepd by the oxidation of acetaldehyde, by the action of CO on methanol or by other methods (Refs 1,5,7 & 8). Dangerous at high temps in contact with oxidizing agents, such as nitric or chromic acids. Its vapors can produce in air a moderately severe expln above 112°F(44.4°)(Ref 3a). Toxicology and fire & expl hazards of acetic acid are discussed in Ref 9. Freezing points of mixts AcOH/H_2O and AcOH/Ac_2O are given in Refs 2 & 3. More information on chemical and physical props of AcOH is given in Refs 5 & 7

Various uses of acetic acid are discussed in Refs 5 & 7. Its principal use in explosives industry is the manuf of cyclonite(RDX) by the Bachmann process. It can also be used for the prepn of high-nitrogen(ca 14%N) nitrocellulose. US specification JAN-A-465, covers the requirements for acetic acid used in Ordnance (see Acetic Acid, Analytical Procedures)

Recovery of acetic acid used in the production of cyclonite and of other nitramines is discussed in Ref 6

Refs: 1)Beil **2**,96(39) & [91] 2)Land-Börnst,HW II,1443; Erg I,749 & Erg II,Teil II, 1472 3a)A.L.Brown,CA **22**,166(1928) 3) ICT **4**,108 4)E.Schjanberg,ZPhysChem **172A**,228(1935) & CA **29**,3589(1935) 5) Kirk & Othmer **1**(1947),56-74 6)HACSIR, BritP 626,926(1949) & CA **44**,3258(1950) 7)Ullmann **6**(1955),778-90 8)Faith,Keyes & Clark(1957),11-20 9)Sax(1957),230

Additional References on Acetic Acid: a)M.Usanovich & S.Abidov, ZhurObshKhim **10**,223-6(1940) & CA **34**,7285(1940) (Nitration of toluene in the presence of acetic acid and nitrobenzene) b)J.Chédin & S.Fénéant, MSCE **32**,92-100(1945)(Molecular composition of HNO_3–AcOH mixtures; studies by Raman spectroscopy) c)J.Chédin et al, MSCE **34**, 289-90(1948)(Mixtures of HNO_3, AcOH, H_2O and metallic nitrates) d)M.Kirsch & C.A. Winkler, CanJRes **28B**, 715-19(1950)(Nitrolysis of hexamine in acetic acid) e)E.D.Hughes et al, JCS **1950**,2406-09(Mechanism of aromatic nitrations in the presence of AcOH) f)S.Fénéant-Eymard, MSCE **37**,297-346(1952) (Physico-chemical studies of AcOH) g) A.V.Titov,ZhObshKhim **24**, 77-9(in Engl) & 78-81(in Rus)(1954); CA **49**,7338(1955)(Formation of slightly stable compd $CH_3COOH \cdot HNO_3$)

Acetic Acid, Analytical Procedures. Acetic acid intended for use in the manuf of expls must comply with the following chemical and physical requirements of Purchase Description PA-PD-572 (superseding Spec JAN-A-465):

a) *Acetic Acid Content*–minim 99.8%, as detd by immersing a glass-stoppered weighing bottle contg 5 ±0.0002 g sample in 250 ml w (previously neutralized to phenol-phthalein by adding few drops of 0.1N NaOH soln) and titrating to a pink end point with 0.1N NaOH

$$\%AcOH = \frac{6.005 \times V \times N}{W}$$

where V = vol of NaOH used in titrating sample, N = normality of NaOH soln and W = grams of sample

b)*Acetic Anhydride Content*–max 0.03%, as detnd by treating a 10 ml sample (measured by a pipette), dissolved in 50 ml w (contained in a 250 ml iodine flask with 10.00 ml of an approx 1.25% Na bisulfite soln) and titrating (after allowing the mixt to stand for 15 mins) the excess of bisulfite with 0.1N std iodine soln, using starch as an indicator

$$\%Ac_2O \text{ in } AcOH = (A - B) \times N \times 0.220,$$

where A = ml of iodine soln used to titrate

10.00 ml of Na bisulfite soln treated as above but without the sample (blank); B = ml of iodine soln used for bisulfite soln contg sample and N = normality of iodine soln

c) *Formic Acid Content*—max 0.15%, as detnd by treating a 10 ml sample (measured by a pipette), dissolved in 100 ml w (contained in a 250 ml iodine flask) with 10 ml of 20% Na acetate soln and 25.00 ml of 0.01 N Na hypobromite soln. After allowing the mixt to stand 15 mins at RT and adding 5 ml of 25% KI soln & 10 ml of concd HCl, the liberated iodine shall be titrated with 0.1N std Na thiosulfate soln

%H COOH in AcOH = (A − B) × N × 0.230,

where A = ml of Na thiosulfate soln used to titrate 25 ml of Na hypobromite soln treated as above but without using the sample (blank); B = ml of thiosulfate soln used to titrate the mixt contg the sample and N-normality of iodine soln

d) *Freezing Point*—min 16.2°, as detd by means of 0° to 50° mercury thermometer graduated to 0.1° and standardized against one having the Bureau of Standards calibration. The test is conducted in 100 ml cylinder immersed in an ice-water bath. After supercooling the sample to ca 1° below the assumed fr p, the cylinder is removed from ice water and its inner side scratched and the contents stirred by means of the thermometer until the supercooled liquid partly solidifies. Then the outside of the cylinder is wiped dry and the thermometer watched very closely. The temp rises quickly and then remains constant for about 30 secs. This temp is taken as the fr p of sample

d) *Lead Content*—max 10 ppm, as detd by evaporating a 5 ml sample on a steam bath, dissolving the residue in 2 ml of 0.1N HCl soln, transferring the soln quantitatively (by rinsing with w) into a 25 ml Nessler tube and filling the tube to the mark with w. A second Nessler tube shall be filled to 25 ml mark with the standard soln (which was previously prepd by dissolving 3.2 mg of lead nitrate and 5 ml of pure AcOH in 1l of w) and 10 ml of satd hydrogen sulfide water added to the contents of each Nessler tube. After allowing the tubes to stand for 10 mins, the color in the 1st tube shall not be darker than that in the 2nd tube

f) *Iron Content*—max 10 ppm, as detd in the manner described for lead detn, except that soln of the residue in 2 ml of 0.1N HCl soln shall be made alkaline by Amm hydroxide soln before pouring it into the 1st Nessler tube and that the standard soln for the 2nd Nessler tube shall be prepd by dissolving 0.43g of $Fe(NH_4)(SO_4)_2 \cdot 12H_2O$ and 5 ml of AcOH in 1 l of w. Same reagent, hydrogen sulfide water, as above is used in both tubes

g) *Chlorides Content*—max 0.001%, as detd by comparing the turbidity produced on mixing in a 50 ml Nessler tube a 10 ml sample, 35 ml w, 3 ml concd nitric acid and 1 ml of 0.1N silver nitrate soln with that produced by 10 ml of standard soln (prepd by dissolving 15 mg of NaCl in 1 l w) with 35 ml w, 3 ml concd nitric acid and 1 ml of 0.1N silver nitrate soln. The turbidity produced in the 1st tube shall be not greater than that in the 2nd one

h) *Sulfates Content*—max 0.001%, as detd by evaporating to dryness a 50 ml sample contg 10 mg of Na carbonate on a steam bath, dissolving the residue in 5 ml of w, filtering into a 10 ml Nessler tube and diluting to the mark with w. A 2nd 10 ml tube shall contain 10 ml of standard soln, previously prepd by dissolving 82 mg of K sulfate in 1 l of w. After adding to each tube 1 ml of 1:20 hydrochloric acid & 1 ml barium chloride soln and allowing to stand for 10 min, the turbidity produced in the 1st tube shall be not greater than that in the 2nd tube

i) *Sulfurous Acid Content*—max 0.001%, as detd by titrating 300 ml of w contg some starch indicator with 0.01N iodine soln to

end point, adding 50 ml of sample and continuing titration to the same end point

% Sulfurous acid = V × N × 0.082,

where V = total ml of iodine soln minus ml used to titrate w and N=normality of iodine soln

Slightly more rigid requirements are for the reagent grade acetic acid (see Ref 2)

For more information on analysis of acetic acid see Refs 1 & 3

Refs: 1)Kirk & Othmer **1**(1947), 68-9 2) ReagentChemicals (1950),20-3 3)Organic Analysis, Interscience, **2**(1954) & **3**(1956) (Various methods are described. Detn of AcOH in anhydride is in v 3, p 24)

Acetic Acid, Azido Derivative (Azidoacetic Acid or Triazoacetic Acid), $N_3 . CH_2 . CO_2H$, mw 101.07, N 41.58%. Col hygrosc plates, mp ca 16°, bp 93° at 3 mm, d 1.354 at 33°. Detonates violently on heating in a capillary tube; expl mildly with evoln of flame on heating on a hot plate. Can be prepd by shaking ethylester of azidoacetic acid with aq 20% KOH soln

Its *silver salt,* $C_2H_2O_2N_3Ag$, N 20.2%, ndls, deflagrates on heating

Refs: 1)Beil **2**,229 & (100) 2)M.Forster & H.Fierz, JCS **93**,76(1908) 3)Yu.Sheinker & Ya.Syrkin,IzvestAkadN,SerFiz **14**,478-87 (1950) & CA **45**,3246(1951)(Ultraviolet,Raman and Infrared spectra) 4)Yu.Sheinker,Dokl-AkadN **77**, 1043-5(1951) & CA **45**,6927(1951) Ultraviolet spectra of azidoacetic acid) 4)J.H.Boyer & J.Hamer, JACS **77**,953(1955) & CA **50**,1826(1956)

Acetic Acid, Diazido Derivative (Azidoacetic Acid Azide or Triazoacetic Acid), $N_3 . CH_2 . CO . N_3$, mw 126.08, N66.66%. Col oil with unpleasant smell; mp explodes; insol in w. Was prepd by treating azidoaceticacidhydrazidehydrochloride with Na nitrite in cold aq soln

Refs: 1)Beil **2**,230 & (101) 2)T.Curtius, et al, Ber **41**, 354 & 1036(1908)

Nitroacetic Acid (Nitroäthansäure or Nitroessigsäure, in Ger), $O_2N . CH_2 . COOH$, mw 105.05, N 13.33%. Ndls, expl on heating; sol in chlf, benz & toluene; insol in petr eth; decompd by w; yields nitromethane when heated with w. Was prepd from the dipotassium salt (see below) and dry HCl in ether (Refs 1 & 3)

Its **dipotassium salt,** $C_2HK_2NO_4$, ndls, sol in w and insol in ethyl & methyl alcs, can expl on contact with w(Ref 2). Can best be prepd by treating nitromethane with KOH (1:1)(Ref 6). Other methods of prepn are given in Refs 1 & 2. It is a powerful expl(Ref 5)

The aq soln of the salt yields with Pb acetate a white ppt and with Ag nitrate and mercurous chloride yel ppts. These salts deflagrate in a flame (the Ag salt the strongest) (Ref 3)

Refs: 1)Beil **2**,225 2)W.Steinkopf, Ber **42**,2029-31(1909) 3)Ibid,**42**, 3925-9(1909) 4)A.Hantzsch & R.Voigt, Ber **45**,108(1912) (Absorption spectra of nitroacetic acid in alc, ether & aq alk solns) 5)D.A.Lyttle & D.I.Weisblatt, JACS **69**,2118(1947) & CA **42**, 114(1948)(The chemistry of nitroacetic acid and its esters) 6)H.Feuer et al, JACS **71**,3079 (1949) & CA **44**, 2915(1950) 7)D.A.Lyttle, C & EN **27**, 1473(1949) & CA **43**,4855(1949) (Approx 3 lb dipotassium nitroacetate in a 10 gal stainless steel can, moistened with w and covered with about 7 lb dry K nitroacetate exploded after 30 mins at RT)

Acetic Acid–Perchloric Acid Mixture. See Perchloric Acid –Acetic Acid Mixture

ACETATES

Ammonium Acetate(Normal), $CH_3COO . NH_4$, mw77.08 Wh deliq crysts, mp 114°, bp decomp, d 1.171 at 25°/4° (see Note). Sol in w & alc, sl sol in acet. Can be prepd by neutralizing acetic acid with ammonia or Amm carbonate. The commercial salt contains some acid salt (see below). The pure salt is used as a lab reagent; etc

Note: According to most chemical handbooks the density of Amm acetate is equal to 1.073. This seems to be impossible if the densities of aqueous solns, as given in Ref 2,p 810, are 1.077 for 40% soln and 1.092 for 50% soln at 16°/4°. The d = 1.171, cited here, was detd by Bilz & Balz (see Ref 1, p 113)

Refs: 1)Beil **2**,107,(47)&[113] 2)Kirk & Othmer **1**(1947),810-11 3)Ullmann **6**(1955), 791 4)CondChemDict(1956),69

Ammonium Acetate(Acid), $CH_3COONH_4CH_3COOH$. Long deliq ndls, mp ca 66°. Sol in w & alc. Can be prepd by distn of the normal salt in acetic acid

Refs: 1)Kirk & Othmer **1**(1947),811 2) Ullmann **6**(1955),791

Calcium Acetate(Acetate of Lime), $(CH_3COO)_2Ca$, mw 158.17. Cryst from w as col ndls contg 2 mols of water of crystn. The transition of the di- to the monohydrate(89.77% of anhyd salt) takes place at 84°. The normal commercial salt contains 82-85% of anhyd salt. The anhyd salt decomp when heated at moderate temp. It is sol in w & alc. Can be prepd by neutralizing the pyroligneous liquors from hardwood distn with Ca carbonate followed by evapn, drying and purification. Until about 1932 it was the only important source for prepn of acetone and acetic acid but the development of the Weizmann fermentation process (yielding acetone and synthetic acetic acid), as well as direct acetic acid processes have relegated the salt to a place of minor industrial importance

Monohydrate of Ca acetate is used as analytical reagent and its requirements when used in military installations are listed in US Spec MIL-C-14000

Refs: 1)Beil **2**,111,(48) & [116] 2)Marshall **1**(1917),340-4 3)Kirk & Othmer **2**(1948) 447/50) 4)Ullmann **6**(1955),792 5)Cond ChemDict(1956),202

Cellulose Triacetate. See under Acetyl Celluloses

Dipotassium Nitroacetate. See p A27

Lead Acetate (Sugar of Lead or Plumbous Acetate)(Bleiazetat or Bleizucker in Ger), $Pb(CH_3COO)_2 \cdot 3H_2O$, mw 379.35. Wh monocl crysts, d 2.55, mp- loses $3H_2O$ at 75°; for the anhyd salt, $Pb(CH_3COO)_2$, mw 325.35 mp ca 280°. Sol in w and glycerin; sl sol in alc, chlf, CCl_4 & benz. Can be prepd by dissolving litharge in hot concd AcOH or by treating a mixt of lead and AcOH with air under press. Its lab method of prepn from red lead oxide (Pb_3O_4), AcOH and chlorine is described in Ref 2

Uses of lead acetate are discussed in Ref 2. US spec MIL-L-15172 lists the requirements of tech grade used in the manuf of antifouling paint for ship bottoms. The material shall be white,crystn and contain 84.89% of $(CH_3COO)_2Pb$ which corresponds to 99% of the trihydrate. A 5 g sample dissolved at RT in 8 ml of recently distd w shall be no more than sl turbid and shall become clear on the addn of 20 ml distd w and 2 drops of AcOH

Note: According to one of the Hercules Powder Co laboratory manuals, lead acetate is used in the prepn of test paper for detection of hydrogen sulfide. Strips of filter paper are dipped in 10% Pb acetate soln and then dried in atm free of H_2S. The moist test paper, in the presence of even traces of H_2S, gives a black color

Refs: 1)Beil **2**,115-6,(50) & [120] 2)Inorg Synth **1**(1939) 47-9 3)Kirk & Othmer **8**(1952), 268 4)CondChemDict(1956),640

Lead Acetates (Basic) are obtained by dissolving lead oxide in aq solns of sugar of lead. White solids, very sol in w. Ullmann (Ref 2) gives their formulae as, $[Pb_2(OH)_2](CH_3COO)_2$ and $[Pb_3(OH)_4](CH_3COO)_2$

Refs: 1)Kirk & Othmer **8** (1952), 268 2Ullmann **6**(1955), 791

Lead Sub-acetate, CH_3COOPb and $(CH_3COOPb)_2 . H_2O$. Wh powd, sol in w. Can be prepd by reaction of lead sub-oxide and acetic anhydride in atm of nitrogen. Its aq soln is used in lab for pptg colloidal substs from solns

Refs: 1)Beil **2,** (50) 2)H.G.Denham, JCS **115,**109-11(1919)

Lead Tetracetate, $Pb(CH_3COO)_4$, mw 443.39. Col monocl ndls, mp 175°, d 2.228; decomp in w to form PbO_2. Can be prepd by adding warm glac AcOH to red lead followed by cooling(Ref 1). It is used as an oxidizer in some chemical reactions (Ref 2)

Refs: 1)Kirk & Othmer **8**(1952),268 2) Ullmann **6**(1955),791-2

Lead Aceto-Bromate, $Pb_2(CH_3COO)_2(BrO_3)_2$, plates, expl when heated to ca 165° or on impact {Beil **2,**[121]}

Lead Aceto-Chlorate, $Pb_2(CH_3COO)(OH)-(ClO_3)_2, 2½H_2O$, scales, expl violently on heating {Beil **2,** [121]

Lead Aceto-Chlorate, $Pb_3(CH_3COO)_2(OH)_2-(ClO_3)_2, 3H_2O$, plates, expl very violently on heating { Beil **2,**[121]}

Lead Aceto-Perchlorate, $Pb_2(CH_3COO)_2-(ClO_4)_2, H_2O$, plates, expl violently on heating or on impact {Beil **2,**[121]}

Lead Aceto-Sodium Perchlorate, $Pb(CH_3COO)_2-NaClO_4$, prisms, explode on heating {Beil **2,** [121]}

Sodium Acetate, $CH_3COO.Na$, mw 82.04. Wh deliq monocl crysts, d 1.528, mp 324°, $n_D^{20°}$ 1.464. Sol in w, sl sol in alc. Can be prepd by neutralizing acetic acid with Na_2CO_3 or with NaOH and heating the resulting trihydrate to remove water of crystn (Refs 1 & 2). Can also be obtained from wood sulfite liquor and NaOH or $Ca(OH)_2$(Ref 2). Its uses are listed in Refs 2,5 & 6. According to Violette (Ref 3) mixts of equal parts of Na acetate with K saltpeter explode violently when heated. The same applies to mixts of Na acetate and saltpeter (Ref 4)

Refs: 1)Beil **2,**107-8,(47) & [113] 2)Kirk & Othmer **12**(1954),599-600 3)Violette,Ann Chim [4] **23,**306(1871) 4)Daniel(1902), 786-7 5)Ullmann **6**(1955),794 6)CondChem Dict(1956),992

ACETIC ANHYDRIDE AND DERIVATIVES

Acetic Anhydride, Ethanoic Anhydride or Acetyl Oxide (abbreviated to Ac_2O), $(CH_3CO)_2O$, mw 102.09, OB to CO_2 −125.4, col liq, fr p −73.1°, bp 139.5°, d 1.0838 at 20°/20°, n_D^{20} 1.3904, sp ht 0.434 cal/g/°C, Q vapzn 66.1 cal/g, fl p 124°F (closed cup), Q_c 431.9 and Qf 130.8 kcal/mol (Ref 2); sol in cold w (12% at 20°), decompd by hot w, sol in alc & eth. Various methods of prepn are given in Refs 1,5 & 18. It has been used in the acetylation of cellulose and other substs,in the manuf of RDX and it can be used, together with HNO_3 and AcOH, in the prepn of high nitrogen content (14%) NC (Refs 3,7 & 13). The physico-chem props of the system HNO_3–AcOH–Ac_2O have been studied(Refs 4,7,11,14, & 15). US Spec JAN-A-459 lists the requirements for Ac_2O used in the manuf of RDX and of some other nitramines. Toxicity, toxicology and fire & expln hazards of Ac_2O are discussed in Sax (Ref 17)

The concn of Ac_2O in air in the range 2.67 to 10.13% is expl. Ac_2O reacts vigorously in contact with w, steam and oxidizing substs. It is particularly dangerous in contact with chromic acid, Na_2O_2 and HNO_3(Refs 5, 16 & 17). An expln of a tank, contg 150 gals perchloric acid ($HClO_4$) and 50-60 gals Ac_2O in an electroplating plant, killing 16 persons and wrecking 116 bldgs,was reported by Kuney (Ref 6). Shock sensitivity and expl response to heat and flame of $HClO_4$–Ac_2O–water mixts have been investigated by Médard et al (Ref 8). The mixt contg 68/32 by vol of $HClO_4/Ac_2O$ is most expl. A mixt contg less than 55% by vol of $HClO_4$ is incapable of

deton or one with $HClO_4$ of d less than 1.50 g/cc has no expl props (Ref 9)

The safe use of Ac_2O–$HClO_4$ as electrolytic polishing bath has been investigated by Hikita & Asaba (Ref 12) and by Médard & Sartorius (Ref 10) resulting in a 3 phase diagram to show the dangerous zones

Refs: 1)Beil **2,** 166,(75) & [170] 2)J. Thomas, ZPhysChem **52,**347(1905) 3)G. Petitpas, MSCE **30,** 245(1943) 4)R.Vandoni & R. Viala, MSCE **32,**80-6 (1945) 5)Kirk & Othmer **1**(1947), 78-87 6)J.Kuney, C & EN **25,**1658-9(1947) & CA **41,**5309(1947) 7)J.Chédin et al,MSCE **35,**fasc 2,53-62(1949) 8)L.Médard et al, RevMet **46,**549-60(1949) & CA **44,**330(1950), 9)P.A.Jacquet,Metal Finishing **47,** No. 11,62-9(1949) & CA **44,** 459(1950) 10)L.Médard & R. Sartorius, MP **32,**179-96(1950) & CA **47,** 9013(1953) 11)J.Chédin & A. Tribot,MSCE **36,**fasc1, 31-6(1951) 12)T.Hikata & T.Asaba, JChem SocJapan,IndChemSect **54,**253-5(1951) & CA **47,**2987(1953) 13)W.G.Harland,JTextile Inst **45,**T678-91(1954) & CA **49,**607-8(1955) 14)T.V.Mal'kova, ZhObshchKhim **24,**1157-64(1954) & CA **49,**2167(1955) 15)T.V. Mal'kova,Referat ZhKhim **1954,** No. 33882 & CA **49,**9373(1955) 16)Ullmann **6**(1955) 804-15(under Essigsäure) 17)Sax(1957) 230-1 18)Faith,Keyes & Clark(1957)21-6

Acetic Anhydride, Analytical Procedures. Material intended for use in the USA for the manuf of expls(such as cyclonite) shall comply with the requirement of Spec JAN-A-459, as determined by the following tests:

a)Color. Prepare 500 ppm of platinum standard by dissolving 1.245 g K_2PtCl_6 and 1.000 g $CoCl_2 \cdot 6H_2O$ in aq HCl(1:5) contained in 1 l volumetric flask and dilute to the mark. Pipette 1 ml of this soln into a 50 ml Nessler tube and dilute with w to the mark. Fill a 2nd Nessler tube with the sample and compare the color of solns in both tubes. The intensity of color of the sample shall be no greater than that of the standard

Save the sample for procedure (b)

b) *Suspended Matter*–none. Observe the sample in the 2nd Nessler tube of proc (a)– there shall be no suspended matter

c) *Acetic Anhydride Content (Purity)*–minim 97.07%. Have two dry glass-stoppered Erlenmeyer flasks, the first 500 ml and the 2nd 250 ml. Pipette 50 ml of approx 0.5N carbonate-free NaOH into first flask and 20 ml of freshly distilled aniline into 2nd flask. Stopper both flasks. By means of Lunge weighing bottle weigh to ±0.0002 g, 0.9 to 1.0g sample into 1st flask and 1.6 to 1.7 g into the 2nd one. The samples shall be added dropwise and the flasks swirled to prevent local overheating. Stopper each flask immediately and, after 3 to 5 mins of standing,wash down the contents of 1st flask with 100 ml w (neutral to phenolphthalein) and titrate with N/2 HCl to phpht end point. Cool 2nd flask, wash down the contents with 100 ml of aq methanol (75:25) (neutral to phpht) and titrate with N/2 carbonate-free NaOH to a phpht end point. Titrate a blank soln consisting of 100 ml of aq methanol and 20 ml aniline using 0.1N carbonate-free NaOH

$$\%Ac_2O = 10.21\left(\frac{V_1N_1 - V_2N_2}{W_1} - \frac{V_3N_1 - V_4N_4}{W_2}\right),$$

where V_1 = ml NaOH added to 1st flask; V_2 = ml HCl used in back titration of contents in the 1st flask; V_3 = ml NaOH used in titrating 2nd flask; V_4 = ml NaOH used in blank; N_1 = normality of NaOH added to 1st flask and used in titrating contents of 2nd flask; N_2 = normality of HCl used in back titration of 1st flask; N_4 = normality of NaOH used in blank; W_1 = g of sample in 1st flask and W_2 = g of sample in 2nd flask

d)*Chlorides*–none, Dissolve 2 ml sample in 20 ml chloride-free w, add 1 ml concd nitric acid,shake and add a few drops of 1N Ag nitrate soln. No turbidity or opalescence shall be observed

e)*Sulfates*–none. Dissolve 2 ml sample in

20 ml sulfate-free w, add 1 ml concd HCl, shake and add a few drops of 10% Ba chloride soln. No turbidity shall be observed

f) *Heavy Metals*–none. Dissolve 10 ml sample in 100 ml w and 5 ml concd HCl. Saturate the soln with hydrogen sulfide gas and note the appearance of a ppt, which is indicative of the presence of Sb, As, Bi, Cd, Cu, Pb, Hg or Sn. Dissolve another 10 ml sample in 100 ml w, neutralize with NH_4OH (using litmus paper indicator) and add 5 ml in excess. Cool the soln, saturate with H_2S gas and note the appearance of a ppt, which is indicative of the presence of Co, Mn, Ni or Zn

Acetic anhydride intended for use as a reagent shall comply with the requirements listed in Ref 3

Analysis of acetic anhydride is also discussed in Refs 1, 3 & 4

Refs: 1)Kirk & Othmer **1** (1947), 86-7 2) Jacobs (1949),652-4 3)Reagent Chemicals (1950), 22-3 4)Organic Analysis,Interscience **3**(1956)

Acetic Anhydride, Azido Derivative (Azidoacetic Anhydride or Triazoacetic Anhydride, $H_3C.OC,O.CO.CH_2,N_3$–not found in Beil or CA through 1956

Acetic Anhydride, Diazido Derivative or Diazidoacetic Anhydride, $N_3.H_2C.OC.O.CO.CH_2.N_3$, mw 184.12, N45.65%. Col oil, bp 110° at 0.2 mm. Indexed in CA as Triazoacetic Acid Anhydride. Prepd by shaking an abs ether suspension of the Ag salt of azidoacetic acid with a soln of azidoacetyl chloride in abs ether

Refs: 1)Beil–not found 2)K.Freudenberg et al, Ber **65**, 1191(1932)

Acetic Anhydride–Perchloric Acid Mixtures. See under Perchloric Acid

Acetic Ether (Essig-äther, in Ger). See Ethyl Acetate

ACETINS AND DERIVATIVES

Acetins are esters obtained by reactions of glycerin with acetic acid. There are mono-, di- and tri-acetins

Monacetin or Glyceryl Monoacetate, $C_3H_5(OH)_2(O_2C.CH_3)$, mw 134.13, OB to CO_2 −71.6%, Mixt of two isomers is a col liq, d 1.206, at 20°/4°, bp 130° at 3 mm, N_D^{20} 1.4499 Q_c^v 595.6 kcal and Q_f^v 214.5(Ref 6); very sol in w or alc; sol in chlf; sl sol in eth or petr eth and insol benz. Can be prepd by heating at 170°, equimolar quantities of anhyd glycerol and glacial acetic acids in the presence of H_3PO_4 or P_2O_5 (Refs 1 & 3). Vender (Ref 2) proposed using water sol acetins as gelatinizing agents for NC in the prepn of smokeless propellents. Acetins were also used in manuf of some dynamites (see also Refs 4 & 5)

Refs: 1)Beil **2**, 146,(69) & [159] 2)V. Vender, Ger P 226,422(1906) & CA **5**, 1517 (1911) 3)H.A.Schuette & P.P.T.Sah, JACS **48**, 3162-26(1926) 4)Kirk & Othmer **7**(1951) 5)226-7 6)P.Tavernier & M. Lamouroux, MP **38**,84(1956)

Diacetin or Glyceryl Diacetate, $C_3H_5(OH)(O_2C.CH_3)_2$, mw 176.17, OB to CO_2 −136.2% OB to CO −72.7%. Mixt of two isomers is a col liq, d 1.177 at 20/20°, bp 175-6° at 40 mm, $n_D^{20°}$ 1.371, Q_c^v 801.8 kcal/mol and Q_f^v 264.8 (Ref 6); sol in w or alc; al sol in eth; very sol in benz. Can be prepd in mixt with other compds by heating at 110°, anhyd glycerol with glacial acetic acid and concd sulfuric acid (Refs 1 & 3). Vender (Ref 2), proposed its use as gelatinizer for NC. According to Refs 4 & 5, diacetin has been used as a solvent, plasticizer and softening agent

Refs: 1)Beil **2**,147,(69) & [160] 2)V. Vender, GerP 226,422(1906) & CA **5**,1517 (1911) 3)H.A.Schuette & P.P.T.Sah, JACS **48**,3161(1926) 4)Kirk & Othmer **7**(1951), 227 5)Durrans(1957),186-7 6)P.Tavernier & M.Lamouroux, MP **38**,84(1956)

Triacetin (TA) or Glyceryl Triacetate, $C_3H_5(O_2C.CH_3)_3$, mw 218.20, OB to CO_2, −139.3%, OB to CO −73.3%. Col liq, fr p ca −78°, bp 258-60°, d 1.163 at 20/20°, $N_D^{20°}$

1.4307, Q_c^v 1008.6 kcal/mol and Q_f^v 314.5 (Ref 6); sl sol in w, miscible with alc, eth, chlf or benz; nearly insol in C bisulfide or ligroin. It is hygroscopic. Can be prepd by treating glycerol with acetic anhydride at 100° (Ref 2) or by other methods listed in Ref 1. Its toxicity and fire & expln hazards are discussed in Ref 7

Triacetin has been used as a solvent for acetocellulose and as a plasticizer in the pyroxylin industry (Ref 5). It was patented by Hale & Cameron (Ref 3) as a flash reducing agent in NC propellants. According to Ref 4, triacetin acts as a gelatinizing and waterproofing agent. It has been used successfully as plasticizer and coolant in single-base propellants and in cast double-base propellants (see Note)

Notes: a) Composition of one of the single-base propellants contg TA is given in Kirk & Othmer **6**,(1951), 83, as follows: NC(12.6%N) 79.0, TNT 15.0, TA 5.0, DPhA 1.0 and graphite (added) 0.2%. Compositions of double-base cast propellants OGK and OIO are given in PicArsn Purchase Description PA-PD-182(1952), which is classified confidential b)Burning characteristics of liq mixt triacetin-metriol trinitrate was studied by D.L.Hildenbrand et al, JPhChem **58**, 1130-3(1954) & CA **49**,2805(1955) c)Burning characteristics of liq mixt triacetin with 2,2-dimethylol-1-propanol trinitrate, also called 2,2-bis(hydroxymethyl)-1-propanol trinitrate was studied by R. Steinberger & K.E.Carder, JPhChem **59**,255-7(1955) & CA **49**,7935(1955)

Refs: 1)Beil **2**,147,(70) & [160] 2)F. Baeyer,GerP 347,897(1919) & JSCI **41**,347A (1922) 3)G.C.Hale & D.R.Cameron, USP 2,035,471(1936) & CA **30**,3650(1936) 4) All & EnExpls(1946), 42 5)Kirk & Othmer **7**(1951),227 5a)Merck(1952),967 6)P. Tavernier & M.Lamouroux, MP **38**, 84(1956) 7)Sax 1957),739-40 8)Durrans(1957),187

Acetins, Analytical Procedures. Analyses of mixts of mono- with diacetin and of mono- with triacetin are described in Ref 1. An infrared method for detg triacetin is described in Refs 2 & 3

Triacetin intended for use in the manuf of US smokeless propellants shall comply with the requirements of Spec JAN-T-301, as detd by the following tests:

a) *Color* Prepare the standard by adding 0.5 ml of 0.1N iodine soln & 100 ml w and visually compare the color in 25 ml Nessler tube with that of sample. The color of sample shall be no darker than that of standard

b) *Specific Gravity*—1.153 ±0.003 at 25/4°, as detd by pycnometer or Westphal balance

c) *Acidity as AcOH*—max 0.005%. Neutralize about 400 ml of 95% ethanol, with 0.1N NaOH soln to faint pink coloration, using 1 to 2 drops phpht indicator, and transfer about half to a 500 ml Erlenmeyer flask contg 100 ml sample. Mix thoroughly and titrate rapidly with 0.1N NaOH to faint pink coloration. Disregard gradual fading of this color

$$\%AcOH = \frac{6.0 \times V \times N}{W},$$

where V=ml of NaOH soln required for titration; N=normality of NaOH soln; W=g of sample (vol × gravity)

d) *Ash*—max 0.002%. Weigh a portion of ca 10 g in an accurately tared small porcelain and evap to near dryness over a low flame or on a hot plate. Ignite the residue to const wt at a red heat, cool in a desiccator and weigh

e) *Ester Content as TA*—min 98.0%. Take two 250 ml flasks which can be fitted by means of ground joints to reflux condensers. Accurately, weigh by means of a Lunge pipette, 1.8 ±0.2g sample into the 1st flask, add 100 ml of N/3 NaOH soln and connect to the 1st reflux condenser. Add to the 2nd flask (blank) only 100 ml N/3 NaOH soln and connect to the 2nd reflux condenser. Boil gently each flask for ca 1 hr with occasional swirling (saponification takes place in the

1st flask). Wash down the sides of each condenser and the ground joints, with about 25 ml w, cool the flasks rapidly to RT and titrate their contents with N/3 sulfuric acid

$$\% \text{ TA} = \frac{7.27 \times (V - v) \times N}{W}$$

where V=ml of acid used to titrate blank; v= ml of sulfuric acid used to titrate excess of NaOH after saponification and N=normality of sulfuric acid

Refs: 1)P. Fuchs, ZAnalChem **121**, 305 (1941) & CA **35**, 6205(1941) 2)A.H.Castelli et al, PATR 2021(1954)(C) 3)A.H.Castelli et al, PATR 2222(1956)(C)

Note: Above classified references 2 and 3 were not used in this description of analytical procedures

Mononitroacetin or Glycerylmonoacetate Nitrate, $C_3H_5(OH)(ONO_2)(O_2C{\cdot}CH_3)$—not found in Beil

Dinitroacetin, Glycerylmonacetate Dinitrate or Glyceroldinitroacetate, called in Beil Glycerin-acetat-dinitrat, $C_3H_5(ONO_2)_2(O_2C{\cdot}CH_3)$, mw 224.13, N 12.50% OB to CO_2 -42.8% Lt yel oil d 1.45 at 15° (Ref 2) d 1.42 at 15° (Ref 3) fr p < −20°, bp 147° at 15 mm (dec); insol in w, benz & CS_2; easily sol in alcs, acet, ether & NG. Can be prepd by nitrating monoacetin with mixed nitric-sulfuric acid or by acetylation of dinitroglycerin. It easily gelatinizes NC at RT, is insensitive to impact but it can be initiated by detonator. Power by Trauzl test 450 cc (Ref 2) and 202 cc(Ref 3)

Note: According to Naoúm(Ref 3) the sample of Vender(Ref 2) probably contained NG

Loss of wt in 24 hrs at 75° was 1.4% (Ref 3)

It was proposed as an antifreeze addition to NG in dynamites (Ref 2)

Refs: 1) Beil **2**, 148 2)V.Vender, SS **2**, 21(1907) 3)Naoúm, NG(1928), 193-7

Mononitrodiacetin, Glyceryldiacetate Nitrate or Diacetin Nitrate, called in Beil, Glycerin-α,β-di-acetat-γ-nitrate, $C_3H_5(ONO_2)(O_2C{\cdot}CH_3)_2$, mw 245.19, N 5.71%. Crysts, mp 18-20°, insol in w. Was prepd by acetylating glycerine-α-mononitrate. It is not mentioned as an expl

Refs: 1)Beil **2**, 148 2)W.Will, Ber **41**, 1120(1908)

Acetoacetyldiphenylamine. See N,N-Diphenylacetamide

Acetocelluloses. See Acetylcellulose

2-(4)-Aceto-1,9-diacetoxy-2,6,8-trinitro-2,4,6,8-tetrazanonane. See 2-(4)-Acetyl-1,9-diacetoxy-2-6,8-trinitro-2,4,6,8-tetrazanonane, under Acetyldiacetoxytetrazanonane

1-Aceto-3,7-dinitro-5-nitroso-1,3,5,7-tetrazacycloöctane. See under Acetotetraza-cycloöctane and Derivatives

1-Aceto-3,5-dinitro-1,3,5-triazacyclohexane. See under Acetotriazacyclohexane and Derivatives

Acetol (1-Hydroxy-2-propanone, Propanolon, Acetylcarbinol or Pyruvic Alcohol) (α-Oxy-β-oxopropan, Oxy-aceton, Acetonylalkohol or Brenztraubenalkohol in Ger), $CH_3CO{\cdot}CH_2OH$, mw 74.08, OB to CO_2 −151.2%, OB to CO −86.4%. Col, pleasant smelling liq, bp 145-7°, d 1.024 at 20°/20°(when freshly distilled), $n_D^{20°}$ 1.4295. Miscible with w, alc & eth. Industrial methods of prepn are given in Refs 1,2 & 3 and the lab prepn is in Ref 4. It is used as a reducing agent in org chem(Ref 5) and as a solv for NC(Ref 6)

Refs: 1)Beil **1**, 821,(418) & [866] 2)E. Holmes, BrP 428,462(1935) & CA 29, 6908 (1935) 3)R.W.McNamee, USP 2,143,383 (1939) & CA **33**, 2914(1939) 4)OrgSynth, CollVol 2(1943), 5-6 5)Hackh(1944), 7 6)CondChemDict(1956),8

ACETONE AND DERIVATIVES

Acetone or Dimethylketone (abbreviated to acet)(2-Propanone, Methylacetyl or Pyroacetic Ether), $CH_3{\cdot}CO{\cdot}CH_3$, mw 58.08, OB to CO_2 −220.4%, OB to CO −137.7%. Col, mobile, flamm liq, frp ca −94.6°, bp 56.5°, d 0.7898 at 20°/4°, $n_D^{20°}$ 1.3591, vap pres 180.3 mm Hg at 20°, sp heat 0.5176 cal/g at 20°, visc 0.00337 cgs units at 15°, Q_c^v 435.3 (Ref 17)

and Q_f 63 kcal/mol.Miscible in all propns with w, alc, eth, methanol, esters and other org solvs. It is a good solvt for NC(Refs 11, 12,16 & 18), cellulose acetate, nitrocompds etc. First obtained in 1595 by Libavius by the dry distn of sugar of lead and in 1805 by Trommsdorff, who distilled Na and K acetates. The correct compn was estbd in 1832 by Liebig & Dumas(Ref 17, p 881)

There are many methods for the prepn of acet of which the dry distn of Ca acetate was the most common until, a few years after WWI, the carbohydrate fermentation method of C.Weizmann was introduced (Refs 31 & 48). By far the largest prodn of acet in the USA is from petroleum-derived propylene by way of isopropanol (Refs 31 & 48). Another method is to pass acetylene and steam over Fe_2O_3-ZnO catalyst at elevated temps(Ref 48). In Germany acet was produced in 95% yield from AcOH by a vapor-phase catalytic process using a cerium oxide catalyst at 400°(Ref 31). There are also other methods of prepn(Refs 1,2,3,4,10,14,31,33,42,47, & 48)

Acetone is very flammable and should not be exposed to heat or flame. Mixts of acet vapor with air are expl if the acet content is 2.55 to 12.8% at RT. The ignition temp of acet vapor in air at 0° is 567° and in oxygen 485° (Ref 20). The expl props and hazards of acet air mixts and precautions against their propagation to expln are discussed in Refs 5,6,7,8,9,19,27 & 28. The exptl detmn of weak shocks in liqs, such as acet, ethanol and ether is discussed in Ref 45. Brooke (Ref 40) detmnd the flash points of acet-water mixts and has shown that they are ignitable even when the acet content is as low as 2%. The fl p of a 2% acet-water mixt is 44.4° and of an 18% soln is 7.1°. The fl p decreases rapidly with an increase in the acet content. In pouring acet down the drain, it is advisible to add enough water to make the acet concn less than 2%

The toxicity and toxicology of acet are discussed in Refs 17,28,31,42 & 46. In general there seems to be no indication that acet produces any toxic effects in workers who use it in well ventilated buildings

Osmotic press data for solns of NC in acet have been obtained by Huggins(Ref 21), vapor tensions of gels by Schultz(Ref 15) and by Calvet(Ref 22), sedimentation rates by Moisimann et al (Refs 24 & 25), viscosity vs NC concn by Wissler (Ref 36), thermodynamic props by Münster (Ref 41), and various other props of NC-acet solns are discussed in Refs 13,23,26,29,32,34,35,38,39 & 44. The absorption spectra of acet are recorded by Pauling (Ref 30) and the reaction with 1,3,5-TNB, in the presence of an alkali, to give a black solid complex has been noted by Kimura (Ref 43)

The principal use of acet in the expl industry is as a solv such as, in the prepn of pentolite from PETN and TNT, in the purification of RDX and other expls, and as a gelatinizer for NC in the prepn of some propellants (eg cordite) (See spec, Ref 49, for Ordn use of acet). Acet is also used extensively in labs as a solv and for the washing of glassware

Refs on Acetone: 1)Beil **1**, 635(335) & [692] 2)V.Meyer & K. Locher, Ber(1) **8**, 216 (1875) 3)M. Berthelot & M.Delépine, CR **130**, 1045 & **131**, 745(1900) 4)Barnett (1919), 85, 87 & 88 5)A.L.Brown, QuartNat FireProtAssoc **21**, 47-54(1927) & CA **22**, 166 (1928) 6)F.Ritter et al, Jahresber CTR **8**, 201-2(1930) & CA **26**, 4474(1932) 7) E.Berl & K.Barth, ZElektrochem **39**, 73-5 (1935) & CA **27**, 2036(1933) 8)G.W.Jones et al, Bur Mines Tech Paper **544**, 26 pp(1933) & CA **27**, 2812(1933) 9)G.W.Jones et al, Bur MinesTechPeper **553**, 24 pp (1933) & CA **27**, 4401(1933) 10)M.Mathieu, CR **199**, 55-7(1934) & CA **28**, 5659(1934) 11)J. Desmaroux & M. Mathieu, MP **26**, 180-203 (1934-5) 12)J.Desmaroux, CR **199**, 148-50 (1934) & CA **28**, 5659(1934) 13)T.H. Durrans & D.G.Davidson, Chem & Ind **1936**, 162-9 & CA **30**, 3116(1936) 14)J.N.Crahay, Ingénieur Chimiste **20**, 13-22(1936) & CA **30**,

7539(1936) 15)G.V.Schulz, Naturiwissenschaften **25**, 346-7 (1937) & CA **31**, 8917 (1937) 16)Y.Inoue et al, Cellulose Ind (Tokyo) **16**, 37-48(1940) & CA **35**, 2715(1941) 17)G.B.Mills & H.Hunt, JPhChem **45**, 1358-9 (1941) 18)E.Calvet, CR **213**, 126-8(1941) & CA **36**, 6073(1942) 19)J.D.Müller-Hillebrand, AutogeneMetallarbeit **34**, 83-90 (1941) & CA **40**, 7631(1946) 20)W.J.Huff, BurMines RI **3669**(1942) & CA **36**, 4337(1942) 21)M.L.Huggins, JACS **64**, 1713(1942) & CA **36**, 5407(1942) 22)E.Calvet, CR **214**, 767-8(1942) & CA **38**, 2546(1944) 23)E. Calvet, AnnFaculté Sci, Marseille **16**, 17-35 (1942) & CA **41**, 2303(1947) 24)H. Moisimann, Helv **26**, 61-75(1943) & CA **37**, 6522(1943) 25)H.Moisimann & R.Signer, Helv **27**, 1123-7(1944) & CA **39**, 3476-7(1945) 26)A.Münster,KollZts **105**, 1-9(1943) & Z Naturforsch 1, 311-20(1946); CA **38**,4494 (1944) & **41**, 4996(1947) 27)M.J.Huff, Bur Mines RI **3745**, 49 pp(1944) & CA **38**, 5407(1944) 28)A.R.Smith & M.R.Mayers, IndBull(NY State Dept of Labor) **23**, 174-6 (1944) & CA **38**, 5613(1944) 29)S.N. Danilov & M.E.Dyn'kin, ZhurObshKhim **15**, 550-64(1945) & CA **40**, 4582-3(1946) 30) L.Pauling, OSRD Rept No 5953(1945) 31)Kirk & Othmer **1**(1947), 88-93 32)J. Chédin & R. Vandoni, MSCE **33**, 205-18(1947) & CA **43**, 4927-8(1949) 33)Giua, Dizionario **1**(1948), 28-33 34)D.Fensom & J.H.Greenblatt, Can JRes **26B**, 215-25(1948) & CA **42**, 4818(1948) 35)S.A.Glickman & L.A.Root, DoklAkadN **65**, 701-4(1949) & CA **43**, 6044-5 (1949) 36)A.Wissler, MakrChem **3**, 5-12 (1949) & CA **43**, 6885(1949) 37)H.Campbell & P.Johnson, JPolymerSci **4**, 427-63(1949) & CA **43**, 8244(1949) 38)S.M.Lipatov & S.I. Méerson, KollZh **12**, 122-30(1950) & CA **44**, 5693(1950) 39)S.Newman, JPhColl Chem **54**, 964-6(1950) & CA **44**, 8740(1950) 40) M.Brooke, ChemAnal **40**, 92(1951) & CA **46**, 9311(1952) 41)A.Münster, JPolymerSci **8**, 633-49(1952) & CA **46**, 7845(1952); ZElectrochem **56**, 899-903(1952) & CA **49**, 7928 (1955) 42)Ullmann **3**(1953)36-41 43) M.Kimura, JPharmSoc(Japan) **73**, 1216-23 (1953) & CA **48**, 12699(1954) 44)P.C. Scherer & N.J.Crookston, JPolymer Sci **14**, 129-34(1954) & CA **48**, 14187(1954) 45) W.M.Flock & D.F.Hornig, JChemPhys **23**, 816-21(1955) & CA **49**,11346-7(1955) 46) Sax(1957), 232-3 47)Durrans(1957) 67 & 112-21 48)Faith,Keyes & Clark(1957), 27-33 49)US Spec JAN-A-465

ACETONE, ANALYTICAL PROCEDURES

Detection of Acetone. The presence of acetone may be detected by the *iodoform test,* which depends on the fact that when acet is treated with iodine and Na hydroxide, iodoform and Na acetate are formed:

$$CH_3{\cdot}CO{\cdot}CH_3 + 3I_2 + 4NaOH = CHI_3 + CH_3COONa + 3NaI + 3H_2O$$

The presence of iodoform is detected by its characteristic odor or by formation of crysts. Other ketones, as well as aldehydes and alcohol interfere and should be detd separately. Description of qualitative test is given in Ref 16, p 619

Other qualitative tests for acetone include: a)*Dénigès' mercuric sulfate test,* described in Refs 2 & 19 b)*Dinitrophenylhydrazine test,* described in Refs 10 & 19 and c) The *Faught sodium nitroprusside test,* described in Ref 16, p 685 (see also Ref 12)

Quantitative Determinations of Acetone. The oldest and still widely used method is the *"Messinger test"*, based on the iodoform reaction described above. This test originally described in 1888 (Ref 1) was also described in detail by Goodwin (Ref 5), Friedmann (Ref 6) and Bonner(Ref 13). Jacobs (Ref 16, pp 685-6) describes the test and its modifications and gives some additional refs. The modification of Baer(Ref 17) described below under "Determination of Acetone in Smokeless Propellants Also Containing Alcohol" was successfully used at PicArsn

The quantitative test, known as the *hydroxylamine hydrochloride test* (Refs 8 & 16) or as the *oximation method* (Refs 15 & 20) is based on the following equation:

$(CH_3)_2\ CO + NH_2OH \cdot HCl \longrightarrow (CH_3)_2C{:}NOH + HCl + H_2O$

In aq soln this reaction goes (in cold) to 95% completion and the same applies to some other lower ketones. For higher ketones it is necessary to heat the mixt to complete the reaction and this might decomp the hydroxylamine hydrochloride

Procedure:

a) Weigh a sample contg acetone into 250 ml vol flask and dil with w to the mark(concn of acet after diln shall be ca 1% b)Take a 10 ml aliquot in a 125 ml Erlenmeyer flask, add few drops of bromphenol-blue indicator (0.1% soln in 30% alc) and neutralize any alkalinity with 0.1N HCl soln c)Weigh out approx 0.5g cp hydroxylamine hydrochloride, dissolve it in 5 ml w, add a few drops of bromophenol indicator and neutralize any free acidity with 0.1N NaOH soln d)Add the hydroxylamine hydrochloride soln to the above acetone soln in 125 ml flask, stopper it and allow to stand for 45 mins e)Titrate the liberated HCl(see the above equation) with 0.1N NaOH and calc the amt of acet, as follows:

$$\%\ \text{Acetone} = \frac{A \times N \times 0.05808 \times 100}{W \times 0.95}$$

Where: A=ml of approx 0.1N NaOH used to neutralize the aliquot; N=normality of NaOH soln and W=total wt of sample

Quantitative tests other than Messinger and oximation methods are described in Refs 7,10 & 11. Detns of acet in propellants are described in Refs 3,4,6,7,13 & 17. The proc of Ref 17 is given below under Determination of Acetone in Smokeless Propellants Containing also Alcohol

Specification Requirements for Acetone Used in Ordnance. Acetone intended for use as a solvent in prepn of propellants and pentolite shall comply with the requirements of US Spec JAN–A–489 and shall be tested according to the procedures outlined below:

a) *Appearance.* Acetone shall be colorless, transparent and show no turbidity when mixed with distilled water in any proportion. This test is conducted in a test tube or cylinder

b)*Distillation Range.* Acetone shall distil completely(reach the dry point) within a range of 1°C. The temp 56.1° shall be included within the distillation range at 760 mm Hg. The method in sect 100.1 of Federal Spec VV–L–791 shall be used

c)*Nonvolatile Matter* – max 0.002 per 100 ml sample, is detnd by evapg a 100 ml sample in a platinum dish on a steam bath, followed by drying the dish to const wt at 105–110°

d)*Permanganate Reduction.* The pink color shall persist for at least 30 mins when 0.5 ml of 0.1N K permanganate soln is added to a 100 ml graduate contg the sample. The graduate is stoppered and the soln is thoroughly mixed before it is allowed to stand at 15°

Note: According to Mr G.D.Clift, the following precautions shall be observed during this test: a)Only glass stoppers shall be used for the sample bottles and the graduate b)The graduate shall be rinsed with HCl, distilled w and a portion of sample just before the test c)Only freshly prepd soln of K permanganate shall be used as the lower oxides present in old solns catalyze the reaction, and discolor the permanganate d) Sample shall not be placed in the sunlight and e)There shall be no oxides of nitrogen or sulfur dioxide present

e)*Specific Gravity* shall be 0 7915 to 0.7935 at 20/20° when detd by a Westphal balance, chainomatic balance, sp gr bottle or pycnometer

f) *Alkalinity as NaOH* – max 0.001%, when detd by titrating a 50 ml sample dissolved in 100 ml water with N/10 standard acid, using methyl red as an indicator

g)*Acidity as AcOH* – max 0.001%, when detd by titrating with N/10 NaOH soln a 300 ml sample(previously reduced by vigorously boiling to 100 ml) dissolved in 300 ml distilled water neutral to phpht

h)*Aldehydes* – max trace, when detd by shaking a mixt of 10 ml acetone and 10 ml w

with 1 cc of the test solution(see below), allowing to stand for 1 hr and filtering the mixt. The filtrate tested with 1 ml of a 10% NaCl soln shall not give more than a trace of turbidity

Note 1: The "test solution" is prepd by mixing 1 ml of NaOH soln(9 g in 100 ml w) with 1 ml of Ag nitrate soln(9 g in 100 ml w), followed by adding dropwise enough Amm hydroxide(d 0.90) to just dissolve the ppt completely

Note 2: Acetone used in manuf of pentolite may contain, according to G.D.Clift, the following amts of impurities: CO_2 –up to 0.05%, mesityl oxide 100,000 ppm, formaldehyde 18500 ppm and tar(such as produced by the action of alkalies on TNT) 1,000 ppm

Acetone Intended for Use as a Reagent shall comply with the requirements and undergo the tests described in Reagent Chemicals (1950),24–5(Ref 18)

Determination of Acetone in Smokeless Propellants Containing Also Alcohol. This method developed by M.Baer of PicArsn is described in Ref 17. Propellant used for this detn contnd NC(13.2%N) 54.6 NG 35.5, Et Centr 0.9,carbon black 1.2, K chlorate(contg 0.5% Mg stearate & 0.5% Mg oxide) 7.6 and volatiles 0.2%. After investigating several existing methods of detn of acetone and finding most of them unsuitable for analysis of the above propellant, Baer decided that the most suitable would be Messinger's method(see Refs 1 & 5), and for alcohol – quantitative oxidation to acetic acid with an excess of $K_2Cr_2O_7$ in H_2SO_4 and titrating the iodine liberated by the excess of dichromate when it is allowed to react with KI

Following is the procedure:

a) Assemble the digestion apparatus, which shall consist of two condensers vertically placed and connected at their tops by an inverted U tube. To the lower end of the 1st condenser is attached a 1500 ml flat bottom flask and the tip of the lower end of 2nd condenser is extended to the bottom of a 250 ml flat bottom flask. All connections shall consist of std ground glass joints b)Cut the sample into ca ⅜" in diam and ½" in length as rapidly as possible, weigh in a stoppered bottle(5 g in case of finished propellant and 25 g in case of green one) and transfer to the 1500 ml flask of the digestion apparatus. Add 500 ml w, 100 ml of 30% NaOH soln, several pieces of porcelain to prevent bumping and a small piece of paraffin to prevent frothing c)To the 250 ml flask of the digestion apparatus(which is marked to indicate vol of 150 ml), add enough w to cover the bottom with a ¼" layer. Connect the digestion app. Immerse this flask in ice-water and let tap w run through the 2nd condenser, but keeping the 1st condenser just full but the w not running d)Heat the 1500 ml flask slowly so that 3–4 hrs will be required to collect 150 ml of distillate. At the end of this period propellant should be completely disintegrated,and if not continue the distillation e)Disconnect the 250 ml flask(receiver) from the 2nd condenser and add 10–15 g of anhyd Na sulfate. Continue to keep the distillate in the ice-bath for 15–20 mins longer and then filter by means of a No 41 Whatman(or equivalent) catching the filtrate in a 250 ml volumetric flask. Rinse the receiver flask and filter paper with two 25 ml portions of w catching the washings in the above vol flask f)Allow the flask and contents to come to RT and fill the flask to the 250 ml mark with w

Estimation of Acetone: g)Pipette 25 ml aliquot to a 250 ml iodine flask contg 50 ml of N/1 NaOH soln, stopper and allow to stand 5 mins h) Add from a burette about 25% excess of 0.1N iodine soln while continually and vigorously swirling the flask. Stopper the flask and allow to stand at least 10 mins (20 mins in cold weather)

Note: If the flask is not swirled vigorously while adding iodine soln and if iodine is not added in 25% excess, the reaction will not go to completion and much iodine remains uncombined. This will require 3 times as much thiosulfate on back titration of

iodine. The excess of iodine may be either calcd or detd by preliminary titration i)Neutralize the NaOH by adding 25 ml of 2N sulfuric acid and then 0.3–0.4 ml in excess.

Note: If a larger excess of acid is added, more thiosulfate will be required to titrate the excess of iodine than necessary. The exact amt of acid necessary for neutralization of caustic may be established by preliminary titration of 50 ml N/1 caustic with 2N acid in presence of phpht indicator
j) Add from a burette, while swirling the flask, 0.05N Na thiosulfate soln until the yel color just remains visible; then add some freshly prepd starch soln and continue titration to the appearance of a bluish color k)Run a blank using the same vol of iodine soln but no sample

$$\% \text{ Acetone} = \frac{(A-B) \times N \times 0.96747}{W}$$

where A = ml of Na thiosulfate soln used to titrate a blank, B = ml Na thiosulfate soln to titrate the sample, N = normality of thiosulfate soln and W = g of sample represented by aliquot portion

Note: Analysis of alcohol is included here because it can be run simultaneously with acetone, using the same distillate

Estimation of Alcohol: g¹)Pipette 25 ml aliquot of procedure (f) to a 250 ml flat bottom flask contg 25 ml w, cool the flask for 15 min in an ice-water bath, add ca 0.20 g of $K_2Cr_2O_7$ (accurately weighed) and 6 ml concd H_2SO_4 h¹)Attach to the flask (by means of a ground glass joint) a reflux condenser. Bring the contents to a boil in 10–15 mins and allow it to boil for 5 mins i¹)Disconnect the flask from the condenser and cool to RT. Dilute the contents to ca100 ml with w, and add 3–4 g KI j¹)Stopper immediately, agitate by swirling and, after allowing to stand 3 mins, titrate the liberated iodine with std 0.1N Na thiosulfate soln in the manner described in proc (j)

$$\% \text{ Alcohol} = \frac{[A - (0.049 \times B \times N)] \times 23.44}{W}$$

where A = g of $K_2Cr_2O_7$ used; B = ml of Na thiosulfate soln used to titrate the sample; N = normality of thiosulfate and W = g of sample represented by aliquot taken

Refs(Acetone, Analytical): 1)A.Messinger, Ber**21**,2366(1888) & JSCI**18**,138(1889) 2) G.Dénigès, JPharmChim**9**,7(1899) & Analyst **24**,92(1899) (Detn of small quantities of acet, such as in air, by treating the sample with a large excess of acidic mercuric sulfate and heating to 100°. An insol compd $(2HgSO_4 . 3HgO)_4 (C_3H_6O)_5$ is deposited. This method is also described in Ref 19) 3)A.Pieroni, AttiAccadLin **27** II,52–7(1918) JSCI **37**,749A(1918) & CA**13**,789(1919) 4) C.F.vanDuin et al, Rec **38**,163–9(1919) & CA **13**,2596–7(1919) 5)L.F.Goodwin, JACS **42**,39–45(1920)(Analysis of acet by the Messinger method was found to be accurate) 6)F.Friedmann,SS **16**,121–3(1921) & CA**16**, 343(1922)(Detn of acet in NG propellants conducted by passing a slow current of CO_2 or N_2 through a U tube filled with fine shavings of sample and immersed in a water bath at 75°. The acet of sample is volatilized and passed into burette filled with 23% KOH soln, where it is absorbed. The soln of KOH is diluted to 100 ml and a 20 ml aliquot is placed in iodine flask where acet is detd by the Messinger method) 7)M.Marqueyrol & P.Loriette,MP**19**,362(1922) & CA**17**,1717 (1923)(Detn of acet based on the addn to a soln contg sample K iodide and Na hypochlorite in the presence of alkali, until the 1st appearance of free iodine, which may be detected by starch-bicarbonate indicator) 8)M.Morasco,IEC**18**,701(1920)(The air contg acet is drawn through bubblers contg 0.2% hydroxylamine hydrochloride soln and the amt of acet is estimated by titrating HCl liberated through the formation of acetoxime, with std NaOH in the presence of methyl orange indicator)(see also Refs 15 & 16) 9)C.A.Adams & J.R.Nicholls, Analyst **54**,

5–9(1929)(Analysis of mixts contg acetone, ethanol & isopropanol) 10)H.A.Iddle & C.E. Jackson,AnalChem **6**,454–6(1934)(Acet, as well as other carbonyl compds, reacts quantitatively with 2,4–dinitrophenylhydrazine with formation of solid hydrazone. This procedure seems to be only approximate) 11)E.K.Nikitin,Zh PriklKhim **9**,1543–6(1936)(in Fr) & CA **31**,2126(1937)(Rapid detn of acetone in w conducted by mixing 1 ml of soln to test with 1 ml of 0.2% furfural and 1 ml KOH soln. Treat in a similar manner 1 ml w contg 0.05% and 1 ml w contg 0.025% serving as standards. Compare the rates of pptn of sample with those of standards and calc concn of acet from the formula given in paper. The accuracy of the method is ±5%) 12)Kirk & Othmer, **1**(1947),92(Analytical procedures and specs for acet) 13)T.G.Bonner, Analyst **72**,434–39 (1947) 14) W.B.Huckabay et al, AnalChem **19**, 838–41(1947)(21 refs)(Optimum conditions for titrimetric detn of traces of acet in liquefied gases) 15)R.Dalbert & J.Tranchant, MP**30**,343–51(1948)(Detn of acet & ethyl acetate in propellants)(36 refs)(After briefly describing and criticizing the principal methods of detg acet in propellants, such as those of Messinger,Marqueyrol & Loriette and Bonner, D & T give,on p 349,their modification of the oximation method described in Refs 8 & 16. D & T claim that their modification gives accurate results and that alcohol and ethyl acetate do not interfere) 16)Jacobs(1949), 619 & 685–7(Estimation of acetone by the iodoform method, by the Messinger method and by the Morasco method, called by Dalbert & Tranchant the *oximation* method)(See also Refs 1,5,8 & 15) 17)M.Baer,ChemLabRept 130,159; PicArsn,Dover,NJ(1950) 18)Reagent Chemicals(1950),24–5 19)A.Boulègue, MSCE **36**,257–8(1951)(Micro detn of acet in air by the method of Dénigès, described in Ref 2 and by the method dinitrophenylhydrazine) 20)J.P.Pillet,MP **36**,267–75(1954) (Anaylsis of mixts contg acet, ethyl acetate and alc employed for prepn of some Fr propellants and for recovery of some discarded propellants. In the method of Pillet, first the density of mixt is detd at 18°, then acetone by oximation(see Ref 15), ethyl acetate by saponification and alc by difference)

Acetoneallylozonide. See Allylacetoneozonide

Acetoneallylperoxide. See Allylacetoneperoxide

Acetone, Azido Derivative(Azidoacetone, Triazoacetone, Azidopropanone or Acetonylazoimide), $N_3 \cdot CH_2 \cdot CO \cdot CH_3$, mw 99.09, N42.41%. Col, very refractive oil bp 33–5° at 1 mm d 1.1132 at 25/4°, n_D^{25} 1.4515(Refs 1 & 2); bp 42–3° at 2 mm (Ref 4),bp 38° at 1 mm (Ref 5); sol in w. Detonates on heating and decomps in storage. Can be prepd by shaking chloroacetone with concd aq soln of Na azide and a small amt of AcOH

Refs: 1Beil **1**, 661 & [720] 2)M.Forster & H.Fierz, JCS **93**, 81(1908) 3)H.Lindemann & H.Tiele, Ber **61**, 1529(1928) & CA **22**, 3598(1928) 4)J.H.Boyer, JACS **73**, 5252(1951) & CA **47**, 490(1953) 5)J.H.Boyer & J.Hamer, JACS **77**, 953(1955) & CA **50**, 1827(1956)

Acetone, Diazido Derivative or 1,3-Diazido-2-propanone, $N_3 \cdot CH_2 \cdot CO \cdot CH_2 \cdot N_3$, mw 140.11, N59.99% – not found in Beil or CA through 1956

Acetoneazidoacetylhydrazide, called in Ger Acetyl-[azidoacet]-hydrazid or Isopropylidene-[azidoacetyl]-hydrazine $(CH_3)_2C{:}N \cdot NH \cdot CO \cdot CH_2 \cdot N_3$, mw 155.16, N45.14%. Wh ndls (from acet), mp 114°. Was prepd from azidoacetic anhydride and acetone. No info on expl props

Refs: 1)Beil **2**,(101) 2)Th.Curtius & A.Bockmühl,Ber **45**,1033(1912)

Acetone-[4-bromphenylhydrazone]-peroxide (Peroxyd des Aceton-p-bromophenylhydrazons, in Ger), $(CH_3)_2C(-O_2)-N{=}NH \cdot C_6H_4Br$, mw 259.16, N10.81%, OB to CO_2 –132.7%, OB to CO –77.1%. Yel,unstable prisms from ligroin, mp 45–47° with decomp; expl on heating. Sol in most org solvents. Can be prepd by passing air through cold, agitated acetone-[4-bromophenylhydrazone], suspended in ligroin(Ref 2)

Refs: 1)Beil **15**,435 & 117 2)P.C.Freer,Ber **30**,737(1897) 3)M.Busch & W.Dietz,Ber **47**, 3289–90(1914)

Acetone Compound, $C_{24}H_{22}N_{18}O_6$, (No structural formula given), mw 658.56, N38.29%, bright yel lfts. Was prepd by pouring acet into ice cold nitric acid (d 1.5), cooling and adding to the resulting oil an excess of ammonia. Its **trinitro derivative,** $C_{24}H_{19}N_{21}O_{12}$, (No structural formula given), mw 793.59, N 37.07%, yel ndls, mp 193–5°, was prepd by dissolving compd $C_{24}H_{22}N_{18}O_6$ in nitric acid (d 1.5) and pouring immediately into water. No expl props of either compd were mentioned

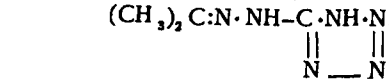
Refs: 1)Beil **1**,647 & 648 2)H.Apetz & C.Hell, Ber **27**,939 & 942(1894)

Acetone Compounds of Pentaerythritol are described by L.Orthner,Ber **61B**,116–18(1928). None of them is an explosive

Acetonediperoxide. See Acetoneperoxide, Dimeric

Acetoneditetrazyl Azide. See under Acetonyl azidotetrazoles

5-Acetonehydrazone-α(1H)-tetrazole, called **Acetone 5-tetrazolylhydrazone** by Benson, (Tetrazolon-iso-propyliden-hydrazon, in Ger),



$(CH_3)_2C{:}N\cdot NH{-}C\cdot NH\cdot N$, mw 140.15,
N 59.97%, OB to CO_2 –137.0%. Cryst, mp 181.5°. Sl sol in w; sol in acet, alc and Et acet. Was prepd from 5-hydrazinotetrazole hydrochloride and acetone in the presence of Na acetate. Its expl props were not examined

Refs: 1)Beil **26**,425 2)J.Thiele & H.Ingle, Ann **287**,237(1897) 3)F.R.Benson, ChemRevs **41**,8(1947)

Acetone Insoluble Test is one of the standard tests for detg the purity of expls and propellants. It is conducted by dissolving a weighed amt of sample(W) in measured vol of acet at RT and filtering the soln through a tared sintered glass crucible(W_1). After rinsing the residue with acet and drying the crucible in an oven to const wt, it is reweighed(W_2)

$$\% \text{ Acetone Insoluble} = \frac{(W_2 - W_1) \times 100}{W}$$

(See also under individual compounds)

Acetonemonotetrazylazide. See under Acetonyl-azidotetrazoles

Acetone, Nitration. Krauz & Stepanek(Ref 1) attempted to prepare tetranitromethane by nitration of acetone, but failed. Instead, they obtained (after treating the resulting product with a silver salt) a very expl solid claimed to be *Ag salt of "acetylmethylnitrolic acid"*, also called *α-nitro-α-isonitroso-acetone.* Hass & Hudgin(Ref 3) nitrated acet, using a vapor-phase nitration technique described in Ref 2. The high-boiling fractn from the nitration gave an odor of acetic acid, an acidic reaction in aq soln, a red color with ferric chloride and a yel salt with Ag nitrate soln, which was water sol and partially decompd on distil. Attempts at its further purificn and prepn of other derivs were unsuccessful, primarily due to instability of the compd. The paper(Ref 3) does not give the compn of high boiling fraction of nitration and does not state whether the substance was solid or liquid
(See also Nitroacetone, described below)

Refs: 1)C.Krauz & J.Stepanek,ChemObzor **10**,137–40(1935) & CA **30**,3403(1936) 2)H.B. Hass, E.B.Hodge & B.M.Vanderbilt, IEC **28**, 339(1936) 3)H.B.Hass & D.E.Hudgin,JACS **76**,2693–4(1955) & CA **49**,8786(1955)

Acetonenitrile. See Acetonitrile

Acetone, Nitro Derivative(Nitroacetone or Nitropropanone), $CH_3\cdot CO\cdot CH_2NO_2$, mw 103.08, N13.59%, OB to CO_2 –85.4%, OB to CO –38.8%. Plates or ndls, mp 49–50°, bp 103–104° at 24 mm or 185–190° with decompn when heated rapidly in small quantities under atm press. Sl sol in w, sol in alc & eth and

very sol in benz. It was prepd in 1899 by Lucas(Ref 4) from iodoacetone and Ag nitrate in ethereal soln at 0°. This is an indirect method of prepn as were the methods of Harries(Ref 5) and Wieland & Block(Ref 6). It was claimed by Henry(Ref 2) that O. de Battice prepd nitroacetone in 1895 in Belgium by oxidation of nitroisopropanol with chromic mixture. Henry described the compd as a col, mobile liq with a sharp odor, d 1.070 at 14°, bp 152° at 767 mm and insol in w. Lucas claimed(Ref 3) that the compd described by Henry was not nitroacetone, but this statement was disputed by Henry(Ref 4). Harries also claimed(Ref 5) that the compd described by Henry is not nitroacetone

More recently, Hass & Hudgin(Ref 7) claimed that they had isolated some nitroacetone from the high-boiling fraction of the vapor-phase nitration of acetone but it is not clear from their paper whether the substance was liq or solid. Hurd & Nilson(Ref 8) prepd nitroacetone as pale-green crysts, mp 47°, by oxidizing 1–nitro–2–propanol with sodium dichromate and sulfuric acid. The yield was 66% of theoretical. The explosibility of this compd was not mentioned

Refs: 1)Beil **1**,661 2)L.Henry,Rec **17**,399–402(1898) 3)A.Lucas,Ber **32**,604 & 3179(1899) 4)L.Henry,Ber **32**,865(1899) 5)C.Harries, Ann **319**,251 & 255(1901) 6)H.Wieland & S. Block,Ann **340**,83(1905) 7)H.B.Hass & E.E. Hudgin,JACS **76**,2694(1954) & CA **49**,8786(1955) 8)C.D.Hurd & M.E.Nilson,JOC **20**,931(1955) & CA **50**,6310(1956)

Acetone Oxime. See Acetoxime

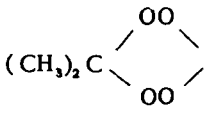
Acetone, Ozonization. According to Schroeter (Ref 1) a product contg active O was prepd by treating acetone with ozonized O or air. No compn was given. Briner & Meier (Ref 2) attempted to ozonize acet in a gaseous form, but instead of prepg an acet ozonide, they obtd CO_2, HCOOH & HCHO. Doevre (Ref 3) conducted ozonization of acet in solns. Schroeter (Ref 4) conducted ozonization of acet in the presence of org catalysts, particularly ether. No compns of resulting products were given

Refs: 1)G.Schroeter, Ger P 495,021(1927) & CA **25**, 1922(1931) 2)E.Briner & R.Meier, Helv **12**, 552(1929) 3)J.Doevre, BullFr [4] **45**, 140(1929) 4)G.Schroeter, GerP 557,516(1933) & CA **28**, 783(1934)

ACETONE PEROXIDES

Two peroxides are known, dimeric and trimeric

Acetoneperoxide, Dimeric or Diacetone Diperoxide(Acetonediperoxide, Dimeric Acetoneperoxide, Acetonedimer Peroxide, Cyclodiacetone Peroxide or Dicycloacetone Peroxide) (Was called in Ger Dimolekuläres-aceton-superoxyd, Polymeres-acetonsuperoxyd or 3.3 6.6–Tetramethyl–1.2.4.5–tetraoxan),



, mw 148.16, OB to CO_2 –151.2%, OB to CO –86.4%. Col prisms(from Et acet), mp 131.5–133°, very volat. Insol in w and dil acids & alkalies, unaffected by boiling in w for 6 hrs. Was prepd by Baeyer & Villiger (Ref 2) by treating a cooled ethereal soln of acet with Caro's reagent(prepd by rubbing K persulfate with concd sulfuric acid and then adding K sulfate). Pastereau(Ref 3) prepd the peroxide by treating acetone with 2% hydrogen peroxide in sulfuric acid soln. Other methods of prepn(including ozonization of acet) are given in Refs 4,5,7,8 and in some of the addnl refs

Phillips(Ref 6) attempted to prepare the dimeric acet peroxide by the method of Ref 2 but obtained the trimeric form instead. However, he succeeded in preparing the dimeric form by using the following method: 5 ml of acetone was mixed with 2 ml of 30% hydrogen peroxide(Baker's Analyzed) and cooled to 5° in an ice bath. Then 3 ml of dil sulfuric acid(4:1), was added at such a rate that the temp rose to 50° but was not allowed to exceed 60°(by temporarily cooling the mixt in an ice bath). The material separated as an oily liq which tended to float on the watery layer. It was purified by dissolving it in ether, washing the ethereal soln three times with w and finally evaporating on a steam bath with a slow current of air. The resulting white solid was dried for

2 hrs and bottled. It explodes violently on heating, impact or friction

Following props(Ref 6) of dimeric acetone peroxide were detd at PicArsn: *action with match flame* – a slight puff; *brisance by sand test* – 30.1 g sand crushed when 0.4 g of peroxide was initiated with 0.2 g MF, vs 48.0 g sand crushed by 0.4 g TNT; *impact sensitivity*, BurMinesApp with 2 kg wt – 7 cm, vs 60+ for TNT; *minimum detonating charge* in sand test – 0.19 g MF; *volatility* – 66.4% loss of wt at RT after 14 days and complete volatilization without residue after 3 hrs at 75°

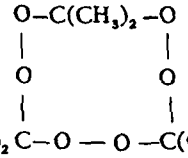
Its toxicity is unknown and fire & expln hazards are moderate(Ref 11)

Rohrlich & Sauermilch(Ref 6a) say that high sensitivity and extreme volatility of dimeric peroxide exclude it from practical consideration. It was recommended, however, by Nahsen (Addnl Ref a) for use in fuzes, detonators and caps and by Thiemann(Addnl Ref c) as an additive to Diesel fuels

Refs: 1)Beil **1**,645 & [714] and **19**,435(under the name of 3.3.6.6– Tetramethyl-1.2.4.5– tetroxan) 2)A.Baeyer & V.Villiger,Ber **32**, 3632(1899) & **33**, 124, 854 & 2480(1900) 3)J.Pastereau, CR **140**,1592(1905) & JCS **88** I, 572(1905) 4)A.Rieche, "Alkylperoxyde und Ozonide," Steinkopf,Dresden(1931), 84(Reproduced by Edwards Bros,Ann Arbor,Mich) 5)A.Rieche & K.Koch,Ber **75**,1016–28(1942) 6)A.J.Phillips, PATR 1202(1942) 6a) M.Rohrlich & W.Sauermilch,SS **38**,98(1943) 7)K.I.Ivanov et al,ZhurObshKhim **16**,1003 (1946) 8)R.Criegee et al,Ann **565**,9–13(1949) 9)Kirk & Othmer **10**(1953)(not found under Peroxides) 10)Tobolsky & Mesrobian(1954), 171 & 179 11)Sax(1957), 234(liq peroxide which is probably a mixt of di- and trimeric peroxides)

Addnl Refs on Dimeric Acetone Peroxide: a)Sprengwerke Dr R.Nahsen, GerP 423,176 (1925) & BritCA, SectB(1926), p 613(Use of acetone peroxides in fuzes, detonators and caps in lieu of MF) b)N.V.de Bataafsche Petroleum Maatschappij, BritP 444,544(1936) & CA **30**,5588(1936); GerP 671,012(1939) & CA **33**,3399(1939)[Acetone peroxides, as well as peroxides of higher mol wt ketones, may be prepd by treating a ketone with hydrogen peroxide (obtained by hydrolysis of $H_2S_2O_8$ or a persulfate in the reaction bath) at low temp in the presence of a strong acid and a stabilizer, such as urea. The resulting peroxide is extracted with gasoline] c)A.E.Thiemann, AutomobiltechnZ **45**,454–7 (1942) & CA **38**,2803(1944) (Acet peroxide dimer and trimer are claimed to be effective ignition promoters when added to Diesel fuels) d)I.N.Nazarov & I.N.Azerbaiev, ZhurObsKhim **18**,414–23(1948) & CA **43**,114(1949)(Dimeric acet peroxide, together with other compds, was obtained on ozonization of 3,3–dimethylallylchloride in methyl chloride soln) e) Y.M.Slobodin et al,ZhurObshKhim **23**,1873–7 (1953)& CA **49**,192(1955)(Dimeric acet peroxide was obtained together with other products on ozonolysis of 5–chloro–2–methyl–2–pentene) f)R.Criegee & G.Lohaus,Ann **583**,6–11(1953) & CA **49**,1588(1955)(Dimeric acet peroxide was obtained by ozonization of freshly prepd tetramethylethylene in Et chloride at –60°) g)M.Kolobielski, CR **237**,1717–18(1953) & CA **49**,1696(1955)(Ozonization of one of the acetylene derivatives gave mixts of a dimeric and trimeric acet peroxides) h)N.A.Milas et al, JACS **77**,2537(1955) & CA **50**(Prepn of diacetone peroxide by ozonization of olefins in the presence of carbonium ions)

Acetoneperoxide, Trimeric or Triacetone Triperoxide (Acetonetriperoxide, Trimeric Acetoneperoxide, Acetonetrimer Peroxide, Cyclotriacetone Peroxide, or Tricycloacetone Peroxide)(Called by Wolfenstein Tricycloaceton-superoxyd and by Rohrlich and Sauermilch Trizycloazetonperoxyd),



$(CH_3)_2C-O-O-O-C(CH_3)_2$,
mw 222.23, OB to CO_2 –151.2%, OB to CO –86.4%. Col volat crysts(from eth), mp 94–5°

(Ref 3); ndls, mp 96.5°(Ref 1,p 714 or crysts, mp 98.5°(Ref 9); d 1.2(Ref 4); volat with w vapor; non-hygroscopic. Insol in w, acids & alkalies and unaffected by boiling in w for 6 hrs; decompd by hot dil sulfuric acid. It is diff sol in methanol, glycerin & isoamyl alc. Its soly in some org solvents at 17° is as follows: absol alc 0.15, ether 5.5, acet 9.15, C disulfide 9.97, C tetrachloride 24.8, trichloroethylene 22.7, benz 18.0, pyridine 15.4, chlf 42.5 & petr eth 7.35%(Ref 13)

Trimeric acetone triperoxide was first prepd by Wolfenstein(Ref 2) from acet, 50% hydrogen peroxide and a small amt of phosphoric acid. This method required 4 weeks. Much simpler and more rapid was the method of Baeyer & Villiger(Ref 3), which consisted of adding(with cooling) concd HCl to a mixt contg equal amts of acet and 50% hydrogen peroxide. More recent methods of prepn are given in Refs 7,9,10,11,13,14 and in some of the additional references. The method of prepn used at PicArsn is described in Ref 10. In this, 5 ml of acet were mixed with 2 ml of 30% hydrogen peroxide(Baker's Analyzed) and cooled to 5° in an ice bath. Then 3 ml of dil sulfuric acid(4:1) was added dropwise, the temp not being allowed to rise above 10° The white flocculent ppt which formed instantly was shaken out with ether and the ethereal soln was washed 3 times in a separatory funnel with cold w. The ether was evapd on a steam bath using a slow current of air and the crysts of peroxide were air dried for 3 hours

The proced used by Ficheroulle & Kovache (Ref 13) consisted of adding in small portions a total of 5 cc of concd sulfuric acid to a small flask contg 16 g of acet (wellcooled in ice w). The flask was shaken vigorously after each addn and the temp was not allowed to rise above 25°. A total of 32 g of 45% hydrogen peroxide was added in small portions while the flask was kept in ice-w and then the mixt was allowed to stand overnight. The crysts were then separated by filtration and, after rinsing them with a large amt of ice w, were dried in a desiccator over anhyd Ca chloride

Trimeric acetone peroxide expld violently on heating, impact or friction. It is highly brisant and very sensitive. It may be detond under water or when it contains up to 25% of moisture(Ref 15)

Its expl and some other props were determined in Germany(Refs 4 & 11), France(Refs 7 & 13), USA(Ref 10) and Russia(Refs 12 & 15)

Following are some properties:

Action of flame, burned violently and sometimes detond

Action of heat, as detd by Patry(Ref 7) by placing small samples of ca 0.005g on a "block Maquenne," the sample melted at 97° and then up to 245° it vaporized without decompn; between 245 & 250°, it either decompd without flame, burned with smoky flame, or deton; between 250 & 285° it deton vigorously; betewen 285 & 305° it behaved in a manner similar to that described for the 245–250° range; over 305° it ignited with a smoky flame without deton

Brisance by the Lead Plate Test was detd by Rohrlich and Sauermilch (Ref 11) with caps contg as top charge: 0.05, 0.1, 0.2 or 0.3 g trimeric peroxide(compressed to 250 kg/cm²), an intermediate charge of 0.3 g PETN(compressed to 250 kg/cm²) and a base charge of 0.5 g PETN(compressed to 500 kg/cm²). The holes punched by these caps were comparable to those produced by No 8 caps

Brisance by the Sand Test was detd by Phillips(Ref 10) using 0.4 g sample initiated with 0.2 g MF. The amt of sand crushed was 34.1 g(TNT 48.0 g)

Burning rate at 1 atm of a highly compressed cylinder of peroxide – 0.95 cm/sec(Ref 12)

Compatibility with explosives. Equal wt of peroxide with PA, TNT, RDX, PETN, tetryl, $KClO_3$, AN, or Sb_2S_3, stored for 40 days at 50°, registered loss of wt equal to about 50% due to complete volatilization of the peroxide; there was no decompn of PA, TNT, etc(Ref 13)

Compatibility with metals. Strips of metals (Cu, Al, Zn, Sn, brass or Fe) stored with peroxide for 15 days showed no signs of corrosion: a slight corrosion was observed with lead(Ref 13)

Detonation velocity -5290 m/sec as detnd in column 6.3 mm diam and d 1.2(Ref 4); 3065 m/sec, as detnd in a column 15 mm diam and with d 0.68(Ref 11); 3750 at d 0.92 and 5300 at d 1.18(Ref 5a)

Friction sensitivity. Extremely sensitive (Ref 4a)

Impact sensitivity with Bur of Mines App and 500 g wt, 10 cm(Ref 10)

Impact sensitivity with the French app called "petit mouton" using 50 g wt, 15 cm for 50% detonations(Ref 13)

Note: Results of impact sensitivity tests show that acetone triperoxide is one of the most sensitive explosives known

Initiation test. A 0.05 g charge of peroxide, compressed at 250 kg/cm², caused PETN to detonate. When compression of the peroxide was increased to 500 kg/cm² partial failures resulted(Ref 11); min chge of peroxide to detonate TNT at d 1.35 in Cu tube 0.16g(Ref5a)

Power by the Trauzl Test. A 10 g sample gave expansion of 250 cc vs 285 cc for TNT(Ref 11)

Minimum detonating charge of MF in the sand test 0.19 g(Ref 10)

Toxicity, Fire & Explosion Hazards are discussed in Ref 18

Volatility. Sublimes even at ord temp(14–18°), losing about 6.5% of its wt in 24 hrs(Ref 11); loses 68.6% of its wt in 14 days at room temp(Ref 10); loses 1.5% in 2 hrs at 50° (Ref 11), loses 100% in 3 hrs(Ref 10). At 100° it volatilizes very rapidly, depositing fine needles on the cover(Ref 11)

Uses. It has been recommended for use in primers, detonators, etc(Refs 13,14 & a), but due to its high volaty and high sensitivity it does not seem very desirable for military use

Refs on Trimeric Acetone Peroxide: 1)Beil **1**,645 & [714] 2)R.Wolfenstein,Ber **28**,2265 (1895) 3)A.Baeyer & V.Villiger,Ber **32**, 3632(1899) & **33**,859 & 2680(1900) 4)Anon, Jahresber CTR **5**,111(1926) & **6**,100(1927) 4a)F.Schoofs & M.Bohet, CA **23**,5008(1929) 5)A.Rieche, "Alkylperoxide und Ozonide," Steinkopf,Dresden(1931)(Reproduced by Edwards Bros, Ann Arbor,Mich) 5a)H.Muraour, Bull Fr[4],**51**,1157(1932) 5b)Pepin Lehalleur (1935),137 6)A.Rieche, "Die Bedeutung der organischen Peroxyd fur die chemische Wissenschaft und Technik," Enke,Stuttgart (1936) 7)M.Patry,SS **32**,177 & 231(1937) 8)W.Dilthey et al, JPrChem **154**,219(1940) 9)A.Rieche & K.Koch,Ber **75**,1016–28(1942) 10)A.J.Phillips,Picatinny Arsenal Technical Report 1202(1942) 11)M.Rohrlich & W.Sauermilch,SS **38**,97–9(1943) 12)A.F.Belyaev & E.E.Belyaeva, DoklAkadN **52**,503–5(1946) 13)H.Ficheroulle & A.Kovache, MP **31**,20–21(1949) 14)C.E.Mavrodi,BritP 620,498 (1949) & CA **43**,6418(1949) 15)K.I.Ivanov et al, ZhurObshKhim **16**,1003(1949) 16)Kirk & Othmer **10**(1953) (not found under Peroxides) 17)Tobolsky & Mesrobian(1954),172 & 179 18)Sax(1957),234

Addnl Refs on Trimeric Acetone Peroxide: a)Sprengstoffwerke Dr Nahsen,GerP 423, 176(1925) & Brit CA,Sect B, **1926**,613 (Use of acet peroxide in detonators, caps and fuzes in lieu of MF) b)A.E.Thiemann,ChZtr **1942II**,2757–8(Acet peroxides are claimed to be effective ignition promoters when added to Diesel fuels) c)R.Acree & H.L.Haller,

JACS **65**,1652–3(1943) (Small quantities of the trimer are claimed to be present in isopropyl alcohol left standing for several years. This peroxide might be a cause of explosions of stored isopropyl alcohol, occasionally reported in literature) d)Gévelot & Gaupilat, FrP 893,941(1944) & CA **47**,8374(1953) (Trimeric acet peroxide combined with PA and RDX gave explosives of high power and velocity of detonation. Such mixts were less sensitive to shock than ordinary primary explosives. A still higher vel of detn may be achieved by replacing the metallic(Cu or brass) container for explosives by a flammable plastic tube which incorporates some explosive, eg NC + black powder) e)F.I.Berezovskaya et al, ZhurFizKhim **18**,321–8(1944) & CA **39**,2024(1945) (Effect of catalytic addns on the decomp of acet peroxide is discussed) f)M.Kolobielski,CR **237**,1717-18(1935) & CA **49**,1696(1955) [Mixt of trimeric and dimeric peroxides may be obtained by total ozonization of 2,2,3,5–tetramethyl–2–(β,β–dimethylvinyl)–2.3–dihydrofuran]

Acetonepicrylhydrazone or Acetone–(2,4,6–trinitrophenylhydrazone) $(CH_3)_2C{:}N{\cdot}NH{\cdot}C_6H_2(NO_2)_3$, mw 283.20, N24.73%, Yel or brn ndls, mp ca 125°, dec ca 130°. Was prepd by heating picryl hydrazine with acetone in alc or AcOH soln. No info on expl props

Refs: 1)Beil **15**,495 2)T.Curtius & G.M. Dedichen, JPraktChem **50**,274(1894)

Acetonetetrazyl Azide. See under Acetonyltetrazoles and Derivatives

Acetonetriperoxide. See Acetone Peroxide, Trimeric

ACETONITRILE AND DERIVATIVES

Acetonitrile, Cyanomethane or Methyl Cyanide (Ethanenitrile or Methanecarbonitrile), CH_3CN, mw 41.05, N34.12%, OB to CO_2 –214.4%, OB to CO –136.4%. Col liq, fr p –41° to –44°, bp ca 82°, d 0.7828 at 20°/4°, $n_D^{16.5°}$ 1.3496, vap press 100 mm at 27°, fl p(Cleveland open cup) 55°F, Q_c^p 304 kcal/mol, Q_f –16 kcal/mol(Refs 2 & 3) Q_c^v detd at PicArsn, 1324 cal/g with w liq (Ref 3a). Miscible with w, alc & eth. Can be prepd by dehydration of acetamide or by other methods. Used as a solvent for many org compds(among them RDX, HMX, etc) and as a starting material for the prepn of some org compds. Its toxicity and fire hazard are discussed in Ref 6. The expl hazard is great when acetonitrile is exposed to heat, flame or chem reactions with oxidizers. It forms an azeotrope with water

Refs: 1)Beil **2**,183,(84) & [181] 2)J.Thomas, ZPhysChem **52**,348(1905) 3)P.Lemoult, CR **148**,1602(1909) 3a)L.E.Newman,PACLR123, 718(1948) (U) 4)Kirk & Othmer **9**(1952),367 5)Merck(1952),8 6)Sax(1957),764 & 888–9

Acetonitrile, Azido Derivative (Azidoacetonitrile or Triazoacetonitrile), $N_3{\cdot}CH_2{\cdot}CN$, mw 82.07, N68.28%. Col liq, bp 53° at 12 mm, deflagrates when dropped on hot plate. Was prepd from chloroacetonitrile and Na azide in aq alc

Refs: 1)Beil – not found 2)K.Freudenberg et al, Ber **65**B,1188(1932) & CA **26**,5071(1932) (no other refs in CA through 1956)

Mononitroacetonitrile, Nitrocyanomethane or Nitromethylcyanide (Nitroethanenitrile) $O_2N{\cdot}CH_2{\cdot}CN$, mw 86.05, N 32.56%, OB to CO_2 –55.8%, OB to CO –18.6%. Yel volat oil, bp–dec on heating, d 1.36 at 18°. Can be prepd by acidifying with sulfuric acid its Amm salt, which in turn can be obtained by treating nitroacetaldoxime(methazonic acid) with thionyl chloride(sulfurous oxychloride), $SOCl_2$ in ether(Ref 2). It can also be prepd by dehydration of nitroacetaldoxime. Its lead block expansion value, according to Blatt (Ref 3),is 90% of PA

Nitroacetonitrile forms sparingly sol salts some of which are expl, eg, **silver salt**, $AgC_2HN_2O_2$, brn ppt, obtained by treating Amm nitroacetonitrile with Ag nitrate

Refs: 1)Beil **2**,227 & (100) 2)W.Steinkopf et al Ber**41**,1048–9(1908) & **42**,619(1909) 3)A.H.Blatt et al, OSRD 2014(1944)

Dinitroacetonitrile or Dinitrocyanomethane (Dinitroethanenitrile), $(O_2N)_2CH \cdot CN$, mw 131.05, N 32.07%, OB to CO_2 –6.1%, OB to CO +18.3%. Solid, expl on heating or impact. It was prepd in impure state in 1861 by Schiskoff on acidifying its Amm salt with aq sulfuric acid and extracting with ether. The Amm salt was obtained by treating trinitroacetonitrile(qv) with hydrogen sulfide: $C_2N(NO_2)_2 + 4H_2S = C_2N(NO_2)_2(NH_4) + 4S + 2H_2O$

Dinitroacetonitrile forms salts, some of which are expl, eg, **silver salt**, $AgC_2N_3O_4$, expl violently on impact

Treatment of dinitroacetonitrile or its Amm salt with fuming nitric acid gave the trinitroacetonitrile described below

Refs: 1)Beil **2**,228–9 2)L.Schischkoff, Ann**119**,249–50(1861)(not in CA through 1956)

Trinitroacetonitrile or Trinitrocyanomethane (Trinitroethanenitrile), $(O_2N)_3C \cdot CN$, mw 176.05,N 31.83%, OB to CO_2 +18.2%, OB to CO +36.4%. Yel volat crysts with pungent odor, mp 41.5° and expl on rapid heating ca 220°(Refs 1 & 2); sol in eth, decomp by w or alc. Was first obtained in 1857 by Schischkoff on treating Na fulminurate with mixed fuming nitric–sulfuric acid in the cold (Ref 3). The same investigator obtained it in 1861 from dinitroacetonitrile and its Amm salt(Ref 3)

According to Blatt(Ref 4) its lead block expansion value is 182% of PA and FI(figure of insensitivity)6% PA

Following props were detd at PicArsn and given in unclassified reports(Refs 5 & 6):

Q_c^v 1324 cal/g with w liq and 982 cal/g with w vapor; impact sensitivity, BurMines app with 2 kg wt 15 cm or less

Trinitroacetonitrile is a very powerful expl, and may be suitable for use in primers and detonators

Refs: 1)Beil**2**,229 2)L.Schischkoff, AnnChim Phys[3],**49**,310(1857) & Ann **101**,215(1857) 3)Ibid, Ann **119**,250(1861) 4)A.H.Blatt et al, OSRD 2014(1944) 5)L.E.Newman, Pic ArsnChemLabRept 123,718(1948) 6)H. Anderson & H.Vaughan, Ibid 123,975(1948)

ACETONYLACETONE AND DERIVATIVES

Acetonylacetone or 1,2–Diacetylethane (2,5–Diketohexane or 2,5–Hexanedione), $H_3C \cdot CO–CH_2 \cdot CH_2–CO \cdot CH_3$, mw 114.14, OB to CO_2 –210.3%, OB to CO –126.2%. Col liq, d 0.9737 at 20°/4°, mp ca –9°, bp 194° at 754 mm, vap press 0.43 mm at 20°, n_D^{20} 1.4232. Sol in w, alc & eth. Can be prepd by gently boiling 2,5–dimethylfuran for 36 hrs with an aq soln of AcOH and sulfuric acid, followed by addn of Na acetate to convert the acid to Na sulfate(Ref 2)

Its *chromium salt* was proposed as a component(up to 3% by wt) of some nitroparaffin-gel compns(such as prepd by blending nitromethane with 10–50% NC) used either as steadily burning rocket fuels or as expls. In the latter case, a sensitizer, eg an org amine, can be added. It is claimed that Cr acetylacetonate improves the ignition of nitroparaffin gels(Ref 3)

Refs: 1)Beil **1**,788,(405) & [841] 2)OrgSynth, CollVol 2(1943),219–20 3)H.Maisner,USP 2,712,989(1955) & CA **49**,14325(1955)

Acetonylacetone,Azido- and Diazido-Derivatives – not found in Beil or CA through 1956

Mononitroacetonylacetone and Dinitroacetonylacetone – not found in Beil or CA through 1956

ACETONYLTETRAZOLES AND DERIVATIVES

5–Acetonyl–α(1H)–tetrazole (C–Acetonyltetrazol or Acetessig–tetrazotsäure, in Ger), $CH_3 \cdot CO \cdot CH_2 \cdot C \cdot NH \cdot N$, mw 126.12, N44.43%,

$$\begin{matrix} & \| & \| \\ & N & N \end{matrix}$$

OB to CO_2 –126.9%. Crysts, mp 114°, easily sol in w or alc. Can be prepd by heating 1,3–dioxotetramethyleneotetrazole–2–carboxamide

with w, eliminating 2 molecules of CO_2 (Ref 2)

Refs: 1) Beil – not found 2)G.Schroeter & E.Finck, Ber **71**,683–4(1938) 3)F.R.Benson, ChemRevs **41**,6(1947)

Acetonylazidotetrazole or Azidoacetonyltetrazole, called by Friederich **Acetonemonotetrazylazide,** $C_4H_5N_4O \cdot N_3$ (no structural formula given in CA), mw 167.14, N 58.67%, OB to CO_2 –90.9%, OB to CO –52.7%. Solid, explodes on heating. Can be prepd by interaction of monochloroacetone with tetrazylazide

It was claimed by Friederich & Dynamit A–G to be a powerful expl, which can be used either alone or in combination with other expls, such as RDX, PETN & tetryl, or as a primary compd in detonators. Usual constituents of primary mixts such as tetracene, Ca silicide, glass powder, Sb sulfide, Pb dioxide, Ba nitrate, etc may be admixed with acetonylazidotetrazole

Refs: 1)W.Friederich, USP 2,170,943(1939) & CA **34**,265(1940) 2)Dynamit A–G, FrP 841, 768(1939) & CA **34**,4574(1940) 3)Dynamit A–G, BrP 510,992(1939) & CA **34**,5664(1940) 4)W.Friederich, GerP 695,254(1940) & CA **35**, 5318(1941)

Acetonylazidoditetrazole, called by Friederich **Acetoneditetrazylazide,** $(N_3CN_4)–H_2C \cdot CO \cdot CH_2–(N_4CN_3)$, mw 276.19 N 71.01%, OB to CO_2 –63.7%, OB to CO –34.8%. Solid, expl on heating. Was prepd by interaction of symdichloroacetone, $Cl \cdot H_2C \cdot CO \cdot CH_2 \cdot Cl$ with 2 mols of tetrazyl azide

It was patented by Friederich & Dynamit A–G for the same purposes as acetonylazidotetrazole

Refs: – same as above

Aceto-Perchloric Acid Mixtures. See Perchloric Acid–Acetic Anhydride–Water Mixtures

ACETOPHENONE AND DERIVATIVES

Acetophenone or Methylphenylketone (Acetylbenzene or Hypnone) (AcPh or MeCOPh), $CH_3 \cdot CO \cdot C_6H_5$, mw 120.14, OB to CO_2 –253.0%, OB to CO –146.5%. Col crysts, mp 20.5°, bp 202° (83.5° at 12 mm), d 1.0266 at 25/25°, $n_D^{20°}$ 1.5337, fl p 221°F (140.5°) (Ref 5). Insol in w but miscible with all common org solvents. According to Kirk & Othmer (Ref 2), it was first prepd in 1857 by Friedel by distn of a mixt of Ca benzoate and Ca acetate. Commercially, acetophenone is prepd by the Friedel–Crafts reaction using benz, Al chloride and acetic anhydride. It is an excellent solvent for NC (Ref 3), as well as for other cellulose esters & ethers (Ref 2). Its toxicity is unknown and its fire hazard is slight when exposed to heat or flame. It can react with oxidizing materials

Refs: 1)Beil **7**,271,(146) &[208] 2)Kirk & Othmer 1(1947),95–7 3)Durrans(1957),186 4)Sax(1957),235

Acetophenone, Azido Derivatives, $C_8H_7N_3O$, mw 161.16, N26.07%. Two isomers are described in the literature: **ω–Azidoacetophenone, Triazoacetophenone, Phenacyl Azide, Benzoylazidomethane,** $N_3 \cdot CH_2 \cdot CO \cdot C_6H_5$ plates (from eth + petr eth), mp 17°. Reacts very explosively with concd sulfuric acid. Was prepd by prolonged shaking of ω–bromoacetophenone with Na azide in aq alc, with cooling

Refs: 1)Beil **7**,(154) 2)M.O.Forster & R.Müller, JCS **97**,140(1910) & CA **4**,1606–7(1910) 3)J.H.Boyer, JACS **74**,4507(1952)

2–Azidoacetophenone or 1–Acetyl–2–azidobenzene, $CH_3 \cdot CO \cdot C_6H_4 \cdot N_3$, crysts (from ligroin), mp 22–22.5°. Can be prepd by treating (2–acetyl–benzenediazo)–hydroxylamide $CH_3 \cdot CO \cdot C_6H_4 \cdot N{:}N \cdot NH \cdot OH$ with dil sulfuric acid. No ref to its expl props

Refs: 1)Beil **7**,[225–6] 2)J.Meisenheimer et al, Ber **60**,1746–7(1927) 3)J.H.Boyer & D.Straw, JACS **75**,2684(1953) & CA **48**,7583 (1954)

Diazidoacetophenone, $C_8H_6ON_6$, mw 202.18, N41.57%. One isomer, called *o-azidophenacylazide*, $N_3 \cdot CH_2 \cdot CO \cdot C_6H_4 \cdot N_3$, wh ndls, mp 37–8° was prepd by Boyer & Straw by treating a dil acid soln of diazotized o-amino-phenacylazide with a sl molar excess of Na azide. Analysis was not attempted because the compd immediately showed signs of decompn

Refs: 1)Beil – not found 2)J.H.Boyer & D.Straw, JACS**75**,2684(1953) & CA**48**,7583 (1954)

Mononitroacetophenones, $C_8H_7NO_3$, mw 165.14, N8.48%. Following isomers are listed in Beil : 2-,3-,4-nitroaceto-phenones, $CH_3 \cdot CO \cdot C_6H_4 \cdot NO_2$, pp 288,(153) & [222–3] and ω-nitro-acetophenone, $(O_2N)CH_2 \cdot CO \cdot C_6H_5$, pp 289 & (153). None of these compds or their salts is expl. The 5-nitro-isomer listed in CA**46**,8630(1952) is actually the 3-isomer listed in Beil

Dinitroacetophenones, $C_8H_6N_2O_5$, mw 210.14%, N13.33%. Following isomers are listed in Beil **7**:

2,4–Dinitroacetophenone, $CH_3 \cdot CO \cdot C_6H_3(NO_2)_2$, yel oil, p 154

3,5–Dinitroacetophenone, $CH_3 \cdot CO \cdot C_6H_3(NO_2)_2$, ndls or plates, mp 82–4°, p 290

4,ω–Dinitroacetophenone, $(O_2N) \cdot CH_2 \cdot CO \cdot C_6H_4(NO_2)$, lt yel plates, mp 148–148.5°, p291. No expl props were reported

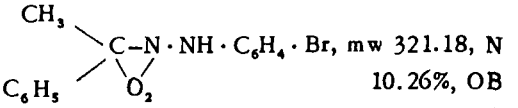
Trinitroacetophenones, $C_8H_5N_3O_7$, mw 255.14, N16.47%, OB to CO_2 –72.1% & OB to CO –21.9%. Only one isomer is described in the literature:

2,4,6–Trinitroacetophenone, $CH_3 \cdot CO \cdot C_6H_2(NO_2)_3$, orange-red crysts, mp 90–2°(dec); sol in hot w with decompn and in most org solvents except eth. Was prepd by the action of an ethereal soln of diazomethane on trinitrobenzaldehyde. Its expl props have not been detd

Refs: 1)Beil **7**,[225] 2)A.Sonn & W.Bülow, Ber**58**,1697(1925) & CA**20**,376(1926)

Tetranitroacetophenone, $C_8H_4N_4O_9$ was not found in Beil or CA through 1956

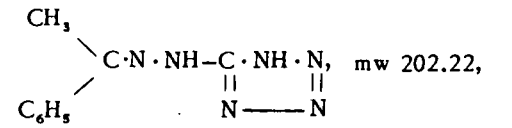
Acetophenone–(4–bromophenylhydrazone)–peroxide, called in Ger Peroxyd des Acetophenon–p–bromphenyl–hydrazon,



CH_3, $C(C_6H_5)(O_2)$–$N \cdot NH \cdot C_6H_4 \cdot Br$, mw 321.18, N 10.26%, OB to CO_2 –162.0%. Yel unstable ndls or prisms, mp 48–9°, expl on heating or standing; sol in most org solvents. Can be prepd by passing air through cold, agitated acetophenone–(4–bromophenylhydrazone), suspended in petr eth. No refs to its expl props

Refs: 1)Beil **15**,437 & (118) 2)P.C.Freer, Ber**30**,737(1897) 3)M.Busch & W.Dietz, Ber**47**,3290–1(1914)

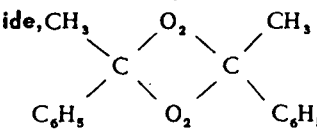
5–Acetophenonehydrazone–α(1H)–tetrazole or 5–[(Methylphenylmethylene)–hydrazine]–α(1H)–tetrazole, called in Ger Tetrazolon–α–phenäthylidenhydrazon or 5–α–Phenäthylidenhydrazinotetrazol,



CH_3, $C(C_6H_5)$–$C(=N-N)$–$NH \cdot N$, mw 202.22, N41.56%, OB to CO_2 –182.0%. Crysts, mp 235°; sl sol in alc & nearly insol in w. Was prepd by treating 5–hydrazino–α(1H)–tetrazolehydrochloride with acetophenone at RT. No refs to its expl props

Refs: 1)Beil **26**,406 2)J.Thiele & H.Inge, Ann**287**,236(1895) 3)F.R.Benson, ChemRevs **41**,8(1947)

Acetophenoneperoxide Dimeric, or Diacetophenone Diperoxide,



C_6H_5, CH_3, O_2, C, C, O_2, CH_3, C_6H_5,

mw 272.29, OB to CO_2 -211.5%. Col crysts, mp 185-6°(Ref 3), 182-3°(Refs 2 & 4), 181-2° (Ref 5). Was first prepd by heating 1-methyl-1-phenyl ozonide in AcOH (Ref 2). Other methods of prepn are given in Refs 3,4, & 5. No refs to its expl props

Refs: 1) Beil–not found 2)C. Harries, Ann **390**, 265-6(1912) 3)W. Dilthey et al, JPraktChem **154**, 234(1940) 4)N. A. Milas et al, JACS **77**, 2537 & 2540(1955); CA **50**, 5512(1956) 5)T. Yokoyama & Y. Yukawa, MemInstSciIndResearch, Osaka Univ **12**, 159(1955)(in Engl) & CA **50**, 16716(1956)

ACETOPHENONEOXIME AND DERIVATIVES

Acetophenoneoxime or Methylphenylketoxime, $CH_3 . C(:NOH) . C_6H_5$, ndls, mp 58.5-59°, is described in Beil **7**, 278-9, (150) & [216]

Acetophenoneoxime, Azido Derivative (Azido acetophenoneoxime or Triazoacetophenoneoxime), $N_3 . CH_2 . C(:NOH) . C_6H_5$, mw 176.18, N 31.80%. Pale yel oil which could not be crystallized. Was prepd from azidoacetophenone and hydroxylamine as described in Ref 2. No refs to its expl props

Refs: 1)Beil **7**, (154) 2)M.O. Forster & R. Müller, JCS **97**, 141-2(1910) & CA **4**, 1607 (1910)

Acetophenoneoxime, Diazido Derivative, $C_8H_7N_7O$–not found in Beil or CA through 1956

Mononitro acetophenoneoximes, $C_8H_8N_2O_3$, mw 180.16, N 15.55%. Several isomers are listed in Beil **7**, 288, 289,(153) & [222]

Dinitroacetophenoneoximes, $C_8H_7N_3O_5$, mw 225.16, N 18.66%. Several isomers are listed in Beil **7**, 290, 291 & (154)

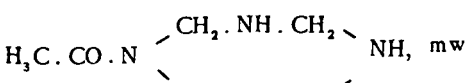
Trinitroacetophenoneoximes, $C_8H_6N_4O_7$, mw 270.16, N 20.74%. Not found in Beil or CA through 1956

Acetophenylamine. See Aminoacetophenone

Acetophenylnitramine. See Nitraminoacetophenone under Aminoacetophenone

ACETOTETRAZACYCLOÖCTANE OR ACETYLOCTAHYDROTETRAZINE AND DERIVATIVES

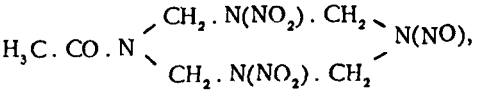
1-Aceto-1,3,5,7-tetrazacycloöctane or 1-Acetyl-1,3,5,7-tetrazacycloöctane,



mw 158.20, N 35.42%. May be considered as the parent compd of derivs which follow

Refs–not found in Beil or CA through 1956

1-Aceto-3,7-dinitro-5-nitroso-1,3,5,7-tetrazacycloöctane or 1-Acetyl-3,7-dinitro-5-nitroso-1,3,5,7-tetrazacycloöctane,

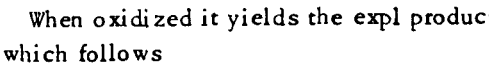


mw 277.20, N 35.30%, OB to CO_2 -66.4%, OB to CO -31.8%. Crysts, mp 180° with frothing, volat completely at 190°. It was prepd by stirring, at 25° for 15 hrs, a suspension of 1,5-methylene 3,7-dinitro-1,3,5,7-tetrazacycloöctane in a mixt of nitrosyl chloride and Ac_2O

When oxidized it yields the expl product which follows

Refs: 1)Beil–not found 2)W. E. Bachmann & N. C. Deno, JACS **73**, 2778(1951)

1-Aceto-3,5,7-trinitro-1,3,5,7-tetrazacycloöctane or 1-Acetyl-3,5,7-trinitro-1,3,5,7-tetrazacycloöctane,



designated as **SEX** and **QDX** and also called **1-Acetyl-3,5,7-trinitroöctahydro-s-tetrazine, 1-Acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine, or Octahydro-1-acetyl-3,5,7-trinitro-s-tetrazocine,** mw 293.20, N 33.44%, OB to CO_2 -57.3%, OB to CO -24.6%. Crysts mp 224.2-224.7° with frothing; can be detond

by a hammer blow (Ref 4). Sl sol in pyridine, acet & nitromethane; nearly insol in alc, benz, AcOH & eth. It is usually formed during nitrolysis of hexamine (Refs 3 & 4), but can also be prepd by other methods, such as oxidation of 1-aceto-3,7-dinitro-5-nitroso-1,3,5,7-tetrazacycloöctane, either with absol nitric acid at 40° or with a mixt of absol nitric with hydrogen peroxide (30% strength) (Ref 6). Goes over (25%) to HMX on treatment with 98% nitric acid at 5° (Refs 3 & 4). X-ray diffraction spectra of SEX are given in Ref 2, UV absorption spectra in Refs 5 & 7 and analytical procedures in Ref 8

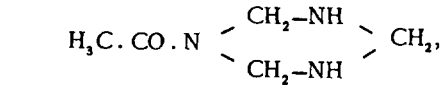
Refs: 1)Beil–bot found 2)A.Soldate & R.Noyes, AnalChem **19,** 442-4(1947) & CA **41,**6105(1947) 3)W.J.Chute et al, CanJRes **27B,** 515(1949) 4)E.Aristoff et al, Ibid **27B,** 533-4(1949) 5)R.N.Jones & D.Thorn, Ibid **27B,** 831(1949) 6)W.E.Bachmann et al, JACS **73,**2778(1951) 7)W.Schroeder et al, AnalChem **23,**1742(1951) 8)E.W. Malmberg et al, AnalChem **25,**901(1953)

Acetotetrazanonanediolacetate and Derivatives. See Acetyldiacetoxytetrazanonane and Derivatives

Acetotoluide or Acetotoludide. See Acetamidotoluene and Derivatives

ACETOTRIAZACYCLOHEXANE OR ACETYLHEXAHYDROTRIAZINE AND DERIVATIVES

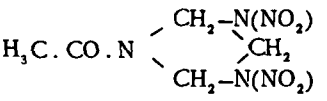
1-Aceto-1,3,5-triazacyclohexane or 1-Acetyl-1,3,5-triazacyclohexane,



mw 129.16, N32.54%. May be considered as the parent compd of the dinitro-deriv which follows

Refs: not found in Beil or CA through 1956

1-Aceto-3,5-dinitro-1,3,5-triazacyclohexane; 1-Acetyl-3,5-dinitro-s-triazine; 1-Acetyl-3,5-dinitro-1,3,5-triazacyclohexane or 1,5-Dinitro-3-acetyl-1,3,5-triazine; designated as **TAX,**



mw 219.16, N 31.9% OB to CO_2 -69.4%, OB to CO -32.9%. Crysts, mp 156-8°, sol in acet, alc and acet-alc mixts. It is one of the products of nitrolysis of hexamine and was first prepd in Canada. It also can be prepd from 3,5-dinitro-3,5-diazapiperidinium nitrate and by other methods described in Refs 4&6. Cyclonite in 38% yield may be obtained by treating TAX with nitric acid as described in Ref 3. Its UV absorption spectra are given in Refs 5&7 and analytical procedures in Ref 8

Refs: 1)Beil–not found 2)W.J.Chute et al, CanJRes **27B,** 515,517(1949) & CA **43,** 9074(1949) 3)E.Aristoff et al, CanJRes **27B,**534-5(1949) & CA **43,**9075(1949) 4)F. Chapman et al JCS **1949,**1640 & CA **44,** 1412(1950) 5)R.N.Jones, G.O. Thorn, Can JRes **27B,**843(1949) & CA 44 2848(1950 6) K.W.Downing & W.J.Downing, JCS **1950,** 2923,2930 & CA **45,** 6443-4(1951) 7)W. Schroeder et al, AnalChem **23,** 1742(1951) & CA **46,**5434(1952) 8)E.W.Malmberg et al AnalChem **25,**901(1953) & CA **47,** 12095(1953)

Acetotrinitrotetrazacycloöctane. See under Acetotetrazacycloöctane and Derivatives

ACETOXIME AND DERIVATIVES

Acetoxime or Acetone Oxime (2-Propanone Oxime or Dimethyl Ketoxime), mw 73.00, N 19.16%, OB to CO_2 -164.2%, Col crysts, mp 61°, bp 136.6°, d 0.97 at 20°/20°, Q_c^p 490.5 kcal/mol, Q_f 12.6 kcal/mol; sol in w, alc, eth & pet eth. Can be prepd by shaking an aq soln of hydroxylamine with acet and extracting acetoxime with ether (Ref 3). The product cannot be obtained in a perfectly dry condition without considerable loss by volatilization (Ref 2). It can be used as a solvent for cellulose ethers; as an intermediate

in org synthesis and as a primer for Diesel fuels

Acetoxime is the simplest **ketoxime.** It occurs in two isomeric forms:

$$R'-\underset{\underset{NOH}{||}}{C}-R \text{ (anti) and } R'-\underset{\underset{HON}{||}}{C}-R \text{ (syn)},$$

where R' is a radical of greater weight than R

Refs: 1)Beil **1**,649,(344) & [716] 2)P. Landrieu, CR **140**,867(1905) 3)OrgSynth, CollVol 1(1941),318-20 4)Hackh(1944), 9 5)Merck(1952), 9 6)CondChemDict(1956), 10

Acetoxime,Azido Derivative (Azidoacetoxime, Triazoacetoxime or 1-Azido-2-propaneoxime), $CH_3 . C(:NOH) . CH_2 . N_3$, mw 114.11, N 49.10%. Col oil, bp 84° at 2 mm with partial decompn. Was prepd from azidoacetone and hydroxylamine hydrochloride in aq soln contg some soda ash. When an attempt was made to distil 50g of azidoacetoxime at 2 mm, about 25 g distilled off at 84° while the residue in the flask gradually darkened and then violently exploded

Refs: 1)Beil **1**,661 2)M.O. Forster & H.E. Fierz, JCS **93**,83(1908)

Acetoxime, Diazido Derivative, $C_3H_5N_7O$– not found in Beil or CA through 1956

Mononitroacetoxime, $CH_3 . C(:NOH) . CH_2 . NO_2$, mw 118.09, N 23.72% is listed in Beil **1**,661

Dinitroacetoxime, $O_2N . CH_2 . C(:NOH) . CH_2 . NO_2$, mw 163.09, N 25.77%–not found in Beil or CA through 1956

ACETOXYDIPHENYLAMINE AND DERIVATIVES

Acetoxydiphenylamine, $CH_3 . COO . C_6H_4 . NH . C_6H_5$. May be considered as the parent compd of di-, tri-, and tetranitro- derivs, listed below

Refs–not found in Beil or CA through 1956

Azidoacetoxydiphenylamine, $C_{14}H_{12}N_4O_2$ and *Diazidoacetosydiphenylamine,* $C_{14}H_{11}N_7O_2$– not found in Beil or CA through 1956

Mononitroacetoxydiphenylamine, $C_{14}H_{12}N_2O_4$– not found in Beil or CA through 1956

Dinitroacetoxydiphenylamine $C_{14}H_{11}N_3O_6$. Several isomers are listed in Beil **13**, 366 & 446

Trinitroacetoxydiphenylamine, $C_{14}H_{10}N_4O_8$, mw 362.25, N 15.47%. Several isomers are described in Beil **13**, 366 & 446, none of them explosive

Tetranitroacetoxydiphenylamine, $C_{14}H_9N_5O_{10}$, mw 407.25, N 17.20%. Following isomer listed in Beil

x,x,2',4'-Tetranitro-4-acetoxydiphenylamine, $CH_3 . COO . C_6H_2(NO_2)_2 . NH . C_6H_3(NO_2)_2$, yel ndls (from aq acet), which brown at 155° & melt at 161°; sol in acet, chlf, benz & AcOH; diff sol in alc; sl sol in eth and insol in ligroin. Was prepd by nitrating 2',4'-dinitro-4-acetoxydiphenylamine with fuming nitric acid. No ref to its expl props

Refs: 1)Beil **13**,532 2)F. Reverdin & E. Deletra, Ber **37**, 1731(1904)

Note: No higher nitrated compds are listed in Beil or CA through 1956

ACETOXYETHOXYTRIAZAHEPTANE AND DERIVATIVES

1-Acetoxy-7-ethoxy-2,4,6-triazaheptane, $CH_3 . COO-CH_2 . NH . CH_2 . NH . CH_2 . NH . CH_2 . O . C_2H_5$, may be considered as the parent compd of the trinitro- deriv described below

Refs- not found in Beil or CA through 1956

1-Acetoxy-7-ethoxy-2,4,6-trinitro-2,4,6-triazaheptane, $CH_3 . COO-CH_2 . N(NO_2) . CH_2 . N(NO_2) . CH_2 . N(NO_2) . CH_2-OC_2H_5$, mw 340.26, N 24.70%, OB to CO_2 –70.5%, OB to CO –32.9%. Crysts, mp 106-7°. Was obtained by Chute et al as one of the products of nitrolysis of hexamine. No refs to its expl props

Refs: 1)Beil–not found 2)W.J. Chute et al, CanJRes **27B**,504 & 513(1949); CA **43**, 9074(1949)

ACETOXYMETHYLTETRAZACYCLOÖCTANE AND DERIVATIVES

1-Acetoxymethyl-1,3,5,7-tetrazacycloöctane, $H_2C-N(CH_2.OOC.CH_3)-CH_2$ / $HN-CH_2-NH-CH_2-NH$, may be considered as the parent compd of trinitro-deriv described below

Refs: not found in Beil or CA through 1956

1-Acetoxymethyl-3,5,7-trinitro-1,3,5,7-tetrazacycloöctane

$H_2C-N(CH_2.OOC.CH_3)-CH_2$ / $O_2N.N-CH_2-N(NO_2)-CH_2-N.NO_2$,

mw 323.23, N30.34%, OB to CO_2 -61.9%, OB to CO -27.2%. Crysts, mp 152° (when heated rapidly). No suitable solvent for its recrystn has been found. It was obtained on nitrolysis and acetylation of DPT (1,5-methylene-3,7-dinitro-1,3,5,7-tetrazacycloöctane)

Acetoxymethyltrinitrotriazacycloöctane reacts with a mixt of HNO_3 and Ac_2O to give a linear tetranitramine, 1,9-diacetoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanone(qv) (scission of the 8-membered ring takes place). When AN was present in the HNO_3-Ac_2O mixt, the ring remained intact and the cyclic tetranitramine, HMX(1,3,5,7-tetranitro-1,3,5,7-tetrazacycloöctane) was formed in good yield

Refs: 1)Beil–not found 2)W.E.Bachmann & E.Jenner, JACS **73**,2773-4(1951) & CA **46**, 2085(1952) 3)W.E.Bachmann & N.Deno, JACS **73**,2778(1951) & CA **46**,2085(1952)

ACETOXYMETHYLTRIAZACYCLOHEXANE AND DERIVATIVES

1-Acetoxymethyl-1,3,5-triazacyclohexane,

$H_2C-N(CH_2.OOC.CH_3)-CH_2$ / $HN-CH_2-NH$,

may be considered as the parent compd of dinitro- deriv described below

Refs: not found in Beil or CA through 1956

1-Acetoxymethyl-3,5-dinitro-1,3,5-triazacyclohexane,

$H_2C-N(CH_2.OOC.CH_3)-CH_2$ / $O_2N.N-CH_2-N.NO_2$,

mw 249.19, N 28.11%, OB to CO_2 -73.8%, OB to CO -35.3%. Crysts, mp 143.7-144.7; sol in acet, insol in w or petr eth. Was obtained by Chute et al as one of the products of nitrolysis of hexamine. No refs to its expl props

Refs: 1)Beil–not found 2)W.J.Chute et al, CanJRes **27B**, 506 & 517-18(1949); CA **43**,9074(1949)

ACETOXYNAPHTHALENE AND DERIVATIVES

Acetoxynaphthalene or Naphthylacetate, called in Beil *Essigsäure-naphthylester*, $CH_3COO.C_{10}H_7$. Two isomers α- and β- are described in Beil **6**, 608, 644, (307, 313) & [580, 600]

Acetoxynaphthalene, Azido Derivative, $CH_3.COO.C_{10}H_6.N_3$ and *Diazido Derivative* $CH_3.COO.C_{10}H_5(N_3)_2$–not found in Beil or CA through 1956

Mononitroacetoxynaphthalene, $C_{12}H_9NO_4$. Four isomers: *2-nitro-1-acetoxy-, 5-nitro-1-acetoxy-, 1-nitro-2-acetoxy- and, 8-nitro-2-acetoxy-naphthalene* are described in Beil **6**, 615, 616, 654 & 655

Dinitroacetoxynaphthalene, $C_{12}H_8N_2O_6$. One isomer, *2,4-dinitro-1-acetoxynaphthalene* is listed in Beil **6**, [587]

Trinitroacetoxynaphthalene, $CH_3.COO.C_{10}H_4(NO_2)_3$, mw 321.20, N13.08%–not found in Beil or CA through 1956

Tetranitroacetoxynaphthalene, $C_{12}H_6N_4O_{10}$, mw 366.20, N15.30%. Following isomer is listed in the literature:

2,4,5,7-Tetranitro-1-acetoxynaphthalene, also called in Beil [2,4,5,7-naphthyl-(1)]-acetat, $CH_3.COO.C_6H(NO_2)_2:C_6H_2(NO_2)_2$. Crysts (from AcOH), mp 163-5°(dec). Was prepd by treating 4-benzeneazo-α-naphthyl-1-acetate with nitric acid (d 1.42). No ref to its expl props

Refs: 1)Beil **6**, [587] 2)R.Meldola & G.T.Morgan, JCS **55**,609(1889)

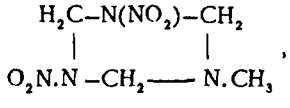
Note: No higher nitrated derivs were found in Beil or CA through 1956

ACETOXYTRIAZAHEPTANE AND DERIVATIVES

1-Acetoxy-2,4,6-triazaheptane, $(CH_3.COO)CH_2.NH.CH_2.NH.CH_2.NH.CH_3$, may be considered as the parent compd of its trinitro-deriv described below

Refs–not found in Beil or CA through 1956

1-Acetoxy-2,4,6-trinitro-2,4,6,-triazaheptane; N,N',N''-Trinitro(acetoxymethylaminomethyl) -(methylaminomethyl)amine; 2,4,6-Triaza-2,4,6-trinitro-heptan-1-ol or MSX, $(H_3C{\cdot}OOC){\cdot}-CH_2{\cdot}N(NO_2){\cdot}CH_2{\cdot}N(NO_2){\cdot}CH_2{\cdot}N(NO_2){\cdot}CH_3$, mw 296.20, N 28.38%. Col rosettes or prisms; mp 153-4°. Was prepd from 1,5-dinitro-3-methyl-hexahydro-1,3,5-triazine,



added along with a soln of AN in 98% nitric acid to AcOH + Ac_2O, stirring and adding w (Ref 2). No ref to its expl props. Ultraviolet absorption spectra are given in Ref 3

This compd was examined in connection with a study of the reaction leading to the production of cyclonite

Refs: 1)Beil–not found 2)F.Chapman et al, JCS **1949,** 1648 & CA **44,** 1412(1950) 3)R.N.Jones & J.D.Thorn, CanJRes **27B,** 835(1949) & CA **44,** 2848(1950)

Acetozone. See Acetylbenzoylperoxide

ACETYLACETONE AND DERIVATIVES

Acetylacetone, 2,4-Pentanedione or Diacetylmethane, $CH_3CO.CH_2.CO.CH_3$, mw 100.11. col liq. d 0.9721 at 25/4° f_p -23.2°, bp 137-140°, $n_D^{18.8°}$ 1.4513. Can be prepd from acetone, ethyl acetate and Na ethylate or by other methods. It is an excellent gelatinizer for NC

Refs: 1)Beil **1,** 777,(401) & [831] 2)L.Claissen & E.Ehrhardt, Ber **22,** 1010 (1889) and many other later refs listed in Beil and in CA

Chromium Salt of Acetylacetone or Chromylacetylacetone, $Cr(C_5H_7O_2)_3$, red-viol crysts, d 1.34, mp 214°, bp 340° (without decompn). Was prepd by Urbain & Debierne by treating chromic nitrate with acetylacetone (Refs 1 & 2). Its UV absorption spectra are given in Ref 3 and crystallographic structure by X-rays in Ref 4

Maisner (Ref 5) claims that incorporation of up to 3% Cr acetylacetonate in rocket propellants prepd by gelling nitroparaffins (such as nitromethane) with NC, renders them easier to ignite. These mixts can vary from syrupy to solid gels, depending on the amt of NC used. When gels are solid (large amts of NC), they are suitable for use as regular propellants. Same mixts can be used as expls, especially when an org amine (such as methylamine) is incorporated to serve as a sensitizer. All these mixts can be prepd at RT

Refs: 1)Beil **1,** 782,(404) & [836] 2)G. Urbain & A.Debièrne, CR **129,**304(1899) & JCS **76,**I,789(1899) 3)G.T.Morgan & H.W. Moss, JCS **105,**200(1914) 4)W.T.Astbury, ProcRoySoc **112A,**449,458(1926) & CA **21,** 842(1927) 5)H.Maisner, USP's 2,690,964 (1955) & 2,712,989(1955); CA **49,**618 & 14325-6(1955)

Acetylacetone, Azido Derivative, $N_3.CH_2.CO.CH_2.CO.CH_3$ and *Acetylacetone, Diazido Derivative,* $N_3CH_2.CO.CH_2.CO.CH_2.N_3$–not found in Beil

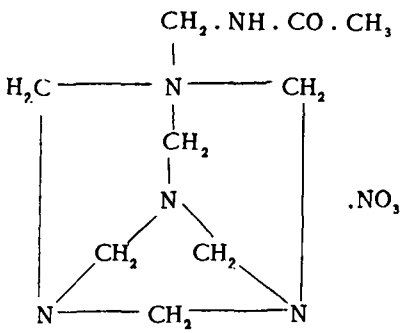
Mononitroacetylacetone, $(O_2N).CH_2.CO.CH_2.CO.CH_3$ and *Dinitroacethylacetone* $O_2N.CH_2.CO.CH_2.CO.CH_2.NO_2$ –not found in Beil

Acetylacetone Peroxide, Polymer, $(C_5H_{10}O_4)_x$, mw $(134.13)_x$, OB to CO_2 -131.2%, OB to CO -71.5%. Glassy syrup, not volatile with steam; very expl. Was prepd by Pastureau from acetyl acetone and hydrogen peroxide in sulfuric acid soln

Refs: 1)Beil **1,** 785 2)J.Pastureau,

BullFr [4], **5**, 228(1909); JCS **96**,208(1909) & CA **4**,191(1910). No other refs in CA 1920-1956

N-Acetylamidomethylhexamethylenetetraminemononitrate; 1-Acetamidomethylhexaminenitrate or 1-Acetamidomethylhexamethylenetetraminenitrate, designated as H_2,



mw 274.28, N 30.64%. Large col plates, mp 183-4° (Ref 4). Its prepn by three different methods was not described until 1951 by Bachmann et al (Ref 4), but the compd was mentioned and used in 1949 (Refs 2 & 3). Ref 2 describes studies of the nitrolysis of H_2 resulting from the prepn of RDX and HMX, while Ref 3 gives UV absorption spectra of "H_2". Neither of the papers discusses the prepn of "H_2" or gives any previous refs on this subject. It seems that Bachmann prepd "H_2" prior to 1949 but did not publish his methods of prepn and physical props, such as mp of "H_2" until 1951 (Ref 4). Spectrophotometric data and the structure of "H_2" are given in Ref 5

Refs: 1)Beil–not found 2)E. Aristoff et al, CanJRes **27B**, 541-5(1949) & CA **43**, 9075(1949) 3)R. Jones & G. Thorn, CanJ Res **27B**,832,853(1949) 4)W. E. Bachmann et al, JACS **73**,2775-7(1951) & CA **46**, 2085(1952) 5)W. Schroeder et al, AnalChem **23**,1741-2(1951), compd No 19

ACETYLALANINE AND DERIVATIVES

Acetyl-dl-alanine or Acetamidopropionic Acid, $CH_3 . CO . NH . CH(CH_3) . COOH$, plates or ndls, mp 132-137.5°. Can be prepd by treating d,l-alanine with acetic anhydride or by other methods

Refs: 1)Beil **4**,394,(495) & [811] 2)A. de Jong, Rec **19**,282(1900) and several other refs in Beil and in CA

Azidoacetyl-dl-alanine, $CH_3.CO.N(N_3).CH(CH_3).COOH$, mw 172.15, N32.55%. Long fine ndls, mp 101°. Was prepd by Freudenberg & Keller from dl-alanin by a procedure described in Ref 2. In the course of prepn of this compd an intermediate, *Azidoacetyl-dl-alanine chloride* was obtained. This chloride could not be purified because it decompd explosively at ca 30°

Refs: 1)Beil–not found 2)K. Freudenberg & R. Keller, Ber **71B**, 334(1938) & CA **32**, 2905(1938)

Note: No nitrated derivs of acetyl-dl-alanine were found in Beil or CA through 1956

Acetylaniline. See Acetanilide

ACETYLBENZOYLPEROXIDE AND DERIVATIVES

Acetylbenzoylperoxide; Benzoylacetylperoxide; Acetozone or Benzozone (formerly called Acetylbenzoyl-superoxyd in Ger) $CH_3 . CO . O_2 . CO . C_6H_5$, mw 180.15. Wh crysts mp 37-41°, bp 130° at 19 mm (might explode); expl violently at 85-100° and also by friction or compression; stable, when dry at RT. but decomps in the presence of moisture, org matter or traces of alc, eth or acids; sl sol in w(0.064 g in 100 ml at 25°) alc & mineral acids; sol in CCl_4, chlf, eth & oils. Was first prepd by Nef(Ref 2) from benzaldehyde and acetic anhydride. Other methods of prepn are listed in Ref 1.

Caruthers (Ref 5) patented a method of prepn in which benzaldehyde and acetaldehyde are caused to react at ca 35° with an O-contg gas in the presence of dibenzoylperoxide

Thermal decompn of acetylbenzoylperoxide is discussed in Ref 3 and the decompn by UV light in Ref 4. Its fire & expln hazard, toxicity and shipping regulations are discussed in Refs 6 & 7

Acetylbenzoylperoxide is used in lab and industry as an oxidation and polymerization catalyst in a number of reactions

Refs: 1)Beil **9**, 179,(93) &[157] 2)J.U. Nef, Ann **298**, 280(1897) 3)F. Fichter & H. Erlenmeyer, Helv **9**,146(1926) & CA **20**, 1385(1926) 4)F. Fichter & E. Willi, Helv **17**, 1173(1934) & CA **29**,1013 (1935) 5)T.F. Caruthers, USP 1,985,886(1935) & CA **29**, 1104(1935) 6)CondChemDict(1956),117 7)Sax(1957), 235

Acetylbenzoylperoxide, Azido Derivative, $N_3 . C_9H_7O_4$–not found in Beil or CA through 1956

Acetyl-(3-nitrobenzoyl)-peroxide or m-Nitrobenzoylacetylperoxide (called by Nef m-nitrobenzoylacetylhydroperoxyd), $CH_3 . CO . O_2 . CO . C_6H_4 . NO_2$, mw 225.15, N6.22%. Col ndls (from hot methanol), mp 68°; expl at higher temps; sol in most org solvents; diff sol in cold methanol & ligroin. Was prepd by treating acetylbenzoylperoxide with fuming nitric acid in the cold

Refs: 1)Beil **9**,381 2)J.U.Nef, Ann **298**, 286(1897)

Note: No later refs were found in CA through 1956

Acetyl-(dinitrobenzoyl)-peroxide, $CH_3 . CO . O_2 . CO . C_6H_3(NO_2)_2$ –not found in Beil or CA through 1956

Acetylbenzylperoxide; Benzylacetylperoxide; Benzylperacetate or Peracetic Acid, Benzylester, $CH_3 . CO . O_2 . CH_2 . C_6H_5$; powder. It was patented in 1927 by Carbide & Carbon Chemicals Co (Ref 2) for use as a catalyst in polymerizing vinyl compds particularly vinyl chloride and acetate. Sax (Ref 4) lists this compd without giving its formula or method of prepn but states that it is a powerful oxidizing agent; its toxicity–details unknown, fire hazard–moderate by spontaneous chemical reaction, expln hazard–moderate when shocked or exposed to heat and disaster control–dangerous; shock will cause deton with evoln of toxic fumes; will react with w and steam to produce heat; can react vigorously with reducing materials

Refs: 1)Beil–not found 2)Carbide & Carbon Chemicals Co, FrP 748,972(1933) & CA **27**, 5755(1933) 3)Tobolsky & Mesrobian (1954)–not found 4)Sax(1957),236

Acetyl Bromide or Ethanoyl Bromide, CH_3COBr, mw 122.96, OB to CO_2 -58.6%, OB to CO -45.5%. Col liq fuming strongly in the air; d 1.663 at 16° mp -96.5, bp 76° at 750 mm, n_D 1.4537 at 15.8°. Miscible with eth, benz & chlf, decomp violently by w and alc. Can be prepd from acetyl chloride and an excess of HBr or by other methods. Its toxicity, fire & explosion hazards are discussed in Ref 3

Refs: 1)Beil **2**,174,(79) & [176] 2)H. Staudinger & E. Anthes, Ber **46**, 1421(1913) 3)Sax(1957),236

ACETYL CELLULOSE AND DERIVATIVES

Acetyl Celluloses (AC) (Acetates of Cellulose or Cellulose Acetates). According to Dorée(Ref 3) the action of Ac_2O on cellulose (called acetylation) should theoretically yield the triacetate $[C_6H_7O_2(OOC . CH_3)_3]n$. Actually, the products of acetylation are a mixture of tri-, di- and mono-acetate. A characteristic property of the lower acetylated acetates is their sol in acetone, whereas the triacetate can absorb acetone only to the extent of causing swelling

Lab and industrial methods of prepn of AC are described in Refs 2,3,4 & 6. AC is used in the manuf of rayon, films, unbreakable

windows (Ref 4), as a component and as an inhibitor coating of rocket propellants (Refs 5 & 7). The Italians claim that AC has the property of slowing down the rate of burning of a propellant and rendering the combstn more uniform

Following are Ital military spec requirements for AC: ash $\leq 0.15\%$, free acid none, foreign matter ≤ 0.1, ether ≤ 0.15, insyl in acet ≤ 1.0 and acidity (calcd as AcOH) $\leq 0.15\%$, detd by 132° Heat Test using a Bergmann-Junk tube

Refs: 1)Beil–not found 2)Heuser(1944), 226 3)Dorée(1947),272-92 4)Kirk & Othmer 3(1949),372-6 5)Capitolato Tecnico Generale per la Fornitura di Esplosivi Propelenti, MD Esercito CTF 35 6)Ott **5**,part 2(1954),767 7)P.Tavernier, MP **38**, 328(1956)(Thermodynamic props of acetylcelluloses) 7)J.A.Rolfe & N.J.Morris, RPE(Rocket Propulsion Establishment), Gr Britain, TechNote No 171, October 1958

Nitroacetylcelluloses (NAC) (Cellulose Acetate Nitrates). Oddo (Ref 2), being dissatisfied with the chem stability of NC's decided to det whether acetylated NC's would be any better. He took 10g of NC (11.66%N), dried it at 90° and gradually introduced 300g of acetic anhydride free of AcOH. After 12 hrs at RT the mixt was heated on a sand bath, under reduced pressure and under a reflux condenser (sealed with a sulfuric acid valve), until about two-thirds of the acetic anhydride was distilled. The residual liq was filtered and poured into a large amt of w. The resulting lt yel product was dried, dissolved in AcOH, filtered and repptd by pouring the soln dropwise with agitation into a large amt of cold w. The dried purified product was white and could be easily pulverized. Its mw was $(290.5)_n$ and N ca 9.65%, which corresponds approx to $C_6H_7O_2(ONO_2)_2(OAc)$, calcd mw $(294.1)_n$. It decomp at 185.5° with the evoln of gas. The product was easily sol in et acet, AcOH & pyridine and could be gelatinized by 1:1-ether/alc

Dorée(Ref 3) prepd several NAC's (some of them with a N content as high as 13.8% and an AcOH content 32.3%) by gradually adding cellulose to an ice-cooled bath contg mixt of acetic anhydride and fuming nitric acid. Krüger (Ref 4) studied the nitration-acetylation of cellulose with mixts of acetic anhydride-nitric acid-acetic acid. Werner (Ref 5) studied a method of prepn of NAC by nitration of fibrous cellulose triacetate with nitric acid contg less than 9% w. He also studied prepn of NAC by acetylation of NC. A brief description of NAC is also given in Ref 6

NAC with N ca 11.5% has been used in Italy as an ingredient of DEGDN propellants (polveri al nitroglicol). Following are Ital military specification requirements (Ref 7): nitrogen content 11.20-11.70%, fineness ≤ 90, acetyl content $\geq 1.50\%$ ash $\leq 1\%$, lime calcd as CaO $\leq 0.30\%$, stability by 80° Abel test > 25 min, by 135° Ger test 50 min and by 131° Bergmann-Junk test ≤ 1.75 cc of NO

Refs: 1)Beil–not found 2)B.Oddo,Gazz **49** II,140-5(1919) & CA **14**,1530(1920) 3) Dorée(1947)305 4)D.Krüger,Cellulosechem **11**,220(1930) 5)K.Werner,AngChem **50**, 127-32(1937)(New method for making and utilizing cellulose triacetate) 6)Heuser (1944),301 7)Capitolato Tecnico Generale per la Fornitura di Esplosivi Propellenti, MD Esercito,1951,CTF 34

ACETYL CHLORIDE AND DERIVATIVES

Acetyl Chloride or Ethanoyl Chloride, $CH_3.CO.Cl$, mw 78.50, OB to CO_2 −91.7%, OB to CO −71.3%. Col liq which fumes in air. It is flam, d 1.1051 at 20°, mp −112°, bp 51-2°, N_D^{20} 1.3898. Miscible with eth, benz, chlf, glacial AcOH & petr eth. Decompd violently by w or alc. Extremely irritating to the eyes. Can be prepd from glacial AcOH and phosphorus trichloride (see also Ref 6) or by other methods listed in Ref 1. Used as an acetylating agent and for the detn of w in organic liquids. Its toxicity, fire and explosion hazards are discussed in Refs 4 & 5

Its nitrocompound is described below

Refs: 1)Beil **2**, 173(79) & [175] 2)L. Orthner & L. Reichel, Organische Chemisches Praktikum, Berlin(1929), 73 3)Merck(1952), 11 4)CondChemDict(1956), 11 5)Sax(1957), 236-7 6)R. D. Coghill, JACS **60**, 488(1938) & CA **32**, 2355(1938)(Explns take place sometimes during this method of prepn. Coghill attributes the formation of phosphine, PH_3, as the cause of such explns)

Azidoacetylchloride or Triazoacetylchloride, $N_3 . CH_2 . CO . Cl$, mw 119.52, N 35.16%. Col pungent smelling liq, decomp by moisture; bp 43-44° at 14 mm & 55-60° at 18 mm; expl at higher temps; d 1.303 at 25°, $n_D^{25°}$ 1.4634. Was prepd by Forster & Müller by the action of phosphoryl chloride on azidoacetate suspended in abs eth (Refs 1 & 2). Other methods of prepn are given in Refs 3 & 4

Refs: 1)Beil **2**, 229 & (101) 2)M. Forster & R. Müller, JCS **95**, 200(1909) & **97**, 1061(1910) 3)E. D. Nicolaides et al, JACS **76**, 2889 (1954) & CA **49**, 10184(1955) 4)F. Huber, JACS **77**, 113(1955) & CA **50**, 804(1956)

Diazidoacetylchloride, $(N_3)_2CH . CO . Cl$—not found in Beil or CA through 1956

Nitroacetyl Chloride, $O_2N . CH_2 . CO . Cl$, mw 123.50, N 11.33%, OB to CO_2 −25.9%, OB to CO ±0%. Liq, fr p −35°, bp 68° at 12 mm; slow distn is accompanied by an expln. Was prepd in poor yield by nitration of ketene in ether, cooled in solid CO_2 + alcohol(Ref 2)

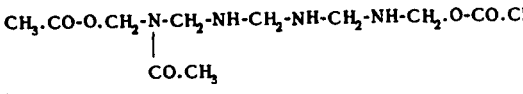
Refs: 1)Beil **2**, not found 2)W. Steinkopf & M. Kühnel, Ber **75B**, 1328(1942) & CA **37**, 4687(1943)

Nitroazidoacetylchloride, $(O_2N)N_3 . CH . CO . Cl$—not found in Beil or CA through 1956

Dinitroacetylchloride, $(O_2N)_2CH . CO . Cl$—not found in Beil or CA through 1956

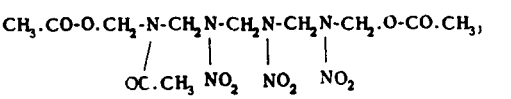
ACETYLDIACETOXYTETRAZANONANE AND DERIVATIVES

2-(4)-Acetyl-1,9-diacetoxy-2,4,6,8-tetrazanonane or 2-(4-)Aceto-2,4,6,8-tetrazanonane-1,9-diol-diacetate,

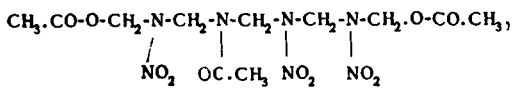


may be considered as a parent compd of trinitrocompd described below

2-(4-)Acetyl-1,9-diacetoxy-4-(2-),6,8-trinitro-2,4,6,8-tetrazanonane; 2-(4-)Aceto-4-(2-),6,8-trinitro-2,4,6,8-tetrazanonane-1,9-diol-diacetate; 1,9-Diacetoxy-2-(4-)acetyl-4-(2-),6,8-trinitro-2,4,6,8-tetrazanonane or H-16



or



mw 425,32, N 23.05%. According to Aristoff et al (Ref 2), this compd was prepd by M. Carmack et al(private communication) when they treated hexamethylenetetramine with nitric acid and acetic anhydride. Its expl props were not investigated. Schroeder et al(Ref 3) give the absorption spectra data and Malmberg et al (Ref 4) the chromatographic data

Refs: 1)Beil—not found 2)E. Aristoff et al, CanJRes **27B**, 526-7(1949) 3)W. A. Schroeder et al, Anal Chem **23**, 1740, 1745 (1951) 4)E. W. Malmberg et al, AnalChem **25**, 903(1953)

Acetyldinitroglycerin. See Glycerin Acetate Dinitrate under Glycerin and Derivatives

Acetyldinitrotriazacyclohexane. See Acetodinitrotriazacyclohexane under Acetotriazacyclohexane and Derivatives

Acetyldinitronitrosotetrazacycloöctane. See Acetodinitronitrosotetrazacycloöctane under Acetotetrazacycloöctane and Derivatives

ACETYLDIPHENYLAMINE AND DERIVATIVES

Acetyldiphenylamine, $C_{14}H_{13}NO$. Its N-acetyl-isomer $(C_6H_5)_2N . CO . CH_3$ is described in Beil **12**, 247,(194) & [144] under the name of Essigsäure-diphenylamid. P. Tavernier & L. Lamouroux, MP **38**, 84 (1956) gives for it Q_c^v 1752 kcal/mol and

Q_f^v 9.64 kcal/mol. Isomers in which acetyl group is attached to the ring, *anilinoacetophenones*, are not described in Beil, although their nitro-, dinitro- and trinitro- derivs are listed in vol 14. One of the anilinoacetophenones, was prepd recently by S.G.P.Plant & C.R.Worthing, JCS **1955**,1279 & CA **50**, 2457(1956) and listed as *4-acetyldiphenylamine*

Azidoacetyldiphenylamine, $N_3.C_{14}H_{12}NO$, mw 252.27,N 22.21%. Following isomer is known: *o-Azido-N-acetyl-diphenylamine or 2'-Azido-N-phenylacetanilide*,

$$CH_3.CO.N\begin{matrix}\diagup C_6H_4.N_3\\ \diagdown C_6H_5\end{matrix},$$

pale yel crysts, mp 99-99.5°, decomp thermally to gums. Was prepd by diazotizing o-amino-N-acetyl-diphenylamine in aq HCl and treating the product with Na azide

Refs: 1)Beil–not found 2)P.A.S.Smith et al, JACS **75**,6336(1953) & CA **49**,7571 (1955)

Diazidoacetyldiphenylamine, $(N_3)_2C_{14}H_{11}NO$–not found in Beil or CA through 1956

Mononitroacetyldiphenylamine, $C_{14}H_{12}O_3N_2$. Its N-acetyl-derivs are listed in Beil **12**, [372,391], while the isomers with acetyl on one of the rings are given in Beil **14**, [29,30]. The latter isomers may also be called *nitroanilinoacetophenones*

Dinitroacetyldiphenylamine, $C_{14}H_{11}N_3O_5$. Three N-acetyl-isomers are described in Beil **12**,720,754 & [391,410], while one isomer with acetyl on one of the rings, 2' 4'-dinitro-4-acetyl-diphenylamine, $CH_3.CO.C_6H_5.NH.C_6H_3(NO_2)_2$, is given in Beil **14**,[32]

Trinitroacetyldiphenylamine, $C_{14}H_{10}N_4O_7$, mw 346.25, N 16.18%. No N-acetyl-isomers are listed in Beil **12**, but two isomers with acetyl on one of the rings are given in Beil **14**,[42 & 47]

Tetranitroacetyldiphenylamine, $C_{14}H_9N_5O_9$, mw 391.25, N 17.90%. Following isomer is described in the literature *2,4,2',4'-Tetranitro-N-acetyl-diphenylamine*, called in Ger Essigsäure-bis[2,4-dinitrophenyl]-amid,

$$(O_2N)_2C_6H_3\diagdown N.CO.CH_3,$$
$$(O_2N)_2C_6H_3\diagup$$

crysts, mp 197°. Was obtained by Pictet on treating N-acetyldiphenylamine with diacetyl-orthonitric acid,$(HO)_3N(O.OC.CH_3)_2$

Refs: 1)Beil **12**,754 2)A.Pictet,Arch-SciencPhysNat,Genève,[IV]**16**,201(1903) & ChemZtr **1903** II, 1109

Pentanitroacetyldiphenylamine, $C_{14}H_8N_6O_{11}$–not found in Beil or CA through 1956

Hexanitroacetyldiphenylamine, $C_{14}H_7N_7O_{13}$, mw 481.25, N 20.38%. Following isomer is described in the literature:

2,4,6,2' 4',6'-Hexanitro-N-acetyl-diphenylamine, called in Ger Essigsäure-bis[2,4,6-trinitrophenyl]-amid,

$$(O_2N)_3C_6H_2\diagdown N.CO.CH_3.$$
$$(O_2N)_3C_6H_2\diagup$$

Lt yel crysts, mp 240° with decompn; starts to blacken ca 200°; sol in ben & acet; insol in ligroin. Was prepd by treating silver salt of hexanitrodiphenylamine with acetylchloride. No refs to its expl props

Refs: 1)Beil **12**,767 2)A.Hantzsch & St. Opolski Ber **41**,1747(1908)

N-Acetyldiphenylhydrazine. See N-Acetyl-hydrazobenzene

ACETYLENE AND DERIVATIVES

Acetylene or Ethine (Ethyne), HC:CH, mw 26.04. OB to CO_2 -307.2%, OB to CO -184.3°. Col gas with garlic odor, fr p –85° at 895 mm, subl p –84° at 760 mm, d 0.91 (air =1.0), Q_f -54.9 kcal/mol. Bruni(Ref 19c) gives bp –23° crit temp +35.4° and temp of triple point –83.6°. Soly in w 1.7 vols per 1 vol of w at RT; soly in acet over the temp range of 0° to 40° and at a partial press of C_2H_2

of 1 atm can be calcd from the equation S = 13000/(T-185.3) -81.3 derived by Brameld & Clark (Ref 7). Soly at higher press is much greater

Acetylene gas burns in air with a very hot luminous flame. When burned in oxygen (as in an oxyacetylene flame) temps of the order of 6000°F(3315°) can be attained. According to Reppe (Ref 20g), acetylene tends to decompose explosively into its elements even at a press of the order of 1 atm, evolving appreciable quantities of heat. Compressing acetylene is a dangerous operation unless a special technique is used (as in loading containers for oxyacetylene welding). For purposes of safe storage, acetylene can be dissolved in acet and kept indefinitely

Mixts of gaseous acetylene and air are extremely expl. In dry air at atm press the expl limits are 2.6 to 77%(or even 80%) of acetylene by vol

Numerous explosions have occurred (see CA from 1907 to present) which were attributed to acetylene or to acetylene liberated from Ca carbide

According to Sax (Ref 23) acetylene is sl toxic and its fire hazard is great when exposed to heat or flame. Its expl hazard is moderate when exposed to heat or flame or when it undergoes spontaneous chem reactions. At high press it may decomp explosively even at moderate temps. It can react vigorously with oxidizing materials and it forms expl compds on contact with Cu or Ag(see Acetylides)

The discovery of acetylene in 1836(or 1837) is attributed to Edmond Davy, but it was not until 1860 that Berthelot definitely identified and named it(Ref 9, p 101 & Ref 16, p 469). The compd obtained by Berthelot from cuprous acetylide was not pure because it contained some vinyl chloride. Acetylene was not produced commercially until Ca carbide was produced in the lab in 1899 by Morehead & Willson, by heating a mixt of lime and coke in an electric furnace. They expected to prepare metallic calcium but obtained the carbide instead

Examination of the material prepd by M & W showed that when it was brought into contact with water, a large amt of gas, identified as C_2H_2, evolved. This gave impetus to the coml production of CaC_2 for use in acetylene generators. At first C_2H_2 was used for increasing the illuminating power and heating value of water gas, but since 1906 it has been utilized for welding and cutting steel. The chemical utilization of C_2H_2 began in Germany in 1910, then in Canada in 1914 and finally in this country. Research in the field of acetylene chem was greatly expanded during WW's I & II and the use of C_2H_2 increased tremendously (Ref 9) The industrial prepn of acetylene from Ca carbide is described in Ref 9, pp 102-7 and Ref 24, pp 34-41

Many other methods for the manuf of acetylene have been developed, especially during and after WW II in Germany, such as from hydrocarbons by the Hüls arc-cracking process (Ref 9, pp 107-10), from hydrocarbons by the Wulff thermal cracking process (Ref 9, pp 110-11) and from methane by its partial combustion (Ref 9, pp 111-12)

In addn to the above mentioned processes for the production of acetylene, several others were developed, of which the Tennessee Eastman process (Ref 22) and the Société Belge de l'Azote (SBA)–Kellogg process (Ref 27) are the most recent

Purification of crude acetylene for lab purposes is described in InorgSynth v 2(1946), 76

Uses: In addition to the extensive use of acetylene in oxyacetylene welding it is used as a starting material for the manuf of inorg and org acetylides as well as many other compds. Some of them such as acetone, acetaldehyde, acetic acid, acetic anhydride, etc are indispensible in the manuf and testing of expls and ammo. Acetylene was also used to manufacture tetranitromethane by the method described in PATR 2510 (1958), p Ger 195, under Tetan

Straight acetylene can be used as an explosive when in liquefied or solidified form (see Acetylene as an Explosive) (See also Acetylene Condensation or Polymerization Products, Acetylene Derivatives, Acetylene Hydroperoxides & Peroxides, Acetylene-Nitric Acid Reactions, Acetylene Reactions, Acetylenic Compounds, Acetylides, Cuprene and Halogenated Acetylenes)

Refs: (Acetylene): 1)Beil **1**,228,(100), [202] & [887] 2)M.Berthelot, AnnChimPhys [3]**67**, 67(1863) 3)J.A.Nieuwland & R.R. Vogt, "The Chemistry of Acetylene," Reinhold,NY(1945) 4)P.Piganiol,"Acétylène, Homologues et Dérivées,"Masson,Paris (1945) 5)R.L.Hasche,ChMetEng **52**,No 10, 116-19(1945)(Acetylene industry in wartime Germany) 6a)W.Reppe, "Advances in Acetylene Chemistry"(as developed at the IG Farbenindustrie A-G), PB Rept 1112(CWS IDR No 4149)(about 1946) 6b)W.Reppe, "Synthesis of Intermediates for Polyamids on Acetylene Basis," PB Rept 25553(about 1946) 7)V.Brameld & M.Clark, JSCI **65**, 58-61(1946) & CA **40**, 3670(1946) 8)A.W. Johnson, "The Chemistry of Acetylenic Compounds,"Arnold,London,Vols 1 & 2 (1946 & 1950) 9)Kirk & Othmer 1(1947), 101-121 10)E.D.Bergmann,"The Chemistry of Acetylene and Related Compounds," Interscience,NY(1948) 11)C.J.Herrly, C&EN **27**, 2062(1949)(The acetylene industry in America) 12)W.Reppe,"Neue Entwicklungen auf dem Gebiet der Chemie des Acetylens und Kohlenoxyds,"Springer,Berlin (1949) 13)J.W.Copenhaver & M.H.Bigelow, "Acetylene and Carbon Monoxide Chemistry," Reinhold, NY(1949) 14)J. W. Reppe "Acetylene Chemistry,"Technical Publications,NY(1949)(Translated from the German by C.A.Meyer & Co,Inc)(PB Rept 18852-s) 15)P.Piganiol,"Acetylene,Homologs and Derivatives,"Mapleton House, Brooklyn (1950)(Translated from the French by F.A. Hessel & J.B.Rust) 16)B.T.Brooks, "The Chemistry of Nonbenzoid Hydrocarbons," Reinhold, NY (1950), p 469-488 17)R.Owens & A.W.Johnson,"The Acetylene Industry and Acetylene Chemistry in Germany During the Period 1939-1945,"HMSO,London(1951) 18) W.Reppe, "Chemie und Technik der Acetylen-Druck-Reaktionen," Verlag Chemie, Weinheim(1952) 19a)M.Konschak,Brennstoff-Wärme-Kraft, **4**,62-6(1952)(Explosive properties of acetylene and safety measures for its storage) 19b)O.Nicodemus & K.Winnacker in E.Weingaertner, "Chemische Technologie," München, **3**(1952), 614-73 19c) G.Bruni,Idracarburi **1953**, Nov 9-12 & CA **50**,5350(1956) 20a)Ullmann **3**,(1953)43-68 20b) E.R.H.Jones, "Acetylene Chemistry," Univ of Notre Dame Press, Indiana(1954) 20c)P.Holemann,R.Hasselmann & G.Dix, "Untersuchungen über die thermische Zündung von Explosibelen Azetylenzersetzungen in Kapillaren,"Westdeutscher Verlag, Köln (1954) 20d)R.Duff et al, JChemPhys **22**, 1618-19(1954) & CA **49**,617(1955)(Studies of detonation of pure acetylene gas in tubes) 20e)W.W.Robertson et al, "Self Combustion of Acetylene," in the "5th Symposium on Combustion",Reinhold,NY(1955), 628-33(9 refs) 20f)E.A.Westbrock et al, "Seif Combustion of Acetylene," in the "5th Symp on Combstn" (1955), 631-37(17 refs) 20g)W.Reppe et al, Ann **596**,6(1955) 21a)R.A.Raphael,"Acetylenic Compounds in Organic Synthesis," Academic Press, NY(1955) 21b)Anon, Explosivst **1956**, 101(Directions for avoiding accidents in handling acetylene) 21c) Forschungsstelle für Acetylen,Dortmund, Explosivst **1956**,141(Investigation of transformations in the explosive decomposition of acetylene) 21d)J.L.Romig et al, Explosivst **1956**,218(Investigation of the course of decomposition of acetonic solns of C_2H_2) 21e)Trzeciak, Explosivst **1956**,219(Sources of danger in the generation of acetylene) 21f)A.Ebert,Explosivst **1956**,245-8(On the prevention of acetylene explosions) 22) Anon,C&EN **35**,32 (Dec 23,1957)[New process for prepn of C_2H_2 designed by Tennessee Eastman Co is briefly described. It involves high temp breakdown of satd hydrocarbons (such as natural gas) conducted in a special furnace (constructed of stabilized zirconium)

which is capable of withstanding temps up to 2500°C. The process is different from any previously used] 23)Sax(1957), 237 24)Faith, Keyes & Clark(1957),34-41 25)F.C.Stehling et al, "Carbon Formation from Acetylene." A paper reported in the 6th Symposium on Combustion, Reinhold, NY(1957),247-54(22 refs) 26)H.H.Nelson, "The Effect of Pipe Diameter on the Thermal Decomposition of Acetylenes." A paper reported in the 6th Symp on Combstn,Reinhold NY(1957),823-27(17 refs) 27)SBA-Kellogg Way to Acetylene, C&EN **36**,15(Jan 13, 1958) [Brief description of the manuf of C_2H_2 from natural gas or naphtha by the process patented by the Société Belge de l' Azote(SBA) using a special burner. This process was adopted in USA by the M.W.Kellogg Co, NY. Ethylene can also be produced by this method] 28) Many papers on acetylene and acetylene chemistry are listed in Chemical Abstracts, especially starting about 1940. There are listed above only the more important papers

Acetylene-Air Mixtures. Various C_2H_2–air mixtures were detonated in rubber balloons by means of central elec detonators. Photographs of spherical explosions thus produced were made with a rotating-drum camera and with a 6000-frame-per-sec movie camera. Deton vels measured by this method agreed with values obtained from measurements in tubes by std techniques. For instance, the mixts contg 12.5% C_2H_2 developed a deton vel of 1920 m/sec and multiple reflected waves were clearly observed (See also Acetylene-Oxygen Mixtures)

Refs: 1)H.Freiwald & H.Ude, CR **236**, 1741-3(1953) & CA **47**,9617(1953) 2)Ibid, ZElektrochem **57**,629-32(1953) & CA **49**, 8602(1955)

Acetylene,Analysis. See Acetylene and Derivatives Analysis, listed after Acetylene Derivatives

Acetylene as an Explosive. When acetylene is liquified it becomes an expl which can be detonated by a blow, spark or a detonator. The same applies to acetylene in the solid state. According to Rimarski & Metz (Ref 3), solid acetylene is an expl of considerable power as detnd by the Trauzl lead block test, although less powerful than common HE's. Its brisance and deton vel (2270 m/sec at d 0.503) are also inferior to the common HE's. Sensitivity to heat, friction, shock and spark are slight. By using solid C_2H_2 with liq air or oxygen, a very brisant expl is obtained. The disadvantages of using solid C_2H_2 lie in the difficulties in storage and transportation

Acetylene may also be used as an expl when in the gaseous state, but for this use it must be previously mixed with some oxygen-contg gas, such as air to create an expl mixt. For instance, gaseous acetylene is used as a blasting expl in cases where materials are desired to be reduced to large pieces (Ref 1). In one application a lead pipe was separated into 3 sections by two light, easily broken partitions. The first sectn (next to the tamping) was charged with CaC_2 in small grains, the second sectn contained water and the third an electric exploder. After inserting the pipe into a borehole and tamping, the first partition was broken from outside by an iron bar thus allowing an influx of air and causing the CaC_2 and H_2O to form acetylene. The resulting press broke the 2nd partition bringing the C_2H_2-air mixt in contact with the exploder which, after a suitable time had elapsed caused the mixt to detonate

According to British regulations issued during WW I, acetylene when liq or when subject to a certain degree of compression, or when in admixture with atm air or oxygen, was deemed an expl (Ref 2)

Stettbacher (Ref 4) gives the following expl props for 74.75/24.55 mixts of liq O_2 with solid C_2H_2: maxim d less than 1, normal gas vol 634 l/kg, heat of expln 2760 kcal/kg, deton vel 6000 m/sec, maxim temp of expl (calcd) 7280° and sensitivity to impact 5 cm with 2 kg wt

The expl decompn of acetylene is discussed in Ref 5 and the effect of mixing with hydrocarbons and other gases upon the explosibility of C_2H_2 in Ref 6 (For more information on explosions of acetylene and its homologs, consult CA under Acetylene)

Refs: 1)Anon, Sprengstoffe, Waffen und Munition **9**,41(1914) & CA **9**, 1115(1915) 2)British Statutory Rules and Orders 1919, No 809, "Acetylene as an Explosive"& CA **14**,468(1920) 3)W.Rimarski& L.Metz, Autogene Metallarbeit, **26**,341(1933); Chem Ztr **1934**,I,803-4 & CA **29**,4942(1935) 4)A. Stettbacher, Protar **8**,91(1942) & CA **37**,1603 (1943) 5)E.A.Blyumberg & D.A.Frank-Kamenetskii, ZhFisKhim **20**,1301-17(1946) & CA **41**,2969(1947) 6)G.W.Jones et al, US BurMinesReptInvest **4196**(1948) & CA **42**, 1739-40(1948)

Acetylene Black. See under Carbon Blacks

Acetylene Chloride or Chloroethyne. See under Halogenated Acetylenes

Acetylene Condensation and Polymerization Products may be obtained by subjecting acetylene to the action of heat, light, electrons, alpha-rays, elec discharge, etc with or without catalysts (Ref 1, {892-3}).One such products is **cuprene**(qv), which is a condensation product of acetylene and not a polymer as it is usually called. The real polymerization product of acetylene is C_6H_6, which was obtained (together with other compounds) in 1866 by Berthelot by heating acetylene in retorts of glass softening at 400-500°(Ref 1,p 232). The same investigator, prepd by silent electrical discharge in acetylene some unidentified products of high mol wt which decompd explosively during their distn (Ref 1, p 232)

Wohl (Ref 2) proposed to use the condensation products obtained from C_2H_2(either by the action of heat or by the silent discharge in the presence of Cu_2O) as comburents for various blasting expls, such as those based on black powder, NG, AN, Amm perchlorate, liq oxygen, etc

Systematic studies of acetylene polymerization were conducted in the laboratories of the duPont Co and the results are described in numerous papers (see Ref 3). Studies of acetylenic polymers from the point of view of their utilization in solid rocket propellants has been conducted by Reaction Motors (see Ref 10). Polymerization under press is described in Ref 4 and some industrial products obtained by polymerizing acetylene are listed in Ref 5

Shimizuya & Kimura(Ref 6) proposed a smoke-producing mixt contg as the principal ingredient a product obtained by treating C_2H_2 polymer (consisting mainly of divinylacetylene) with 5-6 atoms of chlorine at 60-70°. Other ingredients of the smoke mixt are Al, $KClO_3$ and kieselguhr

According to Saito(Ref 7), the acetylene trimers obtained as byproducts in the prepn of CH_2:CH.C:CH by condensation of acetylene are expl. They can be stabilized by hydrochlorination in the presence of a complex salt of CuCl and NH_4Cl to yield additive compds contg 1 or 2 mols of HCl, from which they are separated by distn. Nakagawa(Ref 8) reviews the chemistry of polyacetylenes and gives 25 references. Polymerization of acetylene is also discussed in Ref 9 (See also Cuprene)

Refs: 1)Beil **1**,232,(101),[211] & {892-5} 2)A.Wohl, BritP 145,258 & 145,597(1920) & CA **14**,3533(1920) 3)J.A.Nieuwland, W.H. Carothers and others, "Acetylene Polymers and Their Derivatives," series of articles in JACS **53**,4197-4225(1931); **54**,4066-76 (1932); **55**,786-95, 1094-1101, 1622-31, 2004-12, 2040-51, 2807-17, 4665-70 & 5077(1933); **56**,1167-70, 1625-8 (1934); **57**,1978-84, 2255-59 & 2739(1935); **58**,1747-49(1936) 4)H.W. Starkweather,"Polymerization under High Pressure;"JACS **56**, 1870-74(1934) 5)M. Piganiol,"New Industrial Acetylene Polymerization Derivatives," BullFr **9**,749-58 (1942) & CA **38**,3248(1944) 6)N.Shimizuya & T.Kimura, JapanP 175,984(1948) & CA

45,5341(1951) 7)H.Saito et al, Japan P 172,910(1946) & CA 46,5072(1952) 8)B.T. Brooks, "The Chemistry of Nonbenzoid Hydrocarbons," Reinhold, NY(1950), 474 8a) M.Nakagawa, Kagaku(Japan) 10,658-65 (1955) & CA 49,15721(1955) 9)R.A.Raphael, "Acetylenic Compounds in Organic Synthesis," Academic Press,NY(1955), 105,152 & 159-60 10)Reaction Motors Division of Thiocol Chemical Corp, Denville, NJ, Report RMD-157F(Final Rept) by G.Golub & D.Perry, "Acetylenic Polymers for Solid Propellants" (1959).Contract NOrd 17851(Conf) (Was not used as a source of info)

Acetylenecyanide or Acetylenenitrile. See Cyanoacetylene

Acetylene Derivatives. Under this term are listed compds obtained by substituting the hydrogen atoms of acetylene or its homologs with metals, with halogens or with organic or inorganic radicals. In case of substitution with metals, the compds are called **acetylides** (qv), whereas the other derivs are usually called **acetylenic compounds**

Following examples of acetylenic compds, some of which are expls, were isolated by Italian investigators from mixts obtained by passing acetylene into fuming nitric acid (d 1.52): a)Straw-yel expl substance melting at 78° without decompn; when crystd from benz or petrol eth, copious evoln of nitrous fumes occur and transparent col crysts of a neutral compd, $C_4H_2N_4O_3$, melting at 108°, separates b)Mono-basic acid, $C_4H_3NO_3$ sepg from toluene in large lt yel crysts melting at 149°, form a stable silver salt which expl mildly at 165° c)Diazoimide $C_3H_2N_4O$, a refractive oil explg with great violence on heating

Refs: 1)G.Testoni & L.Mascarelli, Atti Reale Accad Lincei [V]10 I, 442-4(1901); JCS 80 I, 494-5(1901); Gazz 31, 461(1901) & 33 II, 319(1903) 2)A.Quilico & M.Freri, Gazz 60,172-84(1930) & CA 24,3789 (1930)

Note: More refs on acetylene derivatives are given under Acetylene–Nitric Acid Reaction Studies

Acetylene, Acetylenic Compounds and Acetylides; Analytical Procedures.

Hydrogen atoms adjacent to the triple bond of an acetylenic compd are easily replaced by silver, cuprous or mercurous atoms and the resulting metallic derivs are usually insol in w

Following methods are described by Siggia (Ref 7a, pp 48-58)

A)Ammonical silver nitrate method, in which the excess silver is detd volumetrically by 0.1N NH_4CNS soln. Ppt of AgC:CR which forms during this reaction is expl and should be destroyed as described under Silver Acetylide

B)Alcoholic silver nitrate method in which the following reaction takes place: $2AgNO_3 + HC{:}CR \rightarrow AgC{:}CR.AgNO_3 + HNO_3$, the liberated HNO_3 is titrated by std alkali. This method is especially useful for H_2O-insol samples but it can also be used for H_2O-sol samples

C)Ammoniacal silver nitrate method which is applicable to samples contg aldehydes; aldehydes are serious interferences in methods A & B because they reduce the silver ions present

D)Cuprous method, described in Ref 7a, pp 57-8, involves reaction of cuprous chloride with the acetylenic compd in a pyridine soln, according to the equation: $Cu_2Cl_2 + 2HC{:}CR \rightarrow 2CuC{:}CR + 2HCl$. The liberated HCl is titrated with std alkali. This method is not as accurate as silver methods, but it is applicable to samples with which silver cannot be used because of interfering reactions

Piganiol(Ref 8) also describes silver nitrate and cuprous chloride methods, as well as the following additional methods:

E)Potassium iodo-mercurate reagent, prepd by dissolving 66 g $HgCl_2$ and 163 g KI in 160 ml w and adding 125 ml of 10% NaOH soln. Acetylenes give, in alc, ppts $(RC{:}C)_2Hg$ whose mp's are characteristic. For instance, $(CH_3{\cdot}C{:}C)_2Hg$ has mp 203-4°, whereas $(C_2H_5{\cdot}C{:}C)_2Hg$ has mp 162-3°. This method permits detn in some cases of the structure of pptd compd, as does the Raman spectra method

F)Raman spectra method is the only physical method used at present to detect an acetylene compd in a complex mixt. The method is not as rapid as chemical methods, but it gives more valuable info about the entire structure of the molecule (Ref 8)

For detn of acetylene in air or other gases the following procedures can be used:
a) For small concn of acetylenes (up to ca 2%), the measured vol of gas is passed through a cuprous chloride or silver nitrate reagent until an appreciable amt of ppt forms. An apparatus of the Orsat type can be used (Ref 8)
b) For high concns of acetylenes, the gas can be passed through concd H_2SO_4 using the Orsat apparatus. Olefins are also absorbed. If silver nitrate soln is used for absorption of gas, olefins do not interfere but ammonia and carbon disulfide do (Ref 8)
c)Fractionation methods in columns of Podbielniak or McMillan type, operating at slightly above atm press to eliminate the freezing of acetylene in the column (Refs 4 & 7)

In detn of disubstituted acetylenic compds, formation of ppts is observed only when using mercuric salts (chloride, sulfate or nitrate), but this reaction is not specific because some ethylenic compds and compds contg certain oxygen and nitrogen groups also give ppts. This method,however, can be used in conjunction with the Raman spectra method (Ref 8)

According to Piganiol(Ref 8), the problem of detection of various acetylenic compds in a mixt is fairly complicated and must be solved individually for each particular case. Sometimes several methods must be tried before the problem is solved

Following methods may be tried for solving each problem:
a) Detn of carbon and hydrogen atoms by combustion
b)Absorption of C_2H_2 contg gas by 80% sulfuric acid
c) Measurement of d of mixt
d) Use of mercuric cyanide for absorption of some ingredients of mixt, such as divinyl acetylene and tetramers and detn of the amt of carbon in residue

Analysis of acetylenes for impurities is briefly discussed in Kirk & Othmer (Ref 7)

Refs: 1)Beil **1**,237-8,(103-4),[216-17] & {908-9} 2)E.Berl in Berl-Lunge, "Chemisch-Technische Untersuchungen," Berlin **1**(1931), 649; **3**(1932),707 & 722 & U.Stolzenberg, Erg-Bd 2(1939)112 & 121 3)C.Coulson-Smith & A.P.Seyfang, Anal **67**,39-41(1942)(Colorimetric method for estimation of small quantities of acetylene in air) 4)H.P.McKoon & H.D.Eddy, IEC, Anal Ed **18**,133(1946)(Detn of traces of acetylene) 5)C.D.Wagner et al, Ibid **19**,103(1947)(Detn of mono- and dialkylacetylenes) 6)T.A.Geisman et al, Ibid **19**,919-21(1947)(Detn of traces of acetylene in air) 7)Kirk & Othmer **1**,(1947) 114-16 7a)S.Siggia, "Quantitative Organic Analysis Via Functional Groups," Wiley, NY (1949) 8)P.Piganiol, "Acetylene Homologues, and Derivatives," Mapleton House, Brooklyn (1950)(Translated from the French by F.A. Hessel & J.B.Rust), 276-9(Numerous references are given) 9)I.Marszak & M.Koulkés MSCE **36**,421-6(1951)(Detg the true C_2H_2 functioning group by using Ag benzoate) 10)M.Koulkés & I. Marszak, Bull Fr [V]. **19**, 556-7(1952) & CA **46**,10050(1952)(Detg the true C_2H_2 functioning group by using the $AgNO_3$–C_2H_4 diamine complex) 11)Ullmann **3**(1953),59 12)Org Analysis, v 2(1954), 40 & v3(1956), 329

Acetylenedicarboxamide. See under acetylenedicarboxylic Acid and Derivatives

Acetylenedicarboxanilide. See Bis(carboxanilide)acetylene, also called Di(N-phenylcarboxamide)-acetylene

ACETYLENEDICARBOXYLIC ACID AND DERIVATIVES

Acetylenedicarboxylic Acid (Acetylendicarbonsäure or Butindisäure, in Ger; Acide Acetylenedicarboxylique, in Fr), $HO_2C.C{:}C.CO_2H$, mw 114.06, OB to CO_2 −70.1%, OB to CO −14.0%. Plates, mp 178-80°(decomp). Very sol in w, alc and eth;

crysts from solns as a dihydrate. May be prepd by the method of Baeyer from α, α'-dibromosuccinic acid and alc KOH (Ref 3). A modified version of the prepn is given in Refs 4 & 5. Gilman & Haubein(Ref 6) prepd it by stirring vinyl bromide and n-butyllithium in ether for 15 mins and carbonating the resulting milky mixt with dry ice

Its **silver** salt detonates violently from heat or shock as was found by Bandrowski (Ref 2). This property is not mentioned in Beil, although the salt is listed(Ref 1, 2, 802)

Refs: 1)Beil **2**,801,(317) & [670] 2)E.Bandrowski, Ber **10**,841(1877) 3)A.Baeyer, Ber **18**,677-8(1885) 4)H.J.Backer & J.M.Van der Zanden, Rec **47**,778(1928) 5)OrgSynth, CollVol **2**(1943),10 6)H.Gilman & A.H. Haubein, JACS **67**,1421(1945)

Acetylenedicarboxamide or Bis(carboxamide)-acetylene; $H_2N.OC.C{\equiv}C.CO.NH_2$, mw 120.09, N24.99%. Microcrystallinic powder, mp-dec at 294°; sparingly sol in w, alc, eth, chlf & AcOH. Was prepd by treating dimethylacethylenedicarboxylate with ammonia at 10°

Refs: 1)Beil **2**,(317) 2)Ch.Moureau & J. Bongrand, AnnChim(Paris), [9], **14**,12(1920)

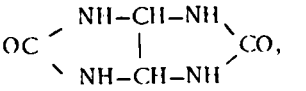
Mononitroacetylenedicarboxamide, (O_2N)-$HN:CO.C{\equiv}C.CO.NH_2$–not found in Beil or CA through 1956

Dinitroacetylenedicarboxamide, $(O_2N).HN.CO.C{\equiv}C.CO.NH(NO_2)$–not found in Beil or CA through 1956

Acetylenedichloride. See under Halogenated Acetylenes

ACETYLENEDIUREIN OR GLYCOLURIL AND DERIVATIVES

Acetylenediurein, Glycoluril or Tetrahydroimidaz[d]imidazole-2,5(1H,3H)-dione (Acetylenediureide; Glyoxaldiureide)(Acetylene diurée, in Fr) {Acetylendiurein, Glyoxaldiurein; α.β; α.β–Diureylen-äthan; Dioxo-hexahydro-[imidazolo-4'.5': 4.5–imidazol]; Glykoluril or Acetylenharnstoff, in Ger},



mw 142.12, N39.43%, OB to CO_2 -101.3%, OB to CO -56.3%. Wh ndls, mp 297° with decompn, decrepitates on heating above mp; Q_c^v 465.4 kcal/mol, Q_c^p 464.5, Q_f^v +115.2 and Q_f^p +118.6 kcal/mol(Ref 5, p 83) It is sl sol in w and nearly insol in ord solvents. Was prepd in 1877 nearly simultaneously by Schiff(Ref 2) and by Böttinger(Ref 3) from glyoxal and urea. Several other methods are listed in Ref 1. Biltz & Schiemann(Ref 4) prepd it by heating a mixture of glyoxal, Na bisulfite and urea in aq HCl. The compd was suggested as an ingredient of propellants (Ref 5)

Its **silver salt,** $Ag_2C_4H_4N_4O_2$ is explosive (Ref 1, p 442)

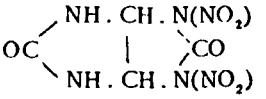
Note: Numbering of acetylenediurein ring includes three different systems: a)Beilstein system b)CA system prior to 1927 and c)CA system since 1927. We are using system c

Refs: 1)Beil **26**,441-2,(131) & [260] 2)H. Schiff, Ann **189**,157(1877) 3)C.Böttinger, Ber **10**,1923(1877) 4)H.Biltz & G.Schiemann, JPraktChem **113**,98(1926) 5)P.Tavernier & M.Lamouroux, MP **38**,67-8 & 83(1956)

Azidoacetylenediurein, $C_5H_5N_7O_2$–not found in Beil or CA through 1956

Mononitroacetylenediurein, $C_4H_5N_5O_4$–not found in Beil or CA through 1956

Dinitroacetylenediurein or Dinitroglycoluril, $C_4H_4N_6O_6$, mw 232.12, N36.21%, OB to CO_2 -27.6%, OB to CO = 0%. One isomer to which Franchimont & Klobbie assigned the structure,



is known. It is a wh pdr which deflagrates at 217° without melting. It was obtained by treating 1 part of acetylene diureine with 5 parts of abs nitric acid. The structure of this compd was not definitely established.

It decompd on heating with w but was not attacked by aq ammonia (Refs 1 & 3)

Another dinitro-compd, called by Franchimont & Klobbie **isodinitroglycolurile**, wh microscop crysts, insol in ordinary org solvents and sol in concd nitric acid was also obtained from acetylenediurein and absol nitric acid. It was distinguished from the first isomer by the fact that the latter was not decomp on heating with w but was decompd by aq ammonia at RT (Refs 1 & 3)

Refs: 1) Beil **26**, 443 2) A. Franchimont & E. Klobbie, Rec **7**, 18-19 & 246(1888) 3) Ibid, **8**, 290-1(1889)

Note: No later refs were found in CA through 1956

Trinitroacetylenediurein, $C_4H_3N_7O_8$ and *Tetranitroacetylenediurein*, $C_4H_2N_8O_{10}$—were not found in Beil or CA through 1956

Acetylene, Halogenated. See Halogenated Acetylenes

ACETYLENE HYDROPEROXIDES AND PEROXIDES

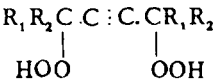
A series of compds which contain both acetylene bonds and peroxide groups, was synthesized by Milas et al by using a modification of the sulfuric acid-hydrogen peroxide method originally described in Refs 1 & 2. The procedure consists essentially in allowing an acetylenecarbinol, $R_1.R_2.C(OH).C:CH$ or glycol $R_1.R_2.C(OH).C:C(OH).R_1.R_2$, in which R_1 & R_2 are various hydrocarbon radicals, to react at low temp with hydrogen peroxide in the presence of sulfuric acid of suitable strength

Following types of compds were prepd:

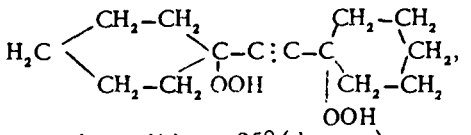
A) **Acetylene hydroperoxides,** $R_1R_2.C(OOH).C:C$,

such as **1,1-dimethyl-2-propynylhydroperoxide or 3-methyl-3-hydroperoxy-1-butyne,** $(CH_3)_2C(OOH).C:CH$, oxygen 16.0%. Liq, bp 42 to 52.2° at 17 mm, d 0.9540 at 25° and $n_D^{25°}$ 1.4295; **1,1-diethyl-2-propynylhydroperoxide or 3-methyl-3-hydroperoxy-1-pentyne,** $(C_2H_5)_2C(OOH).C:CH$, oxygen 14.0%. Liq, bp 38-40° at 5 mm, d 0.9547 at 25°/4°, $n_D^{25°}$ 1.4369

B) **Acetylene dihydroperoxides,**

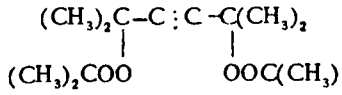


such as **1,1,4,4-tetramethyl-2-butynylene-dihydroperoxide or 2,5-dimethyl-2,5-dihydroperoxy-3-hexyne,** $(CH_3)_2C(OOH).C:C(HOO)C(CH_3)_2$, oxygen 18.4%. Solid, mp 107-9° (decomp); **1,1'-dihydroperoxy-1,1'-dicyclohexylacetylene,**



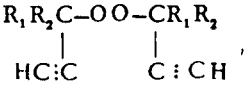
oxygen 12.6%; solid, mp 95° (decomp)

2,5-dimethyl-2,5-di-(t-butylperoxy)-3-hexyne,

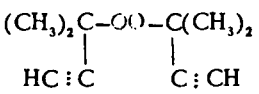


Liq, bp 65-7° at 2 mm, d 0.881 at 25°, $n_D^{25°}$ 1.4219

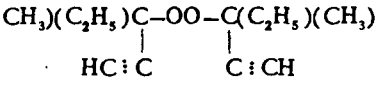
C) **Dialkynyl peroxides,**



such as **bis(1,1-dimethyl-2-propynyl)-peroxide or di-(3-methylbutynyl)-3-peroxide,**



Liq, bp 60° at 76 mm; **bis(1-methyl-1-ethyl-2-propynyl)peroxide or di-(3-methylpentynyl)-3-peroxide,**



Liq, bp 53-55° at 2 mm, d 0.9030 at 25°/4° and $n_D^{25°}$ 1.4390

These peroxides are stable but they can be detonated by means of a blasting cap

Refs: 1) N. A. Milas, USP 2,223807(1940) & CA **35**, 1802(1941) 2) N. A. Milas & D. M. Surgenor, J ACS **68**, 206-7(1946) 3) N. A. Milas

& O.L.Mageli, JACS **74**,1471-3(1952) & CA **48**,545(1954) 4)Kirk & Othmer **10**(1953), 68-9

Acetylene, Manufacture of Tetranitromethane, From. See PATR 2510(1958), p Ger 195, unde Tetan

Acetylene-Nitric Acid Reactions Studies. Reactions between acetylene and nitric acid were studied in Italy as early as 1901 by Baschieri,(Gazz **31** II,462), in 1902 by Testoni & Mascarelli and in 1903 by Mascarelli

A.Quilico, M.Freri and other investigators found that the earlier work was incomplete and questionable and for this reason they repeated some of the earlier work and published a series of papers in Gazz, beginning in 1929(vol 59). The products which they examined, many of them explosive, were prepd by bubbling a slow current of purified acetylene through fuming nitric acid. Following is a selected list of papers and the names of the expl compds prepd by Q & F:

a)Gazz **59**,930-41(1929) & CA **24**,3484(1930) (An expl compd presumably 5-isoxazolecarboxylic acid. This compd was reexamined in 1942 and the results were published in Ref m)

b)Gazz **60**,721-44(1930) & CA **25**,1247(1931) (An expl compd, $C_4H_2N_6O_7$, which was not identified)(See Ref h)

c)Gazz **61**,484-500(1931) & CA **26**,454(1932) (The constitution of an expl compd, $C_4H_2N_6O_7$, was partially established). There was also prepd an expl oil heavier than water, $C_3H_2ON_4$, bp 147° at 155 mm

d)Gazz **61**,759-72(1931) & CA **26**,1606 (1932)(The structure of a previously prepd expl oil, $C_3H_2ON_4$, was partly established)

e)Gazz **61**,970-6(1931) & CA **26**,2978(1932) (Prepn and some props of α-isoxazoleamine hydrochloride and diazoaminoisoxazole)

f)Gazz**62**,436-44(1932) & CA **26**, 5561 (1932)(Monosubstituted derivs of α-isoxazolecarboxylic acid, etc)

g)M.Freri,Gazz **62**,457-63(1932) & CA **26**, 5952(1932)(Some expl derivs of α-**izoxazolecarboxylic acids and of α-methylisoxazolecarboxylic acid**

h)Gazz **62**,503-18(1932) & CA **62**,5953-4 (1932)(The structure of a previously prepd expl compd, $C_4H_2N_6O_7$, was established as $\overline{ON{:}CH.CH{:}CN{:}N.C(NO_2)_3}$. The compd was named **α-isoxazoleazotrinitromethane.** Benzeneazotrinitromethane and its nitrocompd **p-nitrobenzeneazotrinitromethane** were also prepd

i)Gazz **62**,912-27(1932) & CA **27**,1348(1933) (More info on benzeneazotrinitromethane, p-nitrobenzeneazotrinitromethane. Prepn an props of other expl compds are given, such a **β-naphthylazotrinitromethane** and **p,p'-biphenylenebisazotrinitromethane)**

j)Gazz **65**,1203-13(1935) & CA **30**,5219-21 (1936)(Prepn and establishment of structure of eulite. It decompd explosively when heated above its mp, 102.8°)

k)Gazz **66**,278-99(1936) & CA **31**,1805(1937) (Prepn of the **mercuric salt of eulite,** which exploded violently on heating)

l)Gazz **71**,327-42(1941) & CA **36**,771(1942) (Nitro, amino and diazo derivs of isoxazole

m)Gazz **72**,458-74(1942) & CA **39**,2753-4 (1945)(By means of a synthesis of isoxazol derivs with fulminic acid, it was establish that the structure of the acid reported in Ref a is incorrect. It should be 3-isoxazole carboxylic acid)

n)Gazz **76**,3-29(1946) & CA **41**,380-2(1947) (Further investign of the compd described in Refs a and m. Prepn of some derivs of furazan. Silver salts of **4-(3-isoxazolyl)-3-furazancarboxylic acid** and of **3-furazanace 4-carboxylic acid** are explosive)

o)Gazz **76**,30-43, 87-107, 195-9(1946) & CA **41**,382-6(1947)(Further work on eulite)

Acetylenenitrile. See Cyanoacetylene

Acetylene-Oxygen and Acetylene-Air Mixt were examined in Japan and the results ar reported in the following refs:

1)R.Kiama et al, RevPhysChemJapan **23**, 43-8(1953) & CA **48**,8544(1954) (A mixt of 54% C_2H_2 and 46% O, initially at 270° and 10.9 atm, exploded on being compressed

rapidly(0.7sec) to 56.1 atm. No expln was observed with mixts of C_2H_2-air treated under similar conditions) 2)R.Kiyama et al, RevPhysChemJapan **24**,41-8(1954) & CA **49**,12006(1955)(Mixts of C_2H_2 and O or air at 10 kg/cm^2 were rapidly introduced into a heated vessel and the occurrence or nonoccurrence of an expln was noted. The min expln temp for C_2H_2-O mixts was 220-30° and for C_2H_2-air mixts ca 390°. Addn of a small amt of CCl_4 elevated the temp of expln about 25°, while addn of H_2O vapor elevated the temp even more. In both cases the resulting explns were more severe than without the addns) 3)H.Teranishi, Rev PhysChemJapan **25**,58-63(1955) & CA **50** 8207(1956)(Previous work on the expln of mixts of C_2H_2 with O and air at 10 kg/cm^2 press was continued. Explns of C_2H_2-air mixts were less violent than corresponding C_2H_2-O mixts. The addn of small amts of H_2O to C_2H_2 mixts increased the temp of expln while addn of small amts of methanol increased the temp only for a C_2H_2/O ratio greater than 2. N retarded the propagation of expln and an increase in the press of either the air or the O mixts decreased the temp required for spontaneous expln)

Acetylene Ozonide. Briner & Wunenburger (Ref 1) reported that the action of ozone on acetylene carried out in a gaseous phase, resulted in expln, but few crysts of ozonide were obtained when reaction was carried out in soln and at low temps. These crysts could not be properly investigated because they exploded violently a short time after their prepn. Hurd & Christ(Ref 2) conducted ozonization of some acetylene derivs with 5-10% solns of ozone in chlf. Jacobs (Ref 3) conducted ozonization of some disubstituted acetylenes. Dallwigk et al (Ref 4) detd infrared spectra of ozonides of some acetylene derivs

Refs: 1)E.Briner & R.Wunenburger, Helv **12**,786-90(1929) & CA **23**, 5156(1929) 2) C.D.Hurd & R.E.Christ JOC **1**,141(1936) 3)T.L.Jacobs, JACS **58**,2272-3(1936) 4) E.Dallwigk, et al, Helv **35**,1377-84(1952) & CA **46**,8965(1952)

Acetylene Peroxides. See Acetylene Hydroperoxides and Peroxides

Acetylene Reactions are described in the following references:
1)Beil **1**,232-37,(101-3), [211-16] & [892-907] 2)J.A.Nieuwland & R.R.Vogt, "The Chemistry of Acetylene," Reinhold,NY(1945) 3)A.W.Johnson, "The Chemistry of Acetylenic Compounds," Arnold,London, **1**(1946) & **2**(1950) 4)Kirk & Othmer **1**(1947), 101-2 5)E.D.Bergmann,"The Chemistry of Acetylene and Related Compounds," Interscience, NY(1948) 6)J.W.Copenhaver & M.H.Bigelow,"Acetylene and Carbon Monoxide Chemistry," Reinhold,NY(1949) 7)C. Gardner, J.D.Rose, B.Weedon, J.B.Batty, R.A.Gale and others made a special study of acetylene reactions and the results of their work were published in JCS **1949**,780-796 8)P.Piganiol, "Acetylene, Homologs and Derivatives," Mapleton House, Brooklyn (1950)(Translated from the French) 9)B.T. Brooks,"The Chemistry of Nonbenzoid Hydrocarbons,"Reinhold, NY(1950), 474-88

Acetylene Tetrachloride. A misnomer for 1,1,2,2-Tetrachloroethane

Acetylene Tetraurethane. See Ethane Tetraurethane

Acetylenic Compounds are organic compounds contg at least one triple bond -C≡C-. They may be hydrocarbons, alcohols, acids, aldehydes, etc. The acetylenic hydrocarbons include, in addition to acetylene (qv), the higher members, such as allylene or propyne $H_3C.C{\equiv}CH$, crotonylene or butyne-2 $H_3C.C{\equiv}C.CH_3$, valerylene or pentyne-2 $H_5C_2.C{\equiv}C.CH_3$, etc

Considerable research on acetylenic compds was conducted before and during WW II in Germany, especially by W.Reppe et al. In more recent years, research on acetylenic compds was conducted by a group of investigators in Gt Britain: K. Bowden, E.R.Jones, Sir Ian Helbron,

E.J.Haynes, B.C.Weedon, E.A.Braude, F.Sondheimer, M.C.Whiting, H.B.Henbest, T.Y.Shen, E.M.Fowler, G.Eglington, etc. Results of their work were described in numerous papers published in JCS, beginning in 1946. Many acetylenic compds are described in refs listed under Acetylene and Acetylides

Following are some additional references on acetylenic compounds which might be of interest as explosives:
1)C.L.Leese & R.A.Raphael, JCS **1950**, 2729, & CA **45**,3324(1951)(In the course of synthesizing long-chain aliphatic acids from acetylenic compounds, some expl substances were obtained, eg, **methylnonylthiuronium picrate**, $C_{17}H_{27}N_5O_7S$, N 15.7%, mp > 300°, detonated violently on rapid heating 2) J.B.Armitage et al, CA **47**,1034(1953)(In the prepn of monosubstituted derivs of diacetylene, a small quantity of an expl product corresponding to the formula:

$H_2C:CH-CH_2.C:C.C:CH$

was obtained. It was a liquid, bp 42° at 150 mm, $n_D^{19°}$ 1.4038 3) T. Herbertz, Ber **85**,475-82(1952) & CA **47**,1574(1953)(In the syntheses of acetylenic compds, starting with diacetylene, some expl substances were obtained, eg, $ClCH_2C:CCH_2Cl$, liq, bp 110° at 5-6 mm, expl decompn ca 120°

Acetylenic Derivatives. See Acetylene Derivatives

Acetylenic Polymers for Solid Propellants. Title of a series of the Reaction Motors Inc Reports, Project 157. Contract NOrd, 17851 (1957)(Conf) (Were not used as sources of info)

Acetylglycine, Azide (Azidoacetylglycine or Triazoacetylglycine), $CH_3.CO.N(N_3).CH_2.COOH$, mw 158.12, N 35.44%. Explosive, undistillable oil. Was prepd from glycine and azidoacetic acid
Refs: 1)Beil–not found 2)K.Freudenberg & R.Keller, Ber **71B**, 334(1938) & CA **32**,2905(1938)

1-Acetylhexahydro-3,5-dinitro-s-triazine. See 1-Aceto-3,5-dinitro-triazacyclohexane under Acetotriazacyclohexane and Derivatives

N-Acetylhexahydrodiphenylamine. See N-Cyclohexylacetanilide

Acetylhexahydrotriazine. See Acetotriazacyclohexane

ACETYLHYDRAZOBENZENE AND DERIVATIVES

N-Acetylhydrazobenzene or N-Acetyl-N,N'-diphenylhydrazine(Acetic 1,2-diphenylhydrazide),

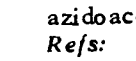
$$C_6H_5.N(COCH_3).NH.C_6H_5,$$

mw 226.27, N 12.38%, OB to CO_2–240.4%. Ndls (from hot alc), mp 159°, Qc 1792.9 kcal/mol(Ref 3); insol in w & alkalies, sl sol in alc & eth. Was prepd from hydrazobenzene and acetic anhydride(Ref 2)
Refs: 1)Beil **15**,244,(64) & [93] 2)D. Stern, Ber **17**,380(1884) 3)A.Pongratz,et al, Ber **77**,651-4(1944) & CA **40**,6068(1946)

Azido-Diazido- and Nitrated Acetylhydrazobenzenes–were not found in Beil or CA through 1956

ACETYLIDES AND CARBIDES (INORGANIC)

Acetylides (Acetylene Derivatives, Inorganic) (Acetylenide or Carbide, in Ger). Acetylides are compds obtained by replacement of one or two hydrogen atoms of acetylene or its homologs or derivatives by a metal. Their structure may be as follows: $HC:CM'$, $M'C:CM$ or $RC:CM'$, where M' stands for a monovalent metal and R for an organic radical, eg $LiC:CH$, $AgC:CAg$, $CuC:CCu$. With divalent metals the formulae would be $C:C$ (M'') or $C:C$ (Ca), eg



Certain *carbides* (but not all) may be regarded as acetylides, eg *calcium carbide*, CaC_2

Note: Kirk & Othmer(Ref 4) list the acetylides

of Ca and of some other metals under carbides

A general method for the prepn of acetylides is to pass C_2H_2 through ammoniacal solns of the corresponding salts (such as nitrates) or of oxides. For the prepn of alkali metal acetylides, the metal (such as Na or K) is dissolved in liq NH_3 and the C_2H_2 is passed through

Most acetylides of the heavy metals are expls, very sensitive to mechanical action. Only one of the acetylides (cuprous acetylide) has found application in industry as an ingredient of electric detonators. Some acetylides, as for instance, that of silver, are probably suitable for use in primers and detonators. They also may be incorporated in LA-based compositions for expl rivets in order to reduce their ignition point (Ref 6)

According to Sax (Ref 6) the toxicity of acetylides is unknown, but their expln hazards are considered to be the same as those of fulminates and azides. The acetylides must be handled with extreme care and in storage they must be kept cool and wet. Metal powders, such as finely divided Cu or Ag, should be excluded. Shipping regulations are the same as for other primary and initiating explosives

Refs: 1)Beil **1**, 238-40, (104-6), [217-20] & {909-14} 2)J. A. Nieuwland & R. R. Vogt, "The Chemistry of Acetylene," Reinhold, NY (1945) 3)Kirk & Othmer **1** (1947), 121-3 4)Ibid **2** (1948), 827-46 5)Karrer (1950), 68 6)Sax (1957), 239-40

The following acetylides and carbides may be of interest

Aluminum Acetylide, $Al_2(C_2)_3$, wh solid, prepd by direct action of acetylene on Al pdr at 450-500°. It is a true acetylide because on hydrolysis it gives C_2H_2

Refs: 1)Beil **1**, [220] 2)J. F. Durand, BullFr [4] **35**, 1141 (1924); JCS **126** I, 1278 (1924) & CA **19**, 632 (1925)

Aluminum Carbide, Al_4C_3, pale yel hexag crysts, mp 2200°, subliming in vacuo at at 1800°; reacts with w giving methane together with less than 10% hydrogen. This reaction was used in Germany for producing the gas employed in galleries for testing expls in regard to their safety for use in gaseous and dusty coal mines (Ref 4). Al_4C_3 was first prepd in 1894 by Moissan (Refs 1, 2 & 3) by heating an intimate mixt of alumina (Al_2O_3) with carbon in an electric arc furnace. Other methods of prepn are also known

Refs: 1)Beil **1**, 59, (7), [11] & {27} 2)H. Moissan, CR **119**, 16 (1894) & JCS **66** II, 450 (1894) 3)Mellor **5** (1924), 870 4)Naoúm, NG (1928), 389 5)Kirk & Othmer **2** (1948), 828 6)Sidgwick, ChemElems **1** (1950), 413-14

Arsenium Carbide, As_2C_6, brn amorph ppt, which expl on warming or gentle rubbing. Was prepd by treating acetylene-bis-magnesium iodide, MgI.C:C.Mg I, in ether with arsenium trichloride

Refs: 1)Beil **1**, [221] 2)E. deMahler, BullFr [4] **29**, 1072 (1921) & JCS **122** I, 101 (1922)

Aurous Acetylide. See Gold Acetylide

Barium Acetylide, also called **Barium Carbide,** BaC_2, greyish solid d 3.75. Was first prepd by Maquenne by treating Ba amalgam with carbon in a stream of hydrogen (Refs 1 & 2). Moissan (Ref 2a) prepd it by heating in an elec furnace mixt of BaO with carbon. Fischer (Ref 3) preferred to heat BaO, or $BaCO_3$ with methane. Vaughn et al (Ref 4) obtained an unstable product, corresponding to an approx formula between BaC_2 and $Ba(C_2H)_2$ by adding a soln of Ba in liq NH_3 to C_2H_2 in liq NH_3. Masdupuy & Gallais (Ref 5) prepd BaC_2, by heating to 120° Ba $(H_2C)_2.4NH_3$, which was obtained by treating Ba in liq NH_3 with acetylene

Refs: 1)Beil **1**, 243 [220] & {913} 2)L. Maquenne, BullFr [3] **7**, 366 (1892) & JCS **62** I, 685 (1892) 2a)H. Moissan, CR **118**, 683 (1894) & JCS **66** I, 314 (1894) 3)F. Fischer, Brennstoffchem **9**, 929 (1928) & CA **23**, 2662 (1929) 4) T. H. Vaughn et al, JOC **2**, 2-3 (1937) & CA **31**, 5751 (1937) 5)E. Masdupuy & F. Gallais, CR **232**, 1837-9 (1951) & CA **45**, 7905 (1951)

Beryllium Acetylide, BeC_2, wh solid, prepd

by passing pure dry C_2H_2 over Be pdr at ca 450°

Refs: 1)Beil 1,[218] 2)J.F.Durand, BullFr [4] 35,1145(1924); JCS 126 I,1278 (1924) & CA 19,632(1925)

Beryllium Carbide, Be_2C, yel or brn-yel crysts, d 1.9 at 15°. Was first prepd by heating beryllium oxide with carbon in an electric furnace (Ref 2). Other methods of prepn are listed in Ref 1. Henry (Ref 3) established its formula as Be_2C

Refs: 1)Beil 1,(7) & {27} 2)P.Lebeau, CR 121,496(1895) & JCS 70 II, 169(1896) 3)L.Henri, CR 121,600-1(1895) & JCS 70 II,169(1896)

Boron Carbide, B_4C; coml prod called "Norbide," mp ca 2375°, d 2.52 is prepd by heating anhyd boric oxide B_2O_3 with carbon in graphite resistance furnace at ca 2500°. Its special interest is due to its remarkable hardness which lies on the Moh's scale betw that of silicon carbide and diamond. Used as an abrasive. Detailed description of this compd is given in Kirk & Othmer 2(1948), 830-4(21 refs)

Cadmium Acetylide, CdC_2(formula is not definitely established). It was prepd by passing pure dry C_2H_2 over pulverized Cd at ca 500°(Refs 1 & 2).Gebauer (Ref 3) prepd two derivs, $CdC_2 . C_2H_2 . CdI_2$ and $CdC_2 . C_2H_2$, both of which partially decompd by hot w but were stable in the air even at 200°

Refs: 1)Beil 1,[220] 2)J.F.Durand, BullFr 35,1142(1924) & CA 19,632(1925) 3)K.Gebauer, ZAnorgChem 176,284(1928) & CA 23,815(1929)

Calcium-1-acetylide, called in Beil Calciumcarbid-Acetylen, $Ca(C_2H)_2$, wh solid which decompd in 5 hrs. Was first prepd either by passing C_2H_2 into soln of Ca in liq NH_3 or by adding Ca–NH_3 soln to C_2H_2–NH_3 soln

Refs: 1)Beil 1,242 & {911} 2)H.Moissan, CR 136,1524(1903) 3)T.H.Vaughn & J. P. Danehy, ProcIndianaAcadSci 44,144-8(1934) & CA 30,428(1936) 4)T.H.Vaughn, et al, JOC 2, 2–3(1937) & CA 31,5751(1937)

Calcium Acetylide or Calcium Carbide, CaC_2, mw 64.10, OB to CO_2 and CaO -75.0%. The pure prod is a wh solid, mp 2300°, d 2.155, sp heat (0 to 2000°)0.28 cal/g, whereas the coml prod ranges in color from steel-grey to red-brn. It exists in four crystn forms of which the tetragonal predominates. It reacts vigorously with w producing acetylene and Ca hydroxide. If only a small amt of w is used, the carbide becomes incandescent and causes an expln of acetylene-air mixt formed on contact of w with CaC_2. Small quantities of expl gas may form in CaC_2 drums during storage, and the opening of such drums by spark producing tools, such as steel chisel or screw-driver, is a dangerous operation. Many methods of prepn are known and listed in Beil. The earliest method is that of Wöhler (Ref 2), who prepd CaC_2 by heating Zn-Ca alloy(previously prepd by Caron) with carbon at very high temp. Invention of an industrial method of prepn–heating quicklime and carbon in an elect arc furnace at 2500-3000° is generally attributed to Willson, who in collaboration with Lord Kelvin prepd CaC_2 in 1892(Ref 3). Slightly later (1894-5) and independently, Moissan prepd CaC_2 by essentially the same process as Willson. Böhm (Ref 4) claimed that he invented a similar process earlier than Willson and applied for patent in 1891, but the patent was not issued until 1895. The electric arc furnace method invented in 1892 is essentially the same as the current method of manuf of CaC_2. Detailed description of the method is given in Refs 5,6,7 & 8. The coml prod contains CaO, graphite and some other impurities

For the lab prepn of pure CaC_2, a small quantity of pure Ca cyanamide is heated in the presence of carbon:

$CaCN_2 + C \rightarrow CaC_2 + N_2$ (see also Ref 5a)

Toxicity, fire & expln hazards, storage & handling and shipping regulations are discussed in Ref 9

CaC_2 is used extensively for the manuf of acetylene, and Ca cyanamide(by fixation of atmospheric nitrogen), as the starting material for making the melamine family of resins, for the manuf of acetylene black and many other purposes

Refs: 1)Beil **1**,242,(105), [218] & {912} 2)F.Wöhler, Ann **124**,220(1862) 3)H. Schweitzer, ZAngewChem **1898**, 411-12 4)L.K.Böhm, ZAngewChem **1899**, 1058-6 5)R.Tausig,"Die Industrie des Calciumcarbids," Knapp,Halle(1930) 5a)H.H. Franck et al, ZAnorgChem **232**,75(1937) & CA **31**,5289(1937) 6)C.H.Aall, "Contribution à l'étude du carbure de calcium industriel," Dunod,Paris(1940) 7)Kirk & Othmer **2**(1948), 834-46 8)Ullmann **5** (1954), 1-43 9)Sax(1957), 425

Calcium Carbide–Ammonia–Acetylene, (called in Ger "Calciumcarbidammoniakacetylen)" $CaC_2 + H_2C_2 + 4NH_3$, wh prism crysts, which become incandescent in contact with w, Cl, CO_2 & SO_2 and yield on heating pure CaC_2. It was prepd by Moissan by passing acetylene into a soln of Ca in NH_3 (cooled at -40 to 80°) until the original blue color disappeared. When the resulting col liq was strongly cooled, or when the NH_3 was allowed to volatilize, wh crysts were obtained. The same method was used by Moissan for prepn of lithium, potassium and sodium carbide derivs

Refs: 1)Beil **1**,242 2)H.Moissan, CR **127**,911-17(1898) & JCS **76** I, 241(1899)

Cesium Hydrogen Acetylide; Monocesium Acetylide or Cesium Acetylide-Acetylene, $CsHC_2$ or $Cs_2C_2.C_2H_2$, wh crysts, mp ca 300, sol in liq NH_3; reacts explosively with some inorg compds. Was prepd by Moissan by treating cesium hydride with acetylene at 100° or by passing acetylene through Cs-NH_3 soln

Refs: 1)Beil **1**, 240 2)H.Moissan, CR **136**, 1217, 1524(1903) & JCS **84** I, 545, 595 (1903)

Cesium Carbide or Dicesium Acetylide, Cs_2C_2, lt brn-red leaflets; reacts explosively with some oxides. Was obtained by Moissan on rapidly heating $CsHC_2$ in vacuo at 300°

Refs: 1)Beil **1**,240 2)H.Moissan CR **136** 1220(1903) & JCS **84** I, 546(1903)

Chromium Carbide, Cr_3C_2, crysts, d 5.62, prepd by Moissan on heating Cr with a large excess of carbon in a crucible in an electric furnace. Another carbide, Cr_4C was also prepd. It is harder than quartz and softer than corundum. According to Kirk & Othmer chromium carbides are not used in industry

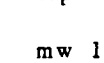
Refs: 1)Beil–not found 2)H.Moissan, CR **119**, 185(1894) & JCS **66** II, 452-3(1894) 3)Kirk & Othmer **2** (1948), 849

Cobaltous Acetylide [Kobalt (II)–acetylenid, in Ger], CoC_2; solid, prepd by treating CaC_2 with $CoCl_2$

Refs: 1)Beil **1**,[220] 2)J.F.Durand,CR **177**, 694(1923) & CA **18**,657(1924)

Copper Acetylide (Kupfer-acetylenid, in Ger) exists in both cuprous, Cu_2C_2, and cupric, CuC_2, forms

Cuprous Acetylide or Dicopper Acetylide (Acetylene Copper or Copper Carbide) [Di-kupfer(1)-acetylenid, in Ger],



mw 151.10

Brick-red amor pdr which expl violently when dry, on heating to ca 120° and also by friction

Note: According to Ref 5, Cu_2C_2 flashes at ca 150°. Klement & Köddermann-Gros (Ref 8, p 213-15) gave for 95% pure prod an expln temp of 170° in air and 265° in a high vac

Cu_2C_2 is sl sol in w and sol in alkalies and aq KCN. The usual method of prepn is to pass dry C_2H_2 into an ammoniacal soln of a cuprous salt in the absence of air. It has been claimed until recently that monohydrate, $Cu_2C_2.H_2O$ is first obtained and this goes,on gentle heating, into the anhyd salt (Ref 4a). Klement & Köddermann-Gros, (Ref 8) prepd C_2H_2 of 95% purity and studied its oxidation products

Morita (Ref 11) prepd cuprous acetylide from a 5% soln of CuCl, and claimed that its ignition temp was 260-70°. This temp was lowered to 100° after the acetylide was oxidized by air. At the same time the color changed to black and the compd became very sensitive to impact. On further oxidn the ign temp rose to 200.° It is presumed that oxidn transformed cuprous acetylide into the cupric compd. Schlubach & Wolf (Ref 9b) in attempting to prep CuC:CH by treating a satd soln of C_2H_2 in w at 0° with an aq soln of $CuSO_4 + NH_4OH + NO.NH_2.HCl$ obtained instead the Cu_2C_2

Vestin (Ref 10) claimed that there is no hydrate of Cu_2C_2 and previously to this Dolgopol'skii claimed that the monohydrate is actually **dicuproacetaldehyde,** $Cu_2CH.CHO$. For its prepn D recommends passing pure C_2H_2 (with exclusion of air) into a soln of CuCl until all the CuCl has reacted. The liq is decanted and the residue washed with abs alc and eth, previously saturated with C_2H_2. The ppt is dried by passing over it dry C_2H_2 at 50° until const wt is obtained. The resulting subst obtained by D was a red-brn powd which expld on heating or on exposure to mech action. When tested on an impact sensitivity apparatus with an 8 kg wt, the subst detond at 30 cm, compared with 15 cm for a coml anhyd prod prepd by passing a mixt of tech gases from a low-temp polymerization of C_2H_2 through the CuCl soln. The latter compd flashed with a bright flame when lightly touched with a glass rod. Its structure was not detd

Cuprous acetylide forms whenever acetylene gas comes in contact with copper, its alloys or some of its salts. As the illuminating gas, made by distn of coal usually contains some acetylene, it should not be conducted through pipes contg Cu. If such pipes are used, great care and caution should be observed in cleaning the pipes inside (Ref 2)

Mixts of Cu_2C_2 with $PbClO_3$ are extremely sensitive to friction (Ref 2) and Cu_2C_2 expl on contact with nitric acid, permanganates, sulfuric acid, bromine or chlorine, etc (Ref 8)

Toxicity, fire & expln hazards and shipping regulations are discussed by Sax (Ref 13)

Cuprous acetylide is the only acetylide which found application in the expl industry. It is used in ign compns for coml elec detonators (Ref 5a)

Chambionnat (Ref 9) in the course of investigation of the possible use of Cu_2C_2 as a fungicide, prepd mixts of Cu_2C_2 with inert subst, such as talcum pdr, and detd their expl props. The tests showed that mixts of talc with as little as 16% Cu_2C_2 can be initiated by an elec spark, but it requires a minimum of 25% Cu_2C_2 for initiation by heat, such as a hot plate. Mixts with as little as 35% Cu_2C_2 can be initiated by rubbing in mortar at temp 55-60°, whereas at RT a minimum of 65-70% of Cu_2C_2 is required.

Cuprous acetylide has been used also for prepn of industrial catalysts (See Cuprous Acetylide Catalyst, which follows)

Refs: 1)Beil **1**, 240(104),[217] & {910} 2)Daniel(1902), 3 3)O.Makowka, Ber **41**, 825(1908) 3a)J.Scheiber et al, Ber **41**, 3818(1908) 4)H.Rupe, JPraktChem **88**, 79(1913) 4a)Thorpe **1**,(1937), 82 5)Dynamit A-G, BritP 528,299(1940) & CA **35**, 7716(1941) 5a)Bebie(1943),50 6)I.M. Dolgopol'skii, ZhPriklKhim **19**, 1281-90 (1946) & CA **41**,6722(1947) 7)V.E.Bromeld et al, JSCI **66**,346-7(1947) & CA **42**, 1740(1948) 8)R.Klement & E.Köddermann-Gros, ZAnorgChem **254**, 202-3, 205-16(1947) & CA **43**, 2112(1949) 9)A.Chambionnat, BullSocSciNatMaroc **28**,77-9(1949) & CA **45**,7791(1951) 9a)E.Jones, ProcRoySoc **198A**, 525(1949) & CA **44**, 2244(1950) (Ignition of copper acetylide by hot wire) 9b)H.H.Schlubach & V.Wolf, Ann**568**, 152 (1950) & CA 44 8313(1950) 10)R.Vestin, SvenskKemiskTidskrift **66**,80(1954) & Beil **1**, {910} 11)N.N.Polyakov, KhimProm **1954** 457-62 & CA **49**, 9260(1955)(Formation of copper acetylides in the low-temp

separation equipment for coke-oven gas) 12)Sh.Morita JSocHighPressureGasInd **19**, 167-76(1955) & CA **50**,6047(1956) 13)Sax (1957), 518

Cuprous Acetylide Catalysts. Cu_2C_2 supported on silica gel, kieselguhr etc can be used as a catalyst in some org reactions

Refs: 1)W.Reppe, "Acetylene Chemistry," Meyer, NY (1949), 80 2)O.Pesta, Mitt-ChemForsch-InstÖsterr **3**, 109-12 (1949) & CA **44**, 4857(1950) 3)W.Reppe et al Ann **596**, 8(1955) & CA **50**, 16771(1956)

Cuprous Acetylide-Chloride, $C_2Cu_2+CuCl+H_2O$, dk violet pdr, deflagrates very weakly on heating in an open flame. Was prepd by passing acetylene through CuCl in 0.2N HCl

Refs: 1)Beil **1**,(104) 2)W.Manchot & J.C. Withers, Ann **387** 270-2(1912)

Note: Some other cuprous acetylide-chloride compds(none of them seem to be expl) were described in Beil **1**,[910] and in the following Scandinavian papers:
1)R.Vestin, ActaChemScand **3**, 650-2(1949) & CA **44**, 1000 (1950) 2)R.Vestin & C.Löfman, ActaChemScand **7**, 398-429(1953) & CA **48**, 13505(1954) 3)R.Vestin et al, ActaChemScand **7**, 745-63(1953) & CA **49**, 449(1955) 4)R.Vestin, SvenskKemTidskr **66**, 65-94(1954) & CA **49**, 3795(1955) 5)R.Vestin et al, ActaChemScand **8**, 533-7(1954) & CA **49**, 10837(1955)

Cuprous Hydrogen Acetylide (Monokupfer-acetylenid, in Ger), CuC!CH. Attempt to prepare this compd by adding with stirring satd soln of C_2H_2 in w at 0° to an aq soln of $CuSO_4 + NH_4OH+HO.H_2N.HCl$ was unsuccessful. Instead of this, the dicopper acetylide, Cu_2C_2 was obtained.

Refs: 1)Beil—not found 2)H.Schlubach & V.Wolf, Ann **568**, 152(1950) & CA **44**, 8313 (1950)

Cupric Acetylide [Kupfer (II)-acetylenid, in Ger], CuC_2, mw 87.56, OB & CO_2 73.1% Black amors ppt, which expl violently on heating, impact or friction. According to Morita (Ref 5) its ignition temp is 100-120° and it explodes on slight impact even under w It was first prepd by Söderbaum (Ref 2), on passing acetylene through an ammoniacal soln of a cupric salt at ca 5°. The compn of the resulting black pdr was 12 $CuC_2 + H_2O$. Durand (Ref 3) prepd CuC_2 by the action of CaC_2 on an aq soln of $CuCl_2$ and purified the resulting ppt with dil AcOH. Nast & Pfab(Ref 6) prepd CuC_2 by treating KC!CH in NH_3 with $[Cu(NH_3)_4](NO_3)_2$ in NH_3

Brameld et al(Ref 4) investigated the formation of copper acetylides from aq solns of various cupric salts and acetylene. The resulting compds appear to fall into two types: a)black amors ppts and b)lustrous, metallic appearing plates

The type a) expl sometimes with a report, sparks and flashes to form black CuO. It could be fired by a drop of HNO_3. The explosibility of this type of acetylide is greater than that of cuprous acetylide. This type includes most acetylides, formed from the more common cupric salts, such as the chloride, sulfate and nitrate, and those formed from copper org salts made alkaline with alkalies other than ammonia, or also with ammonia, provided insufficient ammonia is present to retain all the Cu as a complex salt

The type b) cupric acetylides expl on gentle tapping (sometimes even when touched under soln) with a bright flash and report to form metallic Cu. This type includes acetylides from Cu borate in strongly alkaline soln and from Cu acetate in acidic and strongly ammoniacal solns

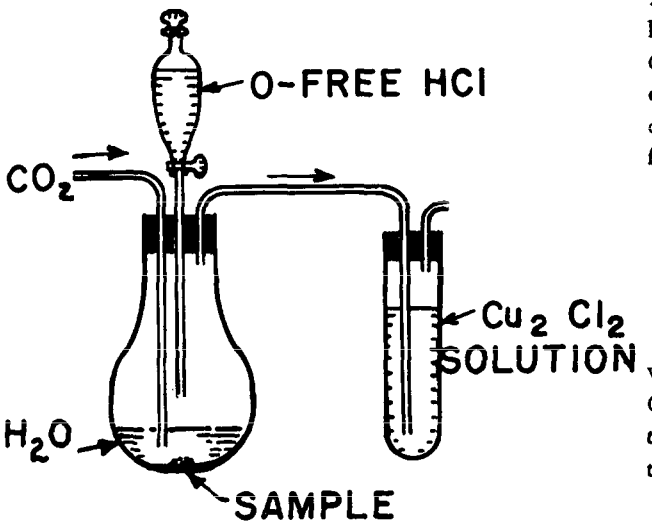
Refs: 1)Beil **1**, 241 & [217] 2)H.G.Söderbaum, Ber **30**, 760 & 814(1897) 3)J.F. Durand, CR **177**, 693(1923) & CA **18**, 657 (1924) 4)V.E.Brameld et al, JSCI **66**, 347-9(1947) & CA **42**, 1740(1948) 5)Sh. Morita, JSocHighPressureGasInd **19**, 167-76(1955) & CA **50**, 6048-9(1956) 6)R. Nast & W.Pfab, Ber **89**, 420-1(1956) & CA **50**, 14604(1956)

Copper Acetylides, Analytical. According

to Dr Hans Walter of PicArsn, the following procedure was developed by the Lindé Co of München:

A) **Determination of acetylene:** a) Assemble an apparatus as represented on the drawing

b) Weigh a sample (W_1 = ca 0.5g)



place it in the flask contg a small amt of O-free w c) Stopper the flask and pass a current of O-free CO_2 or N_2 to remove air

d) Continuing to pass CO_2, add slowly from a separatory funnel O-free concd (20-30%) HCl until the disappearance of amber color e) Collect the liberated C_2H_2 in the test tube contg O-free 5% aq soln of gelatin (used as protective colloid) with some NH_4Cl and Cu_2Cl_2 (free of $CuCl_2$) f) Boil the soln in the flask to expel residual C_2H_2 and collect it also in the test tube

g) Acetylene reacts with Cu_2Cl_2 in the test tube giving Cu_2C_2 which imparts an intense red coloration to the contents of the tube. Determine the amt of Cu_2C_2 in the tube by comparing the color with known standards or by using a colorimeter and calc the corresponding amt of C_2H_2 (W_2)

B) **Determination of total copper:** a) Oxidize the cuprous copper soln in the flask to cupric state by adding $KClO_3$ in slight deficiency to the HCl present b) Boil the soln to drive out chlorine, cool, transfer to a volumetric flask and make up with w c) Take an aliquot, add an excess of KI and titrate the resulting brn soln with Na thiosulfate in presence of starch indicator

d) Calculate copper content (W_3) taking into consideration the following equations:

$2CuCl_2 + 4KI = 2CuI + I_2 + 4KCl$ and

$I_2 + 2Na_2S_2O_3 = 2NaI + Na_2S_4O_6$

C) **Estimation of Cu(I) and Cu(II) in copper acetylide** can be accomplished by the indirect method, which involves solving the following equations

$$x + y + z = W_1$$

$$127.14x + 63.57y = 63.57W_3$$

$$\frac{26.04}{151.16}x + \frac{26.04}{87.59}y = W_2$$

where W_1 is the wt of sample, x is the wt of Cu_2C_2, y the wt of CuC_2, z the wt of impurities, W_3 total wt of Cu(procedure B) and W_2 total amt of C_2H_2(procedure A)

In the method described by Klement & Köddermann-Gros (Ref 4) total Cu is detd by dissolving a sample in HCl, oxidizing Cu(I) to Cu(II) with HNO_3, boiling with H_2SO_4 (until the disappearance of NO_2 fumes), cooling, diluting with w and estimating Cu content electrolytically. For detn of Cu(I) content a 50 mg sample is placed in the flask of the apparatus described on p 204 of Ref 4 and (after removing all air by passing a stream of O-free CO_2) it is treated with 10 ml O-free concd HCl. After evoln of C_2H_4 ceases, 70-80 ml w is added and the soln titrated with 0.1N $KBrO_3$ soln as described by Zintl & Wattenberg(Ref 1). The acetylene content is detd gas-volumetrically in a special apparatus (described on p 205, Ref 4) by treating a sample with 20% KCN soln; Cu_2C_2 $8KCN + 2H_2O \rightarrow C_2H_2 + 2K_3[Cu(CN)_4] + 2KOH$. The water content is detd by heating a sample in a high vacuo to 140° in the presence of P_2O_5

In the method of Voronkov(Ref 3), Cu(II) is reduced to Cu(I) with NH_2OH, the obtained Cu_2C_2 is decompd with HNO_3 and Cu detd

volumetrically. The acetylene content is detd by a rather complicated method which is not described here because its brief description is given in CA **43**, 2972(1949)

Dolgopol'skii et al (Ref 2) proposed two methods of detg copper in Cu_2C_2: a)The dichromate methods involves soln in 15-18% HCl, soln in boiling HNO_3 adding concd alkali(to ppt Cu as hydroxide), soln of the ppt, addn of Seignette salt (turning the color from greenish to lt violet), of boiling with $NH_2OH.HCl$ to ppt Cu_2O, dissolving it in hot $Fe_2(SO_4)_3$ and titrating with $K_2Cr_2O_7$ in the presence $(C_6H_5)_2NH.H_2SO_4$, as an indicator b)The iodometric method involves dissolving a 0.1-0.2 g sample in 15-20% HCl, evaporating the soln to 2-3ml, treating it with ca 25 ml concd HNO_3 and boiling to complete oxidation (color turning from grn to blue); this followed by boiling the soln with concd H_2SO_4 until disappearance of NO_2, cooling, adding ammonia in excess, boiling to remove excess NH , cooling, adding H_2SO_4, adding 40-50ml 10% KI and titrating the soln with 0.25N $Na_2S_2O_3$ using starch indicator. These methods of analysis are also applicable to copper derivs of vinylacetylene and of acetylenedivinyl

Siggia(Ref 5) gives some general methods of analysis of acetylene and acetylides

Caution: Copper acetylides are very expl when dry and should be destroyed, after experiments, in the same manner as described by Siggia for silver acetylide (Ref 5). For this, rinse all pieces of used apparatus with dil nitric acid, dissolve all solid deposits in the same acid and pour the solns into a sink. Dolgopol'skii (Ref 2) advises destroying Cu derivs of acetylene, etc, as well as the technical gas mixts from acetylene polymerization, by treating them in the cold for about 1 hr with a 15% HCl or by heating them to 80-85° for about 1 hr with 10% HCl

Refs: 1)E.Zintl & H.Wattenberg, Ber **55**, 3366(1922) 2)I.M.Dolgopol'skii et al, Zh-PriklKhim **19**, 1281-90(1946) & CA **41**, 6721-2(1947 3)G.Voronkov, ZhAnalKhim **1**, 285-9 (1946) & CA **43**, 4972-3(1949) 4)R.Klement & E.Köddermann-Gros, ZAnalChem **254**, 203-5(1947) & CA **43**, 2112(1949 5)S.Siggia, "Quantitative Organic Analysis via Functional Groups," Wiley, NY(1949), 53 & 55

Gold Acetylide or Gold Carbide(Aurous Acetylide), Au_2C_2, mw 418.42. Yel ppt, mp- expl on rapid heating(various temps are reported ranging from 83° to 157°). Very sol in w, insol in alc. Can be prepd by passing acetylene through an ammoniacal soln of sodium aurothiosulfate $Na_3Au(S_2O_3)_2$, which had been previously prepd by mixing aq solns of $AuCl_3$ and $Na_2S_2O_3$

When thoroughly dried, aurous acetylide readily expl, not only on rapid heating(see above) but also on impact, friction or even when touched with a camel's hair brush. Several explns have occurred during its prepn in various labs. When expld, it produces a flame and leaves a black, powdered residue of gold. Reppe et al (Ref 3) found Au_2C_2 as a suitable catalyst in prepn of some org compds

Refs: 1)Beil **1**, 241 2)J.L.Mathews & L.L.Waters, JACS **22**, 108(1900) 3)W.Reppe et al, Ann **596**, 6(1955) & CA **50**, 16771(1956)

Halogen Substituted Products of Acetylene. See Halogenated Acetylenes under H's

Iron Acetylide (Ferrous Acetylide) [Eisen (II)-acetylenid, in Ger], FeC_2; solid, stable in the air or w at RT; decompd by HCl with evoln of C_2H_2. Was prepd by Durand on treating CaC_2 with ferrous chloride

Refs: 1)Beil **1**, [220] 2)J.F.Durand, CR **177**, 693 (1923) & CA **18**, 657(1924)

Iron Carbide Fe_3C; brilliant wh crysts, ignites when heated, d 7.07 at 16°, not attacked by w even at 150°. Was prepd by Moissan on heating pure iron with sugar-charcoal in an elec furnace

Refs: 1)Beil **1**, (8) & [11] 2)H.Moissan, CR **124**, 716(1897) & JCS **72** II, 375(1897)

Lead Acetylide, PbC_2(probably), mw 231.21. Lt grey powd, stable toward H_2O. Stated to be prepd by adding CaC_2 to an aq soln

of Pb acetate and washing the resulting ppt with dil AcOH(Ref 2)

Montignie(Ref 3) reported unsuccessful attempts to prep PbC_2 by calcining some org salts but he prepd a mixt of PbC_2 with several other compds by dropping a methanolic soln of Pb acetate on CaC_2. He reported that his prod was stable in air but hydrolized by acid or alk solns

Refs: 1)Beil **1**, [220] & {914} 2)J.F. Durand, CR **177**,693-5(1923) & CA **18**,657 (1924) 3)E.Montignie, Bull Fr [5]**2**, 1807-9 (1935) & CA **30**,691(1936)

Lithium Carbide or Monolithium Acetylide, LiC_2; wh crysts, d 1.65 at 18°; was prepd by Moissan on heating Li carbonate with 6 mols of sugar-charcoal in an elec furnace: $Li_2CO_3 + 6C \rightarrow 2LiC_2 + 3CO$(Ref 2). It is a powerful reducing agent and reacts with w in cold to produce pure C_2H_2 and the reaction becomes violent at ca 100° (see also Dilithium Acetylide listed below

Refs: 1)Beil **1**, 238 2)H.Moissan, CR **122**,362-3(1896) & JCS **70** II, 419(1896)

Monolithium Hydroacetylide or Monolithium Acetylide-Acetylene, $LiHC_2$ or $Li_2C_2 + C_2H_2$. It is claimed in Beil **1**, p 238 that this compd was prepd by Moissan (Ref 2). There is some misunderstanding because the compd $LiHC_2$ is not listed in Ref 2, but there is described by Moissan, among other acetylides, the complex, $Li_2C_2 \cdot C_2H_2 \cdot 4NH_3$, called lithium carbide-ammonia-acetylene (see below) which gave on heating Li_2C_2. Campbell et al, (Ref 3) prepd $LiHC_2$ in soln by adding small pieces of Li to liq NH_3 while passing C_2H_2 gas into the mixt until the final soln changed from blue to col. $LiHC_2$ appears to undergo spont decompn during its isolation from liq NH_3, with an approx equimol mixt of $LiHC_2$ and Li_2C_2 being formed with some occlusion of NH_3 vapor. Masdupuy & Gallais (Ref 4) claimed to have prepd $LiHC_2$ from Li, NH_3 and C_2H_2 but Beil **1** {909} states that it exists only in soln

Refs: 1)Beil **1**, 238 & {909} 2)H.Moissan, CR **127**, 911(1898) & JCS **76** I, 241(1899) 3)K.N.Campbell & B.K.Campbell, Proc IndianaAcadSci **50**, 123-7(1940) & CA **35**, 5457(1941) 4)E.Massdupuy & F.Gallais. CR **232**, 1837-9(1951) & CA **45**,7905(1951)

Lithium Carbide-Ammonia-Acetylene, $Li_2C_2 \cdot C_2H_2 \cdot 4NH_3$; wh prism crysts, which become incandescent in contact with w, Cl_2, CO_2 or SO_2. Was prepd by Moissan from Li, NH_3 and C_2H_2 in a similar manner as briefly described under Calcium Carbide-Ammonia-Acetylene. It gave on heating CaC_2

Refs: 1)Beil **1**, 238 2)H.Moissan, CR **127**, 911(1898) & JCS **76** I, 241(1899)

Dilithium Acetylide or Lithium Carbide, Li_2C_2, wh crysts. Was first prepd by Guntz on heating Li to redness in a vac with carbon or in a current of CO or CO_2(Ref 2). Moissan (Ref 3) prepd it by heating lithium carbide-ammonia-acetylene. Tucker & Moody (Ref 4) prepd it by heating Li carbonate and carbon in three types of elec furnaces to produce the reaction: $Li_2CO_3 + 4C \rightarrow Li_2C_2 + 3CO$. The granular carbon type of furnace gave the best results. Other methods of prepn are given in Refs 5 & 6

Refs: 1)Beil **1**, 238,(104) & {909} 2)A. Guntz, CR **123**, 1273-5(1896) & JCS **72** II, 212(1897) 3)H.Moissan, CR **127**, 911(1898) & JCS **76** I, 241(1899) 4)S.A.Tucker & H.R.Moody, JACS **33**, 1479-80(1911) 5)A. von Antropoff & J.F.Müller, ZAnorgChem **204**, 313(1932) & CA **26**, 3417(1932) 6)E. Masdupuy & F.Gallais, CR **232**, 1838(1951) & CA **45**,7905(1951)

Magnesium Acetylide or Magnesium Carbide, MgC_2, tetragonal crysts, which are decompd by w into C_2H_2 and $Mg(OH)_2$. Was first prepd in 1866 by Berthelot (Ref 2) by heating Mg pdr in stream of C_2H_2. Many other methods are listed in Beil, but it seems that heating of Mg pdr to ca 450° in a stream of acetylene is the simplest method (Ref 3). It was claimed that MgC_2 was converted to Mg_3C at ca 500°

Refs: 1)Beil **1**, 421,(105) & {911} 2)M. Berthelot, Ann **139**, 161(1866) 3)H.H. Franck et al, Z AnorgChem **232**, 110(1937)

Manganous Acetylide [Mangan (II)-acetylenid, in Ger], MnC_2; solid, stable at RT in air or w. Was prepd by treating CaC_2 with $MnCl_2$ at 450-500°

Refs: 1)Beil 1,[220] 2)J.F.Durand, CR **177**,694 (1923) & CA **18**,657(1924)

Manganese Carbide, Mn_3C; solid, d 6.89 at 17°; was first prepd by Troost & Hautefeuille. Moissan prepd it by heating in an elec furnace a mixt of Mn_3O_4 with sugar-carbon. Other methods of prepn are given in Refs 1 & 3

Refs: 1)Beil 1,(8), [11] & {28} 2)H. Moissan, CR **122**,421(1896) & JCS **70** II, 423-4(1896) 3)W.R.Myers & W.P.Fishel, JACS **67**,1962(1945)

Mercurous Acetylide [Quecksilber(I)-acetylenid, in Ger], $Hg_2C_2 . H_2O$. Grey ppt, mp-lost some water on heating and then decompd or detond. Can be prepd by treating with acetylene a cold aq suspn of mercurous acetate or by treating Cu acetylide with an aq soln of mercurous nitrate. Both operations should be carried out in the absence of light

Reppe et al (Ref 5) investigated Hg_2C_2 as a possible catalyst for some org syntheses, but found it inferior to Cu, Ag and Au acetylides

Refs: 1)Beil 1,243 & [220] 2)R.T.Plimpton & M.W.Travers, JCS **64**, 264(1894) 3) E.Burkard & M.W.Travers,JCS **81**,1270-1(1902) 4)J.F.Durand, CR **177**,694-5(1923) & CA **18**, 657(1924) 5)W.Reppe et al, Ann **596**,6(1955) & CA **50**,16771(1956)

Mercuric Acetylide [Quecksilber(II)-acetylenid, in Ger], HgC_2, mw 224.63.Keiser(Ref 2) claimed to prep it by passing C_2H_2 through Nessler's soln (an alk mercuric potassium iodide soln). The resulting white, very expl ppt proved later to be the hydrate $3HgC_2 + H_2O$. This hydrate dec at 110° and expl at higher temps. It is insol in w, alc & eth

The anhyd salt, HgC_2, was claimed to be prepd by Durand (Ref 3) by treating an aq soln of $HgCl_2$ with CaC_2. It was a greyish solid which expld on rapid heating leaving a black residue. It also expd when struck sharply

Ferber & Römer (Ref 4) prepd a very expl compd $2HgC_2 . C_2H_2 . H_2O$ by passing C_2H_2 through a satd acetic acid soln of mercuric acetate

Refs: 1)Beil 1,243 & [220] 2)E.H. Keiser, AmerChemJ, **15**,535(1893) & JCS **66** I,61(1894) 3)J.F.Durand,CR **177**,694 (1923) & CA **18**,657(1924) 4)E.Ferber & E.Römer, JPraktChem **139**,277(1934)(not listed in CA)

Mercuric Acetylide + Three Silver Nitrates (Complex), $HgC_2 + 3AgNO_3$. Wh crysts, decomp on heating without expln. Can be prepd by treating $Ag_2C_2 + 6AgNO_3$ with sufficient $Hg(NO_3)_2$ in HNO_3 soln to ppt the $HgC_2 + 3AgNO_3$

Refs: 1)Beil 1, {913} 2)J.A.Shaw & E. Fisher, USP 2,474,869(1949) & CA **43**, 7670(1949)

Nickel Acetylide [Nickel (II)-acetylenid, in Ger), NiC_2; solid, stable at RT in air or w. Was prepd by treating CaC_2 with an aq soln of $NiCl_2$(Refs 1 & 2). It was proposed as a catalyst in some org syntheses (Ref 3)

Refs: 1)Beil 1,[220] 2)J.F.Durand, CR **177**, 693-5(1923) & CA **18**,657(1924) 3)O.Pesta, MittChemForsch-InstIndÖsterr **3**, 109-12(1949) & CA 4857(1950)

Nickel Carbide, Ni_3C; solid, stable up to 380-400°; decompd by dil acids or by superheated steam to methane & other products. Was prepd by interaction of Ni pdr with CO at 200-300°

Refs: 1)Beil 1,{28} 2)H.A.Bahr & Th. Bahr,Ber **61**,2178-83(1928) & **63**,99(1930)

Phosphorus Carbide, P_2C_6, wh amorph ppt, which spontaneously ignites when gently warmed. Was prepd by treating an ethereal soln of acetylene-bis-magnesium iodide, MgI.C:C.MgI with phosphorous trichloride

Refs: 1)Beil 1,[221] 2)E. deMahler, BullFr [4]**29**,1071(1921) & JCS **122** I, 101(1922)

Note: W.Venier, BritP 6705(1906) proposed to use HgC_2 as an ingredient of primer compns, such as $KClO_3$ 57.1, HgC_2 28.6 & sulfur 14.3%

Monopotassium Acetylide or Potassium Hydrogen Acetylide (Ethynylpotassium), KHC_2, mw 64.13. Wh crysts, viol dec on contact with w; ignites & burns with incandescence in atm of Cl_2 or SO_2 in the cold. Was first prepd by Berthelot (Ref 2) on heating potassium in acetylene. Moissan (Ref 3) obtained it by passing C_2H_2 through K liquefied by NH_3 at -40 to -80°. Vaughn et al (Ref 4) by adding a soln of K in liq NH_3 to C_2H_2 in liq NH_3. Other methods of prepn are listed in Ref 1

Refs: 1)Beil 1, 239[217] & {909} 2)M. Berthelot, AnnChimPhys [4]9, 385(1866) 3)H.Moissan, CR 127, 911(1898) & JCS 76 I, 241(1899) 4)T.H.Vaughan et al, JOC 2, 3-4(1937) & CA 31, 5751(1937)

Dipotassium Acetylide or Potassium Carbide, K_2C_2, mw 102.2. Wh crysts, dec vigorously on contact with w. Was obtained in small quant by Moissan on heating monopotassium acetylide: $2KHC_2 \rightarrow K_2C_2 + H_2C_2$. Other methods of prepn are listed in Ref 1

Refs: 1)Beil 1, 239 2)H.Moissan, CR 127, 917(1898) & JCS 76 I, 241(1899)

Monorubidium Acetylide or Rubidium Hydrogen Acetylide, $RbHC_2$ or $Rb_2C_2 + C_2H_2$. Wh hygr crysts, mp ca 300° with sl decompn; stable in dry atm; reacts explosively when heated to 350° in presence of PbO_2. Was obtained by Moissan by passing C_2H_2 through a soln of Rb in liq NH_3. Other methods of prepn are listed in Ref 1

Refs: 1)Beil 1, 239 2)H.Moissan, CR 136, 1217(1903) & JCS 84 I, 545(1903)

Dirubidium Acetylide or Rubidium Carbide Rb_2C_2, wh solid; reacts violently with halogens, liq S and sl heated P with evoln. of flame. Was prepd by Moissan on heating $RbHC_2$ in a vacuum

Refs: 1)Beil 1, 239 2)H.Moissan, CR136, 1221(1903) & JCS 84 I, 546(1903)

Silicon Carbide, SiC, mw 40.10; crysts from pale grn to blk, mp-starts to dec ca 2500° and dec completely ca 2700°, d 3.15-3.20 Was first prepd in 1891 by Acheson by heating SiO_2 with C and essentially the same method is used now. It is an abrasive, which only boron carbide and diamond surpass in hardness. A detailed description of SiC is given in Kirk & Othmer 2(1948), 854-66(49 refs)

Silver Acetylide; Disilver Acetylide or Silver Carbide (Azetylenesilber; Disilberacetylenid or Silberacetylenid, in Ger), Ag_2C_2, mw 239.78. Wh solid expl at 120-140° (Ref 2, p 3); 225° (Ref 6, p 304) for Ag_2C_2 prepd from neutral soln. Was first prepd in 1858 by Quet and in the same year by Vogel & Reischauer (see Ref 2, p 1) by passing a stream of acetylene through an ammoniacal soln of silver nitrate. Stettbacher(Refs 2 & 6a) and other investigators(Refs 3-6) studied props of Ag_2C_2 and compared methods of prepn using ammoniacal silver nitrate solns with those using neutral or slightly acidic solns [see also Disilver Acetylide-Silver Nitrate (Complex)]

A method of prepn recently described(Ref 7) consists of passing a rapid stream of acetylene through an aq N/10 soln of silver perchlorate contg 10% ammonia

Following are some props of silver acetylide which was prepd from ammoniacal solns of silver nitrate: *deton vel* 1880 m/sec at d (not given); *power by Trauzl test* 132 cm^3 at d 1.67(Ref 6, pp 304-5); *minimum initiating charge* for 0.8g tetryl, 0.07g Ag_2C_2 compared to 0.02g for LA(Ref 2,p 4); *heat of expln* 400 cal/g

Taylor & Rinkenbach(Ref 4) investigated four samples of silver acetylide prepd by various methods. The sample B,prepd from an ammoniacal soln of $AgNO_3$, gave an impact sensitivity with 0.5kg wt 15 cm(LA 43 cm); expln temp 177°(LA 383°) and pendulum friction values, fall 33 cm and number of swings 4(for LA 37.5 cm and 12 swings)

Ag_2C_2 is considered to be inferior in expl props to silver acetylide-silver nitrate, which is described below. Ag_2C_2 is more sensitive to shock

Muraour(Ref 5) investigated the action of

the shock of electrons on Ag_2C_2

Refs: 1)Beil **1**,241,(104), [217] & {910} 2)A.Stettbacher, SS **11**,1-4(1916) 3)J. Eggert, Chem Ztg **42**,199-200(1918); Ber **51**,454-6(1918) & JSCI **37**,390A(1918) 4)C. A.Taylor & W.H.Rinkenbach, J FranklInst **204**,374(1927) 5)H.Muraour, Chim & Ind **30**,39-40(1933) & CA **27**,5541(1933) 6)R. Stadler, SS **33**,270, 302 & 334(1938) 6a)A. Stettbacher, NC **11**,227(1940) 7)R.Vestin & E.Ralf, ActaChemScandinavica (Denmark) **3**,101(1949) and Svensk Kemisk Tidskrift (Sweden) **66**,66-78(1954)(33 references) & CA **49**,3795(1955)

Monosilver Acetylide + Silver Chloride (Complex), $AgHC_2 . AgCl$ or Ag_2HC_2Cl, wh weakly expl ppt. Can be prepd by treating an ammoniacal soln of AgCl with an excess of C_2H_2

Refs: 1)Beil **1**,241 2)C.Willgerodt, Ber **28**,2111(1895)

Note: According to R.Vestin & E.Ralf, Acta ChemScandinavica **3**,106(1949) & Beil **1**, {910} this complex does not exist

Monosilver Acetylide + Silver Nitrate (Complex), $AgHC_2 . AgNO_3$ or $AgHC_2NO_3$ Solid, expl violently ca 230°. Can be prepd from C_2H_2 and an aq soln of silver nitrate, as described in Ref 2

Refs: 1)Beil **1**,241 2)C.Willgerodt, Ber **28**,2108-10(1895)

Note: According to R.Vestin & E.Ralf, Acta ChemScandinavica **3**,106(1949) & Beil **1**, {910}; this complex does not exist

Disilver Acetylide + Silver Chloride(Complex), $Ag_2C_2 . AgCl$ or $[Ag_3C_2]Cl$; wh expl ppt. Can be prepd by passing C_2H_2 through an ammoniacal soln of freshly pptd AgCl until about two-thirds of the AgCl is consumed

Refs: 1)Beil **1**,241 2)M.Berthelot & M. Delépine, CR **129**,370(1899) & JCS **76**I,842 (1899)

Disilver Acetylide + Silver Nitrate (Complex) (Silver Acetylide-Silver Nitrate), $Ag_2C_2 . AgNO_3$ or $Ag_3C_2NO_3$, mw 409.67, N3.42%. Wh pdr,d 5.38(Ref 5);mp-deton ca 212°(Ref 6). It is insol in w, alc & eth. Was prepd by passing pure C_2H_2 through a soln of 10 g $AgNO_3$ in 40 ml H_2O and 6 ml HNO_3(d 1.4). After cooling, the wh ppt was filtered from the soln and purified by washing with alc and acet. The yield was 7 to 7.95 g compared with the theoretical 8.04 g(Ref 2). Another method of prepn is to pass C_2H_2 through an aq soln of $AgNO_3$, not stronger than 10% (Refs 3 & 6)

This compd is more powerful and less sensitive than Ag_2C_2. Its rate of deton is higher than that of Ag_2C_2, but the brisance is almost the same. It is extremely sensitive to flame, less sensitive to impact than MF and less sensitive to friction than LA. It detonates according to the equation: $Ag_2C_2 . AgNO_3 = 3Ag(vapor) + CO_2 + CO + 0.5N_2 + 185$ cal(Ref 3, p 338)

Following is additional data on the expl props of this complex: *brisance*(Brisanzwert, calcd by the Kast formula) 94(millions), compared with 107 for LA; *deton vel* 3460 m/sec at d 3.96(LA 5300 at d 4.6); *expln temp* 217° & 265°(LA 315°); *heat of expln* 451 cal/g (LA 268); *impact sensitivity* with a 2kg wt 3.4 cm(LA 3.2 cm); *initiating ability*-comparable to MF and LA; *power*(by Trauzl test) 145cm^3 for a 10g sample(LA 181 cm^3); *spec vol* 200 l/kg(LA 310); *stability in storage at 90°*-satisfactory. It is unaffected by moisture, light and CO; *temp of expln* (calcd by Kast formula given in Ref 3, p 338) 5740° (LA 3450°)

According to Shaw & Fisher(Ref 6a),this complex can be used as a means of detg C_2H_2 in gas mixtures

Refs: 1)Beil **1**,241 & {910} 2)A.Stettbacher, SS **11**,1-4(1916) 3)R:Stadler, SS **33**,271-2, 304-5 & 334-8(1938) 4)A.Stettbacher, NC **11**,227-9(1940) 5)Ibid **13**,26 (1942) 6)J.A.Shaw & E.Fisher, JACS **68**, 2746(1946) 6a)J.A.Shaw & E.Fisher, Anal Chem **20**,533-6(1948) 7)J.A.Shaw & E. Fisher,BritP 616,319(1949) & CA **43**,5175 (1949); USP 2,474,869(1949) & CA **43**, 7670(1949) (Highly explosive $Ag_2C_2 . AgNO_3$

Note: W.Venier, BritP 6705(1906) proposed to use Ag_2C_2 as an ingredient of primer compns, such as MF 41.4, $KClO_3$ 41.4, Ag_2C_2 6.9, K picrate 6.9 & Al 3.4%

complex was obtained, together with non-explosive $Ag_2C_2.6AgNO_3$, during removal of acetylene from coke-oven gases by absorption in an aq soln of $AgNO_3$)

Disilver Acetylide + Six Silver Nitrates (Complex), $Ag_2C_2 + 6AgNO_3$ or $C_2Ag_8N_6O_{18}$, crysts, mp-when heated to 308-327° it melts and then suddenly decomp with evoln of red fumes. Can be prepd by passing C_2H_2 through a 30% soln of $AgNO_3$ in H_2O or N/1 HNO_3. It is not expl

Refs: 1)Beil **1**, {911} 2)J.A.Shaw & E. Fisher, JACS **68**, 2745(1946); USP 2,474,869 (1949) & CA **43**, 7670(1949) 3)Ibid, USP 2,483,440(1949) & CA **44**, 1679(1950)

Disilver Acetylide + Silver Iodide (Complex), Ag_2C_2. AgI or $[Ag_3C_2]I$; grn very expl ppt. Can be prepd from C_2H_2 and an ammoniacal soln of freshly prepd AgI, taken in excess

Refs: 1)Beil **1**, 241 2)M.Berthelot & M. Delépine, CR **129**, 361(1899) & JCS **76** I, 842(1899)

Disilver Acetylide + Two Silver Iodides (Complex), $Ag_2C_2 + 2AgI$ or $Ag_4C_2I_2$; yel ppt which mildly deton when heated in a flame. Can be prepd by reacting C_2H_2 with AgI soln in aq KI in the presence of a small amt of KOH

Refs: 1)Beil **1**, 241 2)M.Berthelot & M. Delépine, CR **129**, 361(1899) & JCS **76** I, 842(1899)

Disilver Acetylide + Two Silver Perchlorates (Complex), $Ag_2C_2 + 2AgClO_4 + 2H_2O$; wh crysts, expl on heating. Can be prepd by treating Ag_2C_2 with concd Ag perchlorate soln

Refs: 1)Beil **1**, {910} 2)R.Vestin & E. Ralf, ActaChemScandinavica **3**, 112(1949)

Disilver Acetylide + Disilver Sulfate Complex, $(Ag_2C_2).Ag_2SO_4$ or $(Ag_3C_2)AgSO_4$, wh ppt, deton weakly in vacuo. Can be prepd by treating C_2H_2 with an excess of silver sulfate in soln

Refs: 1)Beil **1**, 241 2)M.Berthelot & M. Delépine, CR **129**, 361(1899) & JCS **76** I, 842(1899)

Disilver Oxide + Acetylene + Disilver Chromate (Complex), $Ag_2O + C_2H_2 + Ag_2CrO_4$. Or-red crysts, expl ca 157°. Can be prepd from acetylene and a boiling soln of silver bichromate

Refs: 1)Beil **1**, 241 2)M.Berthelot & M. Delépine, CR **129**, 361(1899) & JCS **76** I, 842(1899)

Note: According to R.Vestin & E.Ralf, Acta ChemScandinavica **3**, 104(1949) & Beil **1**, {911} the existence of this compd is questionable

Two Disilver Acetylides + Silver Chloride (Complex), $(Ag_2C_2)_2$. AgCl or $Ag_2C_2 + Ag_3C_2Cl$; yel ppt which expl violently. Can be prepd by treating ammoniacal AgCl soln with HCl and C_2H_2

Refs: 1)Beil **1**, 241 2)M.Berthelot & M. Delépine, CR **129**, 361(1899) & JCS **76** I, 842(1899)

Silver Acetylide, Analytical. The acetylene content can be detd by the same method as described under Copper Acetylides, Analytical, procedure A, except that the sample shall not be exposed to direct light. The silver content can be detd by transferring quantitatively the residue of AgCl in the flask into a tared sintered glass crucible, rinsing it with w and then alcohol, drying it and weighing

R.Stadler, SS **33**, 269-72, 302-5 & 334-38 (1938), briefly outlines analyses of pure and tech silver acetylides and describes methods of detg the expln temp, heat of expln, gases developed on expln, vel of deton, lead block expansion, sensitivity to initiation, friction sensitivity, impact sensitivity, stability in storage and brisance by Kast formula

Silver Acetylide, Destruction can be accomplished by dissolving it in dil nitric acid and pouring the soln into a sink. Another method is to make ammoniacal the mixture

contg solid acetylide and then dissolve the ppt (under hood) in 5% KCN soln. The resulting liq is poured into 5% ferrous sulfate (to destroy the excess cyanide) and then into a sink

Ref: S.Siggia; "Quantitative Organic Analysis via Functional Groups," Wiley, NY(1949), 53 & 55

Monosodium Acetylide or Sodium Hydrogen Acetylide (Ethynylsodium) $NaHC_2$, mw 48.02; wh to yel friable solid, d 1.33; mp expl ca 150° with evoln of gases that catch fire in the air leaving a blk residue which is still very reactive; decomp explosively on contact with w or alc and burns with flame in atm of Cl or Br at RT; it is sol in liq NH_3 and insol in eth & benz. Was first prepd by Berthelot (Ref 2) by heating Na with C_2H_2 and then by Moissan (Ref 3) from Na and C_2H_2 at RT but under pressure. Many other methods of prepn are given in the literature. Its prepn from Na & C_2H_2 in liq NH_3 and from sodamide and C_2H_2 in liq NH_3 are described in Refs 4 - 7

Refs: 1)Beil **1**, 238,(104),[217] & {909} 2)M. Berthelot, AnnChim[4]**9**, 402(1866) 3)H.Moissan,CR **126**, 302(1898) 4)T.H. Vaughan et al, JOC **2**, 2-5(1937) 5)Inorg Synth **2**(1946), 76-81(12 references) 6)Org Synth **30**(1950), 15 7)R.A.Raphael, "Acetylenic Compounds in Organic Syntheses," Academic Press, NY(1955), 193 8)Air Reduction Co, BritP 744,803(1956) & CA **50**, 17358(1956)(Prepn of $NaHC_2$ by treating C_2H_2 at < 110° with Na dispersed in an inert liq such as xylene)

Disodium Acetylide or Sodium Carbide, Na_2C_2, mw 70.00; wh pdr, d 1.575 at 15°, mp decomp ca 400°; insol in common org solvents. It reacts explosively on contact with w and forms expl mixts with oxidizers, phosphorus, some metals, chlorides and iodides. Was first prepd by Berthelot (Ref 2) from C_2H_2 and molten Na. Matignon (Ref 3) prepd it by heating monosodium acetylide to 210-220°. Guernsey & Sherman (Ref 4) describe in detail apparatus and procedure by bubbling acetylene through molten Na. Ylla-Conte (Ref 5) patented in Germany an industrial method of prepn by the action of Na vapors on carbon at high temps produced by an elec arc betn C electrodes in atm of H in a closed chamber. Some other methods are listed in Ref 1, p {909}

Refs: 1)Beil **1**, 239,[217] & {909} 2)M. Berthelot, AnnChim [4] **9**, 402(1866) 3)Matignon, CR **124**, 776(1897) 4)E.W.Guernsey & M.S. Sherman, JACS **48**, 141(1926) 5)J.Ylla-Conte, GerP 526,627(1930) & CA 4808(1931)

Strontium Acetylide or Strontium Carbide, SrC_2; greyish solid, d 3.19; reacts with w and acids with evoln of acetylene. Was first prepd by Moissan (Ref 2) on heating Sr or $SrCO_3$ with carbon in an elec furnace. Franck et al (Ref 3) prepd it by heating Sr or SrO with CO at 1050° & 2000°. Cryst structure is discussed in Ref 4

Refs: 1)Beil **1**, 243 & {911} 2)H.Moissan, CR **118**, 684 (1894) & JCS **66** I, 314(1894) 3)H.H.Franck et al, ZAnorgChem **232**, 109 (1937) & CA **31**, 5289 4)M.A.Bredig, JPhys Chem **46**, 816, 818(1942) & CA **37**, 1918(1943)

Thorium Dicarbide, ThC_2, solid, d 8.96 at 18°. Was prepd by Moissan & Étard on heating in an electric furnace a mixt of powdered thorium oxide and sugar-charcoal made into a paste with turpentine. Other methods of prepn are given in Ref 3

Refs: 1)Beil **1**, {914} 2)H.Moissan & A.Étard, CR **122**, 573(1896) & CA **70** II, 423 (1896) 3)Gmelin, Syst No 55, "Thorium" (1955), 298

Titanium Carbide, TiC; solid mp ca 3140° d 4.97. Can be prepd by heating an intimate mixt of TiO_2(or Ti metal) with carbon in vacuo or in atm of hydrogen. It is used as an abrasive, being slightly softer than WC

Ref: Kirk & Othmer **2**(1948), 848-9

Tungsten Carbide (Wolfram Carbide) WC; crysts, mp 2867, d 15.7. Was first obtained in 1893 by Moissan. Can be prepd by heating W-metal pdr and carbon black to cu 2000° in a pure graphite crucible. It is the most important abrasive for general use

Ref: Kirk & Othmer **2**(1948), 846-8(13 refs)

Uranium Carbides. Moissan prepd in 1896 a gm cryst compd d 11.28 at 18° to which he assigned the formula U_2C_3. This method of prepn consisted of heating a mixt of U_3O_8 with sugar-charcoal in an elec furnace. Later investigators obtained on using Moissan's method U_2C. As none of the compds prepd by Moissan's method was pure, Litz et al (Ref 3) designed a new method of prepn and succeeded in preparing pure UC and UC_2. Both compds are crystalline. UC goes at temp ca 2400° to UC_2

Refs: 1)Beil 1, [28] 2)H.Moissan, CR **122**, 274(1896) & JCS **70** II, 364(1896) 3) L.M.Litz et al, JACS **70**, 1718-22(1948)

Vanadium Carbide, VC, crysts, d 5.36. Was first obtained in 1896 by Moissan on heating vanadium anhydride with carbon in the carbon tube of an elec furnace (Ref 2). Oldham & Fischer (Ref 3) examined some reactions of pure VC obtained from the Vanadium Corp of America. According to Kirk & Othmer (Ref 4), VC was used during WWII in Germany when WC became unavailable

Refs: 1)Beil 1, [28] 2)H.Moissan, CR **122**, 1297(1896) & JCS **70** II, 608(1896) 3) S.E.Oldham & W.P.Fishel, JACS **54**, 3610-12(1932) 4)Kirk & Othmer **2**(1948), 849

Wolfram Carbide. See Tungsten Carbide

Zinc Acetylide (Zinkacetylenid, in Ger), ZnC_2; yel pdr, reacts with w or NaOH soln giving off C_2H_2. Can be prepd by passing dry C_2H_2 over Zn pdr at 450-500°

Refs: 1)Beil 1, [220] 2)J.F.Durand, CR **176**, 992(1923); BullFr [4] **35**, 166 & 1142(1924); JCS **126** I, 602(1924)

Zirconium Carbide, ZrC; grey pdr, not attacked by w, NH_3 or HCl even when heated. It is harder than quartz but softer than ruby. Was first prepd by Moissan et al on heating a mixt of Zr oxide with carbon in an electric furnace

Refs: 1)Beil–not found 2)H.Moissan et al, CR **122**, 651(1896) & CR **70** II, 429 (1896)

Acetyl Laurin (Acetyl Coconut Oil). A compd patented by Woodbridge (Ref 1) for use(in combination with DNT etc)as an ingredient of smokeless propellants. The formula and methods of prepn are not given in the original patent but, according to C.I. Johnson (Ref 2), theproduct designated as acetyl laurin apparently consisted of a mixture of monoacetyl dilaurin, $C_3H_5O_3(CH_3CO)(C_{11}H_2O)_2$ and diacetyl monolaurin, $C_3H_5O_3(CH_3CO)_2$-$(C_{11}H_{23}CO)$ in which the term laurin was used to mean coconut oil. This material is a good plasticizer for NC and a satisfactory propellant was made using it. It also acts as a flash reducer

According to F.R.Schwartz(Ref 3), the compd may be prepd as follows: a)Heat mixture consisting of 639 g coconut oil (1 mol), 188 g high gravity glycerol(98% purity) (2mols) and 0.3-0.5 g CaO(catalyst) at 250°(480°F) with agitation and in an atm of CO_2 b) Periodically, withdraw a small amt of the reaction mixture and test it for miscibility with 90/10-methanol/water soln. As soon as one part of the reaction mixture becomes miscible with 4 parts of the 90/10 methanl-water solvent, stop heating c) Filter while still hot using a filter of Columbia activated carbon and 8 g of Decalite (diatomaceous earth) to remove the CaO catalyst d) Add 150 g of AcOH + 2 g H_2SO_4 to the filtrate and heat under reflux until the acid number is less than 5. This takes 2 to 5 hours e)Distill off the excess AcOH, using a stream of CO_2, neutralize with Na_2CO_3 and filter

Note: Method of prepn of acetyl laurin is described in detail because it is not found in the literature

Another method of prepn of acetyl laurin is to treat triacetin (see under Acetins), in the presence of an alkaline catalyst with lauric acid to replace one of the acetyl groups. The AcOH produced by the reaction can be removed by azeotropic distn using sufficient hydrocarbon solvent to maintain the distn temp at ca 200°

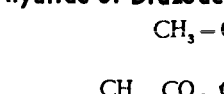
Refs: 1)R.W.Woodbridge, USP 1,854,776 (1932) & CA **26**, 3380(1932) 2)C.I.Johnson, Burnside Laboratory of E.I.duPont Co, Penns Grove,NJ; private communication, Oct 17, 1950 3)F.R.Schwartz, Pic Arsn Dover, NJ; private communication, April 1958

Acetylmethylglucoside. See Methylglucosideacetate

Acetylmethylnitrolic Acid; Pyruvonitrolic Acid or α-Nitro-α-isonitrosoacetone [1-Nitropropanon-(2)oxim-(1), in Ger], $CH_3.CO.C(NO_2):N.OH$, mw 132.08, N21.21–, OB to CO_2 -48.5%, OB to CO -12.1%. Plates, unstable, mp 55-62° with decompn; very sol in w & eth, insol in ligroin. Was prepd by mixing acetone with nitric acid(d1.14) and a little fuming HNO_3 and allowing the mixt to stand for 8 days at RT. An ether extraction gave on evaporn some acetylmethylnitrolic acid(Refs 1 & 2). Krauz & Stepanek (Ref 3) attempted and failed to prep tetranitromethane by nitration of acet. Instead, they obtd (after treating the nitrated prod with silver salt) an expl compd claimed to be the *Ag salt of acetylmethylnitrolic acid,* $CH_3.CO.C(NO_2):N.OAg$ (see also Acetone, Nitration)

Refs: 1)Beil **3**, 621 2)R.Behrend & H. Tryller, Ann **283**, 221-3(1894) 3)C.Krauz & J.Stepanek, ChemObzor **10**, 137-40(1935) & CA **30**, 3403(1936)

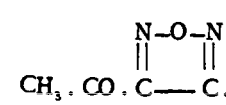
ACETYLMETHYLOXYDIAZOLES, $C_5H_6N_2O_2$, mw 126.11, N22.22%. Following isomers are listed in the literature: **4-Acetyl-5-methyl-1,2,3-oxydiazole; Acetylacetone-diazoanhydride or Diazoacetylacetoneanhydride,**



lt yel oil, does not solidify at -17°; dec ca 90° when distd at 13 mm press and explodes when distd at 760 mm; sol in w, alc & eth. Was prepd by diazotization of the prod obtd on redn of isonitrosoacetylacetone with Zn in dil H_2SO_4

Refs: 1)Beil **27**, 631 & (585) 2)L.Wolff et al, Ann **325**, 175(1902) & **394**, 36-8(1912) 3)H.Staudinger, Helv **4**, 239(1921)

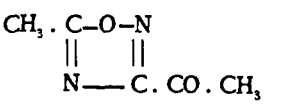
4-Acetyl-3-methyl-1,2,5-oxydiazole or 4-Acetyl-3-methyl-furazan,



liq, bp 154.5° at 743 mm; volat with steam; diff sol in w. Can be prepd by heating α'-acetyl-α-methyl-glyoxime diacetate with w

Refs: 1)Beil **27**, [692] 2)G.Ponzio & G. Ruggeri, Gazz **52** I, 294, 297(1922) & CA **16**, 2676(1922)

3-Acetyl-5-methyl-1,2,4-oxydiazole,



This compd is not described, but its oxime and oximebenzoate are in

Refs: 1)Beil **27**, [692] 2)G.Ponzio & G. Ruggeri, Gazz **53**, 301(1923) & CA **17**, 3874 (1923)

Acetylnaphthylamine. See Acetamidonaphthalene

Acetyl Nitrate or Acyl Nitrate (Nitroacetic anhydride), $CH_3.CO.O.NO_2$, mw 105.05, N13.33, OB to CO_2 -22.8%. Col, hyg, strongly fuming (in air) liq, d 1.24 at 15°, bp 22° at 70 mm; dec ca 60° to oxides of N, TeNM & a yel oil; expl violently on rapid heating or on contact with active oxides such as HgO; dec by w into AcOH & HNO_3. Can be prepd by the action of N_2O_5 on Ac_2O in the cold or by adding anhyd HNO_3 to a slight excess of Ac_2O, followed by distn in vacuo (Refs 1,2,3 & 5). Another method involves treating ketene with anhyd HNO_3 in the presence of inert diluents (such as CCl_4, CH_2Cl_2, etc) in cold (-10 to -40°)(Ref 12). Its solns are prepd by mixing fuming HNO_3 with AcOH & Ac_2O, in the cold. Wibaut (Ref 4) reported an expln of crude acetyl nitrate during its distn in vacuo and König(Ref 15) reported two explns attributed to acetyl nitrate

Acetyl nitrate is a very powerful nitrating agent and can be used for the prepn of anhyd inorg nitrates (Ref 5) as well as nitric esters of alcohols, PE and cellulose (Ref 10). Dis-

cussion on nitrating action of acetyl nitrate is given in Ref 8. With aromatic compds, acetyl nitrate shows a strong tendency to give ortho-substituted products. Thus toluene nitrated with acetyl nitrate gives 88% ortho- and only 12% para-nitrotoluene. Acetylnitrate solns intended for nitration purposes shall be prepd just before use by gradually running fuming nitric acid into freshly prepd mixt of Ac_2O with AcOH. All operations of mixing and nitration must be conducted in the cold, preventing excessive evoln of red fumes. Benzoyl nitrate is also a good nitrating agent

Following are some props of acetyl nitrate given in the literature: Vandoni & Viala (Ref 6) detd the compn of the vapors & partial pressures of acetyl nitrate, Ac_2O,N_2O_5 & HNO_3 at 0°; total press was also measured. Dunning & Nutt(Ref 11) gave freezing points of acetyl nitrate and anhyd HNO_3. Chédin & Fénéant(Refs 7 & 9) gave Raman spectra of mixts Ac_2O & HNO_3 at −10°, as well as some other props. Mal'kova(Refs 13 & 14) investigated the systems Ac_2O-HNO_3 by methods of physico-chemical analysis

Refs: 1)Beil **2**, 171,(79) & [175] 3)A. Pictet & E.Khotinsky, CR **144**, 210(1907); Ber **40**, 1164(1907) & CA **1**, 1117, 1543(1907) 3)J.Houben, "Die Methoden der Organischen Chemie," G.Thieme, Leipzig, 4(1941) 4)J.P.Wibaut, ChemWbl **39**, 534(1942) & CA **38**, 3129(1944) 5)A.Chrétien & G.Boh, CR **220**, 822-3(1945) & CA **40**, 3693(1946) 6)R.Vandoni & R.Viola, MSCE **32**, 80-6 (1945) & CA **42**, 4812(1948) 7)J.Chédin & S.Fénéant, CR **229**, 115-17(1949) & CA **44**, 6274(1950) 8)V.Gold et al, JCS **1950**, 2467-73 & CA **45**, 7538(1951) 9)J.Chédin & S.Feneant, MSCE **35**, 53-62(1950) & CA **46**, 2887-8(1952) 10)J.Chédin & A. Tribot, MSCE **36**, 37-42(1951); Bull Ass Tech Ind Papetière **5**, 435-43(1951) & CA **46**, 3757(1952) 11)W.Dunning & C.Nutt, TrFarad Soc **47**, 15-25(1951)& CA **45**, 6467(1951) 12)M.Reuter, GerP 849, 405(1952) & CA **47**, 4899 (1953) 13)T.Mal'kova, ReferatZhKhim **1954**, No 33882 & CA **49**, 9373(1955) 14)T.Mal'kova, ZhObshchKhim **24**, 1157-64(1954) & CA **49**, 2167(1955) 15)W.König, AngChem **67**, 157 (1955) & CA **49**, 6607(1955)

Acetyl Nitrite (Nitrosoacetic Anhydride), $CH_3.CO.O.NO$ mw 89.05, N 15.73%. Yel liq, dec by direct light, its vapors expl violently. Can be prepd by the action of nitrosylchloride on AcOAg under strong cooling (Ref 2) or by the action of dry $AgNO_2$ on acetyl chloride at −30 to −40°

Refs: 1)Beil **2**, 170 & (79) 2)L. Francesconi & U.Cialdea, AttiRAccadLincei [V], **12** II, 74-5(1903) & JCS **84** I, 788(1903) 3)E.Ferrario, Gazz **40** II, 97(1910) & JCS **98** I, 707(1910)

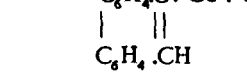
1-Acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocene. See 1-Aceto-3,5,7-trinitro-1,3,5,7-tetrazacycloöctane, under Aceto-tetrazacycloöctane

Acetylperchlorate or Acetyliumperchlorate, $CH_3CO{\cdot}ClO_4$. This compd was usually prepd by treating perchloric acid with an excess of acetic anhydride: $HClO_4 + Ac_2O \rightarrow AcClO_4 + AcOH$. This soln is a very efficient C-acetylating agent (Refs 2 & 3). Schmeisser(Ref 5) prepd $AcClO_4$ by treating Ag perchlorate with acetylchloride in ether

Refs: 1)Beil—not found 2)H.Mackenzie & E. Winter, TrFaradSoc **44**, 169(1947) & CA **42**, 6623(1948) 3)H.Burton & P.Praill, JCS **1950**, 1203 & 2034 4)Ibid, **1953**, 827 (Action of acetyl perchlorate on benzene and related compds) 5)M.Schmeisser, AngewChem **67**, 501(1955) & CA **49** 15590(1955)

Acetylperoxide. See Diacetylperoxide

9-Acetylphenanthrene,



mw 220.3, OB to CO_2 -268.8% OB to Co -152.5%. Bluish fluorescent leaflets (from alc), mp 123°; easily sol in eth, alc & bz, less sol in ligroin. Can be prepd by treating phenanthrene (dissolved in benz) with acetyl chloride in the presence of $AlCl_3$ (Refs 1 & 2). It was tried in France as a possible replacement for centralite in some solventless smokeless propellants (poudres SD) and found to be of interest. On extrusion, the colloid prepd from NC

and NG with 7% acetylphenanthrene and 2% centralite gave smooth, brilliant grains of dark green color. The propellant showed good stability in storage at temp as high as 90°

Refs: 1)Beil **7**(276) & [450] 2)IB Farbenind, GerP 493688, ChemZtr **1930** I, 3486 3)R. Dalbert & H. Fischeroulle, MP **30**, 283-300(1948)

Acetylphenylamine. See Aminoacetophenone

ACETYLPHENYLHYDRAZINE AND DERIVATIVES

Acetyphenylhydrazine, $C_8H_{10}N_2O$ Several isomers are listed in Beil **15**, 236, 241, (62, 63) & [91, 92, 286]

Azidoacetylphenylhydrazine, $C_8H_9N_5O$—not found in Beil or CA through 1956

Mononitroacetylphenylhydrazine, $C_8H_9N_3O_3$. Several isomers are listed in Beil **15**, 458, 464, 478, & [180, 183, 203]

Dinitroacetylphenylhydrazine, $C_8H_8N_4O_5$, mw 240.18, N 23.33%. One isomer, *2,4-dinitro-β-acetylphenylhydrazine* is listed in Beil **15**, 492

Trinitroacetylphenylhydrazine, $C_8H_7N_5O_7$, mw 285.18, N 24.56%. One isomer is described in the literature:

N'-Acetyl-N-(2,4,6-trinitrophenyl)-hydrazine or Acetylpicrylhydrazine, $CH_3.CO.HN.NH.C_6H_2(NO_2)_3$; lt yel ndls (from alc), grn-yel prisms (from dil alc); mp 210° (Ref 2), 223° (Refs 3 & 4) very diff sol in chlf & eth, insol in pet eth. Can be prepd by heating picryl hydrazine in glacial AcOH. No refs to its expl props

Refs: 1)Beil **15**, 496 2)A. Purgotti, Gazz **24I**, 572(1894) & JCS **68** I, 28(1895) 3) Th. Curtius & G.M. Dedichen, JPraktChem, **50**, 272(1894) & JCS **68** I, 30(1895) 4)H. Leemann. & H. Grandmoujin, Ber **41**, 1295 footnote 2(1908)

ACETYLPHENYLHYDROXYLAMINE AND DERIVATIVES

Acetylphenylhydroxylamine, $C_8H_9NO_2$. One deriv, *N-acetyl-N-phenyl-hydroxylamine,* $CH_3.CO.N(OH).C_6H_5$ is described in Beil **15**, 8 &(4). The deriv *O-acetyl-N-phenyl-hydroxylamine,* $CH_3.CO.O.NH.C_6H_5$ was not found in Beil or CA through 1956

Azidoacetylphenylhydroxylamine, $C_8H_8N_4O_2$—not found in Beil or CA through 1956

Mononitroacetylphenylhydroxylamine, $C_8H_8N_2O_4$—not found in Beil or CA through 1956

Dinitroacetylphenylhydroxylamine, $C_8H_7N_3O_6$, mw 241.16, N 17.43%. One isomer, *O-acetyl-N-(2,4-dinitrophenyl)-hydroxylamine* is described in Beil **15**, [11]

Trinitroacetylphenylhydroxylamine, $C_8H_6N_4O_8$, mw 286.16, N 19.58%. One isomer is described in the literature:

O-Acetyl-N-(2,4,6-trinitrophenyl)-hydroxylamine or O-Acetyl-N-picryl-hydroxylamine, $CH_3.CO.O.NH.C_6H_2(NO_2)_3$; dk yel ndls (from alc), mp 130°; insol in w, dissolves in soda soln (red color). Was prepd by treating N-(2,4,6-trinitrophenyl)-hydroxylamine with boiling Ac_2O. No mention of its expl props

Refs: 1)Beil **15**, [12] 2)W. Borsche, Ber **56**, 1942(1923) & CA **18**, 533(1923)

Acetylphenylnitramine. See Nitraminoacetophenone, under Aminoacetophenone

Acetylpicrylhydrazine. See N'-Acetyl-N-(2,4,6-trinitrophenyl)-hydrazine, under Acetylphenylhydrazine

Acetylpicrylhydroxylamine. See O-Acetyl-N-(2,4,6-trinitrophenyl)-hydroxylamine, under Acetylphenylhydroxylamine

ACETYLPYRROLE AND DERIVATIVES

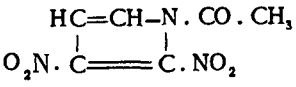
Acetylpyrrole, Oxoethylpyrrole or Methylpyrrylketone, C_6H_7NO. One isomer is listed in Beil **20**, 165 and another in Beil **21**, 271, (280) & [236]

Azidoacetylpyrrole, $C_6H_6N_4O$, mw 163.14, N 42.93%—not found in Beil or CA through 1956

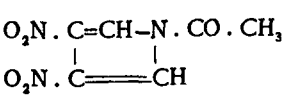
Mononitroacetylpyrrole, $C_6H_6N_2O_3$, mw 154.12, N 18.18%. Two isomers are listed in Beil **21**, 272

Dinitroacetylpyrrole, $C_6H_5N_3O_5$, mw 199.12, N21.10%. One isomer is described in the literature:

3,4-(or 4,5-)Dinitro-2-acetyl-pyrrole,



or



Its monohydrate, yel ndls (from w), mp 106-7°, looses H_2O and then melts at 114°; easily sol in alc, eth & hot benz. Was obtained, along with other products by action of fuming nitric acid on 2-acetyl-pyrrole, or on 4-nitro-2-acetyl-pyrrole. No refs to its expl props

Refs: 1)Beil **21**, 273 2)G.Ciamician & P. Silber, Ber **18**, 1463(1885)

ACETYLSALICYLIC ACID AND DERIVATIVES

o-Acetylsalicylic Acid or Aspirin (o-Acetylsalicylsäure or 2-Acetoxy-benzoesäure, in Ger), $CH_3.COO.C_6H_4.COOH$, mw 180.15, OB to CO_2 -159.9%, OB to CO -79.9%. Col ndls, mp, 135-6°, Q_c^v 859.3 kcal/mol; sol in w, sl sol in eth, very sol in benz. Was prepd by Gerhardt from acetyl chloride and Na salicylate (Ref 2). Can also be prepd by treating salicylic acid with Ac_2O or by other methods (Refs 1). Several salts are described in Ref 3

Its *lead salt,* $(C_9H_7O_4)_2Pb$. mw 565.50, was proposed as an ingredient of some propellent mixts

Refs: 1)Beil **10**, 67,(28) & [41] 2)Ch. Gerhardt, Ann **87**, 162(1853) 2)O.Gerngross & H.Kersarp, Ann **406**, 240-60(1914) 3)Ullmann **1**(1928), 163-4 4)Kirk & Othmer **1**(1947), 124-5

Azidoacetylsalicylic Acid or Salicylic Acid Triazoacetate, $CH_3.COO.C_6H_3(N_3).COOH$, mw 221.17, N19.00%. Col crysts, mp 104°; easily sol in alc,acet, EtOAc, eth, chlf; diff sol in w. Was prepd by treating salicylic acid with azidoacetylchloride in chlf and pyridine. No mention of explosive props

Refs: 1)Beil–not found 2)K.Freudenberg et al, Ber **65B**, 1190(1932) & CA **26**, 5072 (1932)

Mononitroacetylsalicylic Acid or Nitrosalicylic acid Acetate, $C_9H_7NO_6$. Several isomers are described in the literature, none of them is expl

Refs: 1)Beil–not found 2)CA **44**, 131e (1950) **44**, 8339f(1950); **45**, 2475a(1951); **45**, 7550b, 7551c(1951), **46**, 3019c(1952); **46**, 6109a (1952); **48**, 2676cd(1954)

Dinitroacetylsalicylic Acid, $C_9H_6N_2O_8$, mw 270.15, N10.37%–not found in Beil or CA through 1956

Trinitroacetylsalicylic Acid, $C_9H_5N_3O_{10}$, mw 315.15, N13.33%–not found in Beil or CA through 1956

Acetyltetranitroaniline. See 2,3,4,6-Tetranitroacetanilide, under Acetanilide

Acetyltetrazacycloöctane and Derivatives. See Acetotetrazacycloöctane

Acetyltetrazanonanediol-diacetate, Trinitro. See under Acetyldiacetoxytetrazanonane

Acetyltriazacyclohexane and Derivatives. See Acetotriazacyclohexane

Acetyltrinitroaniline. See 2,4,6-Trinitroacetanilide, under Acetanilide

Acetyltrinitrotetrazacycloöctane. See 1-Aceto-3,5,7-trinitro-1,3,5,7-tetrazacycloöctane, under Acetotetrazacycloöctane

Acetylure(Fr). Acetylide

Achema-Jahrbuch 1956/1958, Dechema, Frankfurt A/M(1957)(1068 pp) is a catalogue in Engl, Fr, Ger & Span of European chemical plants, technical institutes, apparatus and instruments

Acid and Base. A general discussion on acids and bases may be found in text books of general, inorg and org chemy. The following selected publications deal mainly with acids and bases:

1)P.Walden, "Salts, Acids and Bases," Translated from the German by L.F.Audrieth, McGraw-Hill, NY(1929) 2)N.Bjerrum, Chem-Revs **16**, 287-304(1935)(Salts, acids and

bases)(20 references) 3)G.N.Lewis, J Frankl Inst **226**, 293-313(1938)(Acids and bases) 4)W.F.Luder, ChemRevs **27**, 547-83(1940) (Electronic theory of acids and bases)(109 references) 5)R.P.Bell, "Acid-Base Catalysis," Clarendon Press, Oxford(1941) 6) W.F.Luder & S.Zuffanti, "The Electronic Theory of Acids and Bases," Wiley, NY (1946) 7)R.P.Bell, QuartRevs **1**, 113-25 (1947)(The use of the terms "acids" and "bases") 8)G.B.L.Smith, "Acid-Base System" in Kirk & Othmer, **1**(1947), 128-137 9)R.P.Bell, "Acids and Bases. Their Quantitative Behavior," Wiley, NY(1952) 10)R.P. Bell, Acid-Base Catalysis and Molecular Structure, 151-210 in "Advances in Catalysis" **4**, Academic Press, NY(1952)

Acid, Abietic. See Abietic Acid

Acid, Acetic. See Acetic Acid

Acid, Adipic. See Adipic Acid

Acid Analyses are given under individual acids, such as acetic, nitric, sulfuric, etc (See also Acidity in Acids)

Acid Anhydrides, Analysis is described in Organic Analysis, Interscience, NY, **3** (1956)

Acid Boiling of Nitrocellulose. Same as Preliminary Boiling of Nitrocellulose. See Nitrocellulose, under Cellulose

Acid, Boric. See Boric Acid

Acide azoteux(Fr), Nitrous Acid

Acide azothydrique (Fr). Hydrazoic Acid (see under Azides, Inorganic)

Acide azotique (Fr). Nitric Acid

Acide carbazotique (Fr). Picric Acid (see under Phenol)

Acide carbolique (Fr). Phenol

Acid Egg or Blowcase is a type of "displacement pump"(qv) used for transferring acids and other corrosive liquids from one apparatus to another by means of compressed air. Its description is given in Refs 1&2. The use of acid eggs is safe for acids having no dissolved org matter, otherwise the gaseous phase in the egg (in the presence of air) may develop into an expl mixt and in case of spark or overheating, result in expln (see also Air Lifts)

Refs: 1)Perry(1950), 1439 2)Riegel, Chem Mach(1953), 172

Acid Elevator with ball valves, operated by air and made of chemicalware, is described by Riegel, ChemMach(1953), 171

Acide métazonique(Fr). Nitromethane

Acide oxypicrique (Fr). Trinitroresorcinol

Acide phénique (Fr). Phenol

Acide de Sprengel (Fr). Sprengel Explosive

Acide styphnique (Fr). Trinitroresorcinol

Acid, Hydrazoic. See Hydrazoic Acid, under Azides, Inorganic

Acid, Hydrochloric. See Hydrochloric Acid

Acidity in Acids. If the acid used in manuf of expls, propellants etc is white nitric, its acidity is detd by dissolving (without loss of fumes) a weighed sample in w and titrating the resulting soln with std NaOH soln in presence of an indicator such as methyl red

$$\% \text{ Acidity} = \frac{R \times N \times 63.016}{W \times 10} = TAN,$$

where R = burette reading, 63.016 = equivalent of nitric acid, W = wt of sample (For abbreviations used in this sectn, see below)

If no other acid is present the above value is equal to TNN and also to ANN. Water content of such acid is equal to 100.00-TAN

If nitric acid is yellow or red it means that some NO_2 is present in addn to HNO_3. On dilution of such acid with w, the following reaction takes place: $2NO_2 + H_2O = HNO_2 + HNO_3$. The resulting nitrous and nitric acids will be included in the value of total acidity of the acid:

$$TAN = \frac{R \times N \times 63.016}{W \times 10}$$

The next step is to det NO_2 content, which is usually done by drowning a sample

under w(without loss of fumes) and titrating the resulting soln with std $KMnO_4$ soln:
$5NO_2 + KMnO_4 + H_2O = KNO_3 + Mn(NO_3)_2 + 2HNO_3$

$$\%NO_2 = \frac{R \times N + 46.008}{W \times 10},$$

where 46.008 is equiv of NO_2

As NO_2 forms equal amts of HNO_2 and HNO_3, only half of it goes for the formation of HNO_3 and the other half must be deducted from TAN

$$ANN = TAN - \tfrac{1}{2}(\%NO_2) \times \frac{63.016}{46.008},$$

where 63.016 is equiv of HNO_3 and 46.008 equiv of NO_2

For detn of acidity in colorless, nonfuming sulfuric acid, an aq soln of a weighed sample is titrated with std NaOH in the presence of an indicator, such as methyl red

$$\%\ Acidity = \frac{R \times N \times 49.041}{W \times 10} = TAS,$$

where 49.041 is equiv of sulfuric acid (½ of mw)

If no other acid is present the above value is equal to TSS and also to ASS. The w content is equal to 100.00–TAS

In case of fuming sulfuric acid (oleum) not contg any other acids, total acidity is higher than 100% and w content will be expressed as a negative value, such as –5.50%. It is customary with such acids to express their acidity in terms of SO_3 as,

$$\frac{R \times N \times 40.033}{W \times 10}$$

As oleums used in manuf of expls freeze at moderate winter temps, they usually contain 4-6% nitric acid which acts as an antifreeze. Outline of analysis of such oleums will be given after brief description of acidity detn in mixed nitric-sulfuric acid

In making MA's from commercial oleums and anhyd nitric acids, nitrogen dioxide present in nitric acids, reacts with sulfuric acid forming nitrosylsulfuric acid (nitroso), as follows: $2NO_2 + H_2SO_4 = HNOSO_4 + HNO_3$. The nitric acid in MA is therefore equivalent to the AN in the original NA, plus the HNO_3 equiv of ½ of the NO_2. The sum of these quantities is termed in the Hercules Manual (Ref 2), "**Available HNO_3**" (see Note b, below). Nitroso reacts with H_2O forming nitrous and sulfuric acids: $HNOSO_4 + H_2O = HNO_3 + H_2SO_4$ and these acids will be included in TA detn by titration with std NaOH soln:

$$TAS = \frac{R \times N \times 49.041}{W \times 10}$$

This value includes all of the free HNO_3, free H_2SO_4 and the nitric and sulfuric acid equivalents of nitroso

Sulfuric acid content can be detd by titrating an aq soln of MA (after removal of NA by evapg a sample in an open dish until the disappearance of nitrogen oxides odor, and adding a few drops of H_2O to break up nitroso) with std NaOH soln:

$$TSS = \frac{R \times N \times 49.041}{W \times 10},$$

where 49.041 is sulfuric acid equiv

This value includes all of the free H_2SO_4, plus the sulfuric acid equiv of nitroso

Nitric acid is usually detd by difference $TNS = TAS - TSS$ and $TNN = TNS \times \frac{63.016}{49.041}$

It can also be detd by the ferrous sulfate method (which gives ANN). This method eliminates detn of sulfuric acid by evapn, because it can be detd by difference knowing total acidity and nitric acid content

Nitroso can be detd by titrating a sample of MA drowned in w (without loss of fumes) with std $KMnO_4$ soln until the appearance of permanent pink coloration: $5HNOSO_4 + 2KMnO_4 + 2H_2O \rightarrow K_2SO_4 + 2MnSO_4 + 5HNO_3 + 2H_2SO_4$

$$\%N\text{-}so = \frac{R \times N \times 63.54}{W \times 10},$$

where 63.54 is nitroso equiv

Sulfuric acid equiv of nitroso is equal to $(\%N\text{-}so) \times \frac{98.082}{127.082} = (\%N\text{-}so) \times 0.7718$ and

nitric acid equiv of nitroso

$$(\%N\text{-so}) \times \frac{63.016}{127.082} = (\%N\text{-so}) \times 0.4959$$

Following is an example of analysis of MA, taken from lab files of Keystone OW, Meadville, Penna:

TAS(by titration with NaOH)	87.3%
TSS (by titration of the residue after avapg HNO_3 from a sample of MA)	42.11%
N-so(by titration with $KMnO_4$)	0.31%

TNS = TAS - TSS = 87.31 - 42.11 = 45.20%

$$TNN = TNS \times \frac{HNO_3}{\frac{1}{2}H_2SO_4} = 45.20 \times \frac{63.016}{49.041} = 58.08\%$$

$$ASS = TSS - (\%N\text{-so}) \times \frac{H_2SO_4}{HNOSO_4} = 42.11 - (0.31 \times 0.7718) = 41.87\%$$

$$ANN = TNN - (\%N\text{-so}) \times \frac{HNO_3}{HNOSO_4} = 58.08 - (0.31 \times 0.4959) = 57.93\%$$

To report: H_2SO_4 41.87, HNO_3 57.93, N-so 0.31 and H_2O(by diff) -0.11%. The sum of actual sulfuric, actual nitric and nitroso is called **total actual acidity (TAA)**

Notes: a)If solids or mud are present, they have to be also reported b)If HCl is present (can be detd by Mohr or Volhard method), the Hercules Manual (Ref 2) gives: "available HNO_3" = TAN -(0.68483 × NO_2) + (1.72812 × HCl) and **actual HNO_3** = TAN -(1.36967 × NO_2) + (1.72812 × HCl)

Analysis of oleum contg nitric acid as an antifreeze is essentially the same as that of MA. Following is an example: TAS (detd by titration with NaOH) 105.92%

ANN(by a ferrous sulfate method) (see Note below) 5.10%

N-so(by titration with $KMnO_4$) 0.30%

Nitric acid equiv of nitroso = 0.30 × 0.4959 = 0.15%

TNN = ANN + 0.15 = 5.10 + 0.15 = 5.25%

$$TNS = 5.25 \times \frac{49.041}{63.016} = 4.08\%$$

TSS = TAS - TNS = 105.92 - 4.08 = 101.84%

Sulfuric acid equiv of nitroso = 0.30 × 0.7718 = 0.22%

ASS = TSS -0.22 = 101.84 -0.22 = 101.62%

H_2O = 100.00 - (101.62 + 5.10 + 0.30) = -7.08%

To report: TAS 105.92, H_2SO_4 101.84, HNO_3 5.25 and N-so 0.30%

Note: Nitric acid in oleum can also be detd by nitrometer method, which gives TNN

Abbreviations: **AN**–actual nitric (unbound acid which actually participitates in nitration), **ANN**–actual nitric as nitric, **ANS**–actual nitric as sulfuric, **N-so**–nitroso(nitrosylsulfuric acid, $HNOSO_4$), **AS**–actual sulfuric, **ASN**–actual sulfuric as nitric, **ASS**–actual sulfuric as sulfuric, **MA**–mixed acid, **N**–normality of std soln, **NA**–nitric acid, **N-so**–nitroso, **R**–burette reading, **SA**–sulfuric acid, **TA**–total acidity, **TAA**–total actual acidity (the sum of actual sulfuric, actual nitric and actual nitroso), **TAN**–total acidity as nitric, **TAS**–total acidity as sulfuric, **TNN**–total nitric as nitric, **TNS**–total nitric as sulfuric, **TSS**–total sulfuric as sulfuric, **W**-weight of sample (See also under individual acids and their mixtures)

Refs: 1)G.D.Clift & B.T.Fedoroff, "A Manual for Explosives Laboratories," Lefax, Inc, Philadelphia, Penna, vol 1(1942), vol 3(1043) 2)Hercules Powder Co.,"Laboratory Manual," Wilmington, Del (1946)

Acidity of Aromatic Nitrocompounds is discussed in text-books on org chem and in the following papers: 1)R.Schaal, CR **239**, 1036-7(1954 & CA **49**, 3624(1955) 2)H.Brockmann & E.Meyer, ChemBer **87**,81-6(1954) & CA **49**,6153(1955)

Acidity in Explosives. The presence of acid in explos is usually due either to mech entrainment of some of the nitrating acid resulting from defects in the manufg process, such as insufficient boiling or washing etc, or to spontaneous decompn of the expl due to heat or aging. In some cases the presence of moisture may cause hydrolysis and the formation of acidic products

The org nitrates, such as NG or NC, are the types most sensitive to acids and most likely to decomp on aging or storage at elevated temps. The presence of very

slight traces of acids in these compds will hasten decompn of the expl, resulting in the evoln of various oxides of nitrogen, and in the presence of moist, in the formn of nitric and nitrous acids. Once the decompn is started it becomes autocatalytic. The decompn reactions are also exothermic so that in badly deteriorated compositions enough heat may develop to cause them to ignite

The mechanically entrapped acids may be sulfuric, nitric, acetic , or any other acid used in the manufg process. These acids will also initiate the decompn of org nitrates as described above

The nitroaromatic compds, such as TNT, are usually quite stable in the presence of traces of acids but not when in contact with metals. The danger from occluded acids in the case of the nitrocompds arises from the reaction of the acid with the metal container in which the expl is usually confined. The metal salts produced by the action of the acids on the metal are capable of reacting with the nitrocompds to form highly sensitive metallo-org compds, the presence of which greatly increases the hazards involved in handling and use. Some expls, such as PA, are normally acidic because of their chem structure, even when pure. In the presence of some metals, highly expl salts(such as picrates) may form

Not all expls and propellants that are acidic show low stability when subjected to the ordinary heat tests. However, such acidic compds usually ignite more readily than neutral substs. In the case of acidic propellants some irregularity in burning may be observed. In many cases such propellants burn completely and exhibit abnormalities in chamber press and range in firing tests

The effect of various acids on the stability of NC was examined by C.Krauz & A. Majrich, ChemObzor **7**, 209-16(1932) & CA **27**, 2812(1933). They found that mineral acids lower the stability of NC as also do some org acids such as aliphatic carboxylic acids and some of their derivatives. Aliphatic dicarboxylic acids have only a slight influence on stability but hydroxy-dicarboxylic acids act also to some extent as stabilizers (See also Alkalinity in Explosives, Antacids and Stabilizers)

Acidity in Explosives Test is one of the std tests for the detn of purity of expls and propellants. A rapid qualitative method consists of touching a sample with a strip of moistened indicator paper, such as blue litmus paper. For quantitative detn of acidity two methods can be used: a) If the sample melts below 100° (such as TNT, DNT, etc) shake it with boiling neutral w, cool, filter the extract and titrate it with std alkali (such as 0.05N NaOH) in presence of an indicator or det the pH of the extract b) If the sample melts above 100° (such as RDX) or does not melt at all (such as NC), dissolve it in neutral acet, add neutral w to reppt the sample, filter the slurry and det the acidity of filtrate either by titration with std alkali or by the pH method

More detailed descriptions are given under individual expls and propellants (See also Alkalinity in Explosives Test and also Angeli & Erani Test)

Acidity of Mixed Nitric-Sulfuric Acids, briefly described under Acidity in Acids, can also be detd by conductometric titration as described by K.K.Savich, ZavodLab **8**, 1059(1939)

Acidity of Nitrating Bath, detn by the use of nomograph is described by Y.Lacroix, MP **37**, 521(1955)

Acidity of Weak Acids, detn by polarographic method is described by I.A.Korshunov et al, ZhAnalKhim, **6**, 96(1951)

Acid Magenta. A mixt of disulfonic and trisulfonic acids of pararosaniline, used as a dye or stain. Was proposed for coating crysts of AN to render them non-hygroscopic and thus make them more suitable for use in expls, propellants and fertilizers. Quantities as low as 0.01-0.03% proved to be sufficient for effective waterproofing

Refs: 1)Hackh(1944),358(under Fuchsin) 2)J.Whetstone, IEC **44**, 2663-7(1952)(15 refs)

Acid, Mixed. See Mixed Acid

Acid, Nitric. See Nitric Acid

Acid, Nitrous. See Nitrous Acid

Acid Number or Acid Value is the number of milligrams of KOH required to neutralize the acidic constituents of one gram of material

Ref: Clark & Hawley(1957), 10

Acido (Ital); **Ácido**(Span). Acid

Acidopentamminecobalt (III) **Salts.** Prepn and some props of several salts are given in OrgSynt CollVol 4(1953), 171-6

Acid, Oxalic. See Oxalic Acid

Acid, Picric. See 2,4,6-Trinitrophenol, under Phenol

Acid Pumps. Various centrifugal pumps as well as some piston pumps are described in Riegel ChemMach(1953), 138-171

Acid Pump. A special type of pump operated by air pressure and manufd by the Tungstone Products Ltd, Market Harborough, England, is described in Riegel, ChemMach (1953), 173-5

Acid Removal in the Manufacture of Explosives is discussed under individual expls

Acid-Resistant Materials. As acid plants are usually attached to plants manufg expls, it is important to have some knowledge of acid-resistant materials, such as metals, bricks, plastics, gaskets, paints, cements and putties, etc.Basic info on this subject may be found in the refs listed below. A common type of acid-proof putty is one consisting of powd red iron oxide and asbestos fiber in a petroleum grease. During WW II, this type of putty was used at many US Ord Plants, including Wabash Ordnance Works

An asbestos putty may be prepd by mixing 50 parts asbestos fiber, 2 ps white lead, 1 p Ba sulfate in a heavy oil or grease. An asbestos cement may be prepd by thoroughly mixing, just before use, a powd asbestos(free from cellulose filling) with a coml Na silicate soln(water glass). This cement may be applied on cracks but not on joints because it becomes as hard as stone on standing. A litharge putty may be prepd by mixing 73 ps of litharge, 8 ps of flock asbestos and 19 ps linseed oil. The mixt sets in about 7 days and resists acids up to about 50% strength. The so-called "German putty" consists of 70 ps quartz flour, 8.5 ps fluosilicate, 1.5 ps clay in 20 ps of water glass (Na_2O 20, SiO_2 60 and H_2O 20%)

A plastic rubber cement which resists most acids may be prepd by dissolving, with gentle heating and stirring, 1 p of rubber (reclaimed) or caoutchouc in 2 ps linseed oil and then adding 1 p of clay

Following are examples of acid-resistant materials available in commerce and suitable for expls plants:
a)DuPont Rubber Putty, manufd at DuPont's Repauno and Carney's Point plants b)Pecora Cement, manufd by Semmet-Solvay Co. This has a silicate base c)Charlab Chemical Putty, manufd by Charlotte Chemical Lab, Inc, Charlotte, NC. This stays plastic and may be applied at joints

Many plastic materials are acid resistant, among them is "Teflon" (polytetrafluoroethylene). This material resists acids but does not withstand high temps. "Silastic" resists nitric acid well. Polyethylene plastic and chlorinated polyethylene are also good acid resistors

For the prepn of acid-resistant cloth some vinyl chloride is dissolved in methyl-ethyl ketone and 10 to 15% of tricresyl phosphate is added to this. The fabric is then impregnated with this soln and the solvent is evapd

Refs: 1)H.Bennett, ed, "The Chemical Formulary," Chem Pub Co, Brooklyn, **4**,28 (1939) & v 6, p 29(1943) 2)G.D.Hiscox & T.O'Connor Sloane, "Fortunes in Formulas," Books,Inc,NY(1947) 3)Perry(1950),453 (Acid resisting cements), pp 1461-1526(Acid resisting construction materials); pp 1526-

34(Acid resistant metals and alloys); pp 1534-36(Acid resistant carbon, graphite, cement, mortar, putty, ceramics and plastics) 4)Kirk & Othmer **8**(1952), 824(Materials of Construction)(See also Ceramics in **3**, pp 575-585; Coatings in **4**, pp 145-89; Corrosion in **4**, pp 487-529; Metallic Coatings in **8**, pp 898-922 and Packing Materials in **9**, pp 762-770) 5)C.P.Bacha, J.S.Schwalje & A.J.DelMastro, "Elements of Engineering Materials," Harper, NY(1957) 6)Annual Reviews of Materials of Construction appear in Chemical & Engineering News and Industrial & Engineering Chemistry 7)Chemical Abstracts, Decennial Indexes, under Acid-Resistant Materials and Sealing Compositions 8)M.H.Sandler, "Second Report on the Development of a Fuming Nitric Acid Resistant Paint," Aberdeen PG, Md, Project No. TB4-006D & 593-32/006(1958)

Acid, Spent. See Spent Acid

Acid, Styphnic. See Trinitroresorcinol, under Resorcinol

Acid Sulfonitric. Same as Mixed Nitric-Sulfuric Acid

Acid, Tartaric. See Tartaric Acid

Acid, Tests. See under individual acids, such as *acetic, hydrochloric, mixed, nitric, sulfuric,* etc

Acids Used in Manufacture and Analyses of Explosives. Most expls (such as PA, TNT, PETN, NG, etc) are prepd by treating org compds with nitric acid in the presence of sulfuric acid or oleum, which binds the H_2O formed during the nitration reaction. Thus, the prepn of NG may be written as follows:

$$C_3H_5(OH)_3 + 3HNO_3 + xH_2SO_4 \rightarrow C_3H_5(ONO_2)_3 + xH_2SO_4 \cdot 3H_2O$$

In the prepn of RDX, nitric acid, glacial acetic acid and acetic anhydride are used. The last two compds serve as dehydrating agents. Mixed nitric-sulfuric acids are also used in the prepn of NC, which is an important component of many smokeless propellants and an occasional ingredient of some dynamites. Acids are also used to neutralize the residual basicity of some expls due to the use of alkalies in purification. For instance, some H_2SO_4 is added to molten TNT prior to flaking to neutralize the slight excess of Na_2CO_3 which is added to crude TNT before "selliting" it. Nitric acid is used to form a number of expls directly by neutralizing some bases, eg, ammonia, methylamine, hydrazine, etc

More info on uses of acids is given under individual acids and explosives

Acid, Waste. See Waste Acid

Acme or Liardet Powder. A blasting expl patented in 1893-4 by Liardet in England and France. It was prepd by thoroughly mixing 1 part wood meal(or pulverized pine needles) and 2 ps PA dissolved in $\frac{2}{3}$ ps of tar at 100°. A preheated mixt of K chlorate and K nitrate was then added to this. Due to the presence of a chlorate, the powder was very sensitive and caused two disastrous expls, one in Australia in 1893 and another in Pittsburgh in 1894

Ref: Daniel(1902), 5 & 406

Aconitic Acid or Propene-1,2,3-tricarboxylic Acid, $(HO_2C).CH_2.C(CO_2H):CH(CO_2H)$ mw 174.11, OB to CO_2 -82.7%, OB to CO -27.6%. Wh to yel crysts, mp ca 195° with decompn, Q_c^v ca 480 kcal/mol; sol in w and alc, very sl vol in ether. Can be prepd by heating citric acid with 2:1-H_2SO_4:H_2O(Ref 2) or by other methods listed in Ref 1. Forms numerous salts. Used in org syntheses and in the prepn of plastics some of which might be used in ord items

Refs: 1)Beil **2**,849(327) & [693] 2)Org-Synth, Coll Vol 2(1943), 12

Acoustics. A branch of science which treats of the phenomena and laws of sound waves (including their production, transmission and effects), and other vibrations in elastic bodies(See also Ultrasonics)

Refs: 1)D.C.Miller, "Sound Waves, Their Shape and Speed," Macmillan, NY(1937) 2)H.F.Olson, "Applied Acoustics," Blackiston, Philadelphia(1939) 3)A.Wood, "Acoustics," Interscience, NY(1944) 4) H.F.Olson, "Elements of Acoustical Engineering," Van Nostrand, NY(1947) 5)L.

L.Baranek, "Acoustic Measurements," Wiley,NY(1949) 6)L.L.Baranek, "Acoustics," McGraw-Hill, NY(1954)

Acoustic Guidance Systems for Missiles. During WWII the Germans utilized the sound produced by airplane engines as a guiding medium. The original acoustic homing device was called "Kranich" and the later version "Pudel." A brief description of these devices is given in PATR 2510 (1958), pGer 83

General description of guidance systems is given by A.S.Locke et al, "Guidance," Van Nostrand, vol 1(1955) of series edited by G.Merrill and entitled "Principles of Guided Missile Design' and in other books listed under "Guided Missiles"

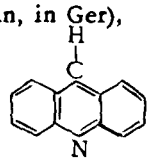
Acqua (Ital). Water

Acrawax. A hard, lt brn synth wax, mp 95-7°; sol in hot alc, toluol, butyl acetate or turpentine, sl sol in mineral oil or mineral spirits, insol in w. A brand name for products, including Acrawax B and Acrawax C, which are modifd fatty acid esters mfd by the Glyco Products Company, Brooklyn, NY. Waxes are used to desensitize expl; with ca 10% incorporated in simple mixt or less is used when wax is applied as coating . Its uses in Ordnance are given in some classified reports

Ref: F.M.Turner, "The Condensed Chemical Dictionary," 4th Ed, Reinhold, NY (1950), 12

ACRIDINE AND DERIVATIVES

Acridine or Dibenzopyridine (2.3:5.6-Dibenzo-pyridin, in Ger),



mw 179.21, OB to CO_2 -272.3%. Rhb crysts (from aq alc), mp 110-1°, bp 345-6°, Q_c^p 1577.8 kcal/mol; sol in alc, eth & CS_2; sl sol in w. Was first obtained in 1870 from crude anthracene(Ref 2). Can be prepd by dechlorination of 5-chloroacridine with hydrogen in presence of Raney nickel or by other methods. It forms nitrocompds, salts and addition compds, some of which may find use in the expl industry

Refs: 1)Beil **20**,459,(171) & [300] 2)C. Graebe & H.Caro, Ber 3,746(1870) 3)A. Albert & J.B.Willis,Nature 157,341(1946) 4)A.A.Morton, "The Chemistry of Heterocyclic Compounds"; McGraw.Hill NY(1946), 326-33 5)Kirk & Othmer, **1**(1947),168-70 6)A.Albert, "The Acridines," Longmans, Green, London(1951) 7)A.Albert, "Acridines," in "Heterocyclic Compounds," edited by R.C.Elderfield, Wiley, NY(1952), 491-563 8)R.M.Acheson & L.E.Orgel, "Acridines," in "Chemistry of Heterocyclic Compounds," edited by A.Weissberger, Interscience, NY(1956)

Azidoacridine, $C_{13}H_8N_4$—not found in Beil or in CA through 1956

Diazidoacridine, $C_{13}H_7N_7$—not found in Beil or in CA through 1956

Mononitroacridine, $C_{13}H_8N\,NO_2$, mw 224.21, N12.50%. Its 2- and 4-nitro isomers are listed in Beil **20**,462-3,(172) & [301-2]

Dinitroacridine, $C_{13}H_7N(NO_2)_2$, mw 269.21, N 15.61%. Its 2,4- and x,x-dinitro are listed in Beil **20**, 463 & (172)

Note: Some nitrated acridines were first prepd by C.Graebe & H.Caro, Ann **158**,275-7(1871), who used warm nitric acid of d ca 1.45 as the nitrating medium. Formulas reptd by G & C are not the same as given in Beil,

Trinitroacridine, $C_{13}H_6N(NO_2)_3$, mw 314.21, N 17.83%—not found in Beil or in CA through 1956

Tetranitroacridine, $C_{13}H_5N(NO_2)_4$, mw 359.21, N 19.50%—not found in Beil or in CA through 1956

Note: C.Graebe & H.Caro, Ann **158**,277(1871) claimed the prepn of tetranitroacridine by nitrating acridine with mixed nitric-sulfuric acid. The compd was in the form of reddish plates having N content 16.38%. This compd did not appear to be tetranitroacridine. No refs to its expl props were made

Acridine Dichromate, $(C_{13}H_9N)_2 . H_2Cr_2O_7$, orange-yel ndls; was prepd in 1871 by treating a salt of acridine (such as nitrate) with $K_2Cr_2O_7$. Its structure was detd by Kahn(Ref 3)

Refs: 1) Beil **20,** 461 2)C.Graebe & H.Caro, Ann **158,**273(1871) 3)S.Kahn Ann **279,**274(footnote)(1894)

Acridine Nitrate, $C_{13}H_9N . HNO_3$, mw 242.23, N 11.57%. Yel crysts, mp-dec, sol in w, Q_c^p 1553.3 kcal/mol(Ref 3). Was first prepd in 1871(Ref 2) by mixing acridine with equim quantity of cold HNO_3(d1.45). Willis (Ref 3) prepd it by adding acridine to normal HNO_3 free of HNO_2

Refs: 1)Beil **20,**461 2)C.Graebe & H. Caro, Ann **158,**273(1871) 3)J.B.Willis, Tr FaradSoc **43,**100(1947) & CA **41,**5008(1947)

Acridine Perchlorate, $C_{13}H_9N . HClO_4$, yel to grn-yel crysts, mp 238°(dec). Can be prepd by treating AcOH soln of acridine with perchloric acid. Its expl props were not reported

Refs: 1)Beil **20,**(171) & [300] 2)K.A. Hofmann et al, Ber **43,**1083(1910) 3)V. Cordier, Monatsh **43,**530(1922) & CA **17,** 1763(1923)

Acridine Picrate, $C_{13}H_9N . C_6H_3N_3O_7$, yel prisms (from alc), mp-dec ca 208°; very sol in w, alc or benz. Can be prepd by rapid crystn from hot alc soln of an equim mixt of acridine and PA. Its expl props were not reported

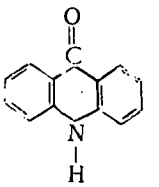
Refs: 1)Beil **20,**461 2)R.Anschütz, Ber **17,**438(1884)

Acridine-1,3,5-Trinitrobenzene Salt, $C_{13}H_9N . C_6H_3N_3O_6$, amber colored ndls (from alc), mp 115°. Was prepd by crystg a mixt of acridine and 1,3,5-TNB from hot alc. Its expl props were not reported

Refs: 1)Beil **20,**(171) 2)J.J.Sudborough & S.H. Beard, JCS **97,**796(1910)

ACRIDONE AND DERIVATIVES

Acridone or 9(10H)-Acridone, called in Beil 9-Oxo-9.10-dihydro-acridin,



mw 195.21, N 7.17%, OB to CO −135.2%. Col crysts, mp 354°; insol in w, sl sol in alc or eth. It was first prepd in 1880(Ref 2) by treating acridine with chromic acid, but its identity was not established until 1892 (Ref 3). Its method of prepn by heating on a water bath N-phenylanthranilic acid with concd H_2SO_4 is given in Ref 4. Can be nitrated to form nitrocompds

Refs: 1)Beil **21,**335,(312) & [280] 2)C. Graebe & H.Caro, Ber **13,**103(1880) 3)C. Graebe & K.Lagodzinski, Ber **25,**1733(1892) & Ann **276,**45(1893) 4)OrgSynth, Coll Vol **2**(1943), 15 5)A.A.Morton, "The Chemistry of Heterocyclic Compounds," McGraw-Hill NY(1946),333 6)Kirk & Othmer **1,**(1947), 169 7)K.M.Acheson & L.E.Orgel, "Acridones," pp 105-198 in "Chemistry of Heterocyclic Compounds," edited by A.Weissberger, Interscience, NY(1956)

Azidoacridone, $C_{13}H_8N_4O$—not found in Beil or in CA through 1956

Diazidoacridone, $C_{13}H_7N_7O$—not found in Beil or in CA through 1956

Mononitroacridone, $C_{13}H_8N_2O_3$, mw 240.21, N 11.66%. Four isomers are listed in Beil **21,**337-8,(313) & [282]

Dinitroacridone, $C_{13}H_7N_3O_5$, mw 285.21, N 14.73%. The following isomers are listed in the literature.

2,4-Dinitroacridone, orange-yel leaflets, mp > 360°

Refs: 1)Beil **21,**338 2)F.Ullmann et al, Ber **40,**4798(1907)

2,7-Dinitroacridone, crysts, mp 360°

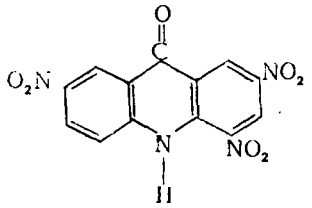
Refs: 1)Beil—not found 2)A.A.Goldberg, BritP 602,331(1948) & CA **42,**8827(1948)

4,5-Dinitroacridone, orange crysts, mp 257-8°

Refs: 1)Beil—not found 2)E.R.Klein & F.N.Lahey, JCS **1947,**1418 & CA **42,**1277 (1948)

Trinitroacridone, $C_{13}H_6N_4O_7$, mw 330.21 N 16.97%, OB to CO_2 −106.6%, OB to CO −43.6%. The following isomer is listed in the literature

2,4,7-Trinitro-9(10H)-acridone,

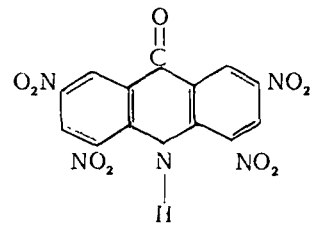


Yel ndls, mp 277°. It was isolated from the products of nitration of 9-aminoacridine

Refs: 1)Beil-not found 2)A.Hampton & D.Magrath, JCS **1949**, 1008-9 & CA **44**, 633 (1950)

Tetranitroacridone, $C_{13}H_5N_5O_9$, mw 375.21, N 18.67%, OB to CO_2 −83.2%, OB to CO −27.7%. The following isomer is described in the literature:

2,4,5,7-Tetranitro-9(10H)-acridone,



Yel crysts, mp > 350° with sublimation (Ref 2), 337° (Ref 3); diff sol in org solvents. Can be prepd by heating thioacridone with fuming nitric acid in a sealed tube at 240° or by other methods. Its expl props were not investigated

Refs: 1)Beil **21**, 338 2)A.Edinger & W. Arnold, JPraktChem **64**, 488(1901) 3)C.W. Pohlmann, Rec **55**, 747(1936) & CA **30**, 7111 (1936)

Acrolein or Acrylic Aldehyde (Propenal, Allyl Aldehyde or Ethylene Aldehyde), CH_2: CH · CHO, mw 56.06, OB to CO_2 −199.8%, OB to CO −114.2%. Col liq, mp −87.7°, bp 52.5°, d 0.8389 at 20°/4°, Q_c^v 391.5 kcal/mol (Ref 3). Very sol in w, sol in alc or eth. Can be prepd by heating glycerin with dehydrating agents such as $KHSO_4$. It is dangerous when exposed to heat or flame (Ref 5)

This poisonous, lachrymatory and highly obnoxious-smelling substance is usually given off as a vapor during blasting operations in the "blow-out shots" (qv)(Ref 2) and has also been isolated from spent acids from the manuf of NG and PETN

Refs: 1)Beil **1**, 725(377) & [782] 2)Marshall 2(1917), 759 3)C.Moureau, JChimPhys **18**, 333(1920) 4)Kirk & Othmer **1**(1947), 173-5 5)Sax(1957), 243-4

ACRYLAMIDE AND DERIVATIVES

Acrylamide (Propenamid or Acrylsäureamid in Ger), CH_2: CH . CO . NH_2; ndls, mp 84-5°, dec < 125°. Can be prepd by saturating a cooled benz soln of acrylic chloride with dry NH_3(Refs 2 & 3)

Refs: 1)Beil **2**, 400,(186) & [388] 2)C. Moureau, BullFr [3] **9**, 417(1893) & JCS **64I**, 695(1893) 3)J.van der Burg, Rec **41**, 23(1922)

Azidoacrylamide, N_3 . CH : CH . CO . NH_2—not found in Beil or in CA through 1956

3-Nitroacrylamide, O_2N . CH : CH . CO . NH_2, mw 116.08, N24.14%; yel crysts, mp 165° (dec). Was prepd by heating a mixt of 3-nitroacrylonitrile and 85% sulfuric acid at 50-55° for 7 hrs, and then pouring the mixt on ice

Refs: 1)Beil—not found 2)H.Shechter, JACS **74**, 5056(1952) & CA **48**, 9912(1954)

3-Nitroacrylnitramide, O_2N . CH : CH . CO . $NH(NO_2)$—not found in Beil or in CA through 1956

Acrylate. A salt of acrylic acid (qv). Acrylates, such as α-methylacrylate, ethylacrylate, etc, are described under the letters M, E, etc

ACRYLIC ACID AND DERIVATIVES

Acrylic Acid or Ethylenecarboxylic Acid

(Acroleic or Propenoic Acid), CH_2:CH.COOH, mw 72.06, OB to CO_2 -133.2%, OB to CO -66.6%. Col liq, d 1.062 at 16°/4°, mp 12.3-13°, bp 141.2°. Miscible with w and alc. A detailed method for its lab prepn is given by Kaszuba, who used acrylonitrile(qv), hydroquinone, powdered Cu and H_2SO_4(Ref 2). The directions given by K should be followed closely, keeping the ingredients well chilled, otherwise a vigorous exothermic reaction (or even an expln) may occur(Ref 4). Other methods of prepn are given in Ref 3. Toxicity, fire and expln hazards are discussed in Ref 6

Note: Some acrylate polymers have been used in Ordnance items

Refs: 1)Beil **2**,397,(186) & [383] 2)F.J. Kaszuba, JACS **67**,1227(1945) & CA **39**, 4051(1945) 3)Kirk & Othmer **1**(1947), 176-80 4)F.J.Kaszuba, C&EN**30**,824(1952) & CA **46**,5319(1952) 5)OrgSynth,CollVol **3** (1955), 30 & 33 6)Sax(1957),244

Acrylic Acid-Trinitrophenylester, such as $C_{11}H_9N_3O_8$, polymerized by heating to ca 100° with dibenzoyl peroxide, yields an exp or readily combustible plastic

Ref: H.A.Bruson & G.B.Butler, USP 2,407, 131(1946) & CA **41**,288(1947)

Azidoacrylic Acid, N_3.CH:CH.COOH–not found in Beil or in CA through 1956

Acryloylazide, CH_2:CH.CO.N_3, mw 97.08, N 43.29%; wh unstable ndls of lachrymatory smell, mp 32 to 35°. Can be prepd by adding with intensive stirring acryloyl chloride in paraffin oil to ice-cooled suspension of NaN_3 in paraffin oil; stirring is continued until chloride is completely consumed (ca 24 hours), and the filtered soln fractionated in vacuo (8-9 mm) to give crude product, which on redistn yields pure compd. Its expl props were not investigated

Refs: 1)Beil–not found 2)T.Lieser, GerP 860,636(1952) & CA **48**,10060(1954)

3-Nitroacrylic Acid, O_2N.CH:CH.COOH, mw 117.06, N 11.97%; yel crysts, mp 136°. Was obtained by dehydrochlorination of 2-chloro-3-nitropropionic acid (mp 78-80°) and also by hydrolysis and deamination of 3-nitroacrylonitrile

Refs: 1)Beil–not found 2)H.Stecher et al, JACS **74**,3055-6(1952) & CA **48**,9912 (1954)

Acrylic Esters, Monomeric are discussed by E.H.Riddle, "Monomeric Acrylic Esters," 221 pp, Reinhold, NY(1954) (See also Methylacrylate and Ethylacrylate)

Acrylic Resins and Plastics are discussed in Kirk & Othmer **1**(1947), 180-4 and in books on Plastics

ACRYLONITRILE AND DERIVATIVES

Acrylonitrile (Propenenitrile or Vinyl Cyanide), CH_2:CH.CN, mw 53.06, N 26.40%, OB to CO_2 -226.2% OB to CO -135.7%. Col liq, d 0.8004 at 25°, frp -82 to 84°,bp 78° (Ref 2), 77.3°(Ref 4); n_D 1.3884 at 25°, Q_c^v 420.5 kcal/mol, fl and fire point 0° ± 2.5° (32°F)(open cup), vap press 100 mm at 22°, sp heat 0.50 ±0.03 cal/g, vap d 1.83(air = 1.0), ignition temp 481° in air and 460°in oxygen (Ref 3); latent heat of evapn 7800 cal/gmol at 0-80°(calcd)(Ref 4). Soly in w 7.4% at 25°(Ref 4); soly in some common org solvent is given in Ref 3; expln range in air 3.05 to 17.0% by vol at 25°(Ref 4); reacts vigorously with oxidizing materials. Was first obtained by Moureau (Ref 2). Current methods of prepn include dehydration of β-hydroxypropionitrile or pyrolysis of cyanoethylacetate(Ref 5). It is somewhat poisonous and sustained exposure to its vapors should be avoided (Refs 4,5,7 & 8). It is manufd on a large scale for use in making oil-resistant artificial rubber of GR-N type, as well as plastics, etc (Refs 3,4,5 & 7). Absorption spectra and some other physical props are given in Ref 4

Refs: 1)Beil **2**,400,(186) & [388] 2) C.Moureau,BullFr [3] **9**,424(1893) & JCS **64** I, 682(1893) 3)W.J.Huff, USBurMines Rept Invest **3669**(1942) & CA **37**,1871(1943) 4)H.S.Davis & O.F.Wiedeman, IEC **37**, 483-5(1945) 5)Kirk & Othmer **1**(1947)

184-9 6)Cyanamid's Nitrogen Chemicals Digest, "The Chemistry of Acetonitrile," American Cyanamid Co NY(1951)(pamphlet; 580 refs) 7)CondChemDict(1956), 19 8) Sax(1957), 244-5

Azidoacrylonitrile, $N_3.CH:CH.CN$—not found in Beil or in CA through 1956

3-Nitroacrylonitrile, $O_2N.CH:CH.CN$, mw 98.06, N 28.57%; yel oil, powerful vesicant and lachrymator, d 1.268 at 20/4°, bp 53-4° at 3.3-3.4mm, n_D 1.4929 at 20°. Was prepd by adding dropwise 2-chloro-3-nitropropionitrile to a stirred suspension of anhyd Na acetate in absol eth. Its expl props were not investigated

Refs: 1)Beil—not found 2)H.Schechter et al JCS **74**, 3056(1952) & CA **48**, 9912(1954)

ACT (Ardeer Cordite Tubular). A propellant manufd at Ardeer plant of Nobel's Explosives Co and later by the Imperial Chemical Industries, Gt Brit

ACT 5 (Ardeer Cordite Tubular No 5). A batch No 320B delivered in 1938 for the Brazilian Navy had to meet the following specification requirements: NC(N= 11.8 ±0.2) 65.0 ±1, NG 29.0±1 and centralite 6.0 ±0.5% Graphite could be added to the extent of 0.2% and max moist content of ACT 5 was 1%

Ref: Adm Alvaro-Alberto, Rio de Janeiro; private communication Oct 14, 1958

ACT 5, Erosion of. The erosive action of ACT 5 as well as of some other propellants was investigated in Brazil by Admiral Alvaro-Alberto (Ref 2) from the point of view of Vieille's erosion theory (Ref 1). This theory was modified using some later data of Muraour and of other investigators. A brief resumé of Alvaro-Alberto's work on erosive action of propellants is given in CA **40**, 2629-30(1946)

Refs: 1)P.Vieille, MP **11**, 156-210(1901) 2)Adm Alvaro-Alberto, Anais da Academia Brasileira de Ciencias(Rio de Janeiro), **14**, 247, 327(1942) & **15**, 39, 187, 329(1943)

Action of Gas Explosions on Solid Propellants. In one series of experiments conducted in Russia, a glass tube, 25 mm in diam and 1.5 m long, was filled (after evacuation) with H-O gas (obtained by electrolysis of H_2O) at atm press and a large cryst (or a solidified drop) of an expl was placed on a piece of iron in the center of the tube. The gas mixt was detond by means of a 15 mg chge of LA placed in a side arm of the tube and the expln was photographed. None of the expls tested(PA, tetryl and PETN) ignited or detond, even when they were preheated to 100°. In the second series of experiments, a steel tube 38 mm in diam and 1 m long was filled with H-O mixts at various pressures, the rest of the conditions were the same as in the first series. None of the expls tested was affected by the expln of the mixts at atm press. At 5 atm, blasting gelatin burned without deforming the tube, but at higher pressures it expld and the tube was blown to bits. Cast PETN did not burn or expl at 6 or 10 atm but it did expl at 15 atm and higher pressures. Powdered PETN exploded at 10 atm and higher. PA was unaffected at 5, 10 & 15 atm, but it burned at 20 & 24 atm and exploded at 30 atm

Ref: K.K.Andréev & V.P.Maslov, Dokl Akad-Nauk **25**, 195-7(1939) & CA **34**, 3495(1940)

Action of Light on Explosives and Propellants. See Light Sensitivity of Explosives and Propellants

Action of Light on Explosives and Propellants, Tests. See under Light Sensitivity Tests

Action of Inorganic and Organic Salts in the Combustion of Carbon in an Atmosphere of Nitric Oxide. See Catalysts in Combustion of Carbon

Action of Radiation. See Radiation Action

Action Time of a Propellant in a Rocket. According to a definition of the Hercules Powder Co, the *action time* is the time interval of that part of the time-pressure curve which is above 10% of the max press of a rocket propellant. This value is

identical with the *10% burning time*, as used at Pic Arsn

Activated Carbon or Charcoal. See Carbon (or Charcoal) Activated

Activated Complex Theory. See Absolute Rate Theory

Activation (or Radioactivation) Analysis (Applications of Radiochemical Methods to Analytical Chemistry)(In collab with T.C.Castorina, PicArsn). *Activation analysis* is the quantitative detn of elements by the measurement of the radioactivity produced in them by nuclear bombardment

Measurement of radioactivity, as an analytical tool became possible after the discoveries of A.H.Becquerel(uranium radiation 1896), Pierre & Marie Curie (polonium & radium in 1898), Sir E.Rutherford (identification of Becquerel rays as consisting of alpha-, beta- and gamma-particles) and of F.Soddy(phenomenon of nuclear disintegration, in 1902)

Since not many natural radioactive elements are in existence analysis by radiochemical methods was rather limited until it became possible to "induce" radioactivity artificially in some of the non-radioactive elements, as was first done in 1934 by I. Curie & F.Joliot(Ref 1). This discovery greatly broadened the application of radiochemical analysis. The first application of artificial radioactivation for the identification of constituents in a mixt was reported by Meinke (Ref 16) to have been done in 1936 by Hevesy & Levi (Ref 2). In the activation method an element undergoes nuclear reactions by means of some source producing sufficiently high thermal neutron flux(preferably by a nuclear reactor) to yield radioactive isotopes. These isotopes are usually unstable and return to their ground state by releasing energy in the form of emitted radiations. By measuring these radiations it is possible to identify, in most cases, one or several components in a mixt. Such nuclear transitions are not affected by the state of chemical combination of the atom so that radiochemical measurement, can, in many cases, be made directly without preliminary separation, by gamma spectrometric methods. At times, however, it does become necessary to resort to wet chemical methods for the more complete identification of the numerous components in the mixt

Because of the extremely high sensitivity of the activation method of analysis detns of trace impurities (as low as 1 part per billion) are made possible. Such analyses can be made only with difficulty, if at all, by conventional chemical methods

Activation analysis now finds wide application in the identification of the rare earths and in the field of metallurgy. It has been also applied to some Ordnance problems, which are being reported in classified literature (Ref 19)

Detailed description of activation analysis can be found in many of the following refs

Refs: 1)I.Curie & F.Joliot, Nature **133**, 201 (1934)(Discovery of phenomenon of induced or artificial radioactivity) 2)G.von Hevesy & H.Levi, Kgl Danske Videnskab Selskab Matfys Medd **14**, 5(1936) 3)O.Hahn, "Applied Radioactivity," Cornell Univ Press, Ithaca, NY(1936) 4)G.von Hevesy & F.A.Paneth, "A Manual of Radioactivity," Oxford Univ Press, London(1938) 5)G.T.Seaborg, Chem Revs **27**, 199-285(1940) (ca 600 refs)(Artificial radioactivity) 6)G.von Hevesy, "Radio active Indicators," Interscience, NY(1948) 7)G.K.Schweitzer, "Radioactive Tracer Techniques," Van Nostrand, NY(1949) 8) G.Friedlander & J.W.Kennedy, "Introduction to Radiochemistry," Wiley, NY(1949) 9)W. F.Bale & J.F.Bonner, Jr, "Determination of Radioactivity," chap 30 in vol 1 of "Technique of Organic Chemistry," edited by A. Weissberger, Interscience, NY(1949) 10)A.C.Wahl & N.A.Bonner, "Radioactivity Applied to Chemistry," Wiley, NY(1951) 11)K.E. Zimen, "Angewandte Radioaktivität," Springer, Berlin(1952) 12)I.M.Kolthoff & E.B.Sandell, "Textbook of Quantitative

Organic Analysis," Macmillan, NY(1952), 659 13)G.B.Cooke & J.F.Duncan, "Modern Radiochemical Practice," Oxford Univ Press, NY(1953) 14)W.J.Whitehouse & J.L.Putnam, "Radioactive Isotopes," OxfordUnivPress, NY(1953) 15)G.Charlot & D.Bézier, "Méthodes Modernes d'Analyse Quantitative Minérale," Masson & Cie, Paris(1955) 16)W.W.Meinke, Science **121**, 177-84(1955) and Rus translation by N.G. Polianskii, UspekhiKhimii **15**,770-80(1956) (Trace element sensitivity; comparison of activation analysis with other methods) 17)G.Charlot & D.Bézier, "Quantitative Inorganic Analysis," translated from the French by R.C.Murray, Methuen Co, London (1957), 295-9 18)L.Meites, H.C.Thomas & R.P.Bauman, "Advanced Analytical Chemistry," McGraw-Hill,NY(1958),344-69 19) Samuel Helf, "Nucleonics Laboratory," Pic Arsn, Dover, NJ(1959); private communication

Activation Energies of Elementary Reactions. See the dissertation of T.A.Vanderslice, Catholic University of America Press, Washington,DC(1956)

Activation Energies of Explosives. It is known that for most expls the following Arrhenius equation holds: log t = E/RT + const, where t is the "induction period" (time lag in secs prior to ign or expln after heating to a temp T in °K), R is the gas constant and E is the activation energy in kcal/mol for the reaction in question

The following table, taken from Ref 1, gives the activation energies for some expls as calcd from the above formula and using T values from tables I to IV of the above work. The value E_2 represents the actvn energy in the lower temp range for those expls which do not follow a straight line relationship over the entire temp range studied, while E_1 is the actvn energy at the higher temp range

Note: The values E are lower than those reported by others, such as in Ref 2

Refs: 1)H.Henkin & R.McGill, IEC **44**, 1394(1952) 2)S.Roginsky, PhysZSow **1**, 640(1932) 3)T.Urbański & Rychter, CR **208**,900(1939) 4)A.J.B.Robertson, TrFaradSoc **44**,677(1948)

Table (Activation Energies)

Explosive	E_1	E_2
Black powder	20.6	–
Diazodinitrophenol	29.0	48.0
Erythritol tetranitrate	22.8	51.0
Ethylenedinitramine	10.0	80-85
Lead azide	21.2	–
Lead styphnate	58.8	–
Mercuric fulminate	20.2	58.0
Nitrocellulose (12.6-13.4%N)	26.5	67.0
Nitroglycerin	22.6	–
Pentaerythritol tetranitrate	22.0	–
Picric acid	27.4	–
Tetryl	14.4	–
Trinitrotoluene*	Not given	

*In Ref 3, the E value for TNT is given as 14 kcal/mol in the temp range 390-450°, while in Ref 4 the value is 32 kcal/mol for a lower temp

Activation Energies of Fuels in High Temperature Combustion are discussed in the paper presented by J. B. Fenn & H. F. Calcotte at the 4th Symposium on Combustion, Williams & Wilkens, Baltimore(1953), pp 231-9(7 refs)

Note: The paper gives, in addition to activation energies of various fuels with stoichiometric amounts of air, the flame temps and the burning velocities

Activator (of a Land Mine). Same as Fuze of a Land Mine, also called Igniter

Active List of Permissible Explosives and Blasting Devices. This list approved by the US Bureau of Mines prior to Dec 31, 1945, may be found in the Bur of Mines Rept of Invest 3910, compiled by J. E. Tiffany & Z. C. Gaugler. There is also a supplement to this report. Earlier Repts of Investigations on the same subject are Nos 3134, 3665 & 3736. No other info on this subject was found in CA through 1956

Active Oxygen is oxygen of an org or inorg compd which is easily liberated in a free state, especially in presence of alkalies and heavy metals, like Pt. Such oxygen is found in peroxy compds, such as dibenzoyl peroxide, Na peroxide, etc. It liberates iodine from KI (Ref 1)

Following method for the detn of active oxygen in dibenzoy peroxide is described in Ref 2

Weigh 0.605 g peroxide into a 125 ml Erlenmeyer flask, add 40 ml acetone and swirl gently until the sample dissolves (takes ca 2 mins). Add 5 ml of KI soln (prepd by dissolving 33 g cp KI in 67 ml H_2O contg small amt of Hg) and swirl for ½ min. Titrate with N/10 Na thiosulfate to a colorless end point

% Active Oxygen = (ml $Na_2S_2O_3$) × 0.1322

(See also Available Oxygen)

Refs: 1) Dr H. Walter, PicArsn; private communication 2) Bulletin No 9 of Lucidol Division, Novadel-Agene Corp, Buffalo, NY (1948)

Active Sheath (Aktive Mantelpatrone). A type of sheath (see Sheathed Explosives), consistg of flameless, gas-producing mixts capable of self-sustenance of their gas-producing character. These sheaths usually consist of NG(with/or without NGc) and inert ingredients, such as $NaHCO_3$, NaCl, Kieselguhr, etc

[See also PATR 2510(1958), p Ger 1]

Actuator, Explosive. See Explosive Actuator

Acyclic Mercaptans Containing 1-5 Carbon Atoms were found to be hypergolic when used in combination with a strong oxidizer such as fuming HNO_3. Such mixts were proposed for use in self-igniting rocket fuels. In order to shorten the ign delay, the oxidizer was mixed with 2-20% by wt of H_2SO_4, NO_2 or $NOHSO_4$. In one example, a 1:3 mixt by vol of *ethylmercaptan*(qv) and an oxidizer contg 15% by wt of H_2SO_4 gave an ign delay of 10 millisec at -40°F

Ref: P. C. Condit & M. A. Pino, USP 2,750,732(1956) & CA **50**, 16110-12(1956)

Acylamine, Nitroso. See Nitrosoacylamines and Diazo Esters

Acylation (Acidylation). A reaction leading to the formation of an org compd contg one or several acyl radicals, RCO-

Refs: 1) Lassar-Cohn, "Arbeitsmethoden für Organisch-Chemische Laboratorien," L. Voss, Leipzig(1924), 5-28 2) Kirk & Othmer **1**(1947), 190

Acyl Hydroperoxides. See Peroxy Acids

Acyl Nitrates and Perchlorates are described by M. Schmeisser, AngewChem **67**, 493-501(1955)

Acyl Peroxides are peroxides contg one or more acyl(RCO-) groups. Such peroxides are described in this work under the names of the corresponding acyl radicals, such as diacetyl peroxide, dibenzol peroxide, etc

Following are some recent refs:

1) K. I. Ivanov et al, ZhObshchKhim **22**, 2126-8 (1952) (in Rus); **22**, 2181-2(1952)(in Engl) & CA **48**, 1257, 5084(1954) 2) K. S. Minsker & L. V. Stupen, ZhObshchKhim **27**, 2875-7(1957) (in Rus); **27**, 2912(1957)(in Engl) & CA **52**, 8085(1958)

Acyl Peroxides Reactions with Phenol were investigated by C. Walling & H. B. Hodgdon, Columbia Univ Tech Rept No 2, Sept 1954-Dec 1956. Project No TBZ-0001, Contract Cu-11-57-ORD-1270

Adams (Explosif). An expl, patented in 1893 in France, contained: K nitrate 54, sulfur 20, flowers of sulfur 13, PA 1, tungstic acid 0.5, HgO 0.5, Sb trisulfide 1 & water 10%
Ref: Daniel (1902), 5

Adamsite. Same as Diphenylaminechloroarsine

Adapter. A metal collar or bushing with external and internal threads. It is screwed into the nose of a projectile, when the nose opening is larger than the diam of the fuze. The adapter serves as the seat for a fuze. The use of adapters permits the attachment of various sizes of fuzes to one particular shell. It also permits the use of a larger opening in the nose of a shell which facilitates the forming and machining of the interior cavity in the expl loaded into the shell. Chemical shells have adapters in order to provide a means of seating the burster casing
Refs: 1) Hayes (1938), 595 2) Dept of the Army, Technical Manual, TM 9-1901 (1950), 379
Note: Specifications for various adapters are listed in the "Index of Specifications and Standards," Dept of the Army, Washington, DC, vol 2, Oct 1958, pp 1-2

Adapter-Booster. A device which consists of a bushing contg a booster charge, usually tetryl pellets. The adapter-booster is screwed deep into the nose or base plug of a bomb. The device is threaded on its protruding end to seat a standard nose or tail bomb fuze
Refs: 1) Hayes (1938), 606 2) Ohart (1946), 217 & 273 3) US Army Spec 50-16-3c (Requirements and tests for adapter-boosters used in bombs)

Adapter, Cluster. See Cluster Adapter

Adde Explosives. Mixts of liq expls, produced by nitrating either xylene, cumene or benz, gelatinized with NC and combined with oxidizing agents (such as nitrates, perchlorates, etc) to yield low-freezing (ca -20°) plastic expls. These expls were patented about 1912 by Symon Adde in Sweden as well as in England. Eg: AN 32, Amm perchlorate 40, liq DNX or TNX 20, NG 5, NC 1 & ferrosilicon 2%
Refs: 1) S. Adde, BritP 13,373 (1912) 2) Colver (1918), 258 & 689

ADE. Designation of Ital time and percussion fuzes used with aerial burst or impact projectiles
Ref: Bureau of Ordnance, Navy Dept, "Italian and French Explosive Ordnance," OP 1668, Washington, DC (1946), 63

Adenine. Same as 6-Aminopurine

Adesivo (Ital). Adhesive

Adhesion is the sticking together of substances in contact with each other. The subject is discussed in the following references:
1) W. Wehl, "The Theoretical Basis of Adhesion," ASTM Proceedings 46 (1946) 2) N. A. deBruyne & R. Houwink, "Adhesion and Adhesives," Elsevier Press, NY (1951) 3) Collective, "Adhesion and Adhesives, Fundamentals & Practice," Symposium, Wiley, NY (1954)

Adhesives are substances (such as glue, plaster, cement, etc) that bind solid materials together. Adhesives are used extensively in Ord items and in the explosives industry. For instance, the solid ingredients (such as AN, K perchlorate, etc) of gelatin dynamites are held together by means of a gel consisting of NG and collodion cotton (see also Binders). In the manuf of large grain rocket propellants, the so-called "inhibitor strips" (qv) are usually attached by means of an adhesive (Ref 4). Adhesives are also used in the packing of ammunition (see under Packing and Packaging)

Testing of various adhesives and adhesive cloths for use in Ord is described in Refs 6 & 8

Refs: 1)P.I.Smith, "Synthetic Adhesives," Chem Pub Co, Brooklyn(1943) 2)T.D.Perry. "Modern Wood Adhesives," Pitman,Chicago (1944) 3)J.Delmonte, "The Technology of Adhesives," Elsevier, NY(1947) 4)A.M. Ball, USP 2,643,611(1953) & CA **47**,9016 (1953)(Inhibitor strips for large-grain rocket propellants are attached by means of an adhesive consisting of NC dissolved in Et lactate, cellosolve, mesityl oxide, Bu acetate or diacetone alcohol. These solvents act as a mutual plasticiser for the material of the inhibitor strip and for the propellant itself) 5)G.Epstein, "Adhesive Bonding of Metals," Reinhold, NY(1954) 6)ASTM Committee D-14 on Adhesives, Standards on Adhesives, Specifications, Physical Tests, Definitions, Philadelphia, Penna(1954) 7) USOrdnance Corps, "Ordnance Materials Handbook ORDP 20-306," Washington, DC (1957) 8)Dept of the Army, "Index of Specifications and Standards," Washington, DC, vol 2(1958), pp 2-3 9)H.A.Perry, "Adhesive Bonding of Reinforced Plastics," McGraw-Hill, NY(1959) 10)J.J.Veliky & M.J.Bodnar, "Adhesives for Bonding Compressed Graphites to Steel," PATR 2604 (1959)

(See also Refs under Adhesion)

Adhesive Tape for Packaging and Packing of Ammunition. US Specification JAN-P-127 and Ammendments cover the requirements of the US Army and Navy. The tape must be pressure sensitive and water-resistant

Following are the tests described in this Spec: wet tensile strength, adhesion at low temp, moisture-vapor transmission rate, water-penetration rate; corrosion, accelerated aging, dry tensile strength and tearing resistance

Adiabatic Compression of Entrapped Gas or Vapor as a Cause of Initiation of Explosives. It has been shown by some Brit investigators that one of the important causes of initiation of sensitive expls by mech action(such as impact) is the adiabatic compression of minute gas or vapor bubbles entrapped by the expls. These tiny air spaces are heated by adiabatic compression and ignite the expls. If precautions are taken to eliminate all bubbles, the expl is comparatively insensitive. These bubbles serve as "hot spots." Another important source of hot spots is the presence of small grit particles(Ref 2, p 3)

This theory was tested by Yoffe(Ref 1) by comparing the energies required to initiate NG and PETN with and without entrapped air. Samples prepared without air in the form of a continuous film required much higher energies of initiation than samples with entrapped air. A simple method of including a gas phase in an expl is to spread it as a small annulus on a flat anvil. When this is struck with a flat hammer, the small amt of gas in the center is trapped and compressed. In these experiments the size of the annulus was such that the initial vol of the gas was ca 5×10^{-5} cc. A more detailed description of the theory of adiabatic compression and methods of testing are given in Ref 2

Refs: 1)A.D.Yoffe, Nature **161**,349(1948) 2)F.P.Bowden & A.D.Yoffe, "Initiation and Growth of Explosions in Liquids and Solids, CambridgeUnivPress; London(1952), 3 & 33-61

Adiabatic Explosion. Since an expln takes place in an extremely short time, it may be considered to be adiabatic. Assuming that an explosive is heated by the decompn of part of the sample and that the rate of decompn increases with increasing temp, R. B.Parlin et al(Ref) derived the equation for the time-temp relationship in a purely thermal expln. A discussion of this equation would require undue space here

Ref: R.B.Parlin et al, OSRD Report 2026 (1943)(Unclassified)

Adiabatic Flame Temperature is the maximum temp produced on combustion (of propellants, pyrotechnic compositions, etc)

assuming no heat is lost to or absorbed from the surroundings. The adiabatic flame temp is a calcd value

Ref: Rocket Fundamentals, Appendix 2, OSRD Rept 3992(1944)

Adiabatic Ignition of Propellants, Pyrotechnic Compositions, etc. When ign of a subst is effected in a highly insulated condition with no gain of heat from or loss of heat to the system, it is called adiabatic ignition. The ign can be initiated by a spark, flame, incandescent wire, etc and the heat developed by these sources must be taken into consideration when calculating the heat of expln or deton from experimental data

Confidential OSRD Rept 4758(1945) by the Explosives Research Laboratory, Bruceton, Pa presents a theoretical treatment of adiabatic ignition and also considerable experimental data. The theory of adiabatic ignition presented applies only to those decompositions which are not autocatalytic

Adiabatic Ignition Test of Propellants was developed during WWII by the Hercules Powder Co at its Radford, Virginia plant. The test is described in confidential Hercules report RD 4 by W.S.Koski, Dec 20, 1943

Adinau Explosive. An Amer pre-WWI expl: Ba nitrate 69.3, TNT 28.7 & Pb chromate 2%

Refs: 1)L. Adinau, USP 1,056,389(1913) & CA **7**, 1612(1913) 2)Colver(1918), 250

ADIPIC ACID AND DERIVATIVES

Adipic or Adipinic Acid (Hexanedioic or 1,4-Butanedicarboxylic Acid)(Hexandisäure, in German), $(CH_2)_4(COOH)_2$, mw 146.14, OB to CO_2 -142.3%, OB to CO -76.6%. Fine, wh crysts or powd, d 1.360 at 25°/4°, mp 152°, bp 337.5°, fl p 385°F (closed cup), vap press 1 mm at 159.5°, vap d 5.04 (air 1.00), Q_c^v 668.6 kcal/mol; reacts with oxidizing materials; sl sol in w or eth and very sol in alc. Can be prepd by several methods. One of them, given in Ref 3, uses cyclohexanol and nitric acid in the presence of NH_4VO_3. The German method using tetrahydrofuran and CO is briefly described in Ref 2. Other methods are given in Refs 1 & 4. The toxicity, fire and expln hazards of adipic acid are discussed in Ref 5. Properties, reactions and current and potential uses of adipic acid and derivs are discussed in Ref 6

Adipic acid is used in the manuf of nylon and of some other plastics. Some of its esters are useful gelatinizers and plasticizers (Refs 4 & 6)

Its salts are called *adipates*

Refs: 1)Beil **2**,649(277) & [572] 2)J.D. Rose, PBRept 25,553, translation of W.Reppe's Report, Ludwigshafen(1941) 3)OrgSynth, CollVol 1(1941), 18 4)Kirk & Othmer **1**, (1947), 153-4 5)Sax(1957), 246 6)Polychemical Dept, E.I.duPontdeNemours & Co, "Adipic Acid and Its Derivatives," Wilmington, Del(1957)(77 pp)

Adipic Acid, Analytical Procedures are described in Organic Analysis, Interscience, NY, vol 2(1954) and vol 3(1956)

Adipic Acid Azide or Adipylazide (Adipinylazide), $N_3 . CO(CH_2)_4CO . OH$—not found in Beil or CA through 1956

Adipic Acid Diazide or Adipyldiazide (Adipinyldiazide), $N_3 . CO(CH_2)_4CO . N_3$, mw 196.17, N 42.84%; col oil solidifying at -1° to wh crysts; bp-expl viol on heating directly or under w; easily sol in alc or eth. Was first prepd in 1915 from adipyl hydrazide, $NaNO_2$ and HCl(Ref 2). Details of prepn are given in Ref 3

Refs: 1)Beil **2**,(278) 2)Th. Curtius, JPraktChem **91**, 8(1915) & CA **9**, 1606(1915) 3)P. Eckert & E.Herr, Kunstseide und Zellwolle **25**, 204(1947) & CA **43**, 1564(1949)

Adipocelluloses and Cutocelluloses are mixts of cellulose with waxy and fatty substs. Cork and some other barks consist principally of adipocellulose, whereas the epidermis of the leaves and twigs of some plants contain cutin(combination of waxes,

fatty acids, resins, higher alcohols, etc) and cellulose. The combination of cutin with cellulose has been called "cuto-cellulose" but its existence (as well as of adipocellulose) as a compd has never been proven. Some investigators believe that adipo- and cuto-cellulose are identical

When treated with concd HNO_3 or mixed HNO_3–H_2SO_4, adipocellulose yields products resembling those obtained by nitration of cellulose and fats

Pulverized adipocellulose (cork, bark, etc) has been used as an absorbent for NG, NGc, etc in coml expls such as dynamites

Refs: 1)Marshall 1(1917), 150 2)Dorée (1947)332 3)Webster's New International Dictionary, Unabridged, Merriam Co, Springfield,Mass(1951), 32 & 652 4)Ott 5,part 1 (1954), 34

Adobe Shooting. Same as Mudcapping or Plaster Shooting (see under Agriculture and Forestry Use of Explosives)

Adsorbents. See Adsorption and Adsorbents

Adsorption and Adsorbents. Adsorption may be defined as the ability of a substance (adsorbent) to hold on its surface, including inner pores or cracks, thin layers of gases, liquids or dissolved substances (adsorbates). Adsorption is a surface phenomenon and should not be confused with **absorption** (qv). Adsorption may be divided into physical and chemical (also called chemisorption). In physical adsorption the forces are those betw the adsorbing surface and the molecules of the adsorbate, and are similar to Van der Waals forces. In chemisorption, which includes ion exchange, the forces are much stronger than those of physical adsorption and depend on chemical bond formation. One of the most effective physical adsorbents is activated carbon. Kieselguhr, formerly used in dynamites, is also an effective adsorbent. Other physical adsorbents include activated alumina, clays, silica, charcoal, sawdust, wood pulp, vegetable meals, carbene and various salts. Some of these substances(sawdust, meals, AN, Na nitrate, etc) are used as absorbents for the liq components of dynamites, while other substs (carbene, charcoal, etc) are used for liq air or liq oxygen expls, such as oxyliquit(qv)

(See also Chemisorption, Ion Exchange and Surface Chemistry & Physics in this Dictionary and Absorption Analysis and Chromatography)

Refs: 1)Kirk & Othmer 1(1947), 206-32 (20 refs) 2)P.Meunier & A.Vinet, "Chromatographie et Mésomérie, Adsorption et Résonnance," Masson,Paris(1947) 3)A. B.Ray, IEC **39**, 12-13 & 32-35(1947)(114 refs) 4)B.L.Harris, IEC **41**, 15-19(1949) (167 refs) and under "Unit Operations" in the January issues of succeeding years 5)Perry (1950), 885-916 6)C.L.Mantell, "Adsorption," McGraw-Hill,NY(1951) 7) J.H.deBoer, "The Dynamical Character of Adsorption," Oxford UnivPress,London(1953) 8)V.R.Dietz, "Bibliography of Solid Adsorbents 1943-1953," USBurStds Circular 566,Govt PrtgOff, Washington,DC(1956)

Adsorption Analysis is discussed in the following books:

1)E.O.Kraemer, edit, "Advances in Colloid Science," Interscience, NY, 1(1942), article by A.Tiselius, "A New Method of Adsorption Analysis and Some of Its Applications" 2)A.Weissberger edit, "Physical Methods of Organic Chemistry," Interscience, NY 5(1951), 1-206

ADT and ADV Propellants are described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959), Unit Nos 394 & 395

Advance Detonation. In 1941, Woodhead (Ref 1) observed that the velocity of detonation of unconfined columns of pressed tetryl pellets was higher by several hundreds m/sec when there was a continuous cylindrical cavity in the column. Later (Ref 2), the same investigator observed that this phenomenon takes place with HE's more sensitive than TNT (such as gelignite, mixt tetryl/TNT, etc) but is not observed with straight TNT. He also discovered that

inside the cavity, under these conditions, a luminous shock wave of fairly const velocity travels nearly twice as fast as the vel of deton of the expl under test. If the cavity is not blocked, this shock wave appears at the open end of the cavity as an intense flame having temporarily a higher speed than when inside the cavity

If this end of the cavity is blocked with a pellet of an adequately sensitive expl, the luminous wave on striking the pellet initiates in it a deton wave which travels in two opposite directions. As the deton initiated in the pellet is ahead of the main deton front, it is called the **advance detonation.** The pellet deton wave which travels toward the oncoming main deton front collides with it and with the luminous shock wave of the cavity producing a sharp increase in the brightness of the flame and a peak in the deton pressure

Some practical applications of tubular charges and the phenomenon of advance detonation are mentioned in Ref 2

Refs: 1)D.W.Woodhead, Nature **160**,644 (1947) 2)Ibid, **183**,1756-7(1959)

"Advanced" Missiles. This term may be applied to missile systems under development for present or future use. These missiles are described in books and papers on rockets, guided missiles, space weapons, missiles of the future, etc. See also R.B.Dow, "Fundamentals of Advanced Missiles," Wiley, NY(1958)

Aerial Bomb, Aerial Torpedo, etc are described in the following refs:

1)Willy Ley, "Bombs and Bombing," Modern Age Books, NY(1941) 2)J.R.Newman, "Tools of War," Doubleday, Doran & Co, NY(1943), 357-62 3)War Department Technical Manual TM9-1900(1945), 131-160

Aerial Burst Fuzes are devices designed to initiate an explosive in a bomb while it is still in flight. They were used by the Germans during WW II

Refs: 1)Dept of the Army, TM 9-1985-2 (1953), 132,168,171 & 174-8 2)PATR 2510(1958), pGer 1-3

Aerochemical Device for discharging many incendiary units simultaneously from a plane or for laying down a shower of a persistent vesicant agent from the air was devised by F.Short, USP 2,422,381(1948) & CA **42**,6117-18(1948)

"Aero" Cyanamid. A trade name for the commercial Ca cyanamide manufd by the American Cyanamid Co

Aerodynamics and Its Application in Ballistics. Aerodynamics is the branch of dynamics which studies the motion of gases, the forces acting on bodies moving through gases(such as air), and the forces involved when gases move past bodies. Aerodynamics is involved in exterior ballistics and in aeronautics

When a projectile flies through a gas(air) it has to overcome some resistance, not only because of friction with the gas but also because some energy has to be spent to compress the air immediately in front of the moving projectile. As a result of this, the air immediately behind the projectile becomes rarified. The combination of compression and rarification constitutes the so-called "shock waves." The existence of these waves were unknown until the French ballistician, General Moisson, in the eighties, published a paper entitled "L'évaluation de la résistance de l'aire," Another French ballistician, Hugoniot, also worked on the subject of the resistance of air and shock waves(1887), but the actual proof of the existance of such waves was produced in 1888 by Mach in Germany, who photographed them

Due to the fact that Hugoniot was the first to formulate the laws of the resistance of air to the flight of projectiles, he is generally considered as the founder of the modern science of "aerodynamics." The principles of aerodynamics are also applied to aeronautics

In earlier works on aerodynamics, it was believed that the resistance of air was a

function of the velocity of the projectile. Sarrau disproved this and stated that the resistance of air is a function of a certain value, now known as the "Mach Number" (nombre de Mach, in French)

Developments in the science of aerodynamics since WWI have been of considerable importance and have resulted in practical improvements in the flight of airplanes, projectiles and rockets

(See also Aerodynamics, Supersonic)

Refs: 1)W.F.Durand, edit, "Aerodynamic Theory," Springer, Berlin, 6 volumes (1934-1936)(Reprinted in 1943 by the Durand Reprinting Committee, California Institute of Technology, Pasadena, Calif) 2)L. Gabeaud, CR **201**, 1460-1(1935)(Shock waves in an aerodynamic field) 3)R.Sauer, "Theoretische Einführung in die Gasdynamik," Springer, Berlin(1943), English translation by R.A.Alpher & F.K.Hill, Edwards, Bros, Ann Arbor, Mich(1947) 4)K.D.Wood, "Technical Aerodynamics," McGraw-Hill, NY(1947) 5)L.M.Milne-Thomson, "Theoretical Aerodynamics," Van Nostrand, NY(1948) 6)A.M. Kuethe & J.D.Schetzer, "Foundations of Aerodynamics," Wiley, NY(1950) 7)H. Hugoniot. Works published in the eighties were reprinted in book form by the MAF, Imprimerie Nationale, Paris(1951) 8)M. Salmon, MAF **25**,805-23(1951)(Aerodynamics and ballistics) 9)O.W.Eschbach, edit, "Handbook of Engineering Fundamentals," Wiley,NY(1952), "Aerodynamics," pp 7-01 to 7-128 10)L.Gabeaud, MAF **26**, 169-252(1952)(Evaluation of aerodynamic resistance of projectiles) 11)M.Rauscher, "Introduction to Aeronautical Dynamics," Wiley, NY(1953) 12)F.I.Frankl & E.Karpovich, "Gas Dynamics of Thin Bodies," translated from the Rus, Interscience, NY(1953) 13)T. von Karman, "Aerodynamics," CornellUnivPress, Ithaca, NY(1954) 14) E.Carafoli, "High Speed Aerodynamics, Pergamon Press, NY(1956) 15)G.Mueller, "Introduction to Applied Aerodynamics," US Naval Institute, Annapolis, Maryland(1957)

Aerodynamics, High Speed. See Aerodynamics, Supersonic

Aerodynamics of Propulsion are discussed in the following books:

1)D.Küchemann, "Aerodynamics of Propulsion," McGraw-Hill, NY(1953) 2)E.A. Booney, M.J.Zucrow & C.W.Besserer, "Aerodynamics, Propulsion, Structures and Design Practice," Van Nostrand,NY(1956)

Aerodynamics, Supersonic (High Speed) is treated in the following refs:

1)H.W.Sibert, "High Speed Aerodynamics," Prentice-Hall, NY(1948) 2)R.Courant & K. Frederichs, "Supersonic Flow and Shock Waves," Interscience, NY(1948) 3)A.Ferri, "Elements of Aerodynamics of Supersonic Flow," Macmillan, NY(1949) 4)E.Miles, "Supersonic Aerodynamics, A Theoretical Introduction," McGraw-Hill,NY(1950) 5) A.E.Bonney, "Engineering Supersonic Aerodynamics," McGraw-Hill,NY(1950) 6)W.F. Hilton, "High Speed Aerodynamics," Longmans, Green, NY(1951) 7)W.R.Sears, "General Theory of High Speed Aerodynamics," PrincetonUnivPress,Princeton,NY(1954) 8)Collective, "High Speed Aerodynamics and Jet Propulsion-Combustion Processes," Series of 12 volumes,published by Princeton Univ Press, Princeton,NJ, beginning in 1955

Aeroelasticity. This subject is treated in the following books:

1)Yüan-chên Fêng, "An Introduction to the Theory of Aeroelasticity," Wiley, NY(1955) 2)R.L.Bisplinghoff et al, "Aeroelasticity," Addison-WesleyPubCo, Cambridge, Mass (1955)

Aerogels are gels in which the liq phase has been replaced by a gaseous phase in such a way as to avoid shrinkage which would occur if the gel had been dried directly from a liquid (Ref 1)

Aerogels of SiO_2, Al_2O_3, MgO, SnO_2 and cellulose were recommended as ingredients (0.1 to 5%) of priming compns, such as: the complex salt (1:1:1-Pb styphnate, basic Pb

styphnate & Pb hypophosphite) 48.5, tetracene 5.0, diazodinitrophenol 7.0, Pb nitrate 19.0, ground glass 19.0, gum arabic 1.0 and aerogel 0.5%. It has been claimed that the addn of aerogels permits the use of more water in the compn than would otherwise be possible without the danger of the ingredients settling out. It also increases safety of handling and improves charging characteristics (Ref 2)

Refs: 1) R.K.Iler, "The Colloid Chemistry of Silica and Silicates," Cornell-UnivPress, Ithaca, NY(1950), 152 2) W.J.Taylor, USP 2,662,818(1953) & CA **48**, 3692 (1954)

Aerojet Engineering Corporation, Azusa, California (A subsidiary of General Tire & Rubber Co). This company has had a number of US Govt contracts, mainly to develop rockets, jatos, rocket fuels, etc

Aerojet Propellants and Other Substances prepd by the Aerojet Corp are described in numerous reports, many of which are classified. Some of these reports are listed under Aeroplex Propellants(qv) (See also pA350)

Aerolit (Aerolit). A Danish permissible expl which had the following approx compn: AN 78.1, K nitrate 7.5, beef suet 2.5, sulfur 8.8, sago flour 1.3, Mn dioxide 1.2 and resin 0.6%

Refs: 1) A.S.Aerolit Co, DanP 19858, abstracted in SS **10**, 295(1915) 2) Marshall **1**, (1917), 392

Aeronautics is the art and science of flying and navigation in the air. The study includes not only the flight of airplanes but also of guided missiles, space ships, rockets, satellites, etc. Following are some recent books on the subject:

1) K.F.Leidecker, "German-English Technical Dictionary of Aeronautics, Rocketry, Space Navigation," S.F.Vanni, NY(1950) 2) B.Kucherov, "Aeronautical Sciences and Aviation in the Soviet Union," A Bibliography, Library of Congress, Washington, DC(1955) 3) W.A.Hefflin, edit, "The United States Air Force Dictionary," Air Univ Press, GovtPrintingOff, Washington, DC(1956) 4) L.L.Beckford, "An ABC of Aeronautics," Cassell, London(1957) 5) H.W.Liepmann & A.Roshko, "Elements of Gas Dynamics," Wiley, NY(1957)

Aeroplex Propellants are solid rocket propellants developed and manufd by the Aerojet Engineering Corp, Azusa, Calif. They consist of a finely divided crystalline oxidizer (such as Amm or K perchlorate) dispersed in a thermosetting resin(such as styrene, methyl acrylate, etc), acting as a fuel. In addition there are binders (such as resins) and other ingredients. Aeroplex propellants differ from the usual NC or NC-NG propellants not only in physical and chemical properties, but also in their methods of manuf

Refs: 1) Aerojet Rept 336(1948), "Investigation of Aeroplex Propellants and Metal Components for Booster Rockets" (Final Summary) 2) Aerojet Rept 410(1949), "Basic Development of the Aeroplex Propellants and Associated Rocket Design and Production" (Volumes 1, 2 and 3) 3) Warren(1958), 11

Note: In addition to the above unclassified Aerojet reports there are many classified reports, such as Nos 426, 444, 462, 475, etc dealing with Aeroplex propellants

A good description of the prepn of a typical Aeroplex Propellant, including a flow-sheet diagram, is given in the confidential report of the 7th Joint Army-Navy-Air Force Meeting on Solid Propellants

Aerosols are colloidal systems with gas(air) as surrounding medium. Eg: smoke, fog or mist. Smoke is used in chemical warfare, as in shells and for producing smoke screens. The term aerosol is also applied to some forms of detergents, emulsifiers and wetting agents. A generator for producing aerosols, smokes, fogs and layers of gas was patented recently in France(Ref 3)

One of the substances which is used as an aerosol is dichlorodifluoromethane. It first made its appearance during WWII in the

so-called "bug-bombs," used extensively by the Armed Forces(Ref 4)

Industrial uses of aerosols include aerosol propellants, antifoam sprays, fire extinguishers and paints. Herzka & Pickthal (Ref 5) describe aerosol propellants, containers, valves, filling methods, lab procedures, emulsified systems, etc

Refs: 1)Webster's New Collegiate Dictionary, Merriam Co, Springfield, Mass (1953),15 2)B.Jirgensons & M.E.Straumanis, "A Short Textbook of Colloid Chemistry," Wiley,NY(1954),389 3)G.Reure & François-Gilbert, FrP 1,070,458 (1954) & MP 38,401-8(1956) 4)Faith, Keyes & Clark(1957),319 5)A.Herzka & J.Pickthal, "Pressurized Packaging(Aerosols)," Academic Press, NY(1958)

Aetna Explosives Company was organized before WWI by the consolidation of several smaller companies including Aetna Powder Co(organized in 1880),Keystone, National Brewester, etc. Then in 1921, the Aetna Explosives Co was sold to the Hercules Powder Co, Wilmington, Del

Ref: Van Gelder & Schlatter(1927),541

AEV Propellant is described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959), Unit No 396 (Conf)

Affusto (Ital); **Affût** (Fr); **Afuste** (Span). Gun carriage; mount

Affut automoteur (Fr). Self-propelled mount

Afosite. A black powder type blasting expl: AN 58-62, K nitrate 28-31, carbon 7-9 & sulfur 2-3%

Ref: Belgrano(1952),174

After Tyne Powder. One of the older Brit "permitted" expls: NG + NGc 9, AN58, MNN 1, wood meal 9, NaCl 22 & water 1%. It passed the Buxton gallery test and its swing was 2.52," vs 3.27" for Gelignite (Brit 60% dynamite, used as a standard)

Ref: Marshall 3(1932),120

After-Flame Ratio (AFR). Bichel investigated the duration of a flame by firing expls at night and photographing the flame through a quartz lens on a moving film. He found in all cases that the flame outlasted the time of deton and named the ratio: duration of deton to duration of flame, the "after-flame" ratio. Experiments with 100 g cartridges of 30 mm diam showed that safety explosives had a very short flame duration and consequently a high AFR. For instance, Ammon-Carbonite had an AFR of 1/7.4 vs 1/883 for blasting gelatin, 1/660 for 75% guhr dynamite and 1/330 for black powder

Ref: Barnett(1919),118-9

After-Separation. In the manuf of NG, the mixt after nitration (consisting of NG + spent acid) is transferred to a lead tank (separator) and allowed to stand so that the lighter NG may rise to the surface. After removing the acid through a stopcock in the bottom of the tank, the NG oil (or NG + NGc) is run to another tank where it is washed with Na_2CO_3 soln and then with water. The oil is then sent to a store-house where it is given further washing. Prior to the days of efficient refrigeration equipment and prior to the use of "separating compounds"(qv), the spent acid still contained an appreciable amt of NG. To remove additional NG, the operation of separation was repeated by allowing the spent acid to stand for 2-3 days. This was done not only to recover as much of the NG as possible for economic reasons, but also to reduce the NG content of the spent acid below the point where subsequent operations would be hazardous. This second separation was called "after-separation." It was conducted in a lead tank (Nachscheider in Ger) similar to the first separator. The NG which floated to the top in the after-separation was skimmed off. The spent acid was then run to another building where it was denitrated with steam

Refs: 1)Marshall 1(1917),211 2)Barnett (1919),34-5

AFU Propellant is described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959),Unit No 397

Agar-Agar (Japanese Gelatin). A yel-white mucilaginous substance (hydrophilic polysaccharide) extracted from some marine algae or sea weeds. It is insol in cold water but sol in hot w, after previous slow swelling (Ref 2). Its aq soln (hydrosol) cannot be easily coagulated by salts (Ref 3)

Agar-agar is used for the prepn of glue, as a culture medium and as a binding agent in some propellant and expl compositions (Ref 1)

Refs: 1)Daniel(1902), 6 & 361 2)Hackh (1944), 22-23) 3)B. Jirgenson & M.E. Straumanis, "A Short Textbook of Colloid Chemistry," Wiley (1954), p 331 4)I.Mizukoshi, JapP 6183(1954 & CA **50**,2093(1956) (Manuf of agar-agar)

Agar-Agar in Analytical Chemistry. According to E.J.Bogan & H.V.Moyer, IEC, Anal Ed **14**,849-50(1942) & CA **37**,575(1943), agar agar may be used as a coagulant for $BaSO_4$ in detn of sulfur or SO_4 by pptn with $BaCl_2$

Agar-Agar Substitutes can be obtained from floridean(red algae) starches

Ref: H.Kirschnick, Seifen-Öle-Fette-Wachse, **82**,39-41(1956)(Review with 16 refs)

Agave. An American aloe of the order *Amaryllidaceae*, widely cultivated in Mexico and Central America for its juice, which is used to make an alcoholic beverage called "pulque." The residual fibrous cellulosic material, after expressing the juice, is suitable for the prepn of cloths, cords, nets, etc. In addition there is a green waste material usually used for fuel and as a diuretic (Ref 3), but which can be dried and nitrated to yield *nitroagave*, an explosive similar to NC. The use of nitroagave in expls was patented by Trench, Faure and Mackie (Refs 1 & 2). The proposed mixt contained nitroagave, NC(dissolved in a volatile solvent), resin, ozocerite, charcoal or soot and glycerin

Refs: 1)BritP 2,742(1876) 2)Daniel (1902),773(under Trench) 3)Hackh(1944), 23

AGB (American Glycerin Bomb) is a time bomb used for blasting of shallow wells. A brief description is given in Blaster's Handbook(1952),449-50. The bomb is also mentioned by Cook(1958),280

Aged Propellants, Solubilities. Solubilities of some aged gun propellants were recently detd by P.E.Gagnon et al and reported in TM 229/59 of ARDE, Canada(1959)

Ageing (Aging) of Dynamites. The term ageing is applied to the total physical changes which dynamites undergo on storage in the course of time. With some dynamites(such as gelatin dynamites) as little as 3 months storage, even at ambient temp, is sufficient to reduce the sensitivity to explosion by influence as measured by the gap test. This decrease in sensitivity might cause misfires of charges in bore-holes and for this reason the problem of ageing is of great importance

The problem of ageing has been studied by many investigators and by such organizations as the US Bureau of Mines. The composition of dynamite is an important factor in ageing. Sawdust and woodpulp are hygroscopic and as they absorb moisture their absorbancy is reduced resulting in exudation of part of the NG. AN is also hygroscopic and its crystallographic form is altered(see under Ammonium Nitrate) resulting in changes in particle size and setting up with loss of sensitivity. Tamping thus becomes more difficult

The above discussion applies to "straight' (non-gelatinized) dynamites, but the greatest changes on ageing take place in the gelatin-dynamites, as was shown by tests conducted at the US Bureau of Mines(Ref 3). According to these tests the decrease in the velocity of detonation and of sensitiveness to explosion by influence of gelatin-dynamites proceeds more rapidly the smaller the diameter of the cartridge and the higher the percentage of explosive oil. For instance, low-freezing 60% gelatin-dynamites aged more rapidly than the corresponding 40% gelatin-dynamites

If dynamite is aged too long, it may become so insensitive that it will burn in the bore-hole instead of detonating. This is very undesirable (in addition to the economics involved) because burning dynamite usually gives off large quantities of nitrogen oxides, which are very poisonous even in small quantities. The "misfire" of an aged gelatin-dynamite may be prevented if the charge is well tamped. This is because good confinement insures better and more complete detonation (Ref 3)

It should be noted here that occasionally gelatin dynamites stored under conditions of excessive humidity may leak NG due to partial pptn of the NC from the NC-NG colloid. Such dynamites are very sensitive and hazardous to handle (Ref 6)

According to Dr I. A. Grageroff, formerly of the Apache Powder Co, Benson, Arizona and of the Keystone Ordnance Works, Meadville, Pennsylvania, the decreased sensitiveness to explosion by influence of aged gelatin-dynamites may be returned close to initial values ("rejuvenated") by prolonged rolling of the cartridges on a flat surface(Ref 5). Many miners are familiar with this method of "rejuvenation" of gelatin-dynamites

There seems to be two main theories of the causes of the ageing of gelatin-dynamites. The first theory, called the "air-bubble theory" (Luftblasentheorie in Ger), attributes ageing to the disappearance of air bubbles, normally present in freshly prepd gelatins. The second theory, proposed by W. A. Hargreaves, formerly Inspector of Explosives in South Australia, states that a gelatin-dynamite has a webbed structure which does not possess sufficient sensitiveness unless there is a considerable amount of free liquid NG between the webs. As soon as this liquid NG disappears (either by the formation of a gel with NC or by segregation, followed sometimes by exudation) the explosive becomes insensitive. In the opinion of Hargreaves(Refs 1 & 2), the gelatin is best prepd by first mixing the NC thoroughly in the cold with part of the NG and then, after gelatinization, adding the rest of the NG. He claimed that in this way some NG remained ungelatinized and the resulting gelatin-dynamite was less liable to age quickly

Dr J. Mayer of Villa María, Argentina, discusses both the above theories and seems to favor the second one(Ref 4)

The following conclusions cited by Dr M are in agreement with Hargreaves' theory: a)Gelatin-dynamites contg NG and soluble (low nitrogen content) NC age faster than those contg either partially soluble or completely insol NC. As an example Dr M cites "Meganit" prepd with insol NC. This explosive shows practically no ageing on long storage b)As NC deteriorates slowly in storage, losing part of its nitrogen, solubility in NG increases, thus "binding" more of the remaining free NG in the explosive. This results in a decrease in sensitiveness c) Substitution of part or all of the NG by a better gelatinizer for NC(such as NGc) results in a more rapid decrease in sensitiveness than the corresponding NG gelatin-dynamites

Note: It should be noted that NGc is much more volatile than NG and some of the ageing might be caused by the loss of some NGc by evaporation

d)Storing of gelatin-dynamite in hot climates causes faster ageing. This may be partly due to an increase of solubility of the NC in the NG or NGc and partly to evaporation of NG or NGc e)Dynamites contg free NG and no soluble NC(such as straight dynamites, ammonia dynamites, etc) show practically no ageing effects f)Gelatin-dynamites which contain, in addition to NC and NG or NG + NGc, some other HE's, which are gelatinizers for NC, such as DNT or TNT, age faster then those contg NC, explosive oils and a non-gelatinizing HE(such as PETN or RDX)

Note: Dr M recommends incorporation of 15-20% RDX in formulations of gelatin-dynamites as one way to prevent rapid ageing, especially in hot climates

This discussion includes only a part of the experimental work conducted by Dr Mayer, for the remainder see his paper(Ref 4)

Author's Note: If Hargreave's theory is valid and the "air-bubble theory" is wrong, what is the explanation for the fact that aged gelatin-dynamites can be "rejuvenated" by prolonged rolling of the cartridges on a flat surface as was reported by I.A.Grageroff. It seems that each theory may be partly right (See also Stettbacher, Explosivst **1954**, 44)

Refs: 1)W.A.Hargreaves, JSCI **33**, 337-40 (1914) 2)Marshall **1**(1917), 365 & 368 3) S.P.Howell & J.E.Crawshaw, US Bureau of Mines Rept of Invest No **2436**(1922) 4)J. Mayer, Explosivst **1953**, 39-41 5)Dr I.A. Grageroff, New York, NY; private communication 6)M.M.Inskeep, PicArsn, Dover, NJ; private communication

Ageing of Mine Explosives Containing Ammonium Nitrate and Chlorides was studied recently in Belgium. The results are reported in Ref. The authors attribute ageing of explosives contg AN and NaCl partly to ionic exchange from NH_4NO_3 + NaCl to NH_4Cl + $NaNO_3$ and partly to a change in particle size as a result of the ionic exchange

Ref: A.Kreyenbuhl & R.Sartorius, Industrie Chimique Belge **20**, Special Number, pp247-57(1955) & CA **50**, 17451-2(1956)

Ageing (Aging) of Propellants. This term is very loosely used, meaning deterioration or degradation in storage and sometimes improvement of physical, chemical and ballistic props which takeplace directly after preparation of the propellant

When cannon or rocket propellant grains prepd by extrusion are allowed to stand for several days to relieve strains or to improve gelatinization in these grains, it is referred in the US as "aging." On the other hand, when some propellants are "annealed" at 140°F in order to relieve stresses from the grains the process is not called aging but "*curing*." The term aging is also used when a propellant is subjected to storage for some time in order to detect any changes in its compn or ballistic props. This is actually a test for *degradation or deterioration of the propellant* (qv)

In GtBritain the term "ageing" has been used for the procedure which involves storing the previously dried (at about 50°) propellant grains in a humid atm for one to two months to raise the moist content of the grains to the desired level. This is necessary because the drying treatment to remove the excess of volatiles results in a too low moisture content. A propellant not subjected to "ageing" would absorb moisture ununiformly in storage and give erratic ballistics. In case of necessity the propellant might be "aged" rapidly by exposing the grains for a few hrs to a hot moist atm. This "artificial" method of "ageing" was claimed to be less satisfactory than the "natural" method

Refs: 1)V.N.Hicks, H.J.Frigand & J.Lerner, PicArsn; private communication 2)Marshall **1**(1917), 325 3)Barnett(1919), 84

Agesid 2. One of the Ger pre-WWI dynamites. It is described in PATR 2510(1958), p Ger 3

Agfa. Abbr for the "Aktiengesellshaft für Anilinfarben," a Ger firm which manuf dyes, fine chamicals, photographic films, reagents, etc

Aggiantive (Ital). Augmenting (charge for mortar or subsidiary artillery charge)

Agglutinant. Same as Binder

Aging of Dynamites. See Ageing of Dynamites

Aging of Propellants in Storage. See Deterioration (Aging) of Propellants in Storage

Agitation. See Stirring and Agitation

AGJ and AGK Propellants are described in confidentail "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959), Unit Nos 398 & 399

Agriculture and Forestry Uses of Explosives. One of the greatest of peacetime usages of expls is for agricultural purposes

According to Cottenet(Ref 4,p 6) expls used in agriculture should possess the following properties: a)Fairly high brisance

and power b)Insensitivity to ordinary shocks and flame c)Good resistance to humidity d)Its residue after explosion should be of value as a fertilizer e)Its cost should be very low

The most suitable expls for agriculture are the AN expls, such as coal mine permissible expls. Aromatic nitrocompds and some dynamites are not as suitable as AN expls but can be used. Expls contg chlorates and perchlorates are not suitable for most agricultural purposes because they develop poisonous gases. Chlorate and perchlorate explosives may be used, however, for clearing passes through jungles and forests and for the destruction of grasses and weeds in vegetable gardens and along railroads and highways

Following are the principal uses of expls in agriculture and forestry:

a)Planting of trees. This is usually done in dry hard ground where digging is difficult. For this purpose, narrow holes, about 30" deep are drilled in the ground and expl charges, ¼ to ½ lb, are fired. This not only enlarges the hole but loosens the surrounding soil so thoroughly that when a young tree is planted its root growth is greatly facilitated (Refs 1 & 3; Ref 4, pp 43-54)

b)Clearing the land of stumps. This is usually done by firing an explosive directly in the woody root of the stump. This shatters the stump and blows it out. If it is desired to remove the stump intact(as for instance in the case of stumps contg valuable substances(such as turpentine or resins) the charges are placed under the roots of the stump (Refs 1,2 & 3; Ref 4, pp 70-78)

c)Clearing the land of stones. This may be done, in the case of small stones, by exploding the charge under the stone. This method dislodges the stone and makes it easier to remove. In the case of larger stones (boulders), one or several charges of expl are placed on top or on the sides of the boulder. Each charge is covered by a thick layer of mud, clay or plaster and for this reason the method is called **mudcapping** or **plaster shooting**. This method, also called **adobe shooting**, breaks the boulder into smaller pieces which are more conveniently handled. The same purpose may be achieved by placing on a boulder one or several "shaped" charges. In some cases a boulder may be broken by exploding a length of detonating cord (eg, one filled with desensitized PETN) wound several times around the boulder and then mudcapped(Ref 2 & Ref 4, pp 55-63). See also Ref 3a

d)Converting rocky ground into tillable land. This may be accomplished by drilling holes in various parts of the rocky ground and exploding charges of brisant and powerful expls. For more information on this subject see Ref 4, 63-66

e)Levelling ground to make it tillable. This is done by exploding charges of various strengths in holes drilled horizontally and vertically in high spots of the ground (Ref 4, pp 67-70)

f)Digging ditches. This may be done by punching, with the aid of an iron bar, a series of holes spaced 18 to 24" and in the line of the desired ditch. After placing one or more cartridges of expl in each hole and placing several extra cartridges in the center hole, the setup is fired by means of a cap placed in the center charge. This method of firing is called the *propagation* method and is suitable only for very wet ground which is free of sand. In cases of dry or sandy ground, the above method does not work and it is necessary to fire the charges simultaneously by means of electric blasting caps placed in each charge (Refs 2 & 3)

g)Drainage of ground (such as in swamps, etc). This may be done either by digging wells(vertical drainage) or by digging draining ditches (horizontal drainage). Both methods are described in Ref 4, pp 89-93

h)Controlling erosion. In rolling country, such as in Georgia and the Carolinas, this may be done by firing charges of dynamite in the soil in order to loosen it. As a result, the waters from heavy rainfalls would soak into the ground instead of running off rapidly and

carrying away the top soil(Ref 2, p 41)
i)Other agricultural uses of explosives include: diversion of the courses of water streams(Ref 4, pp 96-7); digging of wells (Ref 4, pp 100-101); digging of ponds for watering cattle(Ref 4,pp 100-101) and in other rural construction (Ref 4,pp 97-100)
j)**Forestry** uses of explosives. These uses are much the same as those for agricultural purposes: eg, removing trees or tree stumps, etc, described in Ref 4,pp 94-96. Other forest uses of explosives are the construction of roads through forests and lumber camps(Ref 3, p 38) and stripping of bark from trees. This latter is often necessary for the control of insect pests. For this work detonating cord (cordeau) is merely wrapped about the trunk of the tree and fired with a detonating cap. The stripped bark is then removed and burned, together with the insect pests (Ref 2, p 400)
Refs: 1)Daniel(1902),6-7 2)Meyer(1943), 398-400 3)J.J.Berliner & Staff, 840 Broadway,NY 3, Pamphlet No 592 entitled "Explosives" (no date), pp 39-41 3a)J. Taylor, "Detonation in Condensed Explosives," Clarendon Press, Oxford (1952) p 22 4)Jean Cottenet, "Les Explosifs au Service de l'Agriculture," La Maison Rustique, Paris (1956)(104 pp)

A-Gun. Same as Atomic Gun

A-Gun Shell. Same as Atomic Gun Shell

AHH Propellant is described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959), Unit No 400

Aimable Cluster. This consists of a number of incendiary bombs held in a single container. When dropped from a plane, at a high altitude, the container opens a few thousand feet above the ground and allows the bombs to scatter over an area. For this reason it is called the *delay opening type,* and is distinguished from the *quick-opening* or *short-delay type,* which opens and scatters the bombs almost immediately below the airplane. As the parts from the quick-opening clusters constituted a major hazard to the planes which followed in formation flights, the USAF, after 1942, required the use of aimable clusters
Ref: W.A.Noyes, Jr edit, "Science in World War II," Office of Scientific Research and Development, Chemistry, Little, Brown & Co, Boston(1948), 395-6

Air Analysis and Air Sampling. The air of many explosives, chemical and ammunition plants is frequently contaminated with impurities which may be gases, mists, vapors of volatile solvents or high expls, dusts of high expls or other materials, etc. As most of these impurities are more or less toxic, it is necessary to insure that they are never in excess of the amts considered safe by the health authorities for breathing by human beings

For this reason analysis of plant air is of the utmost importance. Air analysis is also necessary for the detection of leaks or other abnormal conditions of manuf

Two general methods for testing air impurities are in use. One involves direct-reading instruments (such as thermal indicators, test paper indicators and other devices described in Ref 16, pp 245-53), the other involves removal of the impurity from a given vol of air and determination of the impurity by a suitable lab method. The second method is more reliable

The simplest and most widely used method of air sampling is to pass a continuous stream of air, by means of a pump or a suction bottle, from a fixed spot in bldg through a volume-measuring device connected to a series of two or three bubble bottles contg a solvent for the impurity to be removed from the air (Ref 16, pp 260-64). Instead of these devices, U-tubes filled with absorbers, such as activated charcoal, or silica gel may be used (Ref 16, 264-7). Sampling is followed by testing the removed impurity by colorimetric or other methods. For instance, either TNT or tetryl produce a red coloration in the presence of NaOH, whereas DNT gives a blue color

Dusts, especially those insol in common

solvents, can be evaluated by passing a measured vol of air through tared filters (paper, asbestos mat or sintered glass) as described in Ref 16, pp 255-6. Collection of dusts can also be made by an electrostatic method (application of the Cottrell precipitator principle)(Ref 16, pp 256-7) or by an *impinger* (a device for collecting dust by impingement at high velocity against a glass surface, followed by entrapment in water or other suitable liquids). Two types of devices, the "standard" impinger and the "midget" impinger are described in Ref 16, pp 257-60

Sometimes "grab samples" of the air are taken in suitable glass containers (sizes up 2500 ml) and transferred to the lab for examination, but this method is not as satisfactory as the continuous sampling briefly described above

In testing the atmosphere of various buildings of a plant manufg expls (as for instance TNT), it is necessary to det one or two ingredients, characteristic of each bldg. For instance, in the so-called "trihouse," it is necessary to know the concn in air of extremely toxic TeNM. It is also desirable to know the concn of TNT. In the bldg where nitric acid is recovered from the mono-waste acid, it is desirable to know the content of TeNM and of nitrogen oxides. In the bldg where spent sulfuric acid is concentrated by passing hot gases, of burning petroleum through the acid, it is desirable to know the content of SO_3 in the air

Some of the analyses are described in the refs listed below, others are given under the descriptions of manuf of individual expls

Refs: 1)O.Martienssen, BritP 237,930 (1924) & CA **20**, 1732(1926)(Apparatus contg an electrically heated catalyst which glows more brightly if combustible or explosive gases are present in the air) 2)H.F.Görlacher, Chem Fabrik **1935**, 329 & CA **29**, 7077(1935)(Apparatus using a thermocouple for detecting expl gases and vapors in air) 3)J.S.Haldane & J.I.Graham, "Methods of Air Analysis," Griffin, London (1935) 4)W.Deckert & B.Prathithavanija, ZAnalChem **113**, 182-9(1938) & CA **32**, 7858 (1938)(Colorimetric procedure using dimethylaniline for detg small quantities of chloropicrin in air, water, etc) 5)I.S.Shereshevskaya, PromOrgKhim **6**, 592-4(1939) & CA **34**, 5017(1940)(Detn of small quantities of aromatic nitrocompds in air using colorimetric and nephelometric methods) 6)J.B.Ficklen, "Manual of Industrial Health Hazards," Service to Industry, West Hartford, Connecticut (1940)(Included are methods for detn of over 90 noxious vapors, gases and dusts) 7)K. Kay, CanJRes **19B**, 86-9(1941) & CA **35**, 3562 (1941)(Analysis of air for the presence of TNT is conducted by drawing the air from one point in the room through two sintered glass bubbling tubes set up in series and contg ca 150 ml acetone, at a rate of 0.5 l/sec, for a period about 1 hr. After a partial concn of acetonic soln by evapn at temp below 82°, the vol of concentrate is measured and 1 ml is withdrawn for test. After adding 0.1 ml of 20% aq NaOH soln to 1 ml of concentrate, the resulting red coloration is compared with standards prepd by dissolving known amts of TNT in measured vols of acetone) 8)W.F. Oettinger, USPublicHealthBull No **271**, Washington, DC(1941), 113(Detn of TNT in air by the method of Kay)(See ref 7) 9)S. S.Pinto & J.R.Fahy, JIndHygToxicol **24**, 24-6(1942) & CA **37**, 847(1943)(Detn of TNT in air by collecting the sample in isopropanol by means of a midget impinger, reducing the TNT to triaminotoluene with $TiCl_3$ and estimating the triamino compd colorimetrically after diazotization and coupling as described in the paper) 10)F. H.Goldman, JInd HygToxicol **24**, 121-2 (1942) & CA **36**, 5349(1942)(Analyses of atmospheric samples contg DPhA, MF, NG, PETN, dimethylaniline, tetryl, TNT and DNT, collected by an impinger in suitable

solvents are described) 11)Kranke & von Gizycki(no initials given), SS **38**,32(1943) & CA **38**,5085(1944)(Detn of nitrocompds, especially TNT, by drawing 50-100 l of air through three wash bottles placed in series, each contg 10 ml of methanol, mixing the contents of the 1st and 2nd bottles, adding to the mixt 2 ml of 2% aq NaOH soln and allowing to stand 10 mins. Meanwhile the contents of the 3rd bottle are tested by adding aq NaOH-no coloration should be produced. The red color of the soln in the 1st and 2nd bottles is compared with standard solns of TNT in methanol. The test is claimed to be very sensitive: 0.5 mg TNT per m^3 may be detected by using a 100 ml sample. If only a qualitative test is required, a piece of filter paper impregnated with 5% NaOH soln is suspended in the air) 12)F.H.Goldman & D.E.Rushing, JIndHygToxicol **25**, 164-7(1943) & CA **37**, 5927(1943)(TNT is detected by passing the air through diethylaminoethanol, as a collecting medium. A red-violet color is produced even by traces of TNT) 13)F.H. Goldman & D.E.Rushing, JIndHygToxicol **25**, 195-6(1943) & CA **37**,5927(1943)(Tetryl is detected by passing the air through ethylaminoethanol as the collecting medium. A red color is produced) 14)Th.E.Cone, Jr, USNavalMedBull **41**, 219-20(1943) & CA **38**,529(1944)(A modification of the Kay method described in Ref 7. The method can be used for the detn of TNT, DNT and tetryl) 15)W.M.Cumming & W.G.D.Wright, BritJIndMed **2**, 83-5(1945) [Colorimetric detn of air-borne TNT, tetryl and DNT. This method is briefly described in CA **39**, 5209(1945)] 16)H.B.Elkins, "The Chemistry of Industrial Toxicology," Wiley,NY (1950), 17-18(Air analysis) and 245-73(Air sampling) 17)E.Effenberger, ZAnalChem **34**,106-9(1951)(Quantitative detn of oxidizing impurities in air) 18)J.Raubal et al, ČeskoslovHygEpidemiolMikrobiolImunol **2**, 300-3(1953) & CA **48**, 13548(1954) (Polarographic detn of TNT in air) 19)M.V. Alekseeva et al "Determination of Harmful Substances in the Atmosphere of Industrial Plants," Goskhimizdat, Moscow,(1954)(in Rus) 20)N.Strafford, G.R.N.Strouts & W.V.Stubbings, "The Determination of Toxic Substances in Air," W.Heffer, Cambridge, England(1956)

Air Blast produced by jets of air suddenly escaping from compressed air pumps, pipes, etc, may cause expl of firedamp

Ref: R.Loison & M.Giltaire, CA **49**, 9279 & 16436(1955)

Air-Blast Effect; Air Blast Energy; Air Blast Impulse and Air Blast Pressure. See under Blast Effects of Air, Earth and Water

Air-Blast Meter. See under Blast Effects, etc

Air-Blast Pressure from Small Charges of Various Explosives are discussed in OSRD Rept 3479(1944)

Air-Blast Pressures from Some Large Bombs are discussed in OSRD Rept 3046(1943)

Air-Burst Effects of the Blast from Bombs and Small Charges are discussed in OSRD Rept 4246(1944)

Air Conditioning is the branch of engineering devoted to the study of factors affecting both the physical and chemical conditions of the atmosphere within any structure. These factors include temp, humidity and motion, as well as distribution of dust, bacteria, odors and toxic gases. In air conditioning it is desirable that all factors be controlled but, if this is not feasible, at least the first three should be simultaneously controlled (see also Refrigeration and Ventillation)

Air conditioning is desirable in the manuf and storing of some expls,eg black powder

Refs: 1)W.H.Carrier et al, "Modern Air Conditioning, Heating and Ventilating," Pitman,Chicago(1940) 2)J.R.Dalzell & C.L.Hubbard, "Air Conditioning, Heating and Ventilating," AmTechSoc,Madison, Wis(1944) 3)Kirk & Othmer **1** (1947), 238-52(9 refs) 4)Perry(1950),776-86 5)Air Conditioning and Refrigerating Machinery Association,Inc,Washington,DC; various publications

"Aircraft and Missile Propulsion." Title of the book by M.J. Zucrow, Wiley, NY (1958)

Aircraft Armament. See Air Warfare and Aircraft Armament

Aircraft Armament Tests are described in the US Ordnance Proof Manual No 16-16

Aircraft Flares. See under Flares

Aircraft Float Lights. See Night Drift Signals

Air, Liquid, Explosives. See Liquid Air and Liquid Oxygen Explosives

Airplane Take-off Apparatus. A device patented by Taylor et al includes a gas-generating charge consisting of a compact smokeless propellant and a separate oxidizing charge comprising one or more compressed masses, formed by casting an O-positive mixt of AN with a chromate compd and a solid nonalk nonoxidizable AN fusion promoter

Refs: 1)J. Taylor & R. Wark, BritP 570, 210 (1945) & CA **40**, 5254 (1946) 2)J. Taylor & R.D.J. Owens, BritP 570, 211 (1945) & CA **40**, 5548 (1946)

Air Pollution (Atmospheric Pollution) is objectionable because it is a danger to the health of people and animals, causes damage to vegetation, corrosion of materials and unpleasant odors

In explosive industries, as for instance in the manufacture of TNT, pollution may be caused by TNT dust, the strongly acid fumes of oxides of nitrogen and sulfur and the vapors of tetranitromethane. All of these are harmful to both man and vegetation

Refs: 1)C.M. Christian, "Air Pollution Literature Review; Elimination of Air Pollution Due to Oxides of Nitrogen from Acid Manufacture at OAC Installations," Project No 56-151, Atlas Powder Co, Wilmington, Del 2)C.E. Lapple, "Dust and Mist Collection," p 1013 in Perry (1950) (numerous refs) 3) L.C. McCabe et al, "Proceedings of the US Technical Conference on Air Pollution," McGraw-Hill, NY (1952) 4)Collective, IEC **44**, 1339-88 (1952) (Symposium on air pollution) (9 papers and numerous refs) 5)A.R. Meetham, "Atmospheric Pollution; Its Origin and Prevention," Pergamon Press, London (1953) 6)C.A. Gosline, Edit, "Air Pollution Abatement Manual," Manufacturing Chemists Assn, Inc, Washington, DC (1954) 7)F.S. Mallette, "Problems and Control of Air Pollution," Reinhold, NY (1955) 8)P.L. Magill, Edit, "Air Pollution Handbook," McGraw-Hill, NY (1956) 9)Clark & Hawley (1957), 25-7 (See also Air Analysis and Sampling)

Air Reduction Sales Company, Murray Hill, New Jersey. This company has had several US Govt contracts, mainly in the field of high explosives, polymers, etc. As their reports are classified confidential no discussion of their work is possible here

Air Resistance to Projectiles in Flight is discussed in the books and papers listed under Ballistics, Exterior and Aerodynamics, as for instance: 1)C. Cranz, "Lehrbuch der Ballistik," Springer, Berlin, vol 1 (1925) 2er Abschnit, 36 2)L. Gabeaud, MAF **15**, 1220 (1936) (Recherche sur la résistance de l'air)

Airdox is a blasting device activated by compressed air and manufd by the Cardox Co, Chicago, Illinois. It is particularly suitable for breaking down coal in fiery mines. Descriptions of various types of Airdox are given in the book of J. Taylor & P.F. Gay, "British Coal Mining Explosives," G. Newnes, London (1958), 137-42

Air Drying Treatment of Propellants. See under Propellants, Manufacture

Air Lifts are devices for lifting liquids by means of compressed air without the use of valves, cylinders, plungers or other mechanisms. The first air lift was invented by Carl Löscher in 1797. Later developments were made by Frizell (1880) and Pohle (1892)

Essentially, an air lift consists of a U tube with legs of uneven length. Liquid (such as an acid) enters the shorter leg and is carried over into the longer leg and then discharged by means of compressed air introduced near the base of the longer leg.

The rise of liquid in the longer leg is due to the fact that the air and liquid mixture is lighter than the liquid alone

The *pulsometer,* one kind of air lift, is a 3-neck, bottle-like pot (made of chemical ware or Pyrex) with the first neck entering one side of the bottle at its bottom, the 2nd neck entering the other side part-way down and the 3rd (middle one) at the top of the pot. Liquid (such as acid) enters through the first neck (bottom) and is met by compressed air entering through the 2nd neck. The resulting air-bubbles lift, under pressure, the liquid through the 3rd neck to the desired height up a vertical tube set in the top outlet

Pulsometers, used in Acid Recovery Houses of explosives plants, are sometimes called *nigger heads.* They serve for transferring the weak nitric acid from the bottom of one absorption tower to the top of the next

Air lifts were formerly used for transporting corrosive liquids from one part of a plant to another, but have now mostly been replaced with acid or alkali resistant centrifugal pumps

Refs: 1) F.C. Vilbrandt, "Chemical Engineering Plant Design," McGraw-Hill, NY (1942), 70 2) J.H. Perry, Edit, "Chemical Engineers' Handbook," McGraw-Hill, NY (1950), 1438 3) E.R. Riegel, Chemical Machinery, Reinhold, NY (1953), 171-3

Air, Thermodynamic Properties are discussed in OSRD Rept 369 (1942) and OSRD Rept 3550 (1944)

Air-Tightness Test. Some explosives, propellants, pyrotechnic compositions and ammunition used at the present time deteriorate much quicker if they are stored under atmospheric conditions (especially in the presence of moisture) than if the containers in which they are stored are air-tight. As air-tight containers are currently in use, it is necessary to test them to determine whether there are any leaks in them. This can be done by pumping air into a closed container until a pressure of 3 to 5 lbs is reached and then noting the variations in the reading of the gauge attached to the air testing apparatus. The pump is connected to the metal container by a special test hole with which all the containers are provided. These holes are closed with air-tight plugs, while the tests are being conducted. The tests should be conducted at least once a year, oftener if there is a suspicion that the container leaks

All containers which are leaking should be repaired, or the contents transferred to air-tight containers. Leaks due to defective covers and gaskets can usually be repaired without removing the contents, but leaks in other parts require the transfer of the contents to air-tight containers and removal of the defective container from the magazine, for repairs. These may be made either in a building containing no explosives or in the open. If it is necessary to repair the containers by soldering, the insides should first be thoroughly cleaned to remove all dust

Note: In opening or closing containers, only nonsparking tools (such as those made of copper alloys or berylium-bronze, etc) should be used

Ref: US Army, Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition," OO Form No 5994, Washington, DC (1928), Sectn XIV, p 4

Air Warfare and Aircraft Armament. War in the air is conducted by aircraft equipped with the following weapons: bombs, guns, machine guns, rockets and guided missiles. General discussion on this subject is given in the following refs: 1) J.C. Boyce, "New Weapons for Air Warfare, Fire Control Equipment, Proximity Fuzes and Air Guided Missiles," Little-Brown, Boston, Mass (1947) 2) S.T. Possony, "Strategic Air Power, the Pattern of Dynamic Security," Infantry Journal Press, Washington, DC (1949) 3) A.P. deSeversky, "Air Power; Key to Survival," Simon & Schuster, NY (1950) 4) R.T. Finke, Ordn **39**, 160-1 (1954) (Aircraft armament)

Aix-la-Chapelle (Poudre d'). One of the older French blasting expls, which was a mixt of

finely pulverized Chile saltpeter (Na nitrate) and carbon
Ref: Daniel(1902) 8

Ajax Powder. A British "permitted" perchlorate explosive: NG 22.5, GC 0.75, TNT + DNT 3.0, WM 10.5, $KClO_4$ 37.5, Amm oxalate 25.0 and H_2O 0.75%; power(swing of ball pend) 2.69", maximum chge 12 oz
Ref: Barnett(1919), 137

AJZ Propellant is described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland (1959), Unit No 404

AK-14 and AK-14(Mod 1) Rocket Fuel Oxidizers are described in confidential "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Md(1959), Unit Nos 1 & 352

Akardit. Ger name for Acardite

A(ko) or Type A Explosive. A Japan expl similar to Ger Hexamit: TNT(or TNAns) 60, HNDhA 24 & Al powder 16%. It was intended for use in torpedo warheads and depth charges to replace the *Type 94* and *Type 97H* Explosives (See Japanese Type Explosives)
Refs: 1) R.A. Cooley et al, PBL Rept 53045(1945), p 9 2) Anon, OpNav 30-3M, "Handbook of Japanese Explosives Ordnance," Washington, DC(1945), 32

Akremite. An explosive, patented by Maumee Collieries Co of Terra Haute, Indiana, suitable for all open operations—stripping, open mining, quarrying, etc. It consists of a commercial grade AN and carbon black shipped in separate polyethylene containers to the place of operation and mixed there as needed. The mixt is put into a blast hole so that there are no air pockets around the expl which is then detonated with a small "booster" charge of a cap-sensitive expl, such as gelatin-dynamite

Since neither the raw materials nor the finished mixture are cap-sensitive expls, they can be shipped at commercial freight rates rather than at the higher rates for expls

The effectiveness of Akremite in actual mining operations was described in Coal Age, May 1955
Ref: Anon, Chem Engrg 62, No 6, 108(1955)

AL-31 Rocket Explosive is described in confidential "Propellant Manual" SPIA/M2, Johns Hopkins Univ, Silver Spring, Md(1959), Unit No 352

Alba, Chemical Co of New York patented, in 1900, a dynamite contg NG, AN, Na nitrate, wood meal & vaseline
Ref: Daniel(1902), 8

Albanite. A white flashless propellant developed during WWII in the USA for use in large Naval guns. The history of its development is given in Ref 1, pp 106-108, and in Ref 2, pp 129-31. It is actually the Brit *Cordite N* (a triple base propellant containing a large proportion of NGu with the remainder chiefly NG & NC) but contg DINA in place of NG. Its composition and some properties were as follows: NC(12.6%N) 20.0, DINA 19.5, NGu 55.0, DBuPh 4.0 & Et Centr 1.5%; to this may be added an amt of K_2SO_4 which varied with the weapon (eg 1.5% K_2SO_4 for the 6 in/47 gun); vol solvent ca 0.2% & moisture <0.1%. For compn contg 1.5% K_2SO_4 the adiabatic flame temp is 2390°K, the potential (C_vT_o) 870 cal/g, the force 98% of pyro propellant and burning rate 75% of pyro

During WW II Albanite and Cordite N were considered among the least erosive propellants

According to Riegel (Ref 3), Albanite is a better flashless propellant than FNH and has low sensitivity to shock
Refs: 1) Summary Technical Reports of Division 8, NDRC, vol 1 "The Preparation and Testing of Explosives," Washington, DC(1946), 106-10 2) W.A. Noyes, Jr, Edit, "Science in World War II, OSRD, Chemistry," Little, Brown & Co, Boston(1948), 24 & 129-31 3) Riegel, IndChem(1949), 709
Additional Refs on Albanite:
A) Explosives Division, Burnside Laboratory, E.I. duPont de Nemours & Co, Inc, OSRD 3726(1944)(Flashless propellant for Navy cannon) B) E.I. duPont deNemours & Co, OSRD 4035(1944)(Analytical procedure

for chem analysis of Albanite) C)E.I.duPont deNemours & Co,Inc, OSRD 5475(1945)(Albanite cannon propellant manufd at Winnipeg Works of Defence Industries Ltd) D)F.H. Westheimer & R.H.Kallenberger, OSRD 5592 (1945)(Substitutes for Albanite) E)E.I. duPont deNemours & Co,Inc,OSRD 6215 (1945)(Final rept on flashless propellant for Navy cannon)

Albionite. According to Daniel(1902),8, it was an expl manufd by the Nobel Explosives Co, Ltd. Its compn is not given

Albit. A Ger blasting expl, such as **Gesteins-Albit** ("Rock-blasting Albit"). It contained Na perchlorate 80, DNN 12, wood meal 3, phenanthrene 3 & NG 2%

Refs; 1)Naoúm, Expls(1927),129 2)PATR 2510(1958), 69

Albite. An expl invented by Bernardini & Manuelli: AN 58.6, NGu 19.1 & GuN 22.3%. It was used during WW II for filling some Ital projectiles

Refs: 1)R.Molina, "Esplodenti," Hoepli, Milano(1930),343 2)Ordnance Sergeant, Aug 1943, p 16 3)All & EnExpls(1946), 149 4)M.Giua, "Dizionario di Chimica," UTET, Torino, 2(1949), 128 & 165 4)C. Belgrano, "Gli Esplosivi," Hoepli, Milano (1954), 131-3

Albumin and Protein, Nitrated. See Protein and Albumin Nitrated

Alcohol (Span). Alcohol

Alcohol. An organic alkyl compd contg a hydroxyl group. It is also the common name for ethyl alcohol or ethanol. This and other alcohols, as well as their nitrated derivs, are described under individual names, such as allyl-, amyl-, butyl-, ethyl-, methyl-, propyl-, etc

Alcohol, Nitration. See under individual alcohols

Alcohols, Heats of Combustion and Formation. See F.D.Rossini, BurStdsJRes **13,** 189(1934) and under individual alcohols

Alcohols, Nitrated, Heats of Combustion and Formation. See R.M.Currie et al, IEC 44,329-31(1952)(15 refs) and under individual alcohols

Alcohols, Nitrated, Preparation from nitroparaffins by condensation with aldehydes in the presence of an alkaline catalysts is discussed by H.A.Aaronson in PATR 1125 (1941)

Alcohol, Tribasic. See Glycerin or Glycerol

Alcool (Fr.or Ital). Alcohol

Aldehyde is an organic compd contg the monovalent –CHO radical. It is also the common name for acetaldehyde. This and other aldehydes are described under individual names, such as benzaldehyde, formaldehyde, etc

Aldehyde-Amine Condensation Products. It was observed in 1850 by Laurent and Gerhardt that benzaldehyde and aniline react with the formation of the compound $C_6H_5CH:NC_6H_5$, now called a *Schiff's base.* It has also been called an *azomethine* because it may be considered as a deriv of azomethine, $H_2C:NH$, also known as *methylenimine.* The Schiff's base may be regarded as the condensation product $C_6H_5CH(OH)\cdot HNC_6H_5$, from which a molecule of H_2O has been eliminated. A few years later (1864), Schiff found that many aromatic and aliphatic aldehydes can condense with aniline in a similar manner. The condensation may be effected by warming the compds together in approximately equimolecular proportions, either diluted with a suitable solvent(such as alcohol or AcOH) or without diluents. The azomethines derived from the lower aliphatic aldehydes and primary aromatic amines are less stable than those from aromatic aldehydes. Only comparatively few aldehydes and amines yield simple addition compdsof the general formula $R.CH(OH).HN.R'$, the majority give the corresponding azomethine $R.CH:NR'$

Formaldehyde reacts with primary and secondary aliphatic aminesbut not with tertiary amines. The reactions with methyl and dimethyl amines proceed as follows: $HCHO + H_2N.CH_3 \rightarrow H.CH(OH).HN.CH_3$, $HCHO + HN(CH_3)_2 \rightarrow HCH(OH).N(CH_3)_2$

The reaction of formaldehyde with

ammonia results in the formation of hexamethylenetetramine, $(CH_2)_6N_4$, used for the prepn of RDX

Some azomethines, eg $H_2C{:}N.C_6H_4CH_3$ have a tendency to polymerize to di- or trimetric forms

For more information on this subject see Refs 2, 3, & 4

Some of the aldehyde-amine condensation products have been recommended as ingredients of nondetonating, deflagrating, expl compns (similar in properties to black powder) for use in the delay combustion-train devices of fuzes. Other ingredients of such compns are non-explosive, such as alkali metal nitrates and charred carbonaceous compounds(Ref 1)

Refs: 1)R.C.Payn et al, BritP 576,107 (1946) & CA **42**, 1424(1948) 2)Hickinbottom(1948), 158-60 3)Walker(1953), 281-90 4)Clark & Hawley(1957), 846-7

Aldehyde-Nitroparaffin Reactions. The reaction between an aldehyde and a nitroparaffin usually proceeds as follows:

$$RCHO + R'NO_2 \rightarrow RCH(OH).CH_2NO_2 \rightarrow RCH{:}CHNO_2 + H_2O$$

The first studies on this subject were apparently made by Haitinger (Ref 1) and then by Priebs(Ref 2). These were followed by Henry(Ref 3), Posner(Ref 4) and others. More recently Boileau(Ref 5) made aldehyde-nitroparaffin reactions the subject of his thesis. By means of these reactions he prepared a number of aliphatic nitroalcohols. He also studied the kinetics of aldehyde-nitroparaffin reactions conducted in a homogeneous medium and in the presence of basic catalysts

Refs: 1)L.Haitinger, Ann **193**, 366(1878) 2)B.Priebs, Ann **225**, 319(1884) 3)L. Henry, CR **120**, 1265(1895) & **121**, 210(1895) 4)T.Posner, Ber **31**, 656(1898) 5)J. Boileau, "Contribution à l'étude de la réaction des aldéhydes sur les nitroparaffines," Thèse, Série n° 2586, presented at the Faculté des Sciences, Université de Paris to obtain the degree of "Docteur ès Sciences Physiques." Imprimerie Nationale, Paris, 6 June 1953(76 pp and 72 refs). This thesis is reprinted in the Annexe of MP **35** (1953)

Adehydes, Oxidation and Ozonization is discussed by H.Wieland & A.Wingler, Ann **431**, 301-22(1923) & CA **17**, 2558(1923)

Aldehydes, Polarographic Method of Determination is discussed by M.B.Neiman, ZhAnalKhim **2**, 135-46(1957)

Aldehydes, Role in Oxidation of Hexane. It was reported by C.F.Cullis, BullFr **1950**, 863-8 & CA **47**, 2689-90(1953) that an expln might take place if a temp of ca 227° is reached during oxidation

Aldol and Aldol Condensation Reactions. An aldol is a compd of the general formula $RCH_2.CH(OH).CHR'.CHO$, where R and R' are alkyl radicals. Aldols were first studied by Butlerov who in 1861 obtained some sugar-like products later proved to be aldols

Aldols can be obtained as result of the so-called *aldol condensation reaction*. This takes place between an aliphatic aldehyde and another aldehyde or ketone. The second aldehyde has at least one $-CH_2CHO$ group. Formaldehyde is the most frequently used aldehyde for this reaction. A small amount of weak alkali serves as a catalyst. The reactions may be represented as follows:

$$RCH_2.CHO + R'CH_2.CHO \rightarrow RCH_2.CH(OH).CHR'.CHO$$ and

$$RCHO + CH_3.CO.CH_3 \rightarrow RCH(OH).CH_2.CO.CH_3 \rightarrow RCH{:}CH.CO.CH_3 + H_2O$$

The first member of the group is *acetaldol* (qv) which condenses on storage to *paraldol*

Some aldols serve for the prepn of explosives: eg, paraldol, which on hydrogenation gives the 1,3-butylene glycol, yielding on nitration the explosive *1,3-butyleneglycol dinitrate*(qv)

Refs: 1)Hickinbottom(1948), 160 & 179 2)Karrer(1950), 164 3)Walker(1953), 164

Aldol Condensation Product of 5-Aminotetrazole. See under Aminotetrazole

5-Aldolimino-(α-tetrazole),

$CH_3 . CH(OH). CH_2 . CH: N-C(=N)-NH-N=N$

mw 155.16, N 45.16%, OB to CO_2 -139.2%, OB to CO -87.6%. Lt-col, shiny leaflets (from alc) or prisms (from water); mp 170°. Fairly sol in w; sl sol in hot alc; insol in eth, acet or chlf. Was prepd by adding to 5-amino-(α-tetrazole) (mp 203°) an excess of anhydrous acetaldehyde, previously distilled in a stream of CO_2 gas. After the resulting foaming had subsided, the mixt was heatedunder reflux for several hrs on a water bath and then cooled. The hardened mass was washed with cold alc and then recrystallized several times either from alc or from w

The *silver salt of aldolimino-tetrazole*, $C_5H_8N_5OAg$, is a mild explosive

Refs: 1)Beil–not found 2)P. Stollé, JPrChem **148**, 218-20(1937) 3)E.R.Benson, ChemRevs **41**,7(1947)

Aldolnaphthylamine Condensation Product (no formula is given). A solid which can be prepd from a soln of a mixt of 1.5 mol of aldol-α-naphthylamine and 0.5 mol of aldol-β-naphthylamine in 1.5 mols of dil HCl with 1.5 mols of acetaldehyde. This was followed by neutralization, filtering, purification and drying

When 2 ps of this mixture were milled for 3 hours with 100 ps of black powder consisting of KNO_3 75, alder charcoal 17 and sulfur 8%, the resulting product burned in fuses at the rate of 250-300 sec/yd (Ref 1), whereas ordinary fuse powder burns at the rate of 120 sec/yd (Ref 2). This aldol-naphthylamine condensation product was proposed as an ingredient of slow burning fuse powders

Refs: 1)R.C.Payn et al, USP 2,423,427 (1947) & CA **41**,6050(1947) 2)Blasters' Hdb(1952),88

Aldonic Acids, Their Derivatives and Nitric Esters. Aldonic Acids are hydroxy-acids of aldoses, which are carbohydrates contg aldehyde groups (pentose, hexose, etc). The acids can be obtained by mild oxidation (with silver salts of bromine) of aldoses, eg:

$$\underset{\text{glucose}}{CH_2OH(CHOH)_4CHO} \xrightarrow{\text{oxygen}} \underset{\text{gluconic acid}}{CH_2OH(CHOH)_4COOH}$$

A number of such compds were prepd and then converted to the corresponding nitric esters by Wolfrom et al of Ohio State Univ while working under a US Ordnance Corps contract(Ref 5 & 6). They found that all the pentanitrates prepd by them could be detonated by gentle heat or by a hammer blow on steel. Thermal stability tests have shown that the aldonamide pentanitrates are more stable than aldonic acid pentanitrates or their methyl esters and that the esters were less stable than the corresponding acids. All of their purified nitrates underwent rapid decompn at their mp's, accompanied by violent bubbling and evolution of nitrogen oxides. No residue was visible after decompn

In a surveillance period of 8 to 10 months the purified nitrates, maintained at 20-35° in a desiccator, exhibited no visible evidence of decompn

Some of the pentanitrates were suggested for use in propellants. To determine the suitability of these nitrates for this purpose, compatibility tests were made with NC (12.6%N). The aldonamide pentanitrate and the methyl ester of aldonic acid pentanitrate were found to be compatible with NC in film formation(employing ethyl acetate as the solvent) and the stability of the film was comparable to that of the free pentanitrate

Aldonamides(such as d-galactonamide and d-gluconamide) were prepd by the method of Glattfeld & Macmillan(Ref 2). This involved treating the corresponding lactones with liquid ammonia and nitration of the products by nitrogen pentoxide, as described by Caesar & Goldfrank(Ref 3). Methods of prepn of individual compds are given in Ref 6

Following are examples of pentanitrates of aldonamides, of aldonic acids and of

methyl aldonates. These compds resemble NC in their explosive and combustive properties

a) *d-Galactonamide Pentanitrate*, $C_6H_8N_6O_{16}$, mw 420.17, N 20.01%, OB to CO_2 0.0%, OB to CO +22.9%. Long, slender crysts, mp 168°(decomp); insol in eth, petr eth, chlf and water; sol in alc, methanol and dioxane. Method of prepn and more information on properties are given in Ref 6

b) *d-Gluconamide Pentanitrate*, $C_6H_8N_6O_{16}$, mw 420.17, N 20.01%, OB to CO_2 0.0%, OB to CO +22.9%. Crysts, mp 147°(decomp); soly characteristics are same as for previous compd(Ref 6)

c) *d-Galactonic Acid Pentanitrate*, $C_6H_7N_5O_{17}$, mw 421.16, N 16.63%, OB to CO_2 +5.7%, OB to CO +28.5%. Crysts, mp 138°(decomp); sol in eth, acet, alc and dioxane; sl sol in chlf; insol in w & benz(Ref 6)

d) *d-Gluconic Acid Pentanitrate*, $C_6H_7N_5O_{17}$, mw 421.16, N 16.63%, OB to CO_2 5.7%, OB to CO +28.5%. Crysts, mp 122°(decomp); soly characteristics are same as for previous compd(Ref 6)

e) *Methyl-d-galactonate Pentanitrate*, $C_7H_9N_5O_{17}$, mw 435.18, N 16.10%, OB to CO_2 -5.5%, OB to CO +20.2%. Crysts, mp 107°(decomp); sol in chlf, alc, eth and acet; insol in light petr eth (Ref 6)

f) *Methyl-d-gluconate Pentanitrate*, $C_7H_9N_5O_{17}$, mw 435.18, N 16.10%, OB to CO_2 -5.5%, OB to CO +20.2%. Crysts, mp 58° (decomp); soly characteristics are same as for previous compd (Ref 6)

Refs: 1)Beil **3**, 2 2)J.Glattfeld & D.Macmillan, JACS **56**, 2481-2(1934) (Preparation of aldonic and saccharic acid amines in liquid ammonia) 3)G.V.Caesar & M.Goldfrank, JACS **68**, 372-5(1946)(Nitration of starches with nitrogen pentoxide in presence of sodium fluoride) 4)Karrer(1950), 325 & 331 5)M.L.Wolfrom et al, US Ordnance Corps Contract No DA-33-019-ORD-163, Project No TB 3-0110-S), Ohio State Univ Res Foudn, Columbus, Ohio. Interim Tech Rept(1951) and Final Tech Rept(1952), p 2 6)M.L.Wolfrom & A.Rosenthal, JACS **75**, 3662-4(1953)(Nitrated aldonic acids) (20 refs)

Aldorfit or Aldorfite [spelled by Escales (Ref 1) *Alldorfit*] is a permissible expl developed and manufd by the SSF(Schweizerische Sprengstoff-Fabrik) A-G, Dottikon, Switzerland. Several formulations are known of which the **Aldorfit-pulverförmig** is the oldest. It contains AN 81, TNT 17, and wood meal 2% (Refs 2,4,5,6 & 7). Its loading d 0.9-1.05, mp-deflgr >360°; Qe 1010 1046 kcal/mol(H_2O vapor), calcd vol of gases at NTP 890 l/kg, temp of expln 2900°, max vel of deton 4500 m/sec, impact sensitivity with 2kg wt 100 cm, Trauzl test value for 10g sample 363-375 cc vs 311 cc for TNT, compression of copper cylinder (7.0 × 10.5 mm) 1.89 mm vs 2.86 mm for TNT (Ref 4, pp 104, 114 & 123; Ref 5, p 133, 144, & 153; Ref 6, p 40). Some props are also given for Aldorfit contg 21.27% TNT(Ref 4, p 14)

A method of prepn of Aldorfit is described in Ref 3

Some recent formulations of Aldorfit contain aluminum, as for instance **Aldorfit LL, aluminiumhaltig**. Stettbacher (Ref 4, p 114 & Ref 5, p 144) reports its Trauzl value as 432 cc but gives no compn

(See also Gelatin-Aldorfit)

Refs: 1)Escales, Ammonspr(1909), 105 2)Naoúm, Expls(1927), 118 3)Stettbacher(1933), 302 4)Stettbacher(1948), 14, 86-7, 104, 114 & 123 5)A.Stettbacher, "Pólvoras y Explosivos," G.Gili, Buenos Aires(1952), 113, 133, 144 & 153 6)A. Stettbacher, Explosivst **1954**, 40 7)Schweiz-Sprengstoff-Fabrik A-G letter dated Dec 10, 1953 to Dr A. Stettbacher, Zürich, Switzerland and transmitted to the authors 8) PATR 2510(1958), p Ger 3

Aleurityl Azide, $C_{15}H_{28}(OH)_3.CO.N_3$; solid mp-dec on slow heating ca 52°, expl when heated rapidly to ca 50°, insol in w & petr eth sol in chlf & alc and sl sol in eth & benz. Was prepd by refluxing aleuritic acid (9, 10, 16-trihydroxypalmitic acid), $C_{15}H_{28}(OH)_3$-COOH with 30% $N_2H_4.H_2O$ in MeOH and treating the resulting aleurityl hydrazide,

$C_{15}H_{26}(OH)_3 . CO . HN . NH_2$ with $NaNO_2$ in dil AcOH

Refs: 1)Beil–not found 2)A.L.Davis & Wm.H.Gardner, JACS **64**, 1902-5(1942) & CA **36**, 5770(1942)

Alexander's Explosive Compositions consisted of naphthalene(with one or more other solid hydrocarbons), K picrate and oxidizers such as KNO_3 or $KClO_3$ in various proportions

Ref: Daniel (1902), 8

Alexander's Primary Explosive consisted of amorphous phosphorus 83 and $Pb(NO_3)_2$ (or other oxidizing metal salt) 17%

Ref: Cundill, MP **5**, 280(1892)

Algin or Alginic Acid is a protein of marine algae and is found in many seaweeds. Its principal source of prepn is as a by-product of the extraction of iodine from kelp, principally from Laminaria digitata. It has been used mainly in Japan, for the prepn of films, fabric dressing, and for thickening jellies. Its soln in Na carbonate can be used as a mucilage (Refs 1 & 2)

Its salt, **sodium alginate,** is used in the manuf of priming compns and in loading ammo. The requirements of the US Army and Navy (Ref 3) are: a)moisture–not more than 20%, when detd as prescribed in paragraph F-4a of Ref 3 b)viscosity of a 1.25% aq soln at 20° shall be not less than 45 secs and not more than 60 secs, when detd as prescribed in par F-4b c) Water and methyl alcohol insolubles shall not be above 1%, when detd as prescribed in par F-4c

Refs: 1)Hackh(1944), 30 2)J.T. Marsh & F.C.Wood, "Introduction to the Chemistry of Cellulose," Chapman & Hall, London (1945), 104 3)US Specification JAN-S-541(1947)

Algodón fulminante or Algodón pólvora (Span)(fulminating cotton). NC with about 13.4% N

Ref: Pérez-Ara(1945), 358

Aliphatic Alkanolamine Perchlorate Salts. These compounds, according to the US NavOrdLab Memorandum 10068, 3/24/1949, are characterized by low mp, good heat stability and moderate sensitivity

Aliphatic Amines and Imines, Nitrated Derivatives. A number of these compds are of interest in the field of explosives and are discussed under the individual compds, such as aminomethane, aminoethane, aminoguanidine, etc

Some aliphatic amines and imines and their nitrated derivs were prepd and investigated before and during WW II by Division 8 of NDRC

Ref: J.R.Johnston et al, OSRD Rept 161 (PBL Rept 31093) (1941)

Aliphatic-Aromatic Nitramines. See individual compds

Aliphatic–Aromatic Nitrocompounds. See individual compds

Aliphatic Nitrocompounds. See individual compds

Aliphatic Peracids (Peroxyacids), called also **Peroxides of the Structure RC(:O)OOH** include performic (peroxyformic), peracetic (peroxyacetic), perpropionic (peroxypropionic), etc acids. They are described in the book by A.V.Tobolsky & R.B.Mesrobian, "Organic Peroxides," Interscience NY(1954), 33-6 & 167. Some of these compds are expl

Aliphatic Peracids (Peroxyacids), Analyses. As these acids have similar props, the same analytical procedures can be applied to all of them. The following two methods have been used for detg the contents of these acids:

a) The method of d'Ans & Frey (Ref 2) based on the procedure of Baeyer & Villiger (Ref 1) consists in titrating of a soln of a peracid with std K permanganate to a pink color adding to the resulting mixt an excess of KI soln and titrating the liberated iodine with std Na thiosulfate

b) The method of Greenspan & MacKellar (Ref 3) is based on the use of std ceric sulfate for the initial hydrogen peroxide

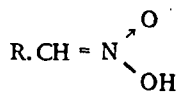
titration, followed by an iodometric detn of the active oxygen present as peracid. It has been claimed that this method gives more reliable results than the method of d'Ans & Frey

Refs: 1)A. Baeyer & V. Villiger, Ber **34,** 854 (1901) 2)J. d'Ans & W. Frey, Ber **45,** 1845(1912) & ZAnorgChem **84,** 145(1913) 3)F. P. Greenspan & D. G. MacKellar, AnalChem **20,** 1061-3(1948)

Alkali and Alkaline Earth Metals explode when brought into intimate contact with Cl, S, O, and some other compds as by a strong impact. This property can be of use in producing explns for blasting or bursting projectiles (Ref 1). In Ref 2 it is stated that the alkali metals act as powerful detonators with org halogen compds such as CH_2Cl_2, CH_2Br_2, CH_2I_2, $CHCl_3$, etc. In Ref 3 are described tests for the reactivity of various combinations of alkali and alkaline earth metals with halogen substitution compds. Many of these systems were found to be highly sensitive to heat or impact and those which are less sensitive react explosively under the influence of a detonator. It is suggested that the expl props of such mixts may be explained by the intermediate formation of small amts of highly expl compds (such as halogen acetylenes) which cause the expl decompn of the entire system

Refs: 1)H. Staudinger, USP 1,547,076(1925) & CA **19,** 2879(1925) 2)H. Staudinger, ZElektrochem **31,** 549-52(1925) & CA **20,** 537(1926) 3)F. Lenze & L. Metz, SS **27,** 255-8, 293-6, 373-6(1932) & CA **27,** 844(1933)

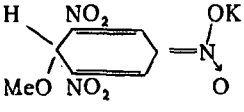
Alkalies, Action on Aliphatic Nitrocompounds. Compds with hydrogen atoms attached to a primary or secondary carbon atom linked to an NO_2 group show weakly acid properties. Such compds dissolve or react slowly in strong alkalies to form salts. This formation of salts is usually comparatively slow. It is inferred that these nitrocompds exist in two forms: a normal nitro form, which is the more stable but less acidic, and an aci(or iso) form which is less stable. The aci form may be written as



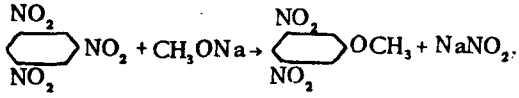
(also called a nitronic acid). This acid form of an aliphatic nitrocompd has not yet been isolated. However, the aci forms of mixed aromatic-aliphatic compds, eg phenylnitromethane and the p-brom derivative, have been isolated(Ref 1). The aci form of phenylnitromethane is acidic, conducts an electric current and dissolves readily in Na_2CO_3 solution

Refs: 1)A. Hantzsch & W. Schultze, Ber **29,** 699 & 2253(1896) 2)Sidgwick(1937),231 et seq 3)Wheland(1949),629 et seq 4) Karrer(1950),135 4)Degering(1950),71

Alkalies, Action on Aromatic Nitrocompounds. Unlike aliphatic nitrocompds, in which C atoms may be primary or secondary, the NO_2 groups in aromatic nitrocompds are attached to tertiary C atoms. As a consequence, the latter cannot have an aci- nitro structure and cannot form nitronic acid salts, although such aci-forms have been postulated. The chemistry of the interaction of alkalies and bases with different types of nitrocompds have not been satisfactorily clarified, although various structures have been postulated for the reaction products in particular cases. In general, in the absence of acidic groups, mononitro aromatic derivs yield no color or at most a yellow or orange. Di- and trinitro derivs, in alcoholic or acetonic solution, usually give decided colors (frequently red and sometimes orange, blue or green). The compds formed are presumed to be addition products of a salt-like nature. Thus trinitrobenzene, in methyl alcohol, when treated with concd aqueous KOH, yields a red expl compd whose formula corresponds to $C_6H_3(NO_2)_3, CH_3OK . H_2O$. The structure



has been proposed. On treatment with acids TNB is regenerated. It has been suggested that since color formation requires more than one NO_2 group, the true structure is a resonance hybrid and involves quinonoid systems. In a number of cases clear cut reactions can be obtained. Thus boiling TNB in methyl alcohol with a solution of Na methoxide proceeds according to the reaction:



yielding 3,5-dinitroanisole

Some reactions of nitrocompds with strong alkalies are quite vigorous. A mixt of powd TNT andpowd KOH inflames when heated to only 80°. Compds formed by the action of alkalies on TNT are very sensitive expls. It has been suggested by some investigators that these substances are metallic salts of $(O_2N)_3C_7H_5 \cdot CH:CH \cdot C_7H_5(NO_2)_3$ or of $(O_2N)_3C_7H_5 \cdot CH_2 \cdot CH_2 \cdot C_7H_5(NO_2)_3$

Na carbonate reacts with TNT to form a black solid which is sol in water or methanol and melts above 200°. This solid is as sensitive to impact and heat as tetryl and is very unstable at 120°(Ref 6)

Note: According to Dr Kostevitch(Ref 2), purification of TNT by an aq soln of Na carbonate may be one of the causes of formation of the so-called "tarry matter" (qv)

K hydroxide in methanol reacts with TNT to form a dark red powder which inflames or expl when heated to 130-150° and has been reported to expl spontaneously on standing at ordinary temp (Ref 6)

It should be noted that substances formed during purification of crude TNT by the action of "sellite" (alkaline aq soln of Na_2SO_3) may also ignite (or explode) spontaneously when in a dry state, as can be seen from the occurrence during WW II at the Keystone Ordnance Plant, Meadville, Pa. When a spent "sellite" liquor from the purification of TNT(red water) was removed from a storage tank, after remaining there for several days, the residue in the tank ignited(or exploded) as soon as it was dried by the heatof the sun. This accident was followed by an investigation of the "red water" residues in other tanks at KOW in order to determine their nature. The deposit collected at that time was very dark red (nearly black) and consisted of a mixture of at least three components: a)a nearly white component soluble in water and insol in methanol(Na sulfate) b)a dark red component sol in methanol and sl sol in water and c)a greyish component insol in methanol or water. The red component was a more sensitive explosive than TNT,but not as sensitive as primary type explosives; whereas the greyish component was an explosive extremely sensitive to heat, friction and shock. Its performance was comparable to ordinary primary explosives. This explosive material was named "Keystonite." Its compn was not detnd due to the shutdown of the plant shortly after the compd was isolated

Refs: 1)G.C.Smith, "TNT and Other Nitrotoluenes," Van Nostrand, NY(1918) 2)M.Kostevitch, "Tarry Matter of Alpha Trinitrotoluene," Part II, Impr d'Art Voltaire,Paris(1927), 8 3)Sidgwick(1937), 259 et seq 4)Davis(1943), 136-7, 147, 149-51 & 170-1 5)Degering(1950), 139 et seq 6) Dept of the Army TM 9-1910(1955), 146

Alkalies, Action on Nitric Esters. Organic nitrates in general are readily saponified by alkaline solns. A simple metathetical reaction to yield the alkali nitrate and alcohol does not take place; instead, as a result of simultaneous oxidation and reduction, alkali nitrite and a variety of products are formed depending on the conditions of the reaction . The resistance of different nitric esters to alkalies varies considerably. Thus, starch nitrate is decomposed much more slowly than cellulose nitrate and amylopectin nitrate still more slowly

Jourdin & Tribot(Ref 4) investigated the

action of alkalies on military grade NC's and found that an attack takes place even by weak alkalies, such as Na carbonate, especially at elevated temps. This attack lowers the stability of the NC

Refs: 1)Naoúm, NG(1928), 122 2)W. Aschford et al, CanJ Res **25B**, 151-8(1947) & CA **41**, 4311-12(1947) 3)Ott, **5**, part 2(1954), 751 4)P. Jourdin & R. Tribot, MP **36**, 65-70 (1954) & CA **50**, 5173(1956)

Alkalies, Action on Stability of Nitrocelluloses. See under Alkalies, Action on Nitric Esters

"Alkali Liability" of Starch or "Alkali Number" of Starch. See under Starch

Alkali Metal Amides, such as potassamide (KNH_2) and sodamide ($NaNH_2$), can be obtained by the action of pure liq NH_3 on these metals. The blue solns of the metals thus formed are the result of the reaction: $M + NH_3 = MNH_2 + 1/2H_2$. Catalysts such as spongy Pt or Fe oxides greatly accelerate the reaction. These amides may serve for the prepn of the very explosive silver amides

Refs: 1) A. Joannis, CR **112**, 392(1892) 2) E. C. Franklin & O. F. Stafford, AmChemJ **28**, 83(1902) & JCS **82I**, 748-9(1902)

Alkali Metals and Halides of Metals, Mixtures. According to J. Cuielleron, BullFr **12**, 88-9(1945) & CA **40**, 4309(1946) mixts of Na or K with halides of metals (except those of the alkali or alkali-earth metals) or metalloids may be exploded by a hammer blow. The same results may be obtained by substituting oxygen-containing compds for the halides

Alkali Metal Ozonates. See under Ozonates

Alkalinity in Explosives and in Propellants. Residual alkalinity in expls and in propellants is undesirable because even traces of alkalies may lower the stability and mp of an expl with a resulting increase in exudation. Alkalies also may form products which are more sensitive than the original expl. Some discussion on this subject is given under "Alkalies, Action on Nitric Esters" and under "Alkalies, Action on Nitrocompounds"

Residual alkalinity is the result of overneutralization of residual acids of the crude expls or of NC (used in propellants) to pH's higher than 7. Neutralization is usually done by aq solns of soda ash or by ammonia but can also be effected by chalk, lime, etc

Refs: 1)M. Copisarow, Chem News **112**, 283-4(1915) & CA **10**, 527-8(1916) 2)G. C. Smith, "TNT and Other Nitrotoluenes," Van Nostrand, NY(1918) 3)M. Kostevitch, "Tarry Matter of Alpha Trinitrotoluene," Part II, Impr d'Art Voltaire, Paris (1927) 4)P. Jourdin & R. Tribot, MP **36**, 65-70 (1954) & CA **50**, 5173(1956)

Alkalinity Test is one of the std tests for detn of the purity of expls and propellants. It is conducted in the same manner as the **acidity test** (qv) except that titration is done by 0.05N aq sulfuric acid instead of std aq NaOH soln. (See also under individual explosives and propellants)

Alkalsit is a Ger blasting expl which is described in PATR 2510(1958), p Ger 3

Alkanes, Nitrated Derivatives (Nitrated Aliphatic Hydrocarbons). The first nitroalkane described in the literature was 1,2-dinitroethane, prepd in Russia by A. Semenov. Since then hundreds of nitroalkanes, some of them explosive, were obtained. The reference given below describes old and new methods of prepn of nitroalkanes. Most of expl nitroalkanes are described in this dictionary under their parent names, such as methane, ethane, propane, etc

Ref: O. von Schickh, AngewChem **62**, 547-56 (1950)(Chemie und Technologie der Nitroalkane)

Alkalites are Belgian industrial safety exps ("explosifs SGP"), which may be sheathed or not. Pepin Lehalleur (Ref 1) gives the following compn: AN 53.0, TNT 14.0, K nitrate 8.5, Al 1.5 & NaCl 23.0%; its charge limite of 900 g is equivalent to 705 g of dynamite No 1 (NG 75, kieselguhr 25); not stable in storage

Dr Deffet (Ref 2) states that current

Alkalite contains: AN 59.0, TNT 12.5 K nitrate 2.0 Al 0.5 & NaCl 26.0%. If a sheath (gaine in Fr) is used, it consists of 140 g of Na bicarbonate per 100 g of Alkalite

Refs: 1)Pepin Lehalleur (1935), 420 2) Dr L. Deffet, Bruxelles; private communication, March 10, 1954

Alkenyl Aromatics of the general formula Ar. CH_2. CH_2. C: CH_2, were prepd by reacting aromatic hydrocarbons (at moderate temp and press) with 1,3-diolefins in the presence of a catalyst(such as boron trihalide satd with an organic carboxylic acid). The purified alkenyl aromatics can be nitrated to yield expl derivs

Ref: W.N. Axe, USP 2,430,660 and 2,430,661 (1947) & CA **42**, 3778(1948)

Alkogol' (Rus). Alcohol

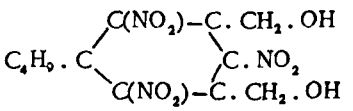
Alkohol (Ger). Alcohol

Alkyd Resins are the reaction products of polyhydric alcohols(such as glycerol, glycols, erythritol, etc) and resinifying polybasic acids(such as phthalic and maleic acids, the dimer of abietic acid, sorbic acid, tartaric acid, etc). The first alkyd resin was prepd by Berzelius in 1847 from glycerol and tartaric acid, but the first resins to become industrially important were the glycerol-phthalate resins introduced in 1901 by W. Smith and used widely at about the time of WWI. Second in importance are the maleic alkyd resins(Refs 1 & 2)

Alkyd resins are finding wide use as substitutes for metals, wood, etc in numerous ordnance items, such as some parts of rocket motors(Ref 3)

Refs: 1)Kirk & Othmer 1(1947), 517-32(48 refs) 2) Clark & Howley(1957), 42-3 3)A. J. Zaehringer & R.M. Nolan, "Missiles and Rockets," March 1958, 69

Note: H. A. Bruson, "Preparation of Polymers Which Might be of Interest in Explosives," in the NDRC Div 8 Interim Rept PT-7, Feb 15 to March 15, 1943 on "Preparation and Testing of Explosives" p 25 claimed that on treating *trinitroterbutylxylene ("musk xylene")* with formaldehyde it is possible to introduce two methylol groups to form a dihydric alcohol,



which might serve as a primary material for prepn of explosive "alkyd resins." It is not stated how these resins can be prepd

Alkyd Resins, Analytical Procedures are given in Org Analysis, Interscience, NY, **2** (1954) and **3** (1956)

α-Alkylacrylonitriles and α-Alkylacrylo-öximes. Both of these groups were prepd and examined in 1950 by Marvel et al (Refs 3 and 4) and a short resumé of their work is presented here

It should be noted that some of the nitriles, CH_2: C(R)CN(where R may by an aliphatic group), were prepd in 1929-1935 in Belgium (see Ref 1) but not by the same method as reported by Marvel et al

Marvel et al also prepd oximes of the general formula CH_2: C(R). CH: NOH by treating α-alkylacroleins with NH_2OH. HCl and an aq soln of Na_2CO_3. The oximes prepd by M et al and by some previous investigators (Refs 1 & 2) are viscous oils which can be purified by distillation. With Et- and iso-Pr-homologs the distn proceeds smoothly but with some higher homologs some decompn and polymerization occurs on heating. Addition of a little hydroquinone facilitates the distillation. In the case of the iso-Pr homolog, hydroquinone prevents a violent reaction which has been observed to take place after bleeding air into the distillation system

In no case did M et al observe the "explosive decomposition" of the metacrolein oxime or of α-ethylacrolein oxime as previously reported in the literature(Ref 2)

Note: M.R. Ross & R. Rolih in a private communication reported to M et al that an explosion took place with α-ethylacrolein oxime

Following are examples of α-alkylacrylonitriles prepd by Marvel et al (Ref 4):

a) **α-Ethylacrolein Oxime**, $CH_2:C(C_2H_5)$. CH:NOH, mw 99.13, N 14.13%, OB to CO_2 −217.9%, OB to CO −137.2%. Viscous oil, bp 78° at 30 mm, $n_D^{21°}$ 1.4820

b) **α-Isopropylacrolein Oxime**, $CH_2:C(iso\text{-}C_3H_7)$. CH:NOH, mw 113.16, N 12.39%, OB to CO_2 −233.3%, OB to CO −148.5%. Viscous oil, bp 63° at 3.5 mm, $n_D^{20°}$ 1.4744

Refs: 1) See Ref 1 in JACS **72**, 5408 2) D. T. Mowry & R. R. Morner, JACS **69**, 1831 (1947) 3) C. S. Marvel et al, JACS **70**, 1694 (1948) 4) C. S. Marvel et al, JACS **72**, 5408-9 (1950)

Alkyl-Aluminum Compounds. See Aluminum Alkyls

Alkylamides and Their Nitrated Derivatives. When an amide of a hydroxyaliphatic monocarboxylic acid of the general formula $HOCH_2.CONRH$ (where R is an alkyl radical) is treated with nitric acid, nitric esters ($O_2NO.CH_2.CO.NHR$) are usually obtained. However, in some cases NO_2 groups are also introduced yielding compds of the general formula $O_2NO.CH_2.CO.N(NO_2)R$

The following compds were patented by Filbert (Refs 1 & 2) for use as ingredients of blasting cap charges:
a) Gluconamide pentanitrate (qv) b) N-2-Hydroxyethylgluconamide hexanitrate (qv) c) N-2-Hydroxyethylglycolamide dinitrate (qv) d) N-Methylgluconamide pentanitrate (qv) e) N-Methyl-N-nitroglycolamide nitrate, described under N-Methyl-N-glycolamide

Refs: 1) W. F. Filbert, USP 2,443,903 (1948) & CA **43**, 1797-8 (1949) 2) Ibid 2,449,843 (1948) & CA **43**, 1797 (1949)

Alkylamines, Alkylarylamines, Arylamines and Their Explosive Derivatives. Alkylamines alkylarylamines and arylamines may form nitrocompds as well as various salts of which the nitrates, perchlorates and picrates may be explosive

For more information on this subject see individual explosives, eg, tri- and tetranitroaniline, ethylenediamine dinitrate, tetryl, etc

Alkylaminoguanidines. See Aminoalkylguanidines and Alkylaminoguanidines

Alkylaminotetrazoles. See Aminoalkyltetrazoles and Alkylaminotetrazoles

Alkylaminotriazoles. See Aminoalkyltriazoles and Alkylaminotriazoles

Alkyl and Aryl Azides are discussed by J. H. Boyer & F. C. Canter in ChemRevs **54**, 1-59 (1954) (See under individual compds, such as allylazide, phenylazide, etc)

Alkylaryl Ureas or Dialkyldiaryl Ureas. See Centralite

Alkylated Benzenes, Nitroderivatives of. Studies were made at Pic Arsn regarding the possibility of using some nitroderivs of alkylbenzene as gelatinizers in smokeless propellants. Although derivs of methylbenzene (toluene), such as DNT and TNT, previously examined, are effective gelatinizing and waterproofing agents when used with NC in smokeless propellants, it was assumed that gelatinization would be improved by using substances which were either liquid (oils) or at least had lower mp's than those of either DNT (ca 70°) or TNT (ca 80°). As the nitroderivs of ethylbenzene melt at much lower temps (DNEtB is liq at RT and TNEtB melts ca 37°), they were chosen for further study. For the results and additional information see under Ethylbenzene and Derivatives

Ref: P. Varrato, PicArsnTechRept R91 (1930)

Alkylated Benzidines, Nitrated Derivatives. Mertens, in 1887, obtained a compd which he considered to be tetranitrodimethylazobenzene (Ref 1). P. van Romburgh proved in 1886, that the substance was tetranitrodimethylbenzidine but he did not det the position of the NO_2 groups (Ref 2). This was done in 1922 by G. van Romburgh, who established the structure as *3,3',5,5'-tetranitrodimethylbenzidine*, $H_3C.HN.C_6H_2(NO_2)_2-C_6H_2(NO_2)_2-NH(CH_3)$ mw 392.28, N 21.43%, red crysts, dec ca 282° (Ref 3) The same investigator prepd *3,3',5,5'-tetranitrodiethylbenzidine*, mw 420.34, N 20.00%, red crysts, mp 248°; *3,3',5,5'-tetranitrodipropylbenzidine*, mw 448.39, N 18.74%, red ndls, mp 200°; *3,3',5,5'-tetranitrodiisopropylbenzidine*, mw

448.39, N 18.74%, red ndls, mp 250°; *3,3',5,5'-tetranitrodiisobutylbenzidine*, mw 476.44, N 17.64%, red crysts, mp 194°; and *3,3',5,5'-tetranitrodiallylbenzidine*, mw 446.36, N 18.91%, orange-red ndls, mp 205°

None of these compds were examined by G.van Romburgh from the point of view of ignitibility or explosibility

Refs: 1)K.H.Mertens, Thesis, Univ of Leyden(1877) & Ber **19**, 2127(1886) 2)P.van Romburgh, Rec **5**, 244(1886) 3)G.van Romburgh, Rec **41**, 38-43(1922) & CA **16**, 1238 (1922)

Alkylation is the process by which an alkyl radical is introduced by addition or substitution into a compd. Description of methods of alkylation may be found in Refs 1,5 & 6. Alkylation reactions assumed great importance during WWII for the prepn of toluene (used for the manuf of the explosive TNT and of DNT, which was used as an ingredient of smokeless propellants), for the prepn of high octane blending agents used in aviation gasoline and for the prepn of materials used in the manuf of synthetic rubbers and plastics. The alkylation reaction was also used for the prepn of cumene (which yields an explosive trinitrocompd on nitration) and of ethylbenzene, which in turn served for the production of styrene and dinitroethylbenzene. The last compd has been suggested as a component of smokeless propellants in lieu of DNT. Ethylbenzene can also be nitrated to trinitroethylbenzene, an explosive slightly less powerful than TNT but not as economical to produce(see under Ethylbenzene)

Explosives have also been prepd from alkylated compds by methods other than nitration. For instance, some explosive primary and secondary dialkyl peroxides were obtained by interaction of alkylmethane sulfonate and hydrogen peroxide(Ref 4, Sept 1957, p 1463)

Some plastics obtained from the products of alkylation can be used in the manuf of various ordnance items

A flow sheet and a brief description of a sulfuric acid alkylation process designed by M.W.Kellog is given in Ref 2

Refs: 1)Kirk & Othmer **1** (1947), 532-50 (67 refs) 2)Anon, "H_2SO_4 Alkylation," ChemEngrg **58**, 212-15(Sept 1951) 3)R. Norris Shreve et al, IEC, Sept issues 1948-1955 4)L.F.Albright, IEC, Sept issues 1956-1957 5)Clark & Hawley(1957),43-4 6)Groggins(1958),804-55

Alkylation, Regeneration of Acid Used in is described in the following papers: 1)J.A.Lee,ChemMetEngrg **53**, 146-9(July 1946)(Recovering alkylation spent acid by the Chemical Construction Corp process) (brief description and a flow sheet) 2) Chemico Bulletin S-107(1946)(Diagrammatic arrangement of the "Chemico" alkylation and regeneration process and a brief description of the process)

Alkyl Azides. Prepn and reactions of some alkyl azides are discussed by J.M.Clegg (Univ of Michigan, Ann Arbor), UnivMicrofilmsPubl No **12555** and Dissertation Abstr **15**, 1310(1955); CA **50**, 259(1956)

Alkyl Boranes. See under Boranes

Alkyldichloroamines. See Dichloroalkylamines

Alkylene. An organic radical derived from an unsaturated hydrocarbon: eg, ethylene, propylene, etc

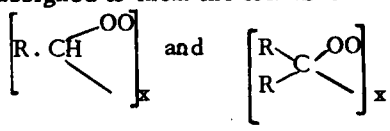
Alkyl Halides are described under individual compds, such as carbon tetrachloride, chloroform, etc

Alkyl Hydrazines are described under individual compds, if they are expls or used in Ordnance. The major physical and chemical props of a large number of alkyl-substituted hydrazines, currently of interest in the rocket propellant field, were detd by R.C.Harshman at the Olin Mathieson Chem Corp, Niagara Falls, NY and discussed in Jet Propulsion **27**, 398-9(1957)

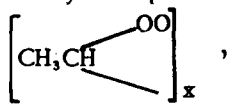
Alkylidene. A divalent organic radical derived from an unsaturated aliphatic hydrocarbon: eg, ethylidene ($H_3C.CH=$), propylidene, ($CH_3CH_2CH=$) etc

Alkylideneperoxide. The name coined by

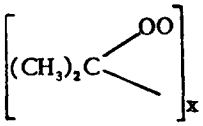
Rieche & Meister for polymeric peroxides derived from alkylaldehydes or alkyl ketones. They assigned to them the formulae:



The simplest known compd for the first group is *ethylideneperoxide*



viscous tar, extremely explosive; whereas the simplest members of the second group are *acetoneperoxides* (dimeric and trimeric)

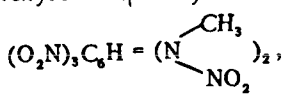


Ref: A.Rieche & R.Meister, Ber **64**, 2335-40(1931)

Alkylized Diamines of the Aromatic Series, Nitrocompounds of. Compounds of this type were patented in France (Ref 1) for use in detonators and as HE fillers for shells, mines, torpedoes and bombs. They may be used either alone or mixed with other explosives or with oxidizers, such as nitrates, chlorates or perchlorates

Among the diamines suitable for the manuf of such explosives may be mentioned phenylene-di(methylamine) and phenylene-ditoluidine

As an example of an explosive prepd from these diamines, Colver (Ref 2) cites *trinitrophenylene-di(methylnitramine)*



which he calls pentanitrodimethylmetaphenylenediamine. It is described in this dictionary as Phenylene-di(methylnitramine), Trinitro

Refs: 1)FrP 391,107(1907) 2)Colver (1918), 711-12

Alkylmercaptosilanes are thioethers of the general formula $(SR)_3SiH$. They are usually prepd by treating the appropriate Na mercaptide with trichlorosilane in benz or toluene soln at ca −70°. The reaction may be written: $3RSNa + HSiCl_3 \rightarrow 3NaCl + HSi(SR)_3$. Attempts to prep tri-tert-butylmercaptosilane, $HSi[SC_4H_9(tert)]_3$, resulted in a serious explosion in one case

Ref: L.Wolinski et al, JOC **16**, 395-8(1951) & CA **46**,423(1952)

Alkylnitramines. The effect of cold 98% HNO_3 on alkylnitramines was detd by A.T.Blomquist & F.T.Fiedorek,OSRD **4134**(1944),1 10. They found that the reactions can be represented as:

a) $R{\cdot}NH{\cdot}NO_2 \xrightarrow{HNO_3} R{\cdot}O{\cdot}NO_2$

b) $R_2{\cdot}NO_2 \xrightarrow{HNO_3}$ no reaction

c) $R{\cdot}N(R'){\cdot}NO_2 \xrightarrow{HNO_3} R{\cdot}O{\cdot}NO_2 + R'{\cdot}O{\cdot}NO_2$

Alkylnitramines, Nitroxy of the general formula $O_2N.O.R.N(R').NO_2$ were proposed as non-volatile plasticizers for triple-base propellants

Refs: J.Kincaid & R.McGill, USP 2,698,228 (1954) & CA **49**, 5846(1955)

Alkyl Nitrates as Liquid Monofuels. The desirability of having a non-expl liq monofuel for application which involves its use in proximity to human life, such as in ATO (assisted-take-off) of aircraft or the starting of airplane engines, was the reason for the Brit investigation of methyl-, ethyl-, propyl-, etc nitrates. It was found that *n-propyl nitrate* is satisfactory for these purposes

Ref: A.C.Hutchison, Report of the ICI(Imperial Chemical Industries), Ltd, Nobel Division, Stevenston, Ayrshire (1950)

N-Alkylnitroanilines were proposed as stabilizers for NC. See under Aniline, Mononitro-

N-Alkyl-N'-(2-nitroxyethyl)ethylene Dinitramines of the general formula, $R.N(NO_2).CH_2.CH_2.N(NO_2).CH_2.CH_2{:}ONO_2$, were recently patented by Blomquist & Fiedorek (Ref 3) for use in propellants as explosive, practically non-volatile plasticizers for NC. These compds can be prepd either by the method of Franchimont & Klobbie (Ref 1), which involves the treatment of

N-alkylethylenedinitramines with ethylene-dibromide, or, preferably, by the method of Wright & Chute (Ref 2). The latter method consists of converting N-alkylethylenediamines by means of ethylene oxide to the corresponding N-alkyl-N'-(2-ethanol)ethylenediamines, followed by treatment with nitric acid and then acetic anhydride in the presence of a chloride or bromide ion:

$$R.NH.CH_2.CH_2.NH.CH_2.CH_2.OH \xrightarrow{HNO_3 + Ac_2O + Cl^- (or\ Br^-)} R.N(NO_2).CH_2.CH_2.N(NO_2).CH_2.CH_2.ONO_2$$

As an example of such a compd the explosive *N-methyl-N'-(2-nitroxyethyl)ethylenedinitramine* is cited

Refs: 1) A. Franchimont & E. Klobbie, Rec **7**, 346-7 (1888) 2) C. F. Wright & W. J. Chute, USP 2,462,052 (1949) & CA **43**, 4286 (1949) 3) A. T. Blomquist & F. T. Fiedorek, USP 2,481,283 (1949) & CA **44**, 4925 (1950)

Alkylolamines, Nitrated Products. The following explosive alkylolamines were prepd and examined in 1944 at PicArsn: a)[Bis(hydroxymethyl)methylamino]methane dinitrate and b)[Tris(hydroxymethyl)amino] methane trinitrate

Both of these compds were found to be unstable even at RT and for this reason unsuitable for military purposes

Ref: H. A. Aaronson, PATR 1412 (1942)

Alkyl Peroxides, Decomposition was discussed in the following refs:

1) E. J. Harris & A. C. Egerton, Nature **141**, 472 (1938) & CA **32**, 7805 (1938) 2) E. J. Harris, Proc Roy Soc **173A**, 126-46 (1939) & CA **34**, 2323 (1940)

Alkyl Silanes or Alkyl Silicanes are compds combining alkyl groups with silicon or silicon hydrides, eg, methylsilane CH_3SiH_3, dimethylsilane $(CH_3)_2SiH_2$, trimethylsilane $(CH_3)_3SiH$, tetramethylsilane $(CH_3)_4Si$ and vinylsilane $(CH_2:CH)SiH_3$. They are very reactive compds and some of them even ignite spontaneously in air. Their mixts with air are usually expl. These compds have been known for about one hundred years, but Ref 1 seems to give the first comprehensive description of their prepn. Several methods of synthesis of alkylsilanes are reviewed in Ref 6. One of the most common methods is the Grignard reaction, which involves the interaction of a suitable alkylmagnesium halide with a halosilane in anhydrous ether:

$$3CH_3MgBr + SiHCl_3 \rightarrow Si(CH_3)_3H + 3MgBrCl$$

In Ref 7 are given the temp-compn limits of spontaneous expln for 9 alkylsilanes with air at atm press, and in Ref 8 are discussed the combustion and expln limits of several alkylsilanes at 1 atm press. The expln temp decreases in the order: tetramethylsilane > trimethylsilane > dimethylsilane > methylsilane > vinylsilane

Some alkylsilanes may be considered as suitable components of liquid rocket fuels

Refs: 1) A. Ladenburg, Ann **164**, 300-332 (1872) 2) A. Stock, "Hydrides of Boron and Silicon," Cornell Univ Press, Ithaca, NY (1933) 3) E. Krause & A. von Grosse, "Die Chemie der Metall-Organische Verbindungen," Gebr Borntträger, Berlin (1937) 4) E. G. Rochow, "The Chemistry of Silicones," Wiley, NY (1946), 32 5) H. W. Post, "Silicones and Other Organic Silicon Compounds," Reinhold, NY (1949) 6) S. Tannenbaum, S. Kaye & G. F. Lewenz, J ACS **75**, 3753-57 (1953), (Synthesis and Properties of Some Alkyl Silicanes) (17 refs) 7) R. L. Schalla & G. E. McDonald, NACA Tech Note No 3405 (1955) & CA **49**, 7248 (1955) 8) R. L. Schalla et al, "Combustion Studies of Alkylsilanes," paper reported in the 5th Symp on Combust, Reinhold, NY (1955), 705-10 (7 refs)

N-Alkyl-N'-(5-tetrazolyl)-ureas and N,N-Dialkyl-N'-(5-tetrazolyl)-ureas, such as N-ethyl-$C_4H_8N_6O$, N 53.83%, mp 223-4° with decompn; N,N-dimethyl-$C_4H_8N_6O$, N 53.83%, mp 287° with decompn and N,N-diethyl-$C_6H_{12}N_6O$, N 45.63%, mp 237°

Although these compds are not expl they contain enough nitrogen to be of some interest as potential gas-producing components for propellants or industrial expls
Ref: L.F. Audrieth & J.W. Currier, "Derivatives of 5-Aminotetrazole," Univ of Illinois Final Rept Part B on "Compounds of High Nitrogen Content," Urbana, Ill, June 15, 1954, pp 75-8 (US Ordn Corps Contract No DA-11-022-ORD-33)

Alkyltetrazylazides, such as methyl-, ethyl-, etc were patented by W. Friederich, USP 2,170,943(1939) & CA **34,** 265(1940) for use as high expls (See under individual compds)

Alkyltrimethylolmethane Triacetates or Alkyl-tris-hydroxymethylmethane Triacetates, $RC(CH_2O{\cdot}OCCH_3)_3$, are compds prepd by acetylation of alkyltrimethylolmethane (Alkyltrisoxymethylmethan in Ger), $RC(CH_2OH)_3$. These acetates were patented as gelatinizers for NC's and acetylcelluloses for use in expls and smokeless propellants. A procedure for the prepn of *methyltrimethylolmethane triacetate,* $CH_3C(CH_2O{\cdot}OCCH_3)_3$, is given in Ref 1. It is a liq, bp 200° at 80 mm
In another patent(Ref 2) it is claimed that the above compds act as stabilizers for NC, NG, etc
Refs: 1) Bombrini Parodi-Delfino, FrP 793,590(1936) & CA **30,** 4517(1936) 2) Paolo Parodi-Delfino, USP 2,096,451(1937) & CA **32,** 357(1938)

Alkyltrimethylolmethane Trinitrates or Alkyl-tris-hydroxymethylmethane Trinitrates, $RC(CH_2{\cdot}ONO_2)_3$, are compds prepd by nitrating alkyltrimethylolmethanes $RC(CH_2OH)_3$. They were patented for use as expls, either alone or in mixts with other substances
Ref: Bombrini Parodi-Delfino, FrP 771,599 (1934) & CA **29,** 929(1935)

Alkynes (Acetylene Series). A group of unsaturated aliphatic hydrocarbons of the general formula C_nH_{2n-2}, contg triple bonds. These compds were also called Alkines

ALL (Propellant) is described in conf "Propellant Manual," SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland(1959), Unit No 406

Alldorfit. Same as Aldorfit

Allégé Explosifs (lightened Explosives)(Explosifs de mine du genre dit allégé) are Fr coal mine expls of low packing density (0.6 to 0.9). For instance, the compn AN 88 and nitropolystyrene 12% has a d 0.65
Ref: L. Médard, MP **34,** 104(1952)

Allenic Compounds are derivs of **allene** or **propadiene,** also called **dimethylenemethane,** $CH_2:C:CH_2$. Allene was probably first prepd in 1865(Ref 2), but the hydrocarbon was not actually identified until 1872 (Ref 3). A rather general method for the prepn of allene by treating 1,2-dihalopropene with Zn dust in alc, was first used in 1888(Ref 4). Many other methods of prepn are known and some of them are described in Ref 5

Numerous haloallenes are known, but the perhaloallenes of the type $X_2C:C:CX_2$ were unknown until a study at the Univ of Calif, LA (Ref 5) was undertaken as part of the US Dept of the Army Contract DA-04-495-ORD-527. It is expected that compds such as *tetrafluoroallene,* $F_2C:C:CF_2$ will be of interest as monomers for both homopolymerization and copolymerization studies and for the prepn of materials similar to Teflon which is the homopolymer of tetrafluoroethylene $F_2C:CF_2$. This study is being continued as of 1958
Refs: 1) Beil **1,** 248,(107),[223] & {922} 2) W. Pfeffer & R. Fittig, Ann **135,** 357(1865) 3) G. Aarland, JPraktChem **6,** 265(1872) 4) G. Gustavson & N. Demjanoff, JPraktChem **38,** 201(1888) 5) T.L. Jacobs & R.S. Bauer, "Chemistry of Allenic Compounds," Tech Rept Univ of Calif, Los Angeles, Calif, (June 1958)(ASTIA Document No 137095)
Note: This report is based chiefly on the dissertation by R.S. Bauer, "The Synthesis and Polymerization of Polyhalogenated Allenes," Univ of Calif, LA(1958)(86 Refs)

Allison Powder. A blasting expl consisting of porous black powder and some NG absorbed in the pores
Refs: 1) Cundill, MP **5,** 281(1892) 2) Daniel (1902), 8

Allophanylazide (Allophansäureazid, in Ger),

$H_2N.CO.NH.CO.N_3$, mw 129.08 N 54.26%. Fine crysts, mp 195°(dec) (Ref 2), 193-4° (dec)(Ref 4); sl sol in alc, nearly insol in w; insol in eth, benz or ligroin. Was first prepd in 1898(Refs 1 & 2) by treating allophanylhydrazide-hydrochloride with aq KNO_2 + HCl in the cold. This procedure was improved by Audrieth et al and a detailed description of the method is given in Refs 3 & 4

Although the azide could not be purified by recrystn because of its instability in warm solns, it could be purified by subliming a small sample at 150° on a hot stage microscope (Ref 3, p 35)

According to the tests conducted at PicArsn, allophanylazide is practically nonhygroscopic (gain in wt at 90% RH and RT 0.19% in 24 hrs). The 100° thermal stability test showed that the azide is appreciably unstable on prolonged heating (loss of wt 5.98% in 24 hrs and 7.15% in 48 hrs). The impact test with a 2 kg wt showed that the azide is insensitive. Contact with a hot wire did not cause the material to inflame to a self-propagating reaction (Ref 3, p 35)

Thiele & Uhlfelder(Ref 2) obtained a white ppt of the explosive *silver salt* by treating an alcoholic soln of allophanylazide with an aq soln of Ag nitrate

Refs: 1)Beil **3**, 129 2)J.Thiele & E. Uhlfelder, Ann **303**, 105-6(1898) 3)L.F. Audrieth & P.G.Gordon, "The Chemistry of Allophanyl Hydrazide and Urazole," Final Rept March 15, 1954, Univ of Illinois, Urbana, Ill, pp 33-41(Contract DA-11-022-ORD-33) 4)L.F.Audrieth & P.G.Gordon, JOC **20**, 247(1955) & CA **50**, 4060(1956)

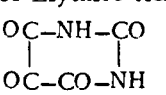
Allophanylhydrazide or Aminobiuret (Allophansäurehydrazid; Kohlensäure-ureidhydrazid or 4-Carbaminyl-semicarbazid, in Ger), $H_2N.CO.NH.CO.NH.NH_2$, mw 118.10, N 47.44%. Wh crysts mp 166° (dec); sol in w, alc & MeOH (Ref 3). Was first prepd in 1898 (Ref 2) in the form of its *hydrochloride*, $C_2H_6N_4O_2.HCl$, by reduction of nitrobiuret with Zn dust in aq HCl. At the same time the *nitrate* and *picrate* were prepd

The free base allophanylhydrazide was prepd by Audrieth et al (Refs 3 & 5) in good yield (80%) by hydrazinolysis of methyl- or ethyl- allophanate under reflux. Some esters and salts of allophanylhydrazide were also prepd (Ref 3, pp 17-27 & 42-6)

Tests at PicArsn showed that allophanylhydrazide is not sufficiently compatible with NC for use in propellants (Ref 4)

Refs: 1)Beil **3**, 100 2)J.Thiele & E.Uhlfelder, Ann **303**, 100-4(1898) 3)L.F.Audrieth & P.G.Gordon, "The Chemistry of Allophanyl Hydrazide and Urazole," Final Rept March 15, 1954, Univ of Illinois, Urbana, Ill, pp 14-59 4)J.P.Picard & W.P. Morton, Letter to Prof F.Audrieth from PicArsn, dated May 1955 (Contract DA-11-022-ORD-33) 5)L.F. Audrieth & P.G.Gordon, JOC **20**, 246(1955) & CA **50**, 4060(1956)

Alloxan or N,N-Mesoxalyl Urea (Pyrimidinetetrone or Erythric Acid of Brugnatelli,



mw 142.07, N 19.72%, mp dec ca 170°. Dark yel crysts, very sol in w and sol in alc. Can be prepd by oxidation of uric acid with HNO_3 (Ref 5) or by other methods (Ref 1)

Several investigators have reported explosions of alloxan after long storage and while attempting to open a glass-stoppered bottle containing alloxan by filing the neck of the bottle in which the stopper was frozen (Refs 2 & 3). One of the reports stated that a bottle of Kahlbaum's alloxan was found to have developed considerable pressure after storage at RT for 1 year (Ref 4)

It was remarked(Ref 6) that if it was possible to nitrate alloxan to the dinitro stage, the resulting product would be a perfectly oxygen-balanced explosive

Refs: 1)Beil **24**, 500, (428) & [301] (Includes several refs of prepn described in Ord Synth) 2)A.S.Wheeler & M.T.Bogert, JACS **32**, 809(1910) & CA **4**, 1906 (1910) 3) E.C.Franklin, JACS **32**, 1362(1910) & CA **4**, 3138(1910) 4)R.A.Gortner, JACS **33**, 85(1911) 5)Karrer(1950),802 6)L.F.

Audrieth et al, "Compounds of High Nitrogen Content," 2nd Quarterly ProgrRept, Univ of Illinois, Urbana, Ill, April 1,1951

Alloys Suitable for Use in Ordnance Plants should be acid resistant, heat resistant and nonreactive with explosives. The most useful alloys in the manuf of acids and expls are various kinds of stainless steels. Among the non-ferrous alloys may be cited Chlorimet 2 and Chlorimet 3. During WWII the Germans used some high temp alloys such as Böhler, Chromadur, Remanit, Sicromal, Thermanit, Thermat, etc

Descriptions of various alloys are given in the following refs: 1)S.L.Hoyt, "Metals and Alloys Data Book," Reinhold, NY(1943) 2)CIOS Rept File No 22-4(1946) 3)W.A. Luce, ChemEngrg **55**, 233 & 238(Feb 1948) 4)F.Johnson, "Alloy Steels, Cast Iron and Non-ferrous Metals," Chem Publ Co, Brooklyn(1949) 5)C.L.Clark, "High Temperature Alloys," Pitman, NY(1953) 6) Anon, "Data on World Wide Metals and Alloys," Engineering Alloy Digest, Inc. Upper Montclair, NJ(1953) 7)J.L.Haughton & A.Prince, "The Constitutional Diagrams of Alloys," Institute of Metals,London(1956) (Bibliography) 8)M.C.Smith, "Alloys Series in Physical Metallurgy," Harper, NY(1956) 9)PATR 2510(1958), p Ger 3

Alloys, Analysis is discussed in the following refs: 1)W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis," Van Nostrand, NY(1939), vol 2, 1348-1509 2)F.Twyman, "The Spectrochemical Analysis of Metals or Alloys, Griffin & Co, London(1941) 3)C.H.Burton, "Analytical Methods for Aluminum Alloys," Aluminum Research Institute, Chicago (1948) 4)M. Jean, "Précis d'Analyse Chimique des Aciers et des Fontes, "Dunod, Paris(1949) 5)I.M.Kolthoff & J.J.Lingane, "Polarography," Interscience, NY(1952), 582-620 6)W.F.Hillebrand & G.E.F.Lundell "Applied Inogranic Analysis," Wiley, NY(1953) 7)G.H.Osborn & W.Stross, "Analysis of Aluminum Alloys," Chapman & Hall, London (1953) 8)E.C.Pigott, "Ferrous Analysis" Wiley, NY(1953) 9)"ASTM Methods of Chemical Analysis of Metals, "Amer Socy for Testing Materials, Philadelphia, Penna (1956) 10)"Review of Analytical Chemistry" in AnalChem, beginning 1953

Allumage (Fr). Ignition or priming (of a grenade)

Allumeur (Fr). Igniter; any burning substance used to touch off a charge; primer (of a grenade)

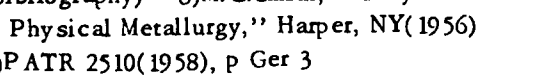
Allumeur de sûreté (F). Safety igniter or fuse

ALLYLACETONE AND DERIVATIVES

Allylacetone or 5-Hexen-2-one, $CH_2:CH\cdot CH_2\cdot CH_2\cdot CO\cdot CH_3$, mw 98.14; col, mobile liq, bp 129.5°, d 0.841 at 20/20°, vap d 3.39(air = 1.00), Q_c 856.5 kcal/mol. Was first prepd in 1877 by heating allyacetic ester with alc KOH(Ref 2). Several other methods of prepn are listed in Ref 1. Its fire hazard is moderate and toxicity is unknown (allyl compds are generally toxic). It can react with oxidizing materials

Refs: 1)Beil **1**, 734,(382) & [792] 2)F. Zeidler, Ann **187**, 35(1877)

Allylacetone Ozonide or Acetoneallyl Ozonide. If the formula of this compd,



is correct, it is an **Ozonide-peroxide,** mw 162.14, OB to CO -118.4%, syrup d 118.4%; expl violently on heating and dec on boiling with w. Was prepd by ozonization of allylacetone

Refs: 1)Beil **1**,734 2)C.Harries & K. Landheld, Ann **343**, 348-9(1905)

Allylacetone Peroxide-Ozonide. Same as previous compd

Allyl Alcohol or Vinyl Carbinol, $CH_2:CH\cdot CH_2OH$, mw 58.08. Col liq with pungent, mustard-like odor; it is irritating to the eyes; mp -50°, bp 96-7°, d 0.8540 at 20°/4°, $n_D^{20°}$ 1.41345, fl p 70°F(open cup). Misc with w, alc, chlf, eth & petr eth. Can be prepd by heating glycerin with formic acid(Refs 1& 2) or by other methods. Used for the manuf of war gas, resins and plasticizers(Ref 3)

It forms an *ozonide* which is very unstable(Ref 1).

Refs: 1)Beil **1**, 436-7, (224-5) & [474-7] 2)OrgSynth,CollVol 1(1944), 42 3)Merck (1952), 34-5 4)Sax(1957),252

Allylamine. See Aminopropene

Allylamine-Diphenylcarbamide Complex. See under Diphenylcarbamide Complexes Suitable as Stabilizers and Gelatinizers in Smokeless Propellants

Allylamine Perchlorate. See under Aminopropene

Allylamine Picrate. See under Aminopropene

5-(Allylamino)-1-amino-α-tetrazole or 5-(Allylamino)-1-amino-1H-tetrazole,

$(CH_2:CH.CH_2).HN.C(=N)-N(NH_2)-N=N$,

mw 140.15, N59.97%. Lt yel ndls, mp 94°. Was prepd from allylthiosemicarbazide, Na azide and Ph oxide as described in Ref 2

Because of its high nitrogen content, it might be of interest as a component of propellant compns

Refs: 1)Beil–not found 2)R.Stollé & E.Gaertner, JPraktChem **132**, 212 & 220 (1931); CA **26**, 1608(1932)

ALLYLAMINOTETRAZOLES

1-Allyl-5-amino-α-tetrazole or 1-Allyl-5-amino-1H-tetrazole,

$H_2N.C(=N-N)-N(CH_2.CH:CH_2)-N$,

mw 125.14, N 55.97%, OB to CO_2 −147.0%, OB to CO −95.9%. Crysts (from w or et acetate) 127 −130.5°, Q_c^v 675.8 kcal/mol and Q_f^v −64.4 kcal/mol(Ref 5). Can be prepd by diazotization of aminoguanidine and cyclization of the resulting 1-allyl-2-azidoguanidine (Ref 2) or by refluxing 5-aminotetrazole, aq NaOH and allylbromide as described in Ref 4. In the latter method, the 2-allyl-5-amino-β-tetrazole was obtained as a by-product

Because of its high nitrogen content, it might be of interest as a component of propellant compns

Note: Henry et al reported that 1-allyl-5-aminotetrazole undergoes a reaction of thermal isomerization with the formation of 5-allylamino-1H-tetrazole,

$(CH_2:CH.CH_2).HN.C(=N-NH-N)=N$,

when treated as described in Ref 3

Refs: 1)Beil–not found 2)W.C.Finnegan, R.A.Henry & E.Lieber, JOC **18**, 779 & 788 (1953) & CA **48**, 7007(1954) 3)R.A.Henry, W.G.Finnegan & E.Lieber, JACS **76**, 88-93 (1954) & CA **49**, 2427(1955) 3)R.A.Henry & W.G.Finnegan, JACS **76**, 928(1954) & CA **49**, 10940(1955) 5)M.M.Williams et al, JPhysChem **61**, 261-3 & 265(1957)

2-Allyl-5-amino-β-tetrazole or 2-Allyl-5-amino-2H-tetrazole,

$H_2N.C(=N-N=N).(CH_2.CH:CH_2)$,

mw 125.14,N 55.97%, OB to CO_2 −147.0%, OB to CO −95.9%. Crysts, mp 67-8°, Q_c^v 682.9 kcal/mol & Q_f^v −67.6 kcal/mol (Ref 3). Was obtained as by-product in the prepn of 1-allyl-5-amino-α-tetrazole from 5-aminotetrazole, aq NaOH and allylbromide, as described in Ref 2

Because of its high nitrogen content, it might be of interest as a component of propellent compns

Refs: 1)Beil–not found 2)R.A.Henry & W.G.Finnegan, JACS **76**, 926(1954) & CA **49**, 10940 (1955) 3)M.M.Williams et al, JPhysChem **61**, 226 & 265 (1957)

ALLYLANILINE AND DERIVATIVES

N-Allylaniline or Phenylallylamine $CH_2:CH.CH_2.HN.C_6H_5$ is listed in Beil **12**, 170, (162) & [96]

N-Allylaniline Azide, $C_9H_{10}N_4$ –not found in Beil or CA through 1956

N-Allylnitroaniline, $C_9H_{10}N_2O_2$, mw 178.19, N 15.72%. The following isomer is known.

N-Allyl-o-nitroaniline, red oil bp 174-5° at 12 mm. Was prepd by oxidation of N-allyl-o-aminoaniline with $FeCl_3$ and p-benzoquinone

Refs: 1)Beil–not found 2)V.C.Barry et al, JCS **1956**, 894

N-Allyldinitroaniline, $C_9H_9N_3O_4$, mw 223.19, N 18.83%. The following isomer is known:

N-Allyl-2,4-dinitroaniline, yel ndls, mp 75-6° Was prepd by treating allyamine with bromodinitrobenzene. Its expl props were not examined

Refs: 1)Beil **1**, 751 2)P.vanRomburgh, Rec **4**, 192(1885)

N-Allyl-trinitroaniline, $C_9H_8N_4O_6$, mw 268.19, N 20.89%. The following isomer is known:

N-Allyl-2,4,6-Trinitroaniline, called by P. van Romburgh **Trinitrophenylallylamine,** $CH_2:CH.CH_2.NH.C_6H_2(NO_2)_3$; solid, mp 80°; was prepd from picrylchloride and allylamine. Its expl props were not examined

Refs: 1)Beil **12**, 765 2)P.van Romburgh, Rec **4**, 192(1885)

Note: In the CA formula index for 1956, the formula $C_9H_8N_4O_6$ is assigned to *allylaminepicrate,* which is evidently in error as it is an addition salt with the empirical formula $C_9H_{10}N_4O_7$ (see under Aminopropene)

N-Allyl-tetranitroaniline, $C_9H_7N_5O_8$–not found in Beil or CA through 1956

Allylazide; 3-Triazopropene or 3-Azido-1-propene, $N_3.CH_2.CH:CH_2$ mw 83.09, N 50.57%. Mobile liq, bp 76.5°, d 0.924 at 25/25°; decomp violently on adding of concd H_2SO_4; its vapor explodes when heated. Was first prepd by heating an alc soln of allyl chloride with an aq soln of NaN_3. Fridman (Ref 3) studied its oxidation reaction, Sheinker & Syrkin (Ref 4) detd its vibrational spectra and Shott-L'vova & Syrkin (Ref 5) its dipole moments

Refs: 1)Beil **1**, 203 & [715] 2)M.O. Forster & H.E.Fierz, JCS **93**, 1177(1908) 3)S.G.Fridman, ZapInstChemUkr **4**, 351-5(1937)(p 356 in Rus & pp 356-7 in Ger) & CA **32**, 5373(1938) 4)Yu.N.Sheinker & Ya.K.Syrkin, IzvestAkadN, SerFiz, **14**, 478 (1951) & CA **45**, 3246(1951) 5)E.A.Shott-L'vova & Ya.K.Syrkin, DoklAkadN **87**, 639 (1952) & CA **47**, 6203(1953)

Allylbenzeneozonide or Phenylallylozonide, $C_3H_5C_6H_5.O_3$, liq, bp 70° at 0.5 mm, d 1.1361 at 20°, n_D 1.5132 at 20°. Can be prepd by ozonization of allylbenzene using the method of Harries (Ref 1) for ozonization of org compds. A detailed description of its prepn is given by Ryffel(Ref 3). Briner et al (Ref 4) detd the following props of allylbenzeneozonide: soly in benz, Raman spectra, UV absorption spectra, dielectric constants and dipole moments

Note: According to Dr H.Walter of PicArs this ozonide is an explosive

A *dimer* of allylbenzeneozonide was obtained from the distn residue of monomer

Refs: 1)Beil–not found 2)C.Harries et al, Ann **343**, 311-75(1906); **374**, 288-368 (1910); **390**, 235-68(1912) & CA **6**, 2754(1912) 3)K.Ryffel, Thesis, Univ de Genève (1939) 4)E.Briner et al, Helv **22**, 927-34 (1939) & CA **33**, 8068(1939)

N-Allyl-N',N'-diphenylurea (Diphenylcarbamylallylamine), $(C_6H_5)_2=N.CO.NH-CH_2-CH:CH_2$, mw 252.30, OB to CO −145.9%. Wh crysts, mp 82-83.9°, Q_C^V 2044 kcal/mol & Q_f^V 7.97 kcal/mol(Ref 3); sol in methanol, acet & alc. Can be prepd by refluxing carbamylurea with allylamine and anhydrous Na_2CO_3(Ref 2). It was suggested as a possible stabilizer or gelatinizer of NC in propellants (Refs 2 & 3)

Refs: 1)Beil–not found 2)R.Levy, MP **32**, 309 & 312(1950) 3)P.Tavernier & M. Lamouroux, MP **37**, 71 & 83(1956)

Allyl Ethers of Carbohydrates. A number of compds, such as allylglycerol, allylglycol, allylmannitol, allylsorbitol, allylpentaerythritol, etc were prepd during and after WWII by Nichols & Yanovsky. They also discussed previous work on this subject and listed several refs. According to them the first compd of this type was triallylglycerol prepd in 1856 by M.Berthelot & S. de Luca

Refs: 1)P.L.Nichols, Jr & E.Yanovsky, JACS, **66**, 1625-7(1944) 2)Ibid, **67**, 46-9 (1945)

Note: Some of the above carbohydrate ethers

were nitrated to form expl derivs, eg *allylpentaerythritol trinitrate* and are described individually

ALLYLGUANIDINE AND DERIVATIVES

N-Allylguanidine, $CH_2:CH.CH_2.NH.C(:NH):NH_2$ has been described in Beil **4,**210 & [664] in the form of salts, among them the *picrate*

N-Allylguanidine Azide, $C_4H_8N_6$—was not found in Beil or CA through 1956

N-Allyl-N' -nitroguanidine or 1-Allyl-3-3-nitroguanidine, $CH_2:CH.CH_2.NH.C(:NH):NH.NO_2$, mw 144.14, N 38.87%; crysts, mp 107-8°. Can be prepd by the interaction allylamine and N-methyl-N'-nitroguanidine by the method B of Ref 3. This high nitrogen compd which may be suitable as a component of propellants was also prepd at the US Naval Powder Factory and described in conf rept (Ref 2)

Refs: 1)Beil—not found 2)US Naval Powder Factory, Indian Head, Md, Quarterly Rept No 2, 1 March to 31 May 1948 3)A.F. McKay, JACS **71,** 1968-9(1949) & CA **43,** 9035(1949)

Allylnitrate, $CH_2:CH.CH_2.ONO_2$, mw 103.08, N 13.59%; col limpid liq with suffocating odor, bp 106-106.8°, d 1.07 at 20/4°, n_D 1.417 at 20°; insol in w. Was prepd by Henry by treating allylbromide with Ag nitrate in alc (Ref 2) and by Desseigne by treating allylalcohol with mixt HNO_3/Ac_2O (Ref 3). It is an explosive

Refs: 1)Beil **1,** 438 2)L.Henry, Ber **5,** 452(1872) 3)G.Desseigne, BullFr **1946,** 98-9 & CA **41,** 92(1947)

Note: Not listed in Beil 3rd Suppl, vol 1, part 2(1958)

Allylnitrite, $CH_2:CH.CH_2.ONO$, mw 87.08, N 16.09%. Liq which does not freeze at −20°; bp 43.5-44.5°, d 0.9546 at 0°; insol in w; its vapor expl at 100°. Was prepd by treating allylalcohol with glycerinenitrate as described in Ref 2. Tarte detd its infrared & UV spectra (Ref 3)

Refs: 1)Beil **1,** 438 2)G.Bertoni, Gazz **15,** 364(1885) & JCS **50** I, 218(1886) 3)P. Tarte, BullBelg, **60,**240(1951) & CA **46,** 826 (1952); JChemPhys **20,** 1570(1952) & CA **47,** 7322(1952)

Note: Not listed in Beil, 3rd Suppl, vol 1, part 2(1958)

Allyl, Nitro-; γ-Nitropropylene or 3-Nitropropene, $O_2N.CH_2.CH:CH_2$, mw 87.08, N 16.09%. Col liq, bp 125-30°, d 1.051 at 21°; sometimes explodes on distn; insol in w, sol in alc & eth. Can be prepd by treating allylbromide with Ag nitrate in ether

Some of its salts are expl, eg the *sodium salt,* $Na.C_3H_4.NO_2$; ndls, easily sol in w; expl when heated above 200°

Refs: 1)Beil **1,**203 2)P.Askenasy & V. Meyer, Ber **25,**1701-3(1892)

Allylnitrolic Acid,

$$CH_2:CH.C\begin{matrix}\diagup NO_2\\ \diagdown\!\!= NOH\end{matrix}$$

, mw 116.08, N 24.14%, OB to CO_2 −68.9, OB to CO −27.6%. Crysts, mp ca 68° (in capillary tube); expl violently ca 95°; sol in w and eth. Can be prepd by the action of nitrous acid ($NaNO_2 + H_2SO_4$) on sodium γ- nitropropylene in aq soln

Refs: 1)Beil **2,** 400 2)P.Askenasy & V. Meyer, Ber **25,**1703-4(1892)

2-Allyloxymethyl-2-hydroxymethyl-1,3-propanediol Trinitrate; Monoallylpentaerythritol ether Trinitrate or Pentaerythritolmonoallylether Trinitrate,

$$O_2NO.H_2C-\underset{\displaystyle CH_2.ONO_2}{\overset{\displaystyle CH_2.ONO_2}{\overset{|}{\underset{|}{C}}}}-CH_2-O\cdot CH_2\cdot CH:CH_2,$$

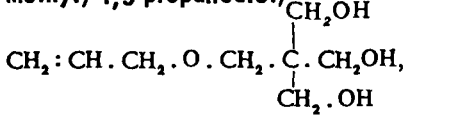
mw 311.22, N 13.50%, OB to CO_2 −64.3%, OB to CO −23.1%. Liq, d 1.373 at 20°/20°, $n_D^{20°}$ 1.4797. Can be prepd by nitrating monoallylpentaerythritol ether. It might be suitable as a gelatinizer for NC (see also Diallylpentaerythritol Ether Dinitrate)

Refs: 1)Beil—not found 2)R.Evans & J.A.Gallaghan, JACS **75,** 1248-49(1953) & CA **49,** 3811(1955)

ALLYLPENTAERYTHRITOL AND DERIVATIVES

Allylpentaerythritol; Monoallylpentaerythritol

Ether or 2-[(Allyloxy)methyl]-2-(hydroxymethyl)-1,3-propanediol,



col liq, bp 148-50° at 1 mm, d 1.135 at 20°/20°, n_D 1.4843 at 20°. Was first prepd in an impure state by Nichols & Yanovsky from PE and allylbromide, as described in Ref 2. Evans & Gallaghan (Ref 3) prepd it in 35% yield by treating a suspension of PE in dioxane with an aq KOH soln followed by the addition of allylchloride

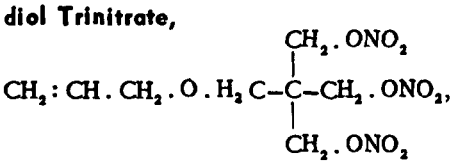
Refs: 1)Beil–not found 2)P.L.Nichols, Jr & E.Yanovsky, JACS **67**, 47-8(1945) 3) R.Evans & J.A.Gallaghan, JACS **75**, 1248-9 (1953) & CA **49**, 3811(1955)

Allylpentaerythritol Azide, $C_8H_{15}N_3O_4$–not found in Beil or CA through 1956

Allylpentaerythritol Mononitrate, $C_8H_{15}NO_6$–not found in Beil or CA through 1956

Allylpentaerythritol Dinitrate, $C_8H_{14}N_2O_8$–not found in Beil or CA through 1956

Allylpentaerythritol Trinitrate or 2-[(Allyloxy)methyl]-2-(hydroxymethyl)-1,3-propanediol Trinitrate,



mw 311.21, N 13.50%. Col liq, d 1.373 at 20/20°, n_D 1.4797 at 20°. Was prepd by nitrating allylpentaerythritol (Ref 4)

The props of this liq explosive were investigated at the US Naval Powder Factory and are discussed in confidential reports (Refs 2 & 3)

Refs: 1)Beil–not found 2)US Naval Powder Factory, Indian Head, Maryland, "Propellant Research and Development Problems," Monthly Progress Rept No 14 (15 Oct, 1947), p 10 (Conf) 3)Ibid, Quarterly Report No 1, Dec 1, 1947 to Feb 29, 1948 (Conf) 4)R.Evans & J.A.Gallaghan, JACS **75**, 1249(1953) & CA **49**, 3811(1955)

Allylpentaerythritol Trinitrate, Polymer is discussed in US Naval Powder Factory conf reports listed as Refs 2 & 3 under Allylpentaerythritol Trinitrate

p-Allylphenylmethylether. Same as Anethole

Allylphthalate. Same as Diallylphthalate

Allylpicrate, Picryllallylether or Allyl-2,4,6-trinitrophenylether, $(O_2N)_3C_6H_2-O-CH_2.CH:CH_2$, mw 269.17, N 15.61%, OB to CO_2 -86.2%. Col crysts, mp 85-90°, Q_c^v 1019.7 kcal/mol. Can be obtained either by the interaction of picrylchloride, allylalcohol & KOH(Ref 2 & 4) or from allyliodide & Amm picrate (Ref 3)

Allylpicrate is an expl about 85% as powerful as TNT as judged by the Trauzl lead block test (Ref 4)

Refs; 1)Beil 6, [281] 2)A,Fairbourne & G.E.Foster, JCS **1926**, 3148 & CA **21**, 1096 (1927) 3)L.C.Raiford & D.M.Birosel, JACS **51**, 1778, footnote 15(1929) 4)A.H.Blatt & A.W.Rytina, JACS **72**, 3274(1950) & CA **44**, 10673(1950)

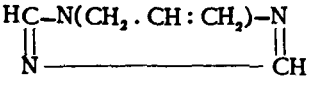
Allyltetrazole, $C_4H_6N_4$–not found in Beil or CA through 1956

Allyltetrazole, Azide, $C_4H_5N_7$–not found in Beil or CA through 1956

Allyltetrazole, Nitrated Derivatives–not found in Beil, or CA through 1956

ALLYLTRIAZOLE AND DERIVATIVES

1-Allyl-sym-triazole or 1-Allyl-1H-1,2,4-triazole



mw 109.13, N 35.51%. Col liq, bp 198°, d 1.056 at 18°; easily sol in w, alc & benz. Was prepd by heating 1,2,4-triazole with Na ethylate soln and allylbromide at 100°

Refs: 1)Beil **26**, 14 2)G.Pellizzari & A.Soldi, Gazz **25** I, 381(1905) & JCS **88** I, 673(1905)

1-Allyl-azidotriazole, $C_5H_6N_6$–not found in Beil or CA through 1956

1-Allyl-nitrotriazole, $C_5H_6N_4O_2$–not found

in Beil or CA through 1956

1-Allyl-dinitrotriazole, $C_5H_5N_5O_4$ – not found in Beil or CA through 1956, but described in confidential Naugatuck Chemical Co Progress Rept of June 15-Aug 15, 1949, p 3, NORD 10121

Allyl Type Alcohols, Polymerized. See under Polymerized Alcohols

Allylene (Propyne or Propine), $CH_3 . C \vdots CH$, mw 40.06. Gas, mp –102.7°, bp –23.2°. Sl sol in w, sol in alc & eth. Its toxicity and fire & expln hazards are discussed in Ref 3

It was reported that an expln occurred when an attempt was made to carry out the reaction $HC \vdots C . CH_2Cl + 2NH_3 = NH_4Cl + HC \vdots C . CH_2NH_2$ in a 1-liter steel vessel with an initial NH_3 pressure of 8 atm. The expln was ascribed to a slow induction period followed by rapid acceleration of the reaction rate (Ref 2)

Refs: 1)Beil **1**, 246,(106), [222] & {919} 2)E.Banik, CA **50**, 14229(1956) 3)Sax (1957), 255

Almatrites (Almatrity in Rus). Commercial expls developed in Russia in 1925. They contain chlorates and perchlorates together with combustible org materials and are claimed to be as stable, but less sensitive than Cheddites. Following are compns and some props of Almatrites: a)*Kaliialmatrit No 55:* K chlorate 88 and combustible (consisting of vaseline 5, paraffin 30 and rosin 65) 12%;d 1.15 and brisance 10.2 mm (compression of lead cylinder) vs 18.0 mm for TNT b)*Natriialmatrit No 19:* Na chlorate 90 combustible (vaseline 5, paraffin 92.5 & rosin 2.5) 10%; d 1.40 and brisance 14.0 mm c)*Ammonalmatrit No 98:* Amm chlorate 89, combustible (vaseline 8, paraffin 27 and rosin 65) 11%; d 1.17 & brisance 16.2 mm

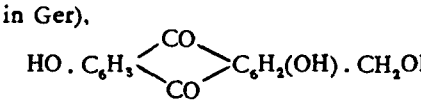
Ref: E.Spital'skii & E.Krause, SS **20**, 120-1 & 134-5(1925) & CA **20**, 1141(1926)

Almidón tetranitrico (Sp). Starch Tetranitrate or Nitrostarch (see under Starch)

Aloe, Nitrated. In 1876 Trench, Faure & Mackie in England, patented explosives contg nitrated aloe (or other nitrated cellulosic material), collodion, charcoal, rosin, ozocerite, etc

Ref: Daniel (1902), 773(under Trench, Faure et Mackie)

Aloeemodine or 4,5,2' - Trihydroxy-2-methyl-anthraquinone (4.5.2' -Trioxy-2-methyl-anthrachinon; Isoemodin; 3-Oxymethylchrysasin or Rhabarberon, in Ger),



orange-red ndls, mp 224-225.5°, is described in Beil **8**, 524,(745) & [565]

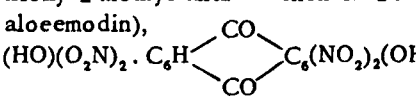
Azidoaloeemodine, $C_{15}H_9N_3O_5$ –not found in Beil or CA through 1956

Nitroaloeemodine, $C_{15}H_9NO_7$–not found in Beil or CA through 1956

Dinitroaloeemodine, $C_{15}H_8N_2O_9$–not found in Beil or CA through 1956

Trinitroaloeemodine, $C_{15}H_7N_3O_{11}$–not found in Beil or CA through 1956

Tetranitroaloeemodine or 1,3,6,8-Tetranitro-4,5,2' -trihydroxy-2-methyl-anthraquinone (Called in Beil 1.3.6.8-Tetranitro-4.5.2' -trioxy-2-methyl-anthrachinon or Tetranitro-aloeemodin),



mw 450.23, N 12.45%. When anhydrous golden-yel ndls (from AcOH); mp- begins to soften ca 285° and then puffs off; its monohydrate, orange-red crysts (from alc), which softens ca 130° and puffs off at higher temp; sl sol in w, sol in alc. Was first prepd in 1841 (Ref 2) under the name of "Aloetinsäure," or "Aloeresinsäure" as one of the nitration products of some aloe derivs. Its composition was established in 1848 (Ref 3). There are other methods of prepn, but the simplest method seems to be nitration of aloeemodine with nitric acid (d 1.5) with cooling as described in Ref 5

Several metallic salts are known of which the Ba salt deflagrates and the Ag salt expl on heating (Ref 4)

Refs: 1)Beil **8**, 525 & (745) 2)E.Schunck, Ann **39**, 4 & 24 (1841) 3)E.Schunck, Ann **65**, 235(1848) 4)C.Finckh, Ann **134**, 236-40

(1865) 5)E.Léger, CR **151**, 1129(1910); CA **5**, 1400(1911) & BullFr [4] **9**, 90(1911)

Tetranitroaloeemodine Nitrate, $C_{15}H_5N_5O_{15}$ – not found in Beil or CA through 1956

Alox 600. A polar wetting agent, one of a series of complex methyl esters of high mol wt alcohols, acids and lactones, manufd by the Alox Corp, Niagara Falls, NY. It is suitable as an additive (1 to 10%) to petroleum waxes (eg *"Aristowax 160-165"* of the Union Oil Co of Calif) to serve as a desensitizer for RDX or other expls, thus replacing previously used beeswax. Such a modified petroleum wax will wet RDX in water in the same way as does beeswax and seems to have about the same desensitizing action as beeswax. A series of such mixts were developed in the USA during WW II, as for instance, *Bruceton Wax No 10*, which consisted of "Aristowax 160–165" 90 and "Alox 600" 10%

Refs: 1)C.Young & K.Coons, "Surface Active Agents," Chem Pub Co, NY(1945) 2)Anon, Summary Technical Report of Division 8, NDRC, "The Preparation and Testing of Explosives," vol 1, Washington, DC (1946), 30

Aloxite. A trade name for an abrasive prepd by fusing alumina (bauxite) in an electric furnace

Note: This abrasive might be of use in primary compns in lieu of glass, etc

Refs: 1)Hackh(1944), 37 2)Webster's New International Dictionary, Merriam Co, Springfield, Mass (1951), 73

Alperox C. Trade name for tech lauroyl peroxide manufd by the Lucidol Division of Wallace & Tiernan, Inc, Buffalo, NY

Alpha-Cellulose is that portion of cellulosic material(pulp, paper, etc) which,after treatment with 17.5% NaOH(mercerized strength) at 20° and diln to 7.3% NaOH,can be separated by filtration. The residue of alpha-cellulose is a good index of the undegraded cellulose content of the material. The alkali treatment removes degraded(oxidized or hydrolyzed) cellulose and short chain material. Some pentosans and hexosans may be included with the alpha-cellulose (See also Beta- and Gamma-Cellulose)

Refs: 1)US Spec JAN-C-216,Dec 7,1945, "Cellulose Woodpulp(Sulfite)" (For use in explosives) 2)Ott, **5**, part 1(1954),12 3)ASTM Standards, Part 7, Test D 588-42, Philadelphia(1955), 843-8(Same as TAPPI Standard Method T 429-48)

Alpha-Compounds, such as α-Mononitronaphthalene, α-Trinitrotoluene, etc are listed under the corresponding parent compds, such as Naphthalene, Toluene etc

Alpha Particles as Initiators of Detonation. According to some investigators α-particles emitted by radium or other sources can, by irradiation, initiate the detonation of very sensitive expls, such as nitrogen iodide, but not of expls such as acetylides or azides. Nitrogen iodide can also be irradiated by fission products (See also Initiation of Explosives by Irradiation)

Refs: 1)G.H.Henderson, Nature **109**, 749 (1922) 2)H.H.Pode, Nature **110**, 148(1922) 3)A.D.Yoffe, Nature **180**, 74(1957) 4)R. C.Ling, PATR 2393(1957) 5)F.P.Bowden, PrRS **246**, 216(1958)

Alphosone. A brand of succinic peroxide (qv), manufd by the Lucidol Division of Wallace & Tiernan, Inc, Buffalo, NY

Alsilite, One of the current Belg high expls ("explosifs brisants") : AN 80, TNT 18 & Al 2%

Ref: Dr L. Deffet, Bruxelles; private communication, March 10, 1954

ALT. A solid propellant for rockets: K perchlorate 76.5 & asphalt base fuel 23.5%. Its props are described in conf rept (Ref 2)

Refs: 1)Armament Engrg(1954), 42 2) Propellant Manual, SPIA/M2, Johns Hopkins Univ, Silver Spring, Maryland (1959), Unit No 297(Conf)

Alto esplosivo (Ital). High Explosive (HE)

Alum. See Alums

ALUMATOL. A Brit expl of the ammonal type. It is practically 80/20 amatol in which part of the AN is replaced by Al powder: AN 77, TNT 20, Al 3%. During WWI alumatol was

used on a considerable scale for filling grenades and trench mortar bombs and as a blasting expl. It was found not very suitable for loading shells. When compressed to d higher than 1.2, there is danger of incomplete deton, while at lower d there is danger from set-back, and full power does not develop (Refs 1 & 2). Some alumatols contained charcoal, as for instance the French composn: AN 65, charcoal 10, TNT 15 & Al 10% (Ref 1)

E.Cheylan studied the behavior of Al in the persence of AN with or without TNT and found the ternary mixtures, Al-AN-TNT, stable when stored for 100 days at temperatures up to 90°. Mixts contg AN contaminated by chlorides proved to be unstable. Cheylan assumes that the instability of some Al contg expls in storage is due to the use of AN containing chlorides (Ref 3).
(See also Aluminum Containing Explosives)
Refs: 1)Marshall **3**(1939), 117 2)All & EnExpls(1946), 58 3)E.Cheylan, MP **30**, 139-41(1948) & CA **45**, 8250(1951)

Alumina. See Aluminum Oxide under Oxides

Aluminized Explosives. See Aluminum Containing Explosives

ALUMINUM (Aluminium in Fr & Ger; Alluminio in Ital; Aluminio in Span; Alumínio in Port; Aluminii in Rus; Aruminiūmu in Japan), Al, at wt 26.98%. Lt silvery metal, very maleable & ductile, mp 660°, bp 2056-7°, d 2.6978 at 25/4°, Q_c (to Al_2O_3) 399.0 kcal, sp heat at 100° 0.2226 cal/g. It does not occur in the free state but its compds, such as feldspar ($KAlSi_3O_8$) mica ($KAlSiO_4$), kaolin clay [$H_2Al_2(SiO_4)_2 . H_2O$], bauxite ($Al_2O_3 . 2H_2O$) are widely distributed as minerals. Al is considered the most abundant metal—it makes up 7.85% of known terrestial matter

Al is nearly insol in w, nitric acid, acetic acid & ammonia; sol in hydrochloric & sulfuric acids and in alkalies

Was first prepd in 1824-5 by H.Oerstead by heating Al chloride with K amalgam and and a few years later, by F. Wöhler who used metallic K as a reducing agent. The first industrial method of prepn was developed in 1854 by H.Sainte-Claire Deville, who used Al chloride and metallic Na, but Al thus produced was impure and very expensive. The first large-scale industrial method was developed in 1886 by Hall in USA (Ref 1) and simultaneously by Héroult in France(Ref 2). This process, which involves electrolytic reduction of Al_2O_3 dissolved in molten cryolite ($3NaF . AlF_3$), has been improved in later years and is known now as the Hall-Héroult method. Detailed descriptions of this and of some other current methods of prepn are given Refs 5, 11,12,15, & 20. As the source of Al_2O_3, the abundant mineral bauxite, $Al_2O_3 . 2H_2O$, is usually used

Al is generally considered non-toxic, except when it is inhaled in the form of dust (Ref 23)(See under Aluminum Dust)

Al possesses a great affinity for oxygen and when finely divided (powdered, flaked, etc) it burns in the air. It burns also when made in the form of a thin ribbon similar to that of Mg. When Al powder is mixed and heated with an oxide of a metal below it in the electromotive series, displacement takes place, for instance in Thermite (qv):

$$Fe_2O_3 + 2Al \rightarrow Al_2O_3 + 2Fe$$

Temps of 3000-3500° are obtained as a result of this reaction

Al has a high heat of combustion, as shown by the equation:
$2Al+1.5O_2 \rightarrow Al_2O_3+393.3$ kcal/mol or 7291 cal/g (Ref 11)

Aluminum is very reactive and some of its reactions proceed with expl violence. Bauer (Ref 7) discussed the danger of expln when Al or its alloys are melted in cast iron crucibles. Kohlmeyer (Ref 8) described an extremely violent expln which destroyed app and injured the observer. The expln occurred when Al powder and Na_2SO_4 in the mol ratio 8:3 were melted at ca 800°. Clogston (Ref 9) discussed fire and expln hazards on heating Al or Mg powders with chlorinated hydrocarbons, such as CCl_4, CH_2Cl_2, $CHCl_3$, etc. Lindeijer (Ref 16) reported a fatal expln on heating a

a mixt of powdered Al with CCl_4. Van Hintel(Ref 18) reported a spontaneous ignition caused by reaction betn Al and trichloroethylene. Charmandarian (Ref 19) reported that the action of powdered Al, Mg, etc on molten AN, hydrazonium nitrate and Na sulfate is very violent and often explosive. Stettbacher (Refs 13 & 17 and many other investigators studied the influence of the addn of Al to expls and this subject is discussed under "Aluminum Containing Explosives"

An important reaction of metallic Al is its behavior with O or water. Under many conditions the reaction is self-stopping because of the formation of an impervious film of Al_2O_3

Explosion of Al dust is discussed under Aluminum Dust and Its Explosion

Uses of Al in industry are too numerous to be described here. They are listed in Refs 5, 11, 12, 15 & 20. The uses of Al powder in expls are discussed under Aluminum Containing Explosives. Al powder is also used in incendiaries and in pyrotechnic compositions. Discussion on uses of finely divided Al and some other metals is given in Ref 16a

Refs: 1)Ch. M. Hall, USP 400,766(1886) 2)P.L.T.Héroult, FrP 175,711(1886) 3) Mellor **5**(1924), 160-73 4)Gmelin, Syst Nr 35,Teil A, Lfg 4(1936) 5)Thorpe **1**(1937), 264-80 6)D.B.Hobbs, "Aluminum," Bruce Pub Co, Milwaukee, Wisc(1938) 7)Th. Bauer, Arbeitschutz No 10, III, 399(1941) & CA **37**, 4664(1943) 8)E.J.Kohlmeyer, Aluminum **24**, 361-2(1942) & CA **37**,5592(1943) 9)C.Clogston, Underwriters Laboratory Bull Research No 34, 5-15(1945) & CA **40**, 209-10 (1946) 10)"Alcoa Aluminum and Its Alloys," Aluminum Co of America, Pittsburgh, Pa (1946) 11)Kirk & Othmer **1**(1947), 591-605 12)Giua, Dizionario **1**(1948), 437-45 13) Stettbacher(1948), 89-90 14)A.vonZeerlender, "Technology of Light Metals," Elsevier, Amsterdam(1949), translated from 1st Swiss edn by A.J.Field 15)Riegel, IndChem(1949), 346-52 16)E.W.Lindeijer,ChemWbl **46**, 571(1950) & CA **46**, 7769(1952) 16a)D.Hart & W.R. Tomlinson, Jr, Metal Progress **59**, 788-92 (1951) & CA **45**, 6844(1951) 17)Stettbacher, Pólvoras(1952), 114-17 18)J.van Hinte, Veiligheid(Amsterdam) **28**, 121-3 (1952) & CA **47**, 10152(1953) 19)M.O. Charmandarian, BullFr **1952**, 975-6 & CA **47**,3740(1953) 20)Ullmann(1953), 331-407 21)Anon, C&EN **32**, 258(1954) & CA **48**, 4838(1954)(Al & Mg dusts react violently with chlorinated hydrocarbons, with Na_2O_2 and $AlCl_3$) 22)Anon, C & EN **32**, 1824(1954) & CA **48**, 9064(1954)(A mixt of Al dust, $AlCl_3$, 2-methylpropane and chloromethane heated in an autoclave, reacted normally at first and then exploded violently) 23)Sax, (1957), 259-60

Aluminum (Analytical Procedures). The Al ion is usually detected in qualitative analysis by the precipitation of $Al(OH)_3$ which results from heating the aq soln with NH_4OH and NH_4Cl. The ppt dissolves on adding a sufficient excess of NaOH. In a quantitative method, the ppt of $Al(OH)_3$ is filtered off, ignited and weighed as Al_2O_3. Alizarin-S,"aluminon"(Amm aurintricarboxylate) and some other reagents give distinctive ppts with the Al ion. Al can also be detd colorimetrically, spectrographically and polarographically. Detailed descriptions of analytical procedures are given in Refs 5-8 & 10-15. An optical method for the study of powdered Al grains intended for use in AN expls is described in Ref 9

Aluminum, in flaked, grained or atomized form intended for use in US ammo should conform to the requirements of specifications listed as Refs 1,2 & 3. The tests and detn include:

a)Optical examination of particles of Al with a 20-30 power microscope to det their form b)Granulation(fineness), usingUS Std sieves conforming to the requirements of Federal Specification RR-S-366 c)Apparent density, using a tared 100 ml graduate d)Material volatile at 105° e)Oil and grease–by extraction with ether f)Silicon g)Zinc h)Iron i)Copper j)Magnesium k)Free

metallic Al l)Alkalinity as $Mg(OH)_2$ m)Grit n)Other metallic impurities. An app and a method for the detn of Al by gas evoln is described in Ref 2

When Al is alloyed with Mg(as for use in some incendiary, tracer of photoflash compns), the tests and detns for such alloys include: a)Granulation, using US Std sieves b) Moisture, by drying in a vacuum desiccator over H_2SO_4 for 24 hrs c)Grease and fats, by extraction with ether d)Aluminum, using 8-hydroxyquinoline reagent e)Magnesium f)Total Al and Mg g)Oxides as Al_2O_3 h)Silicon i)Iron j)Zinc k)Other metals l)Grit

In Ref 2 are described the US requirements and tests for Al powder, superfine

Refs: 1)US Specification JAN-A-289, "Aluminum Powder Flaked, Grained and Atomized" (for use in ammunition) 2)US Spec JAN-A-512, "Aluminum Powdered" (Grained or Atomized)(from secondary metal) (for use in pyrotechnics or in incendiary "thermite") 3)US Spec JAN-A-667, "Aluminum Powder, Superfine" 4)US Spec JAN-M-454, "Magnesium-Aluminum Alloy, Powdered" 5)W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis, "Van Nostrand, NY(1939) 6)H.V.Churchill & R.W.Bridges, "Chemical Analysis of Aluminum," Aluminum Co of America, Pittsburgh, Pa(1941) 7)"Routine Spectrographic Analysis of Aluminum and Magnesium and Their Alloys," Aluminum Co of America, Pittsburgh, Pa(1944) 8)Kirk & Othmer 1 (1947), 595 8a)American Research Institute, "Analytical Methods for Aluminum Testing," Chicago(1948) 9)P.Sartorius, MP **32,** 145-52(1950) 10)I.M.Kolthoff & J.J. Lingane, "Polarography," Interscience, NY(1952) 11)R.S.Young, "Industrial Inorganic Analysis," Wiley, NY(1953), 1-10 12)W.F.Hillebrand et al, "Applied Inorganic Analysis," Wiley, NY(1953), 494-515 13)"ASTM Methods of Chemical Analysis of Metals," Philadelphia, Pa (1956) 14)E.B.Sandell, "Colorimetric Determination of Traces of Metals," Interscience, NY(1959), 219-53 15)F.D. Snell et al, "Colorimetric Methods of Analysis," vol IIA, Van Nostrand(1959), 156-87

Aluminum Alkyls were prepd in 1865 by the action of aluminum on mercury alkyls(Refs 1 & 6)(see also Note below). Later they were made by the action of "electron metal" (alloy of Al and Mg) on a soln of the alkyl halide in ether (Refs 2 & 6). The Al trialkyls are volatile liquids, violently attacked by air or water. Following are examples: *trimethylaluminum* $Al(CH_3)_3$, d 0.752 at 20°/4°, mp 15.0°, bp 126.1°; *triethyl-*, $Al(C_2H_5)_3$, d 0.837 at 20°/4°, mp −52.5°, bp 185.6°; *tri-n-propyl-*, $Al(n\text{-}C_3H_7)_3$. d 0.823 at 20°/4°, mp −107°, bp ca 250° (Refs 3,4,5 & 6). These three compds are inflammable in air and for this reason may be of interest as components of liquid propellants for rockets

Note: The prepn of a compd called "Aethylaluminium" was claimed by W.Hallwachs & A.Schaffarik, Ann **109,** 207(1859) but it was not properly identified and its props (except that it is violently decomp by water) were not detd

Refs: 1)G.B.Buckton & W.Odling, AnnSupl **4,** 109(1865) 2)E.Krause & B.Wendt, Ber **56,**466(1923) 3)A.V.Grosse & J.M.Mavity, JOC 106(1940) 4)A.W.Laubengayer & W.F. Gilliam, JACS **63,** 477(1941) 5)K.C.Pitzer & H.S.Gutowski, JACS **68,** 2204(1946) 6) Sidgwick, ChemElems(1950), 414-15

Aluminum Alkyl Halides of the general formulae $AlRX_2$ and AlR_2X were prepd by Grignard and other investigators by treating Al with alkyl halides: $2\,Al + 3RX = AlRX_2 + AlR_2X$. Most of these compds are liquids or low melting solids which easily catch fire in air and react violently with water. As examples of these compds may be cited *aluminum ethyl diiodide,* $Al(C_2H_5)I_2$ solid, mp 35-7°, bp 158-60° at 4 mm and *aluminum diethyl iodide,* $Al(C_2H_5)_2I$, liq, bp 118-20° at 4 mm

For additional info on prepn and props see:

Refs: 1)W.Hallwachs & A.Schafarik, Ann **109,** 207(1859) 2)V.Grignard & R.L.Jenkins, BullFr [4] **37,** 1376(1925) 3)A.V.Grosse & J.M.Mavity, JOC **5,** 106-121(1940)(12 refs)

4)Sidgwick, ChemElems 1(1950),417-18

Aluminum Alkyl Hydrides. As an example of such compds may be cited *aluminum tetramethyl hydride*, $Al_2H_2(CH_3)_4$, a viscous liq, volat in vacuo and burning explosively with a purple flame in air. It is hydrolyzed by w and is decompd above 160° yielding $Al(CH_3)_3$. Was prepd by the action of an electric discharge on a mixt of $Al(CH_3)_3$ and hydrogen

Refs: 1)W.E.Wiberg & O.Stecher, AngChem **52**,372(1939) 2)Sidgwick,ChemElems **1**,(1950),415-16

Aluminum Azide. See under Azides, Inorganic

Aluminum Block (of Kreulen) is a hollow Al block used for detg the tendency of coal and other materials to spontaneous combustion

Ref: D.Kreulen,Brennstoff-Chem **11**, 261-2 (1930) & CA **25**,394(1931)

Aluminum Block Expansion Test is similar to the Lead Block Expansion Test,but superior to it when testing brisant expls, especially those contg Al. A brief description of this test is given under Trauzl Tests

Aluminum Boride. See under Borides

Aluminum Borohydride. See under Borohydrides

Aluminum Carbide. See Aluminum Acetylide and Aluminum Carbide under Acetylides

Aluminum Chlorate. See under Chlorates

Aluminum Chloride. See under Chlorides

Aluminum Chloride-Nitromethane Complex. See under Chlorides

Aluminum Containing Alloys. Al forms various alloys with other metals. Some of the alloys melt at temps below the mp of either metal(eg, Al-Cu, Al-Ag), others above that of either metal and some at intermediate temps(eg, Al-Sn, Al-Fe, Al-Zn)(Ref 6, p 281). Some alloys contain large percentages of Al while others contain only small percentages. Several Al-contg alloys are listed by Perry(Ref 6, pp 1527-1531). The more common alloys, of which Al is the largest constituent, are tabulated by Thorpe(Ref 2) who also lists 20 books and pamphlets relating to Al and its alloys. The metallurgy of Al alloys is discussed in Ref 5 and the phase diagrams of the most important Al systems are given in Refs 1 & 4. Al and its alloys find wide application in the fabrication industry in castings and the making of wrought products. Alloys for casting generally contain larger amts of added elements than those for wrought products. The nominal compositions of commercial cast and wrought alloys and their typical mechanical and physical props are given in Ref 4. Clark & Hawley(Ref 8) give an example of wrought "age-hardenable" alloys which are modifications of Duralumin or of castable alloys containing Al and ca 12%. Other industrial products of Al alloys are granules of various sizes used for adding to molten steel, for Thermite reactions and for expls. Requirements of the most important alloy of Al(Mg-Al) used in expls are described in a joint Army-Navy Spec(Ref 3). The tests and detns are listed under Aluminum (Analytical Procedures). The alloy of Al with Mg is used in pyrotechnic compositions and as a metal additive to some high explosives

According to Pérez Ara(Ref 9a) the addition of Al-Zn alloy has certain advantages over straight Al because the alloy is less reactive(oxidizable) in the presence of moisture

Examples of tracer and incendiary compns which use Al-Mg alloy in projectile ammo, taken from Ref 7, follow:

Composition, %	Red Tracer		Incendiary	
50/50 Al/Mg alloy	40	37	48.0	48.0
Chlorinated rubber	5	–	–	–
Strontium nitrate	55	56	–	–
Barium nitrate	–	–	50.5	50.5
Polyvinyl chloride	–	7	–	–
Linseed Oil	–	–	1.5	–
Asphaltum	–	–	–	1.5

Refs: 1)Mellor 5(1924), 229-46 2a)Pérez-Ara(1945) 2)Thorpe 1(1937),275-9 3)Spec JAN-M-454(1947) 4)Kirk & Othmer 1(1947), 605-9 5)ASM, "Physical Metallurgy of Aluminum Alloys,"Cleveland, Ohio(1949)

6)Perry(1950),281, 1527 & 1531 7)US Dept of the Army Technical Manual TM9-1910 (1955),292 & 294 8)Clark & Hawley(1957), 47

ALUMINUM CONTAINING EXPLOSIVES OR ALUMINIZED EXPLOSIVES (Explosifs d'-aluminium in Fr; Aluminiumhaltige Sprengstoffe in Ger & Swiss; Explosivi contenenti alluminio in Ital and Explosivos con alumin in Span). The addition of Al to increase the performance of expls was first proposed by Escales (Germany) in 1899 and patented by Roth in 1900(Ref 1). According to Lheure (Ref 2), Führer of Austria, in 1901, proposed to the Fr Govt the use of Al in expls. The following compns were examined in 1902 by the CSE (Commission des Substances Explosives): a)AN 83, charcoal 3 & Al 14% b)AN 70, charcoal 5 & Al 25% and proved to be fairly satisfactory. Better results were obtained when part of the AN was replaced by a HE such as NG, DNN or TNT, as in the explosive designated *Formula 226:* AN 74.5, DNN 6.0, Al 5.0 cellulose 2.0, NG 12.0 & NC(12%N) 0.5%. This compsn was tested in 1925 by the CSE and found to have a CUP(Fr Trauzl test) of 130 (PA 100) and a deton vel of 3400 m/s (Ref 25). Investigations conducted before and during WWI in Austria and Germany with Al-contg expls also gave satisfactory results

Notwithstanding satisfactory performance of aluminized expls, they were not used much as long as the quantity of Al on the market was limited and its cost much higher than of any other ingredient of the expl compn. When these drawbacks were overcome, (some time after WWI), more and more aluminized expls started to be used not only for military purposes but also as industrial expls. This included the use of Al powder in some primary and ignition compns, as was proposed in 1906 by Venier (Ref 3). Other ingredients of such compns were MF, K chlorate, K picrate and Ag acetylide

Although the cost of Al was high at the time of WWI, the Austrians, Germans and to a lesser extent the French, used it in some expls, such as Ammonal (qv). To a much higher extent aluminized expls were used during WWII by all the belligerent nations, especially in underwater ammo, (sea mines, torpedoes, depth charges, etc), where they were found to be most effective. They were also found very effective in Aerial bombs (because their radius of blast damage was greater than with nonaluminized expls) and also in incendiary bombs, flares, photoflash bombs etc

The action of Al in expls was investigated by many persons, inclg Kast(Ref 5), Tonegutti (Ref 8), Muraour (Ref 11), Stettbacher (Ref 12), Schmidt & Haid (of Zentralstelle in Neubabelsberg), as reported by Stettbacher (Ref 12) and by others. It has been claimed that Al does not take part in the actual detonation (Ref 12) but reacts immediately afterward with the products of expln such as CO_2 and H_2O:

$$2Al + 3CO_2 \rightarrow Al_2O_3 + 3CO + 196 \text{ kcal/mol } Al_2O_3$$

$$2Al + 3H_2O \rightarrow Al_2O_3 + 3H_2 + 226 \text{ kcal/mol } Al_2O_3$$

The large amts of heat liberated by these reactions maintain a high pressure of expln for a longer period of time than would be obtained without Al; that is, the pressure-time curves of expls contg Al do not have such high "peaks" as do the corresponding non-aluminized expls but the pressures remain high, lasting 2-3 times as long (Ref 12)

Haid & Schmidt have shown that Al reacts not only with oxygen but also with nitrogen forming a nitride (Al +N = AlN +80 kcal). This means that it is not necessary to make Al expls with a positive oxygen balance, as was done prior and during WWI, but it is better to maintain some negative balance

A further advantage of the addition of Al lies in the fact that Al_2O_3 formed during the reaction does not remain as a solid but vaporizes, thus increasing the overall volume of gases and the pressure. These increases are due to the fact that the bp of Al_2O_3 is only 2980° while the temp developed on expln of HE's is usually above 4500°

According to Médard(Ref 25), important studies of aluminized expls in France were made by Douillet about 1935. At that time he showed that although the binary mixt of AN 82 & Al 18% gave a Trauzl test value (CUP or cup, in Fr) much higher than PA, it was inferior to mixts in which part or all of the AN was replaced by a nitrocompd such as TNT, or pentolite

Stettbacher (Ref 12) gave several tables showing the differences in some expl props of non-aluminized and aluminized expls, from which the data for the table were taken

This table shows considerable increase in E and substantial increases in Q_e and T_e due to the addition of Al, whereas the vols of gases evolved decrease

Muraour (Ref 11) discussed the advantage obtained by adding Al to mixts TNT/HNDPhA. Such mixts without Al were used by the Germans during WWI for filling torpedoes and sea mines. When about 15% Al was incorporated in such mixts, the heat of expln was raised about 40%. It seems that the addn of 15% Al adopted by the Germans for their underwater expls is about optimum

(cont'd on next page)

TABLE

Comparison of Properties of Explosives with and without Aluminum

(Reduction to CO + H_2 in Aluminized Explosives)

Composition	Max Loading d	Gas Vol Evolved l/kg	Heat of Expln Q_e^p kcal/kg	Heat of Expln Q_e^v kcal/kg	Temp of Expln °C T_e^p	Temp of Expln °C T_e^v	Spec Press f atm	Max d of Energy E
TNT	1.62	684.0	950.0	–	–	–	–	1539.0
TNT 81.8 + Al 18.2	1.78	559.0	1472.0	–	–	–	–	2620.0
Erythritetetranitrate	1.70	704.8	1467.7	1486.0	4729.8	4759.0	44080	2496.0
ETeN 67.75 + Al 32.25	1.99	452.4	2350.0	2361.7	–	–	–	4676.0
Penthrinit[NG 74.9%, NC of 12.1%N 4.2% & PETN 20.9%]	1.64	719.5	1597.8	1616.5	4857.2	4885.9	48473	2620.4
Penthrinit 69.+ Al 31	1.93	496.5	2274.0	2286.8	–	–	–	4389.0
Blasting Gelatin[NG 91.3%, NC (of 12.24% N) 8.7%]	1.61	711.1	1612.5	1631.0	4970.2	4998.4	47528	2596.1
BlGel 68.7 + Al 31.3	1.92	488.7	2287.9	2300.6	–	–	–	4393.0
Oxyliquit(liq oxygen 75%, $C_{10}H_8$ 25%)	1.1	610.8	2287.0	2303.0	–	6383.0	38300	2516.0
Oxyl 67.05 + Al 32.95	1.6	410.8	2747.0	–	–	–	–	4395.0

Note: For definition of (f) and (E) see Ref 12

because it is close to the amt required to reduce the CO_2 to CO and the H_2O to H_2

Médard (Refs 25 & 29) detd some props of aluminized expls and gave a table comparing Trauzl test values(CUP in Fr)and in some cases deton velocities of nitrocompds contg from 0 to 40% Al. It seems that in most cases 15 to 20% is the optimum but this amt can be as high as 25% or even 30% when PETN or RDX is present. Among the aluminized expls investigated by Médard were a)*Nn°30:* AN 80.2, TNT 10.6 & Al 9.2%; CUP 132(PA 100) b)*Nn°31:* AN 78.5, pentolite(PETN/TNT-80/20) 12.3 & Al 9.2%; CUP 138 c)*Nn°33:* AN 69, TNT 10 & Al 21%; CUP 146 d)*63–CSE–1949:* AN 67, pentolite(80/20) 12 & Al 21%; CUP 147 *Note:* The first two expls have a positive oxygen balance and lower CUP while the last two have a negative OB and higher CUP

Médard also examined an industrial aluminized expl *Nn° 32:* AN 78, DNClB 12 & Al 10% (Ref 2, p 223), as well as *Sofranex A:* AN 48, NG 40, NC 2, Al 8 & liq DNT 2% (Ref 23, p 218) and *Sevranite N°1:* NH_4ClO_4 31, PETN 48, Al 3 & plasticizer (polyvinylacetate in liq DNT) 18% (Ref 29, p 219)

Le Roux (Ref 31) claimed that compns contg phlegmatized RDX and granulated Al are more powerful and possess higher deton velocities than those contg pulverized Al. They can be easily loaded by compression to d's higher than those with pulverized Al. As an example of such expls may be cited: RDX 80, MNN 5 & granulated Al 15%; CUP ca 155(PA 100), deton vel ca 7350 m/s at d 1.60

Belgrano(Ref 34) gave compns and props (Trauzl test values, gap test values & deton velocities) of a number of Ital aluminized expls. Most of them seem to be too weak for military purposes. An Ital military aluminized plastic HE consisting of RDX 67.2, NG 16.3, Al 12.2, wax 4.1 & unacc 0.2%, is listed in Ref 9. This expl was used during WWII for filling some projectiles. Other Ital, Al-contg military expls of WWII were *Nitramite* and *Trialine-105*

Following is some additional information on the props and uses of Al in expl compns as well as the advantages of such uses:

Baron (Ref 6) reported that replacement in AN-expls of carbonaceous material by a light metal, such as Al, reduces the amt of gas liberated on expln, but the loss in power due thereto is more than compensated by the greater production of heat. Holmes (Ref 7) proposed incorporation of small amts of Al powder in blasting expls consisting of black powder & AN. Schwarzer(Ref 10) proposed incorporation of Al powder in expls consisting of NG & kieselguhr

Stettbacher (Refs 12, 24 & 36), in addition to the previously mentioned advantages gained by the use of Al in expls, gave examples of military aluminized expl compositions developed during WWII, such as: a)German underwater expl contg TNT 62, HNDPhA 23 & Al 15% b)British and American expl contg TNT 42, RDX 40 & Al 18% c)Russian expl contg erythritetetranitrate & Al powder

Cooley et al(Ref 15) & Anon in OpNav 30-3M(Ref 16) discussed the uses of aluminized expls by the Japanese. Belyaev & Nalbandyan (Ref 17) described expl props of gasless mixts of Al with K chlorate. Ratner & Khariton (Ref 18) found that a small addn of Al, such as 5-10%, to AN raised not only the blasting power but also the brisance. When large amounts of Al were added, such as 20%, the brisance was not affected. This was explained by volatilization of the Al_2O_3, which absorbs heat. The blast effect was increased because expansion of the detonation products caused the temp to drop; the Al_2O_3 vapor condensed and its latent heat of vaporization was liberated to enhance the blast effect

All & EnExpls(Ref 19) gives a general discussion on aluminized expls and its use by belligerents during WW II

Shidlovskii(Ref 21) described expl mixts

of powdered Al & Mg with water. Perverzev(Ref 22) reported that the max increase in expl force of a nitroaromatic is achieved when the amt of Al is sufficient to completely reduce CO_2 and H_2O vapor. Goto & Sito (Ref 23) discussed expln and inflammation of Al powder in air (see under Aluminum Dust and Its Explosion)

Dinamite Nobel SA(Ref 26) patented aluminized HE's such as RDX with 10-25% of Al. Polverifici Giovanni Stacchini SA(Ref 27) patented HE's contg Al 5-30, TNT 30-90 & PETN 5-65% and also (Ref 28) expls in which half of the TNT of the preceding patent was substituted by DNN

Tominaga and Kanno (Ref 30) reported that Al powder used in flashlight powders can be partially replaced by CaS_2 or FeS_2 which results in their improvement. The presence of KNO_3 promotes uniform burning and reduces the combustion rate. Stearin and coconut binders are superior to paraffin with respect to promoting uniform combustion. Sakamaki (Ref 33) patented compns for use in electric detonators, such as Pb dinitroresorcinate 55, perchlorate 20, sulfur 10, Al 10 & binder (jelly of CC) 5%. Byers (Ref 35) patented AN-expls contg atomized Al particles (cryst size ca 30 μ) as an activator. An intimate mixt of ingredients was obtained by introducing atomized Al directly into the nitrate crystg bath. The resulting expls were reported to be easily activated

Sartorius(Ref 37) investigated several metals and metalloids as possible replacements of Al in expl compns. None of the straight substances seems to be as satisfactory as Al, even including Mg, but the author thinks that beryllium in alloys is promising and needs further investigation. Silicon, although less satisfactory in expls than Al from the point of view of power & deton velocity, might find application if the price was less than that of Al. Nuhsbaum(Ref 37a) patented a cartridge for an expl charge which contained within a separate casing an admixt of substances having high burning temps, such as powdered Al, Mg, ferrosilicon or red phosphorus. Richardson (Ref 38) patented expl compns consisting of particles of sulfur coated with a liq nitroaromatic and finely powdered AN & Al. Wallerius (Ref 41) claimed that incorporation of 8-12% Al powder together with the necessary amt of inorg nitrate for its combustion in plastic expls based on liq org nitrogen compds, lowered their costs and sensitivity to shock. Frutiger (Ref 42) patented expl compsns claimed to be of high stability and low sensitivity to shock by mixing methylhydrazine perchlorate with 1-2.5% carbonaceous material (such as graphite, starch or woodmeal) and up to 10% of Al powder. To make such expls plastic, gelatinized acetylcellulose may be used as the carbonaceous material

Note: Nav Ord Repts(Refs 43 & 46), being conf, were not used here as sources of info

PATR 2510(Ref 44) listed several Ger aluminized expls used prior to and during WW II

Streng & Kirschenbaum (Ref 45) claimed that an expl consisting of Al powder and a stable oxidizer is rendered more powerful and safer to handle and to store if some water is incorporated

A recent study of the role of Al in expl mixts is that of Cook and co-workers(Ref 42). The low relative "brisance" of aluminized explosives has been attributed in the past to incomplete reaction of Al at the "Chapman-Jouguet plane," and the high blast potential to after-burning of the Al. Thus, early shaped charge studies indicated that Al acts effectively as a diluent as far as the end effect is concerned. More careful studies by Cook showed, however, that Al lowers the detonation pressure and velocity even more than an ideal diluent. The effective endothermic reaction of Al in the deton wave is shown in the following results of deton pressures measured by the shaped charge method:

(See next page)

Explosive	Detonation Pressures Density d, g/cc	Detonation Pressure (atm × 10^{-3})
TNT	1.59	150
80/20-TNT/Al	1.68	140
TNT	0.81	46
80/20-TNT/Al	0.94	45
Composition B	1.71	230
80/20-Comp B/Al	1.81	170
73.2/26.8-Comp B/Al	1.83	155

This table shows that the deton pressures of Tritonal and HBX are smaller than those of TNT and Composition B respectively, even though the d's of the former expls are higher. This is significant in view of the known effect of d on press. The same situation may be observed by compg the deton vel (D), as represented below:

Velocity Comparison (by Cook)

Explosive	d, g/cc	D(m/sec)
TNT	1.59	6910
80/20-TNT/NaCl	1.75	6900
80/20-TNT/Al	1.75	6800
TNT	0.85	4525
80/20-TNT/NaCl	1.00	4400
60/40-RDX/TNT	1.70	7800
45/30/25-RDX/TNT/NaCl	1.77	(7430)[a]
45/30/25 RDX/TNT/Al	1.77	7200
50/40 RDX/TNT	1.00	(5650)[a]
45/30/25 RDX/TNT/NaCl	1.15	(5400)[a]
45/30/25 RDX/TNT/Al	1.15	4600

[a]By linear interpretation of results for TNT/NaCl and RDX/NaCl

These results show that Al lowers the deton val of TNT and of 60/40 RDX/TNT even more than does NaCl which acts as a heat absorbing or endothermic material. Therefore, Al must have a strong endothermic effect at the C-J plane. This would be the result if Al_2 (gas) was to form in appreciable amount in the detonation wave. But if Al_2O_3 (cryst) was the sole Al product, the det vel of the TNT/Al and RDX/TNT/Al mixts would be appreciably higher than the corresponding expls without Al due to the high heat of formation of Al_2O_3(cryst). The only compds of Al which might form in deton besides the oxides are AlN and AlH. Al_4C_3 exists only in the solid state, decompg on vaporization. Cook showed that none of these can be important as deton products and the significant products are therefore considered to be only Al_2O_3(gas), Al_2O(gas) and AlO (gas)

Besides the Al-contg expls described in this section, there are many other expls which are described individually; such as: *Alumatol, Ammonal, Anagon, APX-4A, ASN, Baronal, Berclavit B, Bonit, Borotorpex, Burrowite, DBX, Dentex; German Fillers Nos 15, 19, 105, 109, 110 & 13-113; HBX, Hexa, Hexamit; Japanese Explosives Types 1, 2, 88 & 92; Minex, Minol, Minol 2, Nitramite, Nitrobaronit, Nobel's 704, Novit Pentonal, Sevranite, Sofranex A, Torpex-2, Torpex D-1, Trialen or Trialine 105, Tritonal and UWE*

Some Al can be added with some advantage to practically every explosive compsn provided the Al does not react with any of the compounds. This is the case with Comp A, Comp B, Ednatol, PETN, RDX, TNT, etc

Refs: 1)G.Roth, GerP 172,327 (1900) 2) L.Lheure, MP **12**,125(1903-4) 3)W.Venier, BritP 6,705(1906) & CA **1**, 929-30(1907) 4) R.Förg, "Das Ammonal," Wien(1917)(200 pp) 5)Kast (1921),378-86 6)Ch.Baron, CR **208**,1010-12(1939) & CA **33**, 4423(1939) 7)H. H.Holmes, USP 2,168,030(1939) & CA **33**, 9648(1939) 8)M.Tonegutti, Suppl Technico della Rivista d'Artiglieria e Genio **1941**, 108-17 9)OrdnSergeant Aug 1943, 16 & 18 10)F.Schwarzer, SwissP 228,654(1943) & CA **38**, 4445(1944) 11)H.Muraour, Protar **9**, 62-3(1943) 12)A.Stettbacher, Protar **9**, 33-45, 212-18, 233-42(1943) & CA **38**, 4445 (1944) 13)Davis(1945), 25 14)A.Pérez

Ara(1945), 246 15)R.A.Cooley et al, "Japanese Explosives," PBL Rept 53,045 (1945) 16)Anon, "Handbook of Japanese Explosive Ordnance," OpNav 30-3M(1945), 32 17)A.F.Belyaev & A.B.Nalbandyan, DoklAkadN **46,** 113-16(1945) & CA **40,** 4523 (1946) 18)S.B.Ratner & Yu.B.Khariton, ZhFizKhim **20,** 221-2(1946) & CA **40,** 5919 (1946) 19) All & EnExpls(1946), 31-3, 48, 85-8, 128-30, 134-35 & 144 20)H.Muraour, MAF, **20,**681-4(1946) 21)A.A.Shidlovskii, ZhPriklKhim **19,** 371-8(1946) & CA **41,** 1105 (1947) 22)I.Perverzev, TrudyLeningrad-TeknologInst **1946,** No 12, 47-68 & CA **44,** 6627(1950) 23)L.Goto & E.Suito, Rev-PhysChemJapan, ShinkichiHoribaCommem Vol **1946,** 82-5 & CA **44,** 1707(1950) 24) Stettbacher (1948), 88-90 & 126 25)L. Médard, MAF **22,** 596-600 & 608-10(1948) 26)Dinamite Nobel SA, ItalP 439,931 (1948) & CA **44,** 6130(1950) 27)Polverifici Giovanni Stacchini SA, ItalP 445,601 & 445,602(1949) & CA **45,** 1770 & 3160(1951) 28)Ibid, ItalP 445,603(1949) & CA **45,** 3160(1951) 29)L.Médard, MP **32,** 215-16 (1950) 30)H.Tominaga & T.Kanno, JChem-SocJapan **53,** 106-8(1950) & CA **46,** 8373 (1952) 31)A.Le Roux, MP **33,** 107-11 (1951) 32)L.Médard, MP **33,** 350(1951) 33)T.Sakamaki, JapanP 147('51) & CA **46,** 11690(1952) 34)Belgrano(1952), 187 and tables at the end of the book 35)L. S.Byers, USP, 2,589,532(1952) & CA **46,** 5320(1952) 36)Stettbacher, Pólvoras (1952), 114-17 37)R.Sartorius, MP **34,** 205(1952) 37a)A.Nuhsbaum, AustrianP 927(1952) & CA **47,** 322(1953) 38)W.B. Richardson, USP 2,647,047(1953) & CA **47,** 10852(1953) 39)"Armament Engineering," US Military Academy, West Point, NY (1954), 69-70 40)"Military Explosives," US Dept of the Army Technical Manual TM 9-1910(1955), 184-6, 196-9, 210-12 & 214 41)P.G.Wallerius, SwedP 152,025 (1955) & CA **50,** 7463(1956) 42)F.Frutiger, SwissP 306,061(1955) & CA **50,** 16110(1956) 42)M.A.Cook et al, JPhysChem **61,** 189-96 (1957) 43)"Aluminized Nylon PBX," US Naval Ordnance Laboratory, NavOrd Rept 6067(1957)(Conf) 44)B.T.Fedoroff et al, PATR 2510(1958), pp Ger 3 & 212 45)A.G. Streng & A.D.Kirschenbaum, USP 2,836,484 (1958) & OffGazz **730**(May 1958), 955 46) D.Price, "Current Status of Problems Concerning Aluminized High Explosives," Nav Ord Rept 6238,(6 March 1959)(Conf)

Aluminum Containing Explosives; Trauzl Test Values. According to Davis (Ref 1), the standard Trauzl test does not give reliable results with expls contg Al or other materials which produce high temperatures on detonation. This is because the hot gases of reaction erode the lead inside the Trauzl block (cylinder), thus increasing its volume in addn to the increase produced by expansion of the gases. Médard (Ref 2) claimed that more reliable results are obtained when the French modification of the test is used. The result thus obtained is called the "coefficient d'utilization pratique," abbreviated to CUP or cup. The test is described in Ref 2

Refs: 1)Davis(1943), 25 2)L.Médard, MP **33,** 344-51 (1951)

Aluminum Containing Flares. See Aluminum Flares

Aluminum Containing Rocket Propellants. See Aluminum Dust in Rocket Propellants

Aluminum Cordeau, according to Davis(1943) p 11, is a detonating fuse consisting of Al tubing filled with PA

Aluminum Dust and Its Explosions. Dusts of Al and of some of its alloys (such as Al/Mg) are hazardous materials to handle because they are inflammable and may cause explns or fires. Particularly dangerous are mixts of dusts with gases contg oxygen such as air. Mason and Taylor (Ref 1) reported that the low expl limit for Al dust in dry air is ca 40 mg Al per 1 l air and if the SiO_2 content in air was twice the concn of Al dust, no expln took place. Berger (Ref 3) discussed the danger of expln and fire during grinding of the light metals Al or Mg. Brown (Ref 4) discussed dust expln hazards in plants producing or handling Al, Mg or Zn powders. Sata & Harisaki (Ref 5) studied

the ignition and expln of Al dust in air in various proportions and at different pressures. Fieldner & Rice (Ref 6) reported that tests of explosibility of various dusts, of comparable fineness, showed that Al, pure Fe, Mg, Dow metal, Ti and Zr are more expl than other metals or coal dust. Hartmann & Greenwald (Ref 7) discussed the explosibility of various metallic dusts. Hartmann & Nagy (Ref 8) discussed the effect of relief vents on reduction of pressure developed by dust explns of Al, Mg, etc. Goto & Suito (Ref 9) claimed that the expln or inflammation of Al powder is a chain reaction in which radiation participates to some extent. The designation "radiation chain" was suggested. Schläpfer(Ref 10)studied Al dust-air explns by the method of producing a steady dust flow. Anon (Ref 12) discussed the hazardous nature of Al and other metallic dusts

According to Hart & Tomlinson(Ref 11), while the explosibility of metal powders depends upon many factors, such as ignition, temperature, particle size, particle size distribution, shape, moisture content, energy required for ignition, etc, the metals may be arranged in decreasing order of explosibility of their dusts as follows: a)Zr & Ti(usually shipped under w or alc) b)Mg (less than 200 mesh) c)Mg/Al alloy (less than 200 mesh) d) Al (less than 6 microns) and e)Si

Toxicology, fire & explosion hazards of Al dust are discussed in Ref 13

Refs: 1)R.B.Mason & C.S.Taylor, IEC **29,** 626-31(1937) & CA **31,** 5582(1937) 2) Ibid, **32,** 67-8(1940) & CA **34,** 1486(1940) 3) H.Berger, Metallwirtschaft **19,** 404-11(1940) & CA **34,** 5805(1940) 4)H.Brown, USBurMinesInfCirc **7148**(1941) & CA **35,** 5703 (1941) 5)N.Sata & Yu. Harisaki, BullChemSoc, Japan **18,** 21-30(1943) & CA 4311(1947) 6)A.C.Fieldner & W.E.Rice, USBurMinesInfCirc **7241**(1943) & CA **37,** 6845 (1943) 7)I.Hartmann & H.P.Greenwald, Mining & Met **26,** 331-5(1945) & CA **40,** 2629 (1946) 8)I.Hartmann & J.Nagy, USBurMines ReptInvest **3924**(1946) 9)L.Goto & E.Suito, RevPhysChem, Japan, ShinkichiHoribaCommemVol **1946,** 82-5 & CA **44,** 1707(1950) 10) P.Schläpfer, SchweizVerGasWasserfachMonatsbull **31,** 69-82(1951) & CA **46,** 3762(1952) 11) D.Hart & W.R.Tomlinson, Jr, Metal Progress **59,** 788-92(1951) & CA **45,** 6844(1951) 12)Anon, C & EN **32,** 258(1954) & CA **48,** 4838(1954) 13)Sax(1957), 261-2

Aluminum Dust in Rocket Propellants. The possibility of using Al dust as a fuel ingredient of rocket propellants was investigated by Stettbacher. The dust was mixed with liq hydrocarbons such as benz, mineral oil, etc, and liq oxygen was added as an oxidizer. Other combustible metal dusts, such as Mg and Be, could be incorporated with Al. Heats of combustion of some of these mixtures were given

Ref: A. Stettbacher, Explosivst **1956,** 27

Aluminum Flares are military devices contg pyrotechnic compns which are mixts of finely powdered substances compressed into candles. The most important ingredients in a pyrotechnic compn are the fuel and the oxidizing agent. To these are usually added other materials to intensify the color of the light produced, decrease the burning rate, act as a binder and waterproof the compn

Powdered Mg, Al and alloys of these are the fuels generally used. The oxidizing agent selected is determined by the color, intensity of light and burning rate desired. The nitrates of Ba, Sr, Na and K, the perchlorates of Amm and K, and the peroxides of Ba, Sr and Pb are among the most important oxidizing agents used. Effective color intensifiers are organic chlorine compounds such as hexachlorobenzene, polyvinyl chloride and chlorinated waxes. Polyvinyl chloride, ethyl cellulose, metallic resinates, oils, waxes and asphaltum have been used as binding agents

Flares are used for illuminating purposes in: a) projectiles to illuminate enemy

territory b)trip flares to prevent enemy infiltrations c) airport flares to provide illumination for landing d)parachute flares for observation and bombing operations e)reconnaissance f)bombardment flares for high-altitude bombing and g)tow-target flares for target practice for antiaircraft gun crews

Some typical Al flare compositions and their characteristics follow:

Composition, %	Trip Flare	Airport Flare	Reconnaissance and Landing Flare	Bombardment Flare
Al, powder	21.5	–	–	8
Al, Grade A	–	2.0	–	–
Al, Grade B	–	20.0	26.0	–
Magnesium	–	–	–	36
Barium nitrate	69.5	62.0	66.0	34
Strontium nitrate	–	11.0	–	–
Sodium oxalate	5.0	–	–	20
Sulfur	4.0	3.5	6.25	–
Linseed oil	–	1.5	–	1
Castor oil	–	–	1.75	1
Characteristics				
Candlepower	50,000	60,000	75,000	800,000
Candlepower, per in^2	22,000	50,000	32,000	71,000
Burning rate, in/min	2.7	5.0	3.7	6.1
Explosion temp, °C	600	600+	–	490

Ref: US Dept of the Army TM9-1910, "Military Explosives," April 1955,

Aluminum Hydride. A series of aluminum hydrides, anologous to borohydrides, were described in Ref 1. They include the soluble polymer $(AlH_3)_x$ as well as its insoluble polymer

Occasionally explns have been reported during evapn of ethereal solns of AlH_3 or related compds used as reducing agents in organic reactions. On one occasion, a violent expln occurred when the residue obtained on evapn of a dimethylcellulosolve soln of AlH_3 contaminated with $AlCl_3$ was warmed, but no explns took place when AlH_3 free of $AlCl_3$ was used (Ref 2)

Refs: See top of the right column

Refs: 1)A.E.Finholt et al, JACS **69,** 1199-1203(1947) 2)G.Barbaras et al, JACS **70,** 877(1948) 3)Sidgwick, Chem Elems **1,**(1950), 413

Aluminum, Illuminating Powders. Various mixtures contg Al and oxidizers were prepd and examined in France as pyrotechnic illuminating composns. It was found that although the "illuminating power" (pouvoir éclairant in Fr) of Al-contg mixts is slightly inferior to those contg Mg, the former are much superior to mixts based on Zn, Cu and organic combustible materials including propellants and explosives

The following table gives the absolute and relative illuminating powers of various Al-contg compositions compared with some mixtures of Mg, Zn, Cu and organic compds:

Substance, Composition or Device	Illuminating Power Relative(P_1) Mg = 1.0	Absolute(P) (Lumens × 10^3) seconds/gram
Mg ribbon burning in air	1.00	290
Bulb "Photolita" of Philips, 115v, 250w	ca 0.04	0.009

(Continued on next page)

Substance, Composition or Device	Illuminating Power Relative(P_1) Mg= 1.0	Absolute(P) (Lumens × 10^3) seconds/gram
Mg 56, $NaNO_3$ 39 & binder 5%	0.62-1.01	180-293
Al 49.3 & $NaNO_3$ 50.7%	0.40-0.84	116-244
Al 39.4, $NaNO_3$ 40.6 & MgO 20%	0.87	252
Al 39.4, $NaNO_3$ 40.6 & Al_2O_3 20%	0.74	214
Al 39.4, $NaNO_3$ 40.6 & NaCl 20%	0.44	128
Mg 58 & $KClO_4$ 42%	0.43	125
Al 49 & $KClO_4$ 51%	0.30	87
Mg 29.2 & NH_4ClO_4 70.8%	0.27	78.5
Al 27.7 & NH_4ClO_4 72.3%	0.27	78.5
Al 43.9 & NH_4ClO_4 56.1%	0.45	130
Mg 52.7 & $NaClO_4$ 47.3%	1.23	357
Mg 78.2 & $NaClO_4$ 21.8%	1.67	485
Al 33.7 & $NaClO_4$ 66.3%	0.35	101
Al 51.2 & $NaClO_4$ 48.8%	0.83	241
Mg 37.3 & $KClO_3$ 62.7%	0.25	72.5
Al 30.6 & $KClO_3$ 69.4	0.23	67
Mg 49.1 & $Pb(NO_3)_2$ 50.9%	0.31	90
Al 30.8 & $Pb(NO_3)_2$ 69.2%	0.26	75.5
Zn 65.3 & $KClO_4$ 34.7%	0.013	3.8
Cu 57.5 & NH_4ClO_4 42.5%	0.000091	0.026
S 20.7 & $KClO_3$ 79.3%	0.013	3.8
NC alone	0.0034	0.99
NC 60 & $KClO_3$ 40%	0.044	13
TNN 31.7 & $KClO_4$ 68.3%	0.047	14
Resin 13.7 & $NaClO_4$ 86.3%	0.088	25.5
Woodmeal 27.6 & $NaClO_3$ 72.4%	0.044	13

Ref: P.Tavernier, MP **31,** 309-426(1949, especially tables on pp 368-382

Aluminum-Liquid Oxygen and Liquid Air Explosives. An expln of a mixt Al powder and liq oxygen, injuring 17 persons, occurred during a lecture demonstrating the ignition of such a mixt (Ref 4). The procedure used in the demonstration was one previously described by Cady(Ref 1). After this accident the following letters (Ref 5) dealing with Al-oxygen explosions appeared: a)A.V.Grosse stated that for a number of years at the Research Institute of Temple University they had exploded mixtures of Al powder with oxyger and air and had also detonated Al powde mixed with liq oxygen(Ref 3). They foun that Al powder mixed with a stoichiomet amt of liq oxygen to form Al_2O_3 is a very powerful expl, giving 3.85 times the amt energy of an equal wt of TNT b)A.T.Ba stated that he too had a classroom expl of a mixt of liquid oxygen and Al when c ducting the experiment according to Cad description. However, Bawden had also p formed the experiment about 100 times, o taining a flash each time rather than an

In 1936 David Bruce, a student at the College of the Pacific, did his master's thesis on the study of powdered Al-liqui O_2 reactions. He learned that such a mi would always expl rather than flare up if were enough O_2 to oxidize 83 to 90% of t Al, when ignited with a burning taper. H also learned that such a mixt could be e ploded by an electric spark from a Ford A continuous spark would not ignite the mixt until it reached expl proporns by ev of the liq oxygen. Then an expln rather t a flare was always obtained. This expln noteworthy because a small amount of th mixt produced a powerful shock wave. T above results were not published becaus it was found that similar experiments ha been previously described(Ref 2)

Austin et al (Ref 6) discussed the exp hazard of Al-liq O_2 mixts in detail

Refs: 1)H.P.Cady, JChemEduc **8,** 1027 (1931) 2)G.S.Perrott & N.A.Tolch, Bur Bull **349,** (1932)(Liquid oxygen explosi 3)A.D.Kirshenbaum, Research Institute c Temple University,Phila,Pa, Final Repo on Fundamental Studies of New Explosiv Reactions for Office of Ordnance Researc Contract No DA-36-034-ORD-1489(30 Ap 1936) 4)Anon, C&EN **35,** 90(17 June 19 5)Anon, C&EN **35,** 12 & 14(15 Aug 1957) 6)C.M.Austin et al, JChemEduc **36,** 54-8 (Feb 1959) (15 refs)

Aluminum-Lithium Hydride or Lithium Al uminohydride, $AlLiH_4$, wh solid prepd fr aluminum chloride and lithium hydride in

ethereal soln: $AlCl_3 + 4LiH = LiAlH_4 + 3LiCl$(Ref 1). The compd is a powerful reducing agent, converting $SiCl_4$ into silane, etc(Ref 3). Although $AlLiH_4$ is considerably more stable than aluminum hydride(AlH_3), explns similar to the ones described under aluminum hydride may take place (Ref 2)
Refs: 1)A.E.Finholt et al, JACS **69**, 1199-1203(1947) 2)G.Barbaras et al, JACS **70**, 877(1948) 3)Sidgwick,ChemElems 1(1950), 413

Aluminum (or Magnesium)–Methanol (or Water) Explosives. According to Shidlovskii (Ref 1),mixts of Al and H_2O(2:3) or Mg and H_2O(1:1) are capable of combustion when subjected to intense heat. The Mg mixture can be detonated with a primer while the Al mixt cannot. The same investigator claimed (Ref 2) that on the basis of theoretical calcns of heat evoln, mixtsof Mg or Al with H_2O or alcs are potentially more powerful expls that the usual military materials, with Mg-MeOH giving the max gas evoln. The tests were conducted in bombs or lead enclosures with tetryl detonators to set off the mixts of powdered metal and the liquid. All mixts tested were found to be powerful expls with Mg-H_2O being most sensitive to shock, while Al-H_2O and Mg-MeOH were less sensitive and required a booster

According to an investigation by Médard (Ref 3), mixts of Mg + H_2O, Mg + MeOH or 2Al + $3H_2O$ are comparable in their power to guncotton but they are not able to propagate the deton unless a small quantity of a sensitizer (such as 7%+ of PETN) is incorporated. For instance, the mixt contg Al (powder) 30, H_2O 30 and PETN 40% has a vel of deton of 5140 m/sec at d 1.55, coefficient d'utilization pratique (CUP)(French Trauzl test value) 119(PA 100), and may be detonated by a Briska primer. It is practically insensitive to shock but its exudation, as detnd by the method of Burlot, [MAF **14**, 303(1935)], and its stability make it unsatisfactory for use as a military explosive

Refs: 1)A.A.Shidlovskii, DoklAkadN **51**, 131-3(1946) & CA **40**, 6817(1946) 2)A.A. Shidlovskii, ZhPriklKhim **19**, 371(1946) & CA **41**, 1105(1947) 3)L.Médard, MP **33**, 491-503(1951) 4)A.G.Streng & D.Kirshenbaum, USP 2,836,484(1958)(Aqueous metal powder explosives; eg Al 42, AN 30 & H_2O 28%)

Aluminum Methyl. Same as Trimethyl Aluminum

Aluminum Nitride. See under Nitrides

Aluminum Ophorite. Under this unusual name, an expl mixt was patented and claimed to be suitable for military purposes. It consisted of Al foil finely ground in oil (which was not in excess of 2% of the mixt) and mixed with pulverized alkali and metal perchlorates
Ref: D.B.Bradner, USP 1,775,063(1930) & CA **24**, 5161(1930)

Aluminum Oxide-Silicon Carbide Fiber, developed by the Carborundum Co at Niagara Falls, NY, will withstand temps of 2300°F. The fiber is suitable for insulation of gas-turbines and jet-engine exhaust systems, and its mixt with asbestos will resist fire and reduce heat loss through radiation
Ref: Anon, Common Defence Bulletin,No 143, Washington,DC(Sept 1952)

Aluminum Perchlorate. See under Perchlorates

Aluminum Picrate. See under Picrates

Aluminum Plate Test for Detonators is briefly described under Plate Tests and also in Davis (1943), 26

Aluminum Soap Gels are briefly described in Science in World War II, Chemistry, edited by W.A.Noyes, Jr, Little, Brown & Co, Boston(1948), 411-12: "Properties of Aluminum Soap Gels as Thickening Agents"

Aluminum Soaps of Mixed Isoöctoic Acids when mixed with hydrocarbon fuels produce jellied gasoline suitable for use in flame throwers and incendiary bombs
Ref: L.Cohen, USP 2,741,629(1956) & CA **50**, 11693(1956)

Aluminum Stearate. See under Stearates

Aluminum Stearate Gels. See under Stearates

Aluminum Triethyl. Same as Triethyl Aluminum

Aluminum Tripropyl. Same as Tripropyl Aluminum

ALUMS

Alum is the generic name given to an important group of double sulfates of the general formula $M^1_2SO_4 . M^3_2(SO_4)_3 . 24H_2O$, which is sometimes written $M^1M^3(SO_4)_2 . 12H_2O$, where M^1 is a monovalent metal or group such as Na, K, Li, Rb, Cs, Tl, NH_4, Ag etc and M^3 is a trivalent metal such as Al, Cr, Fe, Mn, In, Co etc (Ref 3). Alums are prepd by mixing aq solns of the corresponding salts and crystallizing out the alum. Alums are all soluble in water and crystallize with 24 moles of water in crysts belonging to the regular systems, usually octahedra or cubic. Some alums, because of their high water of crystn, have been used in commercial expls as cooling agents. Those alums which have been used for this purpose are described below

For more information on alums see Refs 1, 2, 3, 4, & 5

Refs: 1)Mellor **1** (1924), 340-54 2)Gmelin, SystNr **35,** B 1 & 2(1934), 248-279, 378, 453, 509 3)Thorpe **1** (1947),293-6 4)Kirk & Othmer **1** (1947), 653-5 5)Cond Chem Dict(1956), 47

Ammonium-Aluminum Alum, $(NH_4)_2SO_4 . Al_2(SO_4)_3 . 24H_2O$, mw 906.64, mp 92-5°, d 1.64 at 20°/4°, n_D 1.4591. Col octagonal crysts losing 24 H_2O at 200°. The soly of the hydrate in w is given by Locke(Ref 1). Ammonium alum is used in medicine, as a mordant in dyeing, in water purification, in paper sizing and in the dressing of skins (Ref 5) and can be used in expls as a cooling agent. For more information see Refs 2,3,4&6

Refs: 1)J.Locke, AmChemJ **26,** 174(1901) 2)Mellor **5,** (1924),340-57 3)Gmelin,SystNr **35,** B2(1934),508-15 4)Thorpe **1** (1947),296 5)Kirk & Othmer **1** (1947),655 6)Cond Chem Dict(1956),49

Ammonium Chrome Alum (Alum Ammonium Chrome), $(NH_4)_2SO_4 . Cr_2(SO_4)_3 . 24H_2O$, mw 956.72, mp dec 100°, d 1.72; gm or viol crysts. Can be prepd by treating an aq soln of $(NH_4)_2Cr_2O_7$ with H_2SO_4 and a stream of SO_2(Refs 1 & 3). Sol in w, sl sol in alc. It has been used in some expls(eg, Chromeammonite Reinforced)(qv), as an oxidizer, and as a cooling agent. On expln it also evolves fairly large amts of gases, N_2, H_2O and SO_2

Refs: 1)C.A.Taylor & W.H.Rinkenbach, BurMinesBull No **219,** 49(1923) 2)Mellor **11**(1931),452 3)Thorpe **3**(1946),100 4) Cond Chem Dict(1956),279

Ammonium-Iron Alum, $(NH_4)_2SO_4 . Fe_2(SO_4)_3 . 24H_2O$, mw 964.40, mp 40°, d 1.71. Violet octagonal crysts, sol in w, insol in alc. Was prepd by mixing molecular proporns of ferric and ammonium sulfates and concentrg the soln spontaneously. It has been proposed as a standard in titrations(Ref 1) and probably can be used as a cooling agent in expls

Refs: 1)L.L.de Konnick, BullBelg **23,** 222 (1909) 2)Thorpe **7,** (1948), 60 3)Cond Chem Dict(1956), 476

Potassium-Aluminum Alum (Kalinite), $K_2SO_4 . Al_2(SO_4)_3 . 24H_2O$, mw 948.75, mp 92°(loses $18H_2O$ at 64.5°), d 1.76 at 26°/4°. Col monocl crysts, sol in w. Its anhydrous salt, $KAl(SO_4)_2$, mw 258.19, d 2.75 at 20°, when heated with carbon produces "Homberg's Pyrophorus," a flammable compd contg K sulfide(Ref 3). It has also been used in expls, such as Clark's Powder(qv)

Refs: 1)Mellor **5**(1924),343 2)Thorpe **1** (1947), 294 3)Kirk & Othmer **1**(1947), 655 4)Cond Chem Dict(1956), 52

Potassium-Chrome Alum(Chrome Alum), $K_2SO_4 . Cr_2(SO_4)_3 . 24H_2O$, mw 998.86, mp 89°, d 1.813. Viol crysts turning gm on melting; in aq sol the change occurs at ca 78°(Ref 1). The satd soln at 18° contains 28.2% $K_2SO_4 . Cr_2(SO_4)_3 . 24H_2O$ of which 51.8% exists as a violet salt (Ref 2). Chrome alum is used in paper making, photography, dyeing, printing and tanning. It has also been used in an expl called Chromeammonite (qv)

Refs: 1)Mellor **11**(1931),454 2)Thorpe **3**(1946),100 3)Cond Chem Dict(1956),279

Potassium-Iron Alum (Ferric Potassium Sulfate or Iron Alum), $K_2SO_4 . Fe_2(SO_4)_3 . 24H_2O$, mw 1006.5, mp 33°, d 1.806. Prepd by mixing equi molecular amts of ferric and K sulfate and concg the soln spontaneously. It forms fine violet octahedra crysts, liable to decomp to a brown deliquescent mass. Iron alum is sol in w, insol in alc. This

alum is used in dyeing and in calico printing and probably can be used as a cooling agent in expls. If caustic potash is added to a soln of the alum and the brown liquid allowed to evaporate, yel-brn crysts of $5K_2SO_4 \cdot 2Fe_2(SO_4)_2 \cdot 16H_2O$ separate. These crysts have the peculiar optical props of tourmaline (Ref 1)

Refs: 1)Mellor **14**(1935),340-4 2)Thorpe **7**(1948),60 3)Cond Chem Dict(1956),479

Sodium-Aluminum Alum (Soda Alum), $Na_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$, mw 916.55, mp 61°, d 1.675 at 20°/4°, n_D 1.43884. Col octahedra or monoclinic crysts, occurg naturally as the mineral mendozite. It is produced from Al sulfate by adding a clear soln of Na sulfate. Soda alum is highly sol in w (Ref 2), sol in dil acid and insol in alc. It has been used in baking powder manuf and in the prepn of matches

Refs: 1)Mellor **5**,(1924),342 2)M.Nousseron & P.Gravier, BullFr [4] **51**, 1383(1932) 3) Thorpe **1**(1947),296 4)Kirk & Othmer **1** (1947),655 5)Cond Chem Dict(1956),53

Alundum. A pure crystalline Al_2O_3 in granular form, d 3.9-4.0, mp 2030-2050°, hardness on the Mohs scale 9. It has been used as an abrasive, a basic refractory material, as a component of low-expansion glasses, as a filtering material and for chemical apparatus. It is probably suitable as a component of primer compns in lieu of glass, etc

Refs: 1)Hackh(1944),41 2)Kirk & Othmer **1**(1947),640-1 & 646-9

Alvisi patented beginning 1898 in Italy and England a series of expls based on ammonium perchlorate, such as *cremonites, kratites,* etc

Ref: Daniel(1902), 17

Am—added to a Fr name or abbrev of a propellant means that amyl alcohol was used as a stabilizer. For instance, $BFAm_2$ stands for "poudre B, fusil, amyl alc 2," means a NC propellant with 2% of amyl alcohol, for use with rifles. Such propellants are no longer manufd because amyl alcohol proved to be a poor stabilizer

Ref: Pascal(1930),234

Amalgam is a liquid, semiliquid or solid alloy of mercury with Na, Ag, Ca, Li, NH_4, Au, etc. The most important amalgam is that of Na and it will be a liquid when the amt of Na is less than 1.25%, or a solid when the amt of Na is higher. It can be prepd by gradually adding small pieces of Na to Hg under kerosene or mineral oil while avoiding a rise in temp, (Refs 3 & 4). A good description of amalgams is given in Thorpe's (Ref 2). Na amalgam is easier to handle than Na metal; it is used as a reducing agent. Other uses of amalgams are given in Ref 2.

Dowling (Ref 1) pointed out the danger of expln in amalgam barrels (*Note:* The type of amalgam is not indicated in Ref 1)

Refs: 1)W.R.Dowling, CA **5**, 2240(1911) 2)Thorpe **1**(1937), 298-300 3)InorgSynth **1**(1939),5-18 4)Kirk & Othmer **1**(1947),447

Amasite. An expl patented in the early 1900's by the Société Anonyme des Explosifs Favier, Vilvorde, Belgium and permitted for use in England: NH_4ClO_4 32 to 36, $NaNO_3$ 29 to 33, NB 30 to 36 & agar-agar 0.15 to 0.5%

Ref: Escales, Chloratspr(1910),163

Amatex. According to Ref 1 there are several composns known under this name, such as *Amatex 5*, which consists of AN 25, TNT 50 & RDX 25%. More important is *Amatex 9:* AN 50, TNT 41 & RDX 9%, which was used by the Brit during WWII in large GP, MC, HC & A/S bombs. It was prepd by mixing 60/40 amatol with 15% Comp B. The latter was added to eliminate the tendency of large amatol-filled bombs toward low order detonation. The sensitivity and brisance of Amatex 9 was slightly higher than that of amatol 60/40

According to Jiménez(Ref 2), Amatex is an amatol sensitized by a small amt of HNDPhA

Refs: 1)All & EnExpls(1946), 133 2)J.M. Jiménez, "Explosivos," Ediciones Ejército, Madrid(1951), 28

AMATOL

"Military Nitrate of Ammonia"

[Called Amatol or Füllpulver(Fp) No 13 & No 13a in Germany; Amatol in GrBrit, Fr & Rus; Amatolo in Spain; Amatola in Italy and Shotoyaku in Japan](Fr abbreviation NT)

Amatols are expl mixts of AN with TNT in various propns. They were invented in 1915(Refs 4 & 6) by the Brit in order to extend the available supply of TNT which was very scarce at that time. Two mixts were used by the British: an 80/20 amatol (a plastic mass resembling wet brown sugar) and a 50/50 amatol (a cast mass resembling cast TNT). The 1st figure refers to AN, the 2nd to TNT. (The Germans who also adopted amatols had the 1st figure referring to TNT and the 2nd to AN.) The US Govt, shortly after its entrance into WWI authorized the use of above amatols for loading HE shells. The first mixt was loaded, while hot, either by extrusion or pressing, whereas the second mixt could be cast-loaded, which was an advantage. Amatols were cheaper than TNT and, on explosion, produced greater volumes of gas per unit weight. The addition of AN, rich in oxygen, results in more complete combustion of the TNT. For this reason the smoke produced by the deton of amatol is of a light yellowish-white color in contrast to the heavy black smoke produced by straight TNT. All amatols have lower velocities of deton and brisance than TNT, but are more powerful as judged by Ballistic Mortar and Trauzl tests. The impact sensitivity of amatols is comparable to that of TNT, but with increasing proportions of AN amatols become more difficult to deton. All amatols are hygroscopic and in the presence of moisture attack metals such as copper, brass, bronze and lead(Ref 14)

Explosive Properties. Some explosive properties of the more important amatols are given in the table shown on the following page

Hackel(Ref 5) reported that with a 2kg hammer there was no difference in impact sensitivity between amatol and straight TNT (value about 60cm) but with larger weights amatol proved to be more sensitive. For one explosion in ten trials using a 5kg wt, Hackel obtained the following results in cm: TNT 40–42, 10/90 AN/TNT 27, 40/60 21, 50/50 19, 60/40 15, 70/30 18, 80/20 24–26 & 90/10 39

Smith(Ref 7) claimed that the explosive action of amatol is increased by incorporating into it (by absorption) a highly inflammable liquid such as benzene or gasoline. A method for increasing the density of a charge of amatol in which the percentage of the TNT is less than that required to make the amatol flow at a temp within 10–20° above the mp of TNT has been reported by Snelling(Ref 8)

Systematic measurements of the influence of boundary conditions on the detonation velocity of 60/40 amatol charges of finite radii have been made by Copp & Ubbelohde (Ref 15). Various physico-chemical conditions which control the thermal decomposition and the rate of energy release were investigated by the method of Dautriche. A summary of mean deton vels (D) for cast 60/40 amatol confined in cylindrical tubes of steel, lead and cardboard (which approximates an unconfined chge) is given in their paper. Results of their work have shown that the grist size of AN and the boundary conditions, have a marked influence on the value of D(mean vel of deton at a given chge radius), but not on the value of Do(vel of deton at infinite chge radius)

Preparation of Amatols

80/20 Amatol. AN(US Spec JAN–A–175), previously ground by running through a crusher, dried to contain not more than 0.25% moisture and screened to remove foreign materials and to obtain required size(see Note), was heated to 90–95° in a mixing kettle, provided with a steam jacket and mechanical agitation. To this was added gradually, with constant agitation, the calcd amt of molten TNT at ca 95° and the mixt thoroughly blended by continuing the agitn for at least 15 mins, while maintaining the temp at 95°. At the end of this period the hot amatol was transferred to the loading apparatus(Refs 6 & 10)

Explosive Properties of Amatols (Taken mostly from Ref 19)

Composition & Properties	Amatol 80/20	60/40	50/50	45/55	40/60
Composition: AN	80	60	50	45	40
TNT	20	40	50	55	60
Nitrogen Content	31.7%	28.4%	26.8%	25.9%	25.1%
Oxygen Balance to CO_2	+1.20%	-17.6%	-27.0%	-31.7%	-36.4%
Oxygen Balance to CO	+11.06%	+2.13%	-2.32%	-4.55%	-6.78%
Color	Lt buff	Lt buff	Buff	Buff	Buff
Melting Point, °C	–	–	81	–	–
Density, g/cc	1.46	1.61	1.59	–	1.54
Detonation Velocity, m/sec	5080*	5500*	5600*	6470	6500
Detonation Velocity, ft/sec	–	–	19,680	–	–
Brisance by Sand Test, assuming 100% for TNT	75	85	86–90	–	95
Explosion Temperature, °C	280–300	270	254–265	–	–
Heat of Explosion, cal/g at C_v	1004	–	950	–	920
Heat Tests	Slightly less stable than TNT				
Impact Sensitivity Test, 2kg wt, assuming 100cm fall for TNT	90–95	95–100	93–100	–	93–100
Pendulum Friction Test		Unaffected			
Power(by Ballistic Mortar Test or by Trauzl Test), assuming 100% for TNT	130	128	120–125	–	120
Power by Trauzl Test, assuming 100% for PA	–	–	–	112	–
Rifle Bullet Test		Unaffected			
Energy of Air Blast	–	–	84	–	–
Energy of Shock in Water	–	–	94	–	–
Shaped Charge Efficiency	–	–	54	–	–

*According Ref 15, vel of deton of 60/40 amatol charged at 1.50 in a steel tube 17 mm diam is 6060 m/sec when prepd with a finely ground AN, vs 5860 m/sec when using a coarse AN. Evans (Ref 156) gives for 60/40 amatol 5600 m/sec at d 1.6, for 50/50 amatol 5850 at d 1.6 and for 80/20 amatol 5200 at d 1.6

Note: In order to obtain a mixt sufficiently plastic to consolidate well on loading without separation (leaking) of molten TNT, it is necessary to use AN of proper granulation. American practice was to use AN, which met the following requirements: Through a No 10 US Std Sieve – not less than 99%, through a No 10 & on No 35 – 32 to 48% and through a No 100 – 15 to 30%

50/50, 60/40 and 40/60 Amatols. AN(US Spec JAN–A–175), previously ground, dried (to contain not more than 0.25% moisture) and screened to remove foreign materials, was heated to 90–95° and gradually added to calcd amt of molten TNT in a kettle, provided with a steam jacket and mech agitn. The rate of addn was such that no segregation or lumping of AN took place. Agitn was continued at 90–95°, until thorough blending and uniform fluidity were achieved. Then the mixt was cooled to ca 85° and ready for cast loading(Refs 6 & 10)

Notes: Mitra & Ram(Ref 14) conducted studies of the optimum conditions for crystn, drying and caking of AN and detnd the effects on amatol fluidity. Some of their concln s were as follows: a) the cryst form of AN is of little importance but spherically shaped crysts make satisfactory amatols b) crystn of AN at a temp of 160°, followed by drying at 100 gives a product suitable for pouring 65/35 amatol c) it is of importance to keep the moist content of AN below 0.15% before mixing with TNT. Médard & Le Roux(Ref 16a) reported that in prepn of various AN expls(including the amatols) the best vel of deton, sensitivity to ignition and coeff of self-excitation are obtained when a heavy wheel (5 tons) is used for pulverizing AN

Shell-Loading with 80/20 Amatol. Part of the mixt maintained in the kettle at 90–95° (See above under Preparation) was transferred to the hopper of an extruder which was provided with a stirrer and jacket heated with steam at 3–5 lbs pressure. The extruding machine consisted of a steel tube in which a worm screw rotated slowly. This machine was counterweighted so that the amatol was forced into the shell under a definite limited pressure. The shell, previously cleaned inside and uniformly coa with a special varnish, was placed at the mouth of the extruder and filled to within inches of the top. After removing the she a cavity for a "booster surround" was f by driving or pressing a hardwood plug i the nose of the shell. After removing the the cavity was filled with molten TNT. A this has solidified, a booster cavity was drilled as described under TNT. Extrusic was carried out automatically and no one allowed in the building while the shell w ing filled. No tools containing copper ma used, which means that brass or bronze e ment, as customarily used in the loading straight TNT, was excluded. After filling first shell, the density of the amatol cha was determined as prescribed by US Arm Spec 50–15–3A. It should not be below 1

Note: The density was detnd by weighin the empty shell before loading (W_e), wei ing it filled with water (W_w), (making all ance for the surround cavity), thoroughly ing the shell and reweighing it after load by extrusion with TNT (W_t). This gives d (of amatol) = $\frac{W_t - W_e}{W_w - W_e}$

If d is below 1.38, the pressure in the truder is increased until the desired d is tained(Refs 6 & 10 and US Army Spec 50 15–3A)

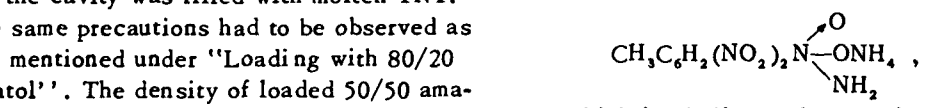
Note: H.Graham et al(Ref 13) described procedure for filling shell with 80/20 am which produced d's up to 1.47. In this pr the amatol was first flaked by passing it through milling rolls heated to about 100 with space between the rolls about 0.019 and then cooled to RT. The empty shell(bomb), nose down, was stemmed with an hammer for 15 to 20 mins while the amatc flakes were added in 20 increments. This method of filling, it was claimed, would ford the advantage of uniform distribution which was never obtained by other metho of filling

Shell-Loading with 50/50 Amatol. A part of molten amatol was brought to a temp ca 85° and poured into the shell through a tightly fitting funnel, called a riser, so that the level of the charge before solidification came within approximately 1" below the bottom of the booster casing. After allowing the charge to solidify, with occasional breaking of the crust until the central portion of the pour was still slightly mushy, a second pour was made to the desired height. Immediately after the second pour a rod was inserted through the second pour until it came into contact with the first pour. When the charge had cooled, the rod was removed with a twisting action and the cavity was filled with molten TNT. The same precautions had to be observed as was mentioned under "Loading with 80/20 Amatol''. The density of loaded 50/50 amatol had to be >1.50 and it was detnd as described under 80/20 amatol or in US Army Spec 50–15–15C(Refs 6 & 10)
Note: This method of loading is applicable to any castable amatol, such as 60/40 and 40/60

Recovery of TNT from Scrap Amatol. During WWI, a method was developed and used for the recovery of TNT from amatol scrap. The recovered TNT, however, was often discolored and of doubtful quality due to the low grade TNT and AN used in making amatol. During WWII, the TNT specified for amatol was of higher quality and since the AN was made from synthetic ammonia, it did not contain impurities which reacted with TNT to affect its color or purity. Therefore, the process formerly used which involved the extraction of the AN with hot water, filtering and graining the molten TNT, yielded a Grade I TNT from scrap 50/50 amatol complying with the minimum requirements of the US Army(Ref 9)

Stability of Amatols. According to Ref 17a, p 183, the vacuum stability of 50/50 amatol is a little less than that of TNT at temps of 100 and 120°, there evidently being very sl reaction betw TNT and AN at those temps. At temps below the mp of TNT(ca 80°), there is no evidence of reaction. After storage at 50° for 3 months, there is no change in the sensitivity brisance or stability

Inasmuch as some amatols prepd in France showed instability, an investigation on stability of various mixts of AN and TNT was conducted after WWII at the Laboratoire Centrale des Poudres, Paris(Ref 16). The results showed that mixts of military grade TNT and pure AN decomposed with the evolution of ammonia. This attacked the TNT to form various unstable colored compds, some of them containing as much as 21.5%N, compared to 18.45%N for TNT. One such compd of brownish-red color was claimed to be:



which is similar to that previously obtained by Korczinski(Ref 1) as a result of the reaction of ammonia with TNT

Also, due to the hygroscopic nature of AN, amatols are very unstable in storage(Ref 1), unless it is possible to exclude moisture. At 90% RH and 30°, 80/20 amatol would contain ca 61% moisture in 2 days. This not only lowers the sensitivity and vel of deton to a low order but results in failure to detonate. In the presence of Fe, hydrolysis of moist AN may take place with the formation of NH_4OH, which reacts with TNT to form an exudate of a brown oily material igniting at 67°. This can be detected by discoloration of the explosive and the odor of NH_3. In this case the shell cannot be safely washed out with steam and it is necessary to use cold water

To prevent the corrosion caused by contact of amatol with metal, it was an American practice to coat the insides of shells with acid-proof black paint prior to loading, and to prevent moisture entering amatol in loaded shells a seal was formed by pouring some molten TNT on top of amatol. This TNT served as a booster surround

Bourjol(Ref 17) investigated samples of amatol stored for 25 years in Zn boxes and

found that considerable deterioration of TNT took place. Lab experiments have shown that if Al is used in lieu of Zn, the TNT remains unaffected

Exudation of TNT from Amatol Shells. The study of exudation began in the USA shortly after WWI because it was observed that some amatol and TNT loaded shells were exuding a brown oil(Refs 2 & 3). Samples of the exudadate were collected at Pic Arsn and the mechanism and significance of exudate formation was the subject of an exhaustive study. Exudation of oil from TNT and amatol filled shell was found to be purely a physical phenomenon resulting from the effect of elevated temperature upon TNT containing impurities. The danger of such a condition lies not in the explosive properties of the exudate but in the decreased density of the charge (due to formation of cavities), making possible a premature explosion upon set-back when the shell is fired. (Cavitation is more pronounced in straight TNT than in amatols.) The TNT oil when present in the booster charge also desensitizes the booster so that duds may result. The presence of gas within TNT or amatol shell is due to a chemical reaction between TNT and alcohol. This reaction is not progressive and will continue only so long as there is alcohol present. Exudation is not as serious a problem from the standpoint of safety as is commonly supposed. Yet, it is not desirable to issue for use shells showing exudation since there is a danger of explosion on unscrewing boosters with explosive collected in the screw threads (Ref 2)

Destruction of Amatols. Scrap amatols or amatol-loaded small bombs or projectiles, may be destroyed by burning in beds not more than 3" thick, as described in Ref 19a, p 316 or in the US Ordnance Safety Manual ORDM 7–224, C7, pp 27–13 to 27–15. Amatol loaded in ammo may be destroyed by detonation as described in ORDM 7–224, C7, pp 27–16 to 27–19. In case of large shells and bombs it is preferrable and less dangerous to remove the amatol by steaming, disposing of AN soln into a stream and burning off the removed TNT

Uses of Amatols

France. Although amatols are described in several Fr papers (Refs 16, 16a & 17), there is no info on their uses in France (see also Refs under Amatol, Analysis)

Germany. The following modified amatols with low TNT content were used during WWI for cast-loading some ammo (Ref 4, p 173): a) AN 60-65, Na nitrate 10, dicyandiamide 5 & TNT 25-20% b) AN 65-67, Na nitrate 12-10, Na acetate 3 & TNT 20%. According to Ref 14a, the following compn was used during WW II: AN 40-45, TNT 50 & RDX 10-5%. Several modified amatols, No 39,40 & 41 are listed on p 4 of Ref 19a. On pp 47-8 of the same Ref are listed: a) 40/60 amatol (called in Ger Füllpulver No 13 or Fp 60/40)- used in GP, SAP & A/P bombs and shells b) 50/50 amatol (called in Ger Füllpulver No 13a or Fp 50/50)- used in GP bombs and land mines, such as the Tellermine c) 70/30 amatol (called in Ger Fp 30/70)- used in some A/P bombs d) 60/40 amatol (called in Ger Füllpulver No 88 or Fp 4/60)- used in some shells, grenades and radio-guided bombs e) 95/5 amatol (Fp 5/95, in Ger)- use is not known (See also Ref 12, p 82)

Great Britain. The 80/20 and 60/40 amatols, invented in 1915 at the Research Dept, Woolwich,soon became the main fillings for HE shells in the Brit Land Service (Ref 4, p 152 & 171). According to Ref 12, the use of amatols during WW II was as follows: a) 60/40 amatol - in GP, Medium Capacity, High Capacity & A/P bombs; also in depth charges, rockets, grenades, land & sea mines b) 80/20, 70/30, 60/40 or 50/50 in shells of all types

Italy. The 80/20, 70/30 and 60/40 amatols were used for filling various kinds of shells, bombs, grenades and bombs (The 60/40 was known as "esplosivo 60/40") (Ref 12, 13a & 16b). According to Ref 16b, the 90/10 amatol

was used in mining. Its props were: Trauzl test value 365 cc, deton vel 2500 m/sec and gap test value (ditanza colpo, in Ital) 2.0 cm

Japan. The 50/50 amatol known as shotoyaku was used in some bombs and projectiles (Ref 11, p 27). According to Ref 12, the use of amatol was limited due to the shortage of TNT. It had been reported in Naval mines

Russia. According to Blinov (Ref 15a), two types of amatol were used during WWII: a) 80/20 amatol - in many types of ammo, such as 76.2 & 107 mm HE shells, 82, 107 & 120 mm mortar shells, and 122 mm HE howitzer shells b) 50/50 amatol in some ammo and to a lesser extent than 80/20 amatol. A similar expl call Ammoksil (qv) contained TNX in lieu of TNT

United States of America. The US Govt shortly after its entrance into the WWI authorized the use of 50/50 amatol for shells from 75 mm up to and including 4.7", and 80/20 amatol for shells from 4.7" up to and including 9.2" (Ref 6, p 124). According to Ref 12, the use of amatols during WW II was as follows: a) 50/50 and (80/20 amatols in LC (light case)), GP and SHP bombs b) 50/50, 60/40 and 80/20 amatols were used in various shells. During the early part of WW II, some 65/35 amatol was used in some shells and bombs (Ref 19a, p 182)
Note: The rapid production during WW II of a huge supply of TNT (obtained by nitration of plentiful petroleum toluene), removed the necessity of using AN as a substitute for TNT. Another factor contributing to the disuse of amatols as military expls was the appearance during WW II of materials more powerful than TNT, such as PETN and RDX, as well as their binary mixts pentolites, cyclotols, etc

The use of cast amatols, such as: AN 35, TNT 34, Na nitrate 30, chalk 0.9 & stearic acid 0.1% was recently patented for military and civlian applications (Ref 17b)

References on Amatols: 1) A.Korczinski, BullAcadSciKrakow **1908**, 633-448 & JCS **94**, 977-8(1908) 2)F.Hawks, "Exudation from TNT and Amatol", ArOrdn **5**,611-2(1924) 3) PA ResRept Nos R-15(7 Feb 1928), R-25 (31 May 1928), R-32(8 Aug 1928) and R-41(4 Dec 1928) 4)Marshall **3**(1932), 152,171 & 173 5)J.Hackel, Wiadomsci Techniczne-Uzbrojenia **No 38**, 519(1937) & MAF **18**,769-72(1939) 6)Anon, WarDeptTM 9-2900(1940), 124-7 7)A.Smith, BritP 538,920(1941) & CA **36**, 3670 (1942) 8)W.O.Snelling, USP 2,275, 569(1942) & CA **36**, 4340(1942) 9)F.H.Vogel, PATR 1225 (1934)(Recovery of TNT from amatol scrap) 10)Anon, WarDept TM 9-1904 (1944), 113-14 & 147-51 11) Anon, NavyDept. OPNAV 30-3M(1945) 12) All&EnExpls(1946), 80 & 82 13)H.Graham et al, CanChem & Process Ind **30**, 37-41(1946) & CA **42**, 4753 (1948)(Bomb filling with 80/20 amatol) 13a) Mangini(1947), 225 14)B.N.Mitra & R.Ram, JSciIndRes(India),**7B**, 163-6 (1948) & CA **43** 6417(1949) 14a)T.Urbański, Przemysl-Chemiczny, **27**(IV), 487(1948) 15) J.L.Copp & A.R.Ubbelohde, TrFaradSoc **44**, 646-69 (Sept 1948) 15a)Blinov, vl (1948), p 19 15b) W.M.Evans, ProcRoySoc **204 A**, 14(1950) 16) F.M.Lang & J.Boileau, MP **34**, 181-7(1952) 16a)L.Médard & A.LeRoux, MP **34**, 195-203 (1952) 16b)Belgrano(1952), 289 17)G. Bourjol, MP **36**, 41-5(1954) & CA **50**, 2173 (1956) 17a)Anon, USDept of the Army TM 9-1910 & Dept of the Air Force TO 11A-1-34, "Military Explosives" (1955), 182-4 17b) C.H.Winning, USP 2,736,724(1956) & CA **50**, 6796(1956) 18)Sax (1957), 266 (Fire & expln hazards and toxicity of 80/20 amatol) 19) PATR 1740(1958), 1-11 19a)PATR 2510 (1958), p 4 (Amatols) & pp 47-8(Füllpulver No 13, No 13a and Füllpulver 30/70 & No 88) 20)Cook(1958), 18, 54 & 307

Additional Refs on Amatol:

a)Van Gelder & Schlatter (1927), 954-6 (Amatol, also called **Military Nitrate of Ammonia**, was manufd in the US during the latter part of WWI. Judging by the amts of its ingredients producd at the same period: AN 95,500,000 lbs and TNT 101,800,000

lbs, the amt of amatols produced was probably ca 150,000,000 lbs, because most of the TNT produced went for the manuf of amatols) b)A.Stettbacher, Explosivst **1954,** Nr 3/4, p 40 (Properties for 50/50 amatol: d 1.5-1.55, gas vol at NTP 930 l/kg, Qe 840 kcal/kg with H_2O vapor, temp of deton 2640°, max vel of deton 5000 m/sec and impact sensitivity 90 cm with 2 kg wt) c)M.A.Cook et al, JPhysChem **59**, 675-80(1955)(Deton vel and wave shape were measured as a function of charge diam for 50/50 amatol and a loosely packed mixt of 50/50 AN/TNT as compared with pure AN and Comp B contg AN. Vels of deton of amatol and loose mixt AN/TNT were practically the same for identical diams of chge, ranging from ca 4700 m/sec for diam 3.81 cm, 5000-6000 m/sec for diam 5.04 cm and 6000-6500 m/sec for diams 7.62-17.78 cm)

Amatol, Analytical Procedures

Identification of Amatol. a) Place about 0.05 g of previously pulverized unknown material in a 5-cc beaker, add 2 to 3 cc of distilled water, stir for 5 mins and observe the color of aliquot. It is colorless in case of amatols b) Test the aliquot with a strip of Universal pH indicator paper;there shall be no change in color c) Add a drop of Nessler's reagent – brown ppt in case of amatol. If the test is negative, the substance is not amatol

If the above test is positive confirm the identity of amatol by one or several of the following tests: A) Place about 0.05 g of unknown material in an indenture of a white porcelain spot-test plate and add 2-3 drops of 65 to 68% aq soln of ethylenediamine and stir – the color of soln shall be maroon B) Repeat the test using a new 0.05 g sample and 3-4 drops of DPhA soln (1g in 100 cc of concd CP sulfuric acid), stir and wait 1 min; the color of soln shall be dirty green C) Repeat the test using a new sample, an equal amt of thymol and 3 drops of concd sulfuric acid; stir the mixt and wait for 5 min – green coloration indicates the presence of amatol (Ref 5)

Note: The same colorations are obtained in with ammonals

Analysis of Amatol

Moisture. a) Weigh to $^1/_{10}$ mg a dry 50 ml Pyrex crystallizer covered with a ribbed watch glass, introduce a previously pulverized sample of ca 5 g and obtain exact total wt of the crystallizer, cover and sample b) Heat for 2-3 hrs at a temp not above 75°, cool in a desiccator and reweigh. The diff in wt divided by wt of sample and multiplied by 100 gives % moisture

Note: The ribbed cover is used to catch the small amt of TNT which sublimes on heating

TNT by Benzene Solution. a) Weigh to $^1/_{10}$ mg a dry sintered glass crucible, place ca 2g of thoroughly pulverized sample and reweigh to $^1/_{10}$ mg b) Insert the crucible into the stopper of a heavy walled filtering flask placed on a steam bath, fill the crucible to ¾ with hot benz, cover with a watch glass, allow to stand for ½ min and apply gentle suction c) Repeat the operation several times, using a total of 75-100 ml benz, in order to estract all the TNT d) Dry the crucible to const wt, cool and weigh. Loss in wt = TNT + moisture e) Crystallize the TNT from the benz soln and det the mp of one or several crysts, using Fisher-Johns or other apparatus

Ammonium Nitrate. a)In the manner described above, extract 2 grams of the original sample, by running through the sintered glass crucible a total of 150 ml of water at 84° b) Evaporate the combined filtrates to a small volume, transfer quantitatively to a tared small dish and continue evapn just to dryness, at temps not higher than 100° c) Leave overnight in a desiccator and rinse the residue 2-3 times with anhydrous ether in order to remove traces of TNT. d) Dry the dish with AN and weigh (Ref 1)

Miaud & Dubois (Ref 3) and previously Bourgoin (Ref 2) devised a rapid method for detn of AN in amatols by comparing index of refraction at 20° of its aq soln with a curve (or table) giving relation between concn of AN and index of refraction)

Miaud (Ref 4) devised a rapid method for detn of TNT in amatols by comparing density of its benzolic soln with a table giving relation between densities of TNT in C_6H_6 and % TNT in C_6H_6

Refs: 1)Anon, War Dept TM 9-2900(1940), 126-7 2)L.Bourgoin, Anales de l'Acfas (Canada) **9**, 90-1(1943) & CA **40**, 1317(1946) 3)P.Miaud & P.Dubois, MP **32**, 225-9(1950) 4)P.Miaud, MP **32**, 227-38(1950) 5) Anon, Dept of the Army TM 9-1910 & Dept of the Air Force TO 11A-1-34(1955), 269-7

Note: According to N.Liszt of PicArsn, the following method is recommended for analysis of mixts of TNT with nitrates: a)Quantitatively transfer an accurately weighed sample (ca 1g) to a dry, tared, sintered glass extraction thimble and extract with anhyd methylene chloride CH_2Cl_2, using a Soxhlet or equivalent apparatus into a tared flask placed on a water bath b)Adjust the temp of the bath so that the solvent drips from the end of the condenser at the rate of 2-3 drops per second c)When extraction is complete, evaporate the liquid in the flask to dryness under stream of dry air and then in a vacuum desiccator to const wt d)Subtract the wt of flask from tot wt, thus obtaining the wt of TNT e)Subtract the wt of TNT from wt of sample (ca 1g), thus obtaining the wt of AN (or its mixt with materials insol in methylene chloride) f)Dry the thimble for TNT extraction and transfer its contents quantitatively into 300 ml Erlenmeyer flask using water g) Determine AN content as described in Spec JAN-A-175, Par F-4j or identify AN by one of the color reactions such as with DPhA(dirty green), thymol (green) or other reactions described in the books on analytical chemistry h)If it is suspected that AN is mixed with Na nitrate, use the procedure described by N.Liszt in PicArsnGenLabRept 54-H1-1718(1954) and included in Spec MIL-C-13879(ORD)(1954)

Nonaqueous Titrimetric Method of Analysis of Compositions contg AN, Na nitrate and TNT was developed by H.L.Herman and described in PATR 2384(1956). This method was critically evaluated by N.S.Garman, PicArsn GLR 57-H1-109(1957) and found to be more rapid than the one described in Spec MIL-C-13879 but slightly less accurate. It was recommended to include this method as an alternate in Spec MIL-C-13879

Amatoxol. A HE contg AN 80 and Toxol (TNT 70 & TNX 30) 20%. Its power by the Trauzl test 118 (PA 100), rate of deton 5100 m/sec and sensitiveness to impact 110+(PA 100)

Ref: Dr L.R.Littleton, Washington,DC; private communication

Amber (Succinum), $C_{10}H_{16}O$(?),d 1.07 to 1.09, hardness 2 to 2.5. A fossilized, bituminous resin which is derived from an extinct variety of pine. It is pale yel to brn or red-brn and varies from transparent to opaque. It is used as a semi-precious stone or in experiments on static electricity (See also Note under Amberites)

Refs: 1)Hackh(1943),41 2)CondChemDict (1956),55

Amberites. Fast burning smokeless propellants manufd by Curtis and Harvey Ltd in Gt Britain beginning in 1891. The original propellant, described by Cundill(Ref 1), consisted of insol NC 40-47, NG 40-30 and paraffin 20-23%, together with a small quantity of shellac soln

Daniel (Ref 2) gives the following compositions for *Amberite No 1:* NC(insol) 40-47, NC(sol) 20-23 and NG 40-30%. For its prepn the mixture of 13% and 12%N nitrocelluloses together with NG was granulated and treated on the surface with a volatile solvent so that only the sol NC was gelatinized. On drying the grains, the two types of NC were cemented and the surface of the grains hardened. Small quantities of paraffin, shellac or linseed oil could be incorporated as moderants

The following compositions contained no NG: a)*Amberite No 2A*:NC(insol) 13.0, NC (sol) 59.5, Ba and K nitrates 19.5, paraffin 6.1 and vol matter(mostly H_2O) 1.9% (Ref 2) and b)*Amberite No 2b*:NC(insol) 53.2, NC (sol) 24.1, Ba and K nitrates 10.8, paraffin 9.6 and vol matter (mostly H_2O) 2.3%(Ref 2)

Beginning in 1894, WM(woodmeal) was included in the formulation and the resulting compn was called "*Blasting Amberite*". In 1899, the incorporation of charcoal and calcined WM was started (Ref 2)

In Refs 3,4 & 5, the formulation of an *Amberite* used as a shot-gun propellant is given as follows: NC 71.0, Ba nitrate 18.6, K nitrate 1.2, WM 1.4, vas 5.8% and vol matter 2.0%. In Ref 6, the following properties of a *British Amberite* are given: Q_c^v 745 cal/g and total vol of gases evolved per gram at NTP 791 ml of which 156 ml is water vapor

Refs: 1)Cundill, MP **5**,281(1892) 2)Daniel (1902), 17-18 3)W.MacNab & A.E.Leighton, JSCI **23**, 293(1904) 4)Marshall **1**(1917),327 5)Barnett(1919),86 6)Thorpe **4**(1946),530 7)PATR 2510(1958), p Ger 4

Note: The name *Amberite* is also used for the compressed amber(qv) scrap used for electrical insulation

Ref:Hackh(1944),42

Amberlac: Trade name for a rosin modified oxidizing type phthalic alkyd resin or modified polyester type resin manufd by Rohm & Haas, Phila Pa.

It was used by Aerojet Engrg Corp, Azusa, Calif in some experimental smokeless propellants. Eg: a)*RL-210* propellant- Amberlac 75 & Paraplex AP-31 25% and b)*RL-223* propellant- Amberlac 85 & Duraplex 15%

Refs: 1)Aerojet Engineering Corp Rept No 192(1946), 16-17 2)CondChemDict(1956),55

Amberlite. Trade name for resorcinol type resins used for wood adhesives and manufd by the Rohm & Haas Co, Phila 5,Pa. Also a trade-mark name for insoluble crossed-linked polyelectrolytes (ion-exchange resins). Used for water conditioning and other purposes

Ref: Cond Chem Dict(1956), 56

Note: It may be used as a binding agent in propellent or expl composns

Amberol. Trade name for oil-soluble phenol formaldehyde – maleic glyceride resins. Used in paints, varnishes, lacquers, etc

Ref: Cond Chem Dict(1956), 56

Note: It may be used as a binding agent in propellent or expl composns

Âme(Fr). Bore(of a firearm)

Âme de canon(Fr). Cannon bore

American Ammonium Nitrate Dynamites. See under Ammonium Nitrate Dynamites

American Ammonium Nitrate Explosives. See under Ammonium Nitrate Explosives

American Ammonium Nitrate Gelatins. See under Ammonium Nitrate Gelatins

American Chemical Society (ACS). Organized in 1876 and located at 1155 16th St,NW.Washington,DC. Publishers of the JACS, C&EN, IEC, CA, Anal Chem, JPhChem, JAg & FChem, and JOC

Refs: 1)Hackh(1944), 42 2)Library of Congress, Scientific and Technical Societies of the United States and Canada, NAS and NRC, Washington 25,DC(1955), 40-43 3)R.S.Bates, Scientific Societies in the United States, Columbia Univ Press,NY(1958),100, 143, 177, 178, 179, 181, 195 & 202

American Dynamite of 1894 contained NG and a mixture of sieved coke and Ca acetate as the absorbent
Refs: 1)Callahan & Higgins, USP 525,188 (1894) 2)Daniel(1902), 18

American Dynamites, Gelatinized. According to Davis(Ref 2), blasting gelatin (qv) is not used very widely in the US; the somewhat less powerful gelatin dynamite or simply *Gelatin* finds much greater use. Gelatin is essentially a straight dynamite in which a gel, consisting of NG (or NG+NGc) with 2 to 5.4% NC is used instead of liq NG or a liq mixt of NG+NGc. The resulting composn is elastic similar to blasting gelatin

The composn of typical American 30 to 70% strength gelatins used in 1915 were given by Munroe & Hall(Ref 1) and also by Davis(Ref 2). They contained 23 to 60% NG (or NG+ NGc), 0.7 to 2.4% NC, 62.3 to 29.6% Na nitrate, 13 to 7% combustible material (wood pulp in those with 50 and 60% NG, wood pulp and in some cases rosin and sulfur in other grades) & Ca carbonate 1%

Bebie(Ref 3) gives two examples: a)NG 62.5, NC 2.5, Na nitrate 27.0 and wood pulp 8.0% and b) NG 36, NC 2, vegetable meal 2, nitrates and/or perchlorates 52 & nitrocompounds(of toluene, naphthalene or diphenylamine) 8%. Bebie also cites *Gel-Coalites* as a brand of gelatinous permissible expls manufd by the Atlas Powder Co of Wilmington, Del, but does not give their composns
Refs: 1)C.E.Munroe & C.Hall, USBurMines Bull 80(1915) 2)Davis(1943),344-5 3)Bebie (1943), 73-4

American Dynamites, Low Freezing, Non Gelatinized, are a series of "straight" dynamites which do not freeze at temps prevailing in the USA in winter. They consist of 20 to 60% of mixed nitric esters (NG with NGc) absorbed on wood pulp(or other carbonaceous materials) and mixed with sufficient Na or K nitrate to maintain any desired oxygen balance
Note: Instead of NGc, nitrosugars may be used as antifreezing components. In European practice dinitrochlorohydrin, tetranitrodiglycerin and liquid aromatic nitrocompounds have been used
Refs: 1)Davis(1943),333-4 2)Bebie(1943), 94

American Dynamites, Low-Freezing, with Ammonium Nitrate. See under Ammonium Nitrate Dynamites

American Dynamites, Ordinary. A series of non-gelatinized, so-called "straight" dynamites or dynamites with active base, reported to contain: NG 15-60, combustible materials (wood pulp, sawdust, ivory nut meal, raw corn flakes, sulfur, etc for grades below 40% NG or wood pulp alone for other grades) 20-14, Na nitrate 64-23 & Ca or Mg carbonate (antacid) 1%. Deton velocs are between 4500 and 6250 m/sec depending on the NG content

The "standard" 40% straight dynamite used at the USBurMines in comparative tests contains: NG 40, Na nitrate 44, wood pulp 15 & Ca carbonate 1% (Ref 2 and Ref 3, p 333)

Refs: 1)C.E.Munroe & C.Hall, USBurMines Bull **80** (1915) 2)C.A.Taylor & Wm.H. Rinkenbach Ibid **219**(1923) 3)Davis(1943), 333 & 338-9 4)Bebie(1943),140

American Dynamites, Ordinary, with Ammonium Nitrate. A series of non-gelatinized or "straight" dynamites reported to contain: NG 15-35, AN 15-30, Na nitrate 51-24, combust material(mixt of WP, flour and sulfur) 18-10 & Ca carbonate or Zn oxide 1%
Refs: 1)C.E.Munroe & C.Hall, USBurMines Bull **80**(1915) 2) Davis(1943), 341

American Electroplastics Corporation Explosive, according to the analysis made at PicArsn, contained: AN 70.97, NS 21.56, Al metal 4.50, oil 2.94 and Pb tetraethyl 0.03%. Its brisance, as detnd by the Sand Test 24.3 g (vs 34.3 g for the Trojan Demolition Expl and 28.5 g for 80/20 amatol); impact test with 2 kg wt 14 cm(vs 25 cm for tetryl and 17 cm for PETN); 75° International Test, 100° Heat Test and 120° Vacuum Stability Test—sl more satisfactory than for the Trojan Demolition Expl. It is more difficult to initiate than the Trojan Explosive

The Electroplastics Corp expl was considered unsatisfactory for military use because of its fairly high sensitivity and low brisance value
Ref: PATR 1117(1941)

American Explosive. A non-gelatinized permissible explosive manufd by the American Cyanamide and Chemical Corp, New York, NY
Ref: Bebie(1943), 20(The compn is not given)

American Forcite Powder Co was organized in New Jersey in 1883 by H.A.deCastro. In 1913 it became the property of Atlas Powder Co, Wilmington, Del
Ref: VanGelder & Schlatter(1927), 453 & 465

Americanite. A very powerful liquid expl invented in 1890 by Smolianinoff and tried successfully in the US for loading shells up to 203 mm. There were no prematures at a muzzle velocity of 654 m/sec. The expl consisted of NG 80 to 97 and a liq alcohol 20 to 3%
Ref: Daniel(1902), 19

American Permissible Explosives. See under Permissible and Permitted Explosives

American Powder, also called **White German Powder.** See Augendre Powder

American Smokeless Propellants, listed in Marshall, v 1(1917), 327, contained a)GC 80 sol NC 19.5 & urea 0.5 and b)GC 80, sol NC 10, NG 9 & urea 1%. Urea was later replaced by diphenylamine as a stabilizer

American Stability Tests at 65.5° and 80°. See under Stability Tests

American Table of Distances for Storage of Explosives, as revised and approved by the Institute of Makers of Explosives, September 30, 1955, is given by Sax(1957), 154-7 and Cook(1958), 354-6

Amiante(Fr); **Amianto**(Ital & Span). Asbestos

Amidation is the process of forming an amide

Amide(Explosif), designated also as *l'explosif amylacé,* was patented in 1886 in France. It contained "sulfurless black powder" (AN+ KNO_3+charcoal) 32-60 & NG 68-40%
Ref: Daniel(1902), 20
Note: The above "sulfurless black powder" was similar in compn to Amide Powder described below

Amide Powder (Amidpulver, in Ger). One of the earliest types of Ger smokeless artillery propellants. The original compn patented in 1885 by Gäns contained AN 35-38, K nitrate 40-46 & charcoal 14-22%. Its formulation was modified several times until a powder which was flashless and nearly smokeless was obtained. The improved compn: AN 37, K nitrate 14 & charcoal 49% was used during WWI as a cannon propellant. Amide powder left only a small residue on combustion. This was attributed to the formation and ignition of potassamide which is formed on burning the powder
Refs: 1)Thorpe 1(1937),304 2)Davis(1943), 49 3)Bebie(1943), 20-21 4)PATR 2510 (1958), p Ger 4
Note: According to Daniel(1902), 20, Amidpulver was exported from Germany to England under the name of *Chillworth Special Powder*

AMIDES, IMIDES AND DERIVATIVES (Inorganic)

Amides and Imides, Inorganic. An inorganic *amide,* also called *ammonobase,* is a compd in which one hydrogen in ammonia is replaced by a metal, as for instance *sodamide,* $NaNH_2$. An inorganic *imide* is a compd in which two hydrogens are replaced by metals, eg, *lead imide,* PbNH

Metallic amides and imides can be pptd from liq ammonia solns of certain metallic salts by the action of potassium amide, KNH_2

Some amides and imides are explosive, eg, *silver amide* and *lead imide* (Compare with Nitrides) (See also Polyamides)
Refs: 1)Mellor 8(1928), 252-68 2)Franklin (1935), 33 3)Hackh(1944), 42 4)Kirk & Othmer 1(1947), 667-71

Alkali Amides, such as potassamide and sodamide are described individually. A general description of the prepn and props of various alkali amides is given by F.W. Bergstrom & W.F.Fernelius, ChemRevs 12, 43-179(1930). Alkali amides can be used for the

prepn of other metallic amides, some of them expl(see Auric Imidoamide, Cadmium Amide Silver Amide, etc)

Auric Imidoamide or Gold Amide-Imide $Au(:NH)NH_2$ or $HN:Au.NH_2$. Powder, extremely expl and sensitive. Was first obtained in the Middle Ages by alchemists and named *aurum fulminans (fulminating gold).* It was prepd by treating gold oxide with an ammoniacal soln of a salt of gold. Fulminating gold does not expl when wet and must be stored under w to avoid accidents. A number of explosions with the dry salt have been reported in the literature. When fulminating gold is kept at 100° for several hrs, it becomes so sensitive that it scarcely can be touched without exploding (Ref 1)

Franklin (Ref 2) prepd it by treating potassium-auri-bromate with potassamide in liq NH_3 soln

Mellor (Ref 1) stated that if aq ammonia is added to a soln of auric chloride, a mixt of fulminating gold and a yel solid identified as *auric imidochloride,* HN:AuCl is formed and the chloride cannot be removed even by prolonged digestion with aq ammonia. The mixt is expl

Refs: 1) Mellor **3**(1923),582-3 2)Franklin (1935), 57

Cadmium Amide, $Cd(NH_2)_2$, wh powder expl when rapidly heated. Can be prepd by treating Cd iodide or KCd cyanide with a soln of potassamide in liq NH_3

When heated to 180° in vacuo, Cd amide loses NH_3 leaving Cd nitride, Cd_3N_2, a black amorphous powder which expl violently when brought in contact with w

Ref: Mellor **8**(1928), 261

Fulminating Gold. See Auric Imidoamide

Fulminating Silver. See Silver Amide

Gold Amide–Imide. See Auric Imidoamide

Lead Imide, PbNH. Orange-red ppt which expl violently when heated or or coming in contact with w, dil acids or liq NH_3. It is obtained when PbI_2 (or some other Pb salt) is brought together with K amide in liq NH_3 soln: $PbI_2 + 2KNH_2 = PbNH + NH_3 + 2KI$

Refs: 1)E.C.Franklin, JACS **27**,842(1915) 2)Mellor **8**(1928), 265 3)Franklin(1935),61 & 325-6

Potassium Amide or Potassamide, KNH_2. Col, delq leaflets, mp 239°. Was first prepd ca 1810 by Gay-Lussac and Thénard by heating metallic K in an atmosphere of NH_3. When metallic K is brought in contact with liq NH_3 it slowly dissolves to form an intensely blue soln, which gives the amide on standing for several weeks (or even months). The reaction can be greatly accelerated by sunlight or by the presence of some substances acting as catalysts. A relatively minute amt of Pt black, placed in a liq NH_3 soln of metallic K, causes an immediate and fairly vigorous reaction resulting in the formation of KNH_2 in the course of a few minutes

According to Mellor (Ref 1, p 255), KNH_2 reacts vigorously with w and the reaction may be accompanied by inflammation

Potassamide can serve for the prepn of other metallic amides and imides, some of them expl, eg $AgNH_2$ or PbNH

(See also Ref under Alkali Amides)

Refs: 1)Mellor **8**(1928),253-5 2)E.C.Franklin, JACS **27**,830(1905) 3)C.A.Kraus & E.J.Cuy, JACS **45**,712(1923) 4)Franklin(1935),53-4

Silver Amide, $AgNH_2$, bulky wh ppt which darkens on exposure to air and shrinks in vol; very expl and its sensitiveness to deton is not materially changed by lowering the temp to −190° (Ref 4). Can be prepd by mixing a soln of potassamide with Ag nitrate (or iodide) in liq NH_3, followed by washing by decantation and careful drying (Refs 2,4 & 5). Franklin (Ref 2 & 5) warned that $AgNH_2$ can expl on the slightest provocation, shattering test tubes containing the material and tearing holes in several layers of strong towelling wrapped around the tube for the protection of the operator

It seems that silver amide is identical with the compd previously known as *fulminating silver of Berthollet* (l'argent fulminant de Berthollet in Fr and Bertholett' schen Knallsilber in Ger), also called by Mellor (Ref 1) *silver imide* or *silver nitride.* This compd,

as a dark extremely expl solid,was first mentioned, but not identified, by J.Kunckel, in 1767. Berthollet prepd it in 1788-9 by treating pptd Ag oxide with concd aq ammonia and described some props. Several other investigators prepd this expl and various formulae were assigned to it, such as Ag_2N and Ag_3N(see Ref 1), until the present formula $AgNH_2$ was established
Refs: 1)F. Raschig, Ann **233**, 93(1886) 2) E.C.Franklin, JACS **27**, 833(1905) 3)Mellor **3**(1923), 381 4)Mellor **8**(1928), 259 5) Franklin(1935), 57 & 319-25

Sodium Amide or Sodamide, $NaNH_2$. Wh crysts, mp 206.4°; dissolves in liq NH_3 and is vigorously hydrolyzed by H_2O. Was first prepd ca 1810 by Gay-Lussac and Thénard by the action of NH_3 gas on molten metallic Na heated to 300°. Can also be prepd by dissolving metallic Na in liq NH_3 and then placing in the soln a a spiral of Fe wire, which catalytically accelerates the otherwise very slow reaction: $Na + NH_3 = NaNH_2 + H$

Mixts of sodamide with nitrates and chlorates expl when triturated (Ref 1, p 255)

When finely divided sodamide was exposed to air in the presence of a little w, a yel-red solid was formed identified as *sodium amidoperoxide*, $NaNH_2O_2$. It is stable in dry air but decomp by moisture (Ref 1, p 255)

Fused $NaNH_2$ dissolves metallic Mg, Zn, Mo, W, quartz, glass, many natural silicates and many other substances
Refs: 1)Mellor **8**(1928),253-5 2)F.W.Bergstrom & W.C.Fernelius, ChemRevs **12**, 67(1933) & **20**,413(1937) 3)Franklin(1935), 54-5 4)InorgSynth **1**(1939) 74-7 & **2**(1946), 128-35

Nitramide or Nitroxylamide, $O_2N.NH_2$, mw 62.03, N 45.17%. Wh solid, mp 72-5° with decompn; puffs off on rapid heating. Sol in w (slowly dec) and in common solvents, except petr ether. Was first, prepd in 1890 by Mathieu-Plessy but not properly identified. Thiele & Lachman prepd it in 1894(Refs 1,2 & 3) from nitrourethane, $O_2N.NHCOO.C_2H_5$, and described its props. Since then nitramide was prepd by various investigators, mostly by hydrolysis and decarboxylation of potassium –N– nitrocarbamate (Ref 4,5&6). Only small quantities should be prepd at a time and kept in a desiccator placed in a refrigerator because the compd is unstable, although it does not expl at ord temp

Thiele & Lachman (Refs 2 & 3) prepd alkali nitramidates and ammonium nitramidate; but could not analyze them because they existed for only a few seconds. A little more stable was *mercuric nitramidate*, $O_2N.NHg$. This was obtained on treating an aq soln of nitramide with a soln of mercuric nitrate in HNO_3
(See also Ref under Alkali Amides)
Refs: 1)J.Thiele & A. Lachman, Ber **27**, 1909-10(1894) 2)Ibid, Ann **288**, 297-302 (1895) 3)Mellor **8**(1928), 268-9 4)Franklin (1935), 154-9 5)InorgSynth **1**(1939), 68-73 6)Davis(1943), 369 7)Kirk & Othmer **1**(1947), 410 8)R.N.Jones & G.D.Thorn, CanJRes **27B**, 829(1949)(UV absorption spectra of nitramide, nitramines, etc)

Amides and Imides, Organic. An *organic amide* is a compt contg the monovalent $-CO.NH_2$ radical (eg, acetamide $CH_3.CO.NH_2$, oxamide $H_2N.CO.CO.NH_2$, benzamide $C_6H_5CONH_2$, etc). An *organic imide* is a compd of the general formula R_2NH in which R is an acyl radical such as CH_3CO-. Another type of imide is a compd derived from acid anhydrides in which one oxygen is replaced by NH, as for instance $C_2H_4<^{CO}_{CO}>NH$ (succinimide)

These compds are not explosive but some of their nitrocompds (*nitramides)* are. For instance, the silver salt of nitroderivatives of *acetamide* (qv) are explosive(See also Urea or Carbamide, Formamide and Polyamides)
Refs: 1)Beil **2**,26, 226, 545,(20, 100, 237) & [36, 509] 2) Beil **9**, 195, (96) & [163] 3) Hackh(1944),42 & 432 4)Kirk & Othmer **1** (1947),668-71 5)Hickinbottom(1948),68, 228-31 & 281-2 6)Text books on Organic Chemistry, such as Karrer, Fieser & Fieser, etc

Amides and Imides, Organic. Analytical Procedures. Some procedures are described in Organic Analysis, Interscience, NY, vol 3(1956)

and in Shriner, Fuson & Curtin(1956), 220-21

Amides and Imides, Organic, Nitrated are compds of the general formula $R.NH.NO_2$ and $R_2N.NO_2$, in which R is an acyl radical, such as CH_3CO-. Most of these compds are unstable and sensitive to moisture. A general description of organic nitramides may be found in the books on Organic Chemistry, such as Gilman vol 4(1953), 979-81. Some nitramides are described individually or as a class in the following refs:
Refs: 1)H.J.Backer, "Ahrens Sammlung Chemischer und Chemisch-technischer Vorträge" **18** 359-474(1912), translated by H. Stone, Ohio State University, Columbus, Ohio (Historical and prepn & props of numerous nitramides and nitramines) 2)R.Adams & C.S. Marvel, OSRD Rept **86**(1941) (Historical survey of nitramides, synthesis of intermediates and their nitration to the final products. Only few of the prepd nitramides were of interest as expls or as components of expl compns) 3)Series of papers by A.H.Lamberton, C. Holstead, J.Barrott, I.N.Denton & others, entitled "Nitramines and Nitramides" in JCS **1951**, 1282-89; **1952**, 1886-94; **1953**, 1998-2005; **1953**, 3341-49; **1953**, 3349-52; **1954**, 2391-95; **1955**, 1655-57; **1955**, 3997-4002

Amidines or Aminoimines are compds contg the monovalent radical $-C(:NH)NH_2$, as for instance, *acetamidine*, $CH_3C(:NH)NH_2$. They are cryst solids, sol in alc and eth. Some of these compds have been known for about ¾ of a century but they were not studied intensively until after WWII, when papers by P.Oxley, W.F.Short, M.W.Partridge, T.D. Robson, J.Miller and others appeared in the Journal of the Chemical Society, beginning in 1946. The compds described by the above authors were not investigated from the point of view of their explosiveness, but one might suspect that some of them are expl

General methods of prepn of amidines are given in Ref 3, p 671. With organic and inorganic acids, amidines form salts which are more stable than the amidines. For instance, *acetamidine* (ethaneamidine or α-amino-α-iminoethane) $CH_3.C(:NH).NH_2$, is an unstable solid melting ca 166-7° with decompn. Its *hydrochloride,* which is prepd by the action of HCl gas on acetonitrile in abs alc cooled in ice-salt, reacts with $AgNO_3$ to give *acetamide nitrate* $C_2H_6N_2+HNO_3$, crysts, mp 148° (dec). The *picrate of acetamidine* $C_2H_6N_2+C_6H_3N_3O_7$, mw 287.19, N 24.39%, mp 252°, is an explosive
(See also Aminoguanidine, Biguanide, Cyanoguanidine, Guanidine and Guanyl urea)
Refs: 1)Beil **2**,185,(85) & [183] 2)OrgSynth, Coll Vol **1**(1944), 5-7 3)A,J,Ewins & J.N. Ashley, USP 2,364,200(1944) 4)Kirk & Othmer **1**(1947), 671-2

4-Amidino-1-(nitrosoaminoamidino)-1-tetrazene. One of the names for Tetracene, also called 1-Guanyl-4-nitrosaminoguanyltetrazene

1-Amidino-3-nitrourea. See Nitroguanylurea under Guanylurea

Amidino-semicarbazide, 1-Nitr-. See 1-Ureido-3-nitroguanidine

Amidino-3-thio-semicarbazide, 1-Nitr-. See 1-Thio-ureido-3-nitroguanidine

Amidoazaurolic Acid. See Aminoazaurolic Acid

Amidocarbonic Acid. See Carbamic Acid or Aminoformic Acid

Amidogène. Two mixts were known under this name: a)A black-powder type expl patented in 1882 by Gemperlé, and manufd for some time in Switzerland, was prepd by mixing the following ingredients in moist condition: K nitrate 73, sulfur 10, bran or starch 8, charcoal 8 & Mg sulfate 1% b)A dynamite-type expl contained NG 70-75, AN 4-7, paraffin 3-10 & powdered charcoal or coal 18-13%. This mixt was hygroscopic and its NG exuded
Refs: 1)Cundill, MP **5**, 346(1892) 2)Daniel (1902), 20-21 3)Thorpe **4**(1940), 463

Amidoguanidine. See Aminoguanidine

Amidon (Poudre à l'), a blasting expl patented in 1884, consisted of black powder mixed with 2 to 5% of starch (Compare with "Starch Powder") *Ref:* Daniel(1902), 21

Amidon nitré(Fr). Nitrostarch

Amidotriazole of Thiele. See 3-Amino-asym-triazole

Amidpulver. See Amide Powder

Amilol. Same as Amylphthalate

Aminated (Aminized) Cellulose or Amino-cellulose. By attaching amino-groups to a cellulose molecule(such as cotton), products are obtained which react differently than the untreated celluloses. Due to the presence of amino groups the aminated celluloses combine with acidic substances, such as acid dyes, some flame-proofing agents, rot-resisting compounds,and acidic explosives, such as 2,4-dinitrochlorbenzene, 2,4,6-trinitrochlorbenzene, etc

A series of papers on amination of cellulose appeared after WW II in various journals (see Refs)

Note: It may be possible to form rapid-burning materials suitable for propellants, fuses, quick matches, etc by combining aminated cellulose with an oxidizer, eg, aminocellulose nitrate or perchlorate (See also Aminoethylcellulose)

Refs: 1)L.Vignon, CR **112**,487(1891) (Direct addition of the amino nitrogen to cellulose was reported but the results have not been reproducible, according to Ref 2) 2)T.S. Gardner, JPolymSci **1**,121 & 289(1946) 3) J.D.Guthrie, Textile Research J **17**,625(1947) 4)C.L.Hoffpauir & J.D.Guthrie, Ibid **20**,617 (1950) 5)W.A.Reeves & J.D.Guthrie, Ibid **23**,522(1953) 6)W.A.Reeves et al, Ibid **23**, 527(1953)

Aminated Cellulose Acetate Esters. Prepn and props of cellulose acetate esters contg amino nitrogen are described in a paper by T.S.Gardner, JPolymSci **1**,121-6(1946) and in some refs listed in that paper

Amination is the process of forming an amine (qv). This may be done by any one of the reactions described in textbooks on Organic Chemistry(see Ref 3). The most important method is the reduction of nitrocompds or cyanides. Other methods, such as the hydrolysis of nitriles, or the replacement of hydroxyl by an amino group, are not used commercially

The process of reduction of nitrocompds was discovered in 1842 by N.Zinin, a prof at the Univ of Kazan',Russia. He prepd aniline by reducing nitrobenzene with $(NH_4)_2S$. A less expensive reducing agent, Fe and dil acid, was proposed in 1854 by Béchamp. The reduction process by Fe and acid was used on a large scale by Perkin. With cheap aniline the synthetic dyestuff industry was born

Amination by reduction may be achieved by using the metals Fe, Sn, Zn (in acid or neutral solns), Zn or Fe (in strongly alkaline solns), Zn (in weak alk soln), sulfides, $FeSO_4$ & hydrosulfites (in alkaline solns), hydrogen in the presence of catalysts, etc. These methods are described in Refs 1,2 &4

The greatest industrial use of amination by reduction was made during WWII in Germany (mostly by IG Farbenindustrie), as well as in Gt Britain and the USA

Although the amination process does not produce any explosives, it is mentioned here because it can be used for transforming some discarded explosive nitrocompds into non-explosive amines, which might be useful as dye intermediates or other purposes. For instance, during WWII, some nitroxylenes were reduced to produce mixed xylidines for use with aviation gasoline. The reduction method can also be used for the harmless destruction of discarded explosives. For in, stance, at least one of the US Ordnance installations destroyed unwanted TNT by placing it in shallow basins dug in the ground and adding iron scrap in water acidified with sulfuric acid

Refs: 1)J.Werner, IEC, Sept 1947-1957, under "Unit Processes Review" 2)Kirk & Othmer **1**(1947),673-702 3)Hickinbottom(1948), 105, 281-4, 344-50 & 402-4 4)Groggins(1958), 129-203 & 388-485

AMINES

Amines are compounds derived from ammonia by substituting hydrocarbon radicals (R) for

hydrogen. They may be primary RNH_2, secondary $\begin{matrix}R\\R\end{matrix}\!>\!NH$ (called also imines), tertiary $\begin{matrix}R\\R\end{matrix}\!>\!N\text{-}R$ or quaternary $\begin{matrix}R\\R\end{matrix}\!>\!N\!<\!\begin{matrix}R\\OH\end{matrix}$ amines. Their prepn and props are given in Organic Chemistry books, Chemical Encyclopedias and Chemical Dictionaries.
(See Refs 1,2,3a & 5)

Amines have been used for the following purposes related to expls,propellants, pyrotechnic compositions and fuels: a)Stabilizers for NC propellants(eg, diphenylamine, nitrodiphenylamine and phenyl-β-naphthylamine, the last examined in France(Ref 3) b)Sensitizers for some expl and propellent compns (Ref 4 and addnl ref d) c)Smoke-producing compds (Addnl Ref d) d)Liquid fuels for rockets in conjunction with strong oxidizers, such as nitric acid(some of these are hypergolic) (Refs 6 & 8 and addnl refs e,g,i,k & l) e)Additives to gasoline and other motor fuels (Addnl refs a,b,c & f)

Refs: 1)Kirk & Othmer **1**,702-17 2)Hickinbottom (1948),105, 281-4 & 292-332 3)L.Médard, MP **32**,305-7(1950) (Phenyl-β-naphthylamine as a stabilizer for smokeless propellants) 3a)Degering(1950),199-221 & 292-321 4)H. Maisner, USP 2,712,989(1955) & CA **49**,14325-6(1955) (An organic amine was proposed as an addn to nitroparaffin-gel rocket propellants to make them expl, eg, a rocket propellant consisting of $MeNO_3$ blended with 10-50% NC and contg Cr acetylacetonate up to 3% by wt became an expl after incorporating some amine) 5)Clark & Hawley(1957), 68-9 6)L.R.Rapp & M.P.Strier, JetPropn **27**,401-4(1957) (The effect of chemical structure on the hypergolic ignition of amine fuels) 7)V.Migrdichian, Organic Synthesis, Vols 1 & 2, Reinhold, NY (1957) 8)R.Fuchs, Explosivst **1958**, 89-97 (Aliphatic and aromatic primary, secondary and tertiary amines were examined from the point of view of their hypergolicity when used with strong nitric acid as an oxidizer. Various devices for such investigations are discussed)

Addnl Refs on Amines: a)SODC, BritP 530, 597 (1940) & CA **36**, 893(1942) (Secondary amines as blending agents in high octane number motor fuels) b)SODC, BritP 559, 051(1944) & CA **39**,4750(1945) (Primary or secondary isoalkylamines in which the isoalkyl groups contain from 4 to 5 carbon atoms, as antiknock blending agents for high octane motor fuels) c)A.G.Mazurkiewicz & V.G. Oberholzer, JInstPetroleum **32**,685(1946) & CA **41**,5281(1947) (Aromatic amines as antiknock addns to motor fuels) d)H.E.Britt, USP 2,420,416(1947) & CA **41**,4874(1947) (An exceedingly dense white smoke is produced when a liq amine such as $C_2H_4(NH_2)_2$ is treated with a volatile org acid, such as CH_3COOH) e)V.L.King, USP 2,474,183(1949) & CA **43**,6811(1949) (Rocket propellants are obtained by treating alkyl-substituted mononuclear aromatic amines with 95% or stronger HNO_3. Such mixts ignite spontaneously even when the amines are dissolved to the extent of 20% by vol in low-boiling hydrocarbons such as gasoline or light fuel oils. Hydrocarbon solns remain miscible and do not freeze at temps of the order –40°) f)A. Beranger, FrP 942,418(1949) & Ca **45**,1337 (1951) (The octane number of gasoline may be increased by adding to it a mixt of o-toluidine, triethanolamine, $Fe(CO)_5$ and cholesterol) g)P.F.Winternitz & D.Horvitz, JAmRocket Soc No **85**,51-67(1951) & CA **45**, 8771(1951) (Amines are superior to the corresponding alcs as fuels in rocket propellants) h)VGFAG, GerP 841,000(1952) & CA **47**,3867(1952) (Amines for prepn of surface active substances) i)J.D.Clark,Ordn **36**,661-3(1952) & CA **48**,11062(1954) (Amines as fuels for liq rocket propellants) j)H. Maisner, USP 2,690,964(1954) & CA **49**,618 (1955) (Amines may be added as sensitizers to gelled liq nitroparaffins used in expls or propellants, eg, nitromethane contg 5-75% NC is sensitized by the addn of some amine. Cr acetylacetonate may be incorporated to obtain easier ignition) k)L.R.Rapp & M.P. Strier, Jet Propulsion **27**, 401-4(1957) (A systematic study of the relationship betw

chem structure and hypergolic ignition of numerous amine fuels with WFNA oxidizer was conducted at Reaction Motors, Inc, Denville, NJ in order to determine the suitability of such amines as rocket fuels) 1) Warren (1958),,25 (Amines as rocket fuels. The most important compds are aniline and unsym-dimethylhydrazine, abbr UDMH. Less important are methylamine and diethylenetriamine)

Amines and Imines, Analytical Procedures are described under some individual compds (such as aniline) and in the following refs: 1)Siggia(1949), 65-73 2)Org Analysis **3** (1956), 129-201 3)Shriner, Fuson & Curtin (1956), 222-4

Amine, Catalyzed Nitration. A series of papers, on this subject, were published in CanJRes **26B** (1948) by the following investigators: 1)W.J.Chute et al, pp 89-103 (Dinitroxydiethylnitramine) 2)G.E.Dunn et al, pp 104-113 (Relative Basicity of Secondary Amines in Acetic Acid) 3)W.J.Chute et al, pp 114-137 (The Ease of Nitration Among Aliphatic Secondary Amines) 4)J.C.MacKenzie et al, pp 138-153 (The Role of Electropositive Chlorine in the Nitration of Lysidine) 5) G.S.Myers & G.F.Wright, pp 257-270 (The Nitration of Aliphatic Dialkylchloramines) 6)K.K.Carroll & G.F.Wright, pp 271-283 (The Nitration of Di-n-octylamine) 7)C.N.Smart & G.F.Wright, pp 284-293 (A New Method of Preparation of Primary Nitramines) 8)T.Connor et al, pp 294-308 (The Medium Used in Nitration of Secondary Aliphatic Amines) (See also "Amines, Nitrated and Nitrited")

Amines, Complexes. A number of amine complexes with cupric chlorates, perchlorates and bromates were examined before WWII in France and described in:

Refs: 1)J.Amiel, CR **199**, 51-3(1934) & CA **28**, 5361(1934) 2)J.Amiel, CR **200**, 672-4 (1935) & CA **29**, 2469-70(1935) (Compare with Ammines)

Amines, Condensation With Aromatic Chlorine Compounds. Amines can be condensed with aromatic Cl derivs and the resulting compds nitrated to explosives. For instance, dinitrochlorobenzene reacts with 2 equivalents of aniline to form dinitrodiphenylamine and aniline hydrochloride. The DNDPhA may be nitrated to tetra- and hexanitrodiphenylamines (Ref,p 185). In some cases an expl can be obtained in one operation without further nitration. For instance, the action of potassium methylnitramine on picryl chloride produces the explosive 2,4,6-trinitrophenylmethylnitramine, known as tetryl:

$$(O_2N)_3C_6H_2Cl + KN{<}^{CH_3}_{NO_2} \rightarrow (O_2N)_3C_6H_2N{<}^{CH_3}_{NO_2} + KCl$$

(Ref,p 175)

Ref: Davis(1943),175 & 185

Amines Halogenated. See Halogenated Amines

Amines Nitrated and Nitrited. The various types of amines have been converted into derivs contg oxynitrogen groups. In some cases the desired derivs can be produced directly from the amine by the action of nitric or nitrous acids or their anhydrides but more often indirect syntheses are required. Many amine derivs contg oxynitrogen groups are explosive. If a hydrogen of an NH_2 group is substituted by an NO or an NO_2 group the resulting compds are known correspondingly as *nitrosamines* (or N-nitrosamines), $R_2N.NO$ and *nitramines*(or N-nitramines), $R.NH.NO_2$. There are also *nitrosoamines*(or C-nitrosoamines) and *nitroamines*(or C-nitroamines) in which the NO and NO_2 groups are attached to C atoms, eg, $ON.C_6H_4.NH_2$ and $O_2N.C_6H_4.NH_2$. The corresponding derivs of secondary amines are called *nitrosimines, nitrimines, nitrosoimines* and *nitroimines*

In *amine nitrates*, the HNO_3 group is connected to a molecule of an amine to form an addition salt, such as $R.NH_2.HNO_3$ or $R_2NH.HNO_3$. The term amine nitrate is also applied to amines contg nitroxy(ONO_2) groups, such as $O_2NO.CH_2.R.NH_2$. In order to distinguish between nitrates and nitrate esters, the amines contg ONO_2 groups are called *nitroxyamines*

Some nitrated and nitrited amines may be

combinations of the above types, such as nitraminonitrates, nitraminonitro compds, nitraminonitroxy compds, nitronitrosamines, etc *Note:* There is frequently a lack of clarity in the nomenclature of nitrated compounds used by various authors. For instance, eight papers in the JCS **1949,** pp 1631-58 (see Addnl Refs on Amines, Nitrated) are entitled "Studies on Nitroamines", but they all actually deal with Nitramines

The literature on the various types and individual oxynitrogen derivs (expl and non-expl) was considerable even before WW II. Since many nitrated amines are expl (the nitro, nitramino and nitrate groups are explosophores), WW II served as a strong stimulus for a more intensive study of them, so that today the published material in this field is so extensive that a listing of more than a small number of the most significant refs is not practicable in this dictionary. Some of the more general articles on nitrated amines are given below, while other articles are given as Addnl Refs on Amines, Nitrated

Individual amines and their nitrated derivs used in expl, propellent and pyrotechnic compns are described separately under their own names, such as: cyclonite, nitroguanidine, tetryl, etc

Attention is also directed to the section on "Nitration" edited by W.deC.Crater in the annual series entitled "Unit Processes Reviews" in the September issues of IEC beginning in 1948 and ending in 1955. The review was resumed in 1957 by W.R.Tomlinson,Jr

Inasmuch as the nitramines are the most important of the nitrated amines, it would be appropriate to say a few words about them. According to Backer(Ref 1) some nitramines were prepd as early as 1869 by Griess,but they were not identified as such until much later when Zincke repeated some of Griess' work. In 1877, Mertens of Holland prepd an explosive by nitration of dimethylaniline but he did not establish its structure. In 1883, van Romburgh, also of Holland, proved that the compd prepd by Mertens was a nitramine. After this, van Romburgh worked with aromatic nitramines while Franchimont(first alone and then with Klobbie and van Erp) studied aliphatic nitramines. Considerable work was done, beginning about 1890, in Germany and in Italy. In later years, work on nitramines was conducted not only in Holland and Germany but in other countries as well. Great advances were made during and after WW II, particularly in the USA (where most of the work is still classified) in Canada(where many papers have been published in Canad JRes and JCS) and in Russia

Aromatic nitramines are much easier to prepare than the aliphatic nitramines. For this, the aromatic amine can be treated with concd nitric acid either alone or in the presence of a dehydrating agent such as sulfuric acid or acetic acid-acetic anhydride(Ref 2, p 320)

In the prepn of aliphatic nitramines, the indirect methods are preferable, as for instance, dehydration of amine nitrates with acetic anhydride. The presence of a chloride ion acts as a catalyst

For more information on the prepn of nitramines, see Refs 3 & 4 and individual compds listed under corresponding amines (See also Amines, Catalyzed Nitration)

Refs: 1)H.J.Backer, Ahrens Sammlung Chemischer und Chemisch-Technisher Vorträge **18**,359-474(1912) (Translated by H.Stone of Ohio State Univ) (General review of nitramine chemistry) 2)Hickinbottom(1948) 3)A.H. Lamberton, Quart Revs **5**, 75-98(1951) ("Some Aspects of the Chemistry of Nitramines") 4)Gilman**4**(1953)

Addnl Refs on Amines, Nitrated: a)A.P.N. Franchimont,Rec **16,**226-8(1897) (Contribution to the knowledge of aliphatic nitramines) b) J.Pinnow, Ber **30**,833-43(1897) (Aromatic nitramines and nitrosamines) c)E.Bamberger, Ber **30**,1248-63(1897) (Alkylnitramines)(earlier refs are given) d)G.R.Clemo & J.M.Smith, JCS **1928**,2414-22 & CA **23**, 117-18 (1929) (Nitration of substituted tertiary aromatic amines. One of thé compds prepd was tetryl) e)Dynamit A-G,BritP 384,966(1931) (Aliphatic

nitrated mono- and polyamines, such as methylaminenitrate and ethylenediaminedinitrate, are melted in mixts with AN to be cast in shells, etc) f)P.Naoúm & R. Sommerfeld, USP 1,968,158(1934) (Nitrates of aliphatic nitramines used in cast AN explosives) g)A.E. Shouten, Rec **56**,541-561(1937) {Prepn and props of some explosive nitramines, such as 1,2-bis-[(2,4,6-trinitrophenyl)-nitramino]-ethane and 1,2-bis-[N-(4-methyl-2,6-dinitrophenyl)-nitramino]-ethane} h)W.L.C.Veer, Rec **57**,989–1015 (1938) {Prepn and props of several explosive nitramines, such as 1,3-bis(2',4',6'-trinitrophenyl-nitramino)-propane; 1,3-bis [N-(4'-methyl-2',6'-dinitrophenyl)-nitramino]-propane; 1,3-bis[N-(4'-chloro(or bromo)-2',6'-dinitrophenyl)-nitramino]-propane; 1,3-bis[N-(5', chloro(or bromo)-2',4',6'-trinitrophenyl)-nitramino]-propane; etc} i)K.F. Waldkötter, Rec **57**,1294–1310(1938) {Prepn and props of some explosive nitramines, such as: N-(2,4,6-trinitrophenyl)-N-nitro-β-aminoethyl nitrate; N-[4-chloro(or bromo)-2,6-dinitrophenyl]-N-nitro-β-aminoethyl nitrate; N-[5-chloro(or bromo)-2,4-dinitrophenyl]-N-nitro-β-aminoethyl nitrate; etc} j)P.P. Shorygin et al,Zh ObshKhim **8**,986–90(1938) & CA **33**,3781(1939) (Nitration of aromatic amines) k)E.Macciotta et al, Series of articles in Gazz and other Italian journals beginning 1930 and ending 1947[See CA **24**, 4279–80(1930); **26**,1585–6(1932); **27**,4528 (1933); **31**,3889 & 4965(1937); **33**,8592(1939); **36**,1593–5(1942) and **41**,4115(1947)] l)G. Römer, Report on Explosives, PBL Rept 85, 160(1946) (Aliphatic nitramines 1,7-dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane and 1,9-dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane obtained as by-products in the manuf of RDX)[See PATR 2510(1958) under Aliphatic Nitramines of WW II and Hexogen, E-Salz and KA-Salz Processes] m)Collective, Series of papers on the prepn and props of nitramines in CanJRes **26B**,pp 89–103, 114–37, 257–70, 271–80, 284–93 and 294–308(1948) & CA **42**,4918,4919,5843 and 5844(1948) n)L.Ilеger,Przemysl Chem **4**, 522–4(1948) & CA **43**,4218(1949) (A review on the prepn of nitroamines) o)Collective, CanJ Res **27B**,218–37,462–8, 469–74, 489–502, 503–19 & 520–44(1949) & CA **43**,8354–5, 9072–75(1949) (Series of papers on the prepn of some explosive nitramines by nitrolysis of hexamethylenetetramine) p)Collective, JCS **1949**,1631–58 & CA **44**,1410–13(1950) (Eight papers on the prepn and props of several nitramines, called in these papers "nitroamines". The compds in these papers which contain about or more than 14% nitro- or nitroxynitrogen are listed individually in this dictionary because they may be potential components of explosive propellent or pyrotechnic compns) q)HACSIR (Canada), BritP 615,419 & 615,793(1949) (Nitramines and their preparation, mostly polymethylene polynitramines, eg 3,7–dinitropentamethylenetetramine, cyclotetramethylenetetranitramine, cyclotrimethylenetrinitramine, 1,9-diacetoxypentamethylene-2,4,6,8-tetranitramine, 1,9-dinitroxypentamethylene-2,4,6,8-tetranitramine, etc) r)G.F.Wright & W.J.Chute, USP 2,461,582 and 2,462,052 (1949) & CA **43**,4286(1949)(Nitramines) s) A.T.Blomquist & F.T.Fiedorek, US P 2,481,283(1949) & CA **44**,4925(1950)(Prepn and props of some nitroalkylnitramines) t)A.T.Blomquist & F.T.Fiedorek, US P 2,485,855(1949) & CA **44**,3516(1950)(Prepn and props of some nitramines and nitrated aminoalcohols) u)J.A.Harpham et al, JACS **72**,341–3(1950) & CA **45**,1048–9(1951) (Prepn and props of linear secondary polynitramines, some of which are explosive, eg (1,8-dicyclohexylnitramino-3,6-dinitro-3,6-diazaoctane) v)K.W. & W.J. Dunning,JCS **1950**,2920–28(1950) & CA **45**,6642–44(1951) (Methylenenitramines) w)L.Berman et al, CanJChem **29**,767–76(1951) & CA **46**,2084 (1952)(Nitrolysis of hexamethylenetetramine) x)W.E.Bachmann et al, JACS **73**,2769–73 (1951) & CA **46**,2084(1952)(Cyclic and linear nitramines formed by nitrolysis of hexamine) y)Collective, JCS **1951**,1282–89 & CA **46**,

2513(1952); JCS **1952**,1886–94 & CA **47**, 1044(1953); JCS **1953**,1998–2005 & CA **48**, 10540(1954); JCS **1953**,3341–49 & CA **49**, 8311(1955); JCS **1953**,3349–52 & CA **49**, 8311(1955); JCS **1954**,2391–5 & CA **49**, 9511(1955); JCS **1955**,1655–7 & CA **50**,5514 (1956); JCS **1955**,3997–4002 & CA **50**,7715(1956) (Ptepn and props of nitramines, some of which are explosive) z)G.F.Wright & W.J. Chute, CanP 479,928(1952)& CA **50**;8747 (1956) (Conversion of secondary amines to nitramines) aa)J.M.Patterson, Northwestern Univ, Evanston, Ill, Univ Microfilms Pub No **6234**(77pp), Ann Arbor, Mich; Dissertation Abstrs **13**,990–1(1953) & CA **48**, 3930(1954) (Prepn and chemistry of hydroxylamino-and aminonitrocompds) ab)G.S. Salyamon et al, Sbornik Statei Obshchei Khimii(Russia) **2**,1315–24(1953) & CA **49**, 4554(1955) (Structure of aromatic nitramines) ac)G.F.Wright & W.J.Chute, USP 2,678,927 (1954) & CA **49**,7606–7(1955) (Prepn and props of expl nitramines, such as cyclotetramethylenetetranitramine) ad)J.Cason,Jr, USP 2,686,804(1954) & CA **49**,2075(1955) [Secondary nitramines prepd by nitrating secondary amines or their nitrates are proposed for use as explosives, eg Bis(2–nitroxy-ethyl)-nitramine] ae)T.M.Cawthon,Jr, Princeton Univ, Princeton, N J, Univ Microfilms, Publ No **13675**,103pp, Ann Arbor, Mich; Dissertation Abstrs **16**,247–8(1956) & CA **50**,7593(1956) (Kinetics and mechanism of thermal decompn of amine nitrates) af) OSRD Reports on Nitramines: **65**(1941), **158**(1941), **159**(1941), **393**(1942), **540**(1942), **560**(1942), **800**(1942), **819**(1942), **820**(1942), **907**(1942), **915**(1942), **950**(1942), **979**(1942), **1044**(1942), **1089**(1942), **1134**(1942), **1234** (1943), **1711**(1943), **1733**(1943), **1734**(1943), **1803**(1943), **2054**(1943), **3567**(1944), **4099** (1945), **4134**(1944), **5186**(1945), **5398**1945), **5943**(1945), **5944**(1945), **5945**(1945), **6126** (1945), & **6628**(1945) ag)Office of Technical Information Reports: PBL 31089(1942) & PBL 18,867(1944) ah)Picatinny Arsenal Technical Reports: **414**(1933), **1166**(1942) & **1177**(1942)

Amines, Nitrated and Nitrited, Analytical Procedures. Many of the procedures used for the analytical determination of amines are applicable to nitramines and nitrosamines(see Refs under Amines). Detn of the nitro-group in nitramines is given in Org Analysis **2**(1954),78,80, & 85 *Addnl Refs:* a)I.V.Grachev, ZavodLab *12*,434 (1946) & CA **41**,1177(1947) (Direct detn of nitrosamines) b)C.J.Ovenston & C.A.Parker, JSCI **66**, 394–5(1947) & CA **42**,2771(1948) (Detn of nitrosamine content of propellants stabilized with symdiethyldiphenyl urea) c)K. Kohlrausch, ActaPhys-Austriaca **1**,292(1948) & CA **42**,6665(1948) (Raman spectra of nitramines and nitrosamines) d)C.A.Parker, JSCI **67**,434(1948) & CA **43**,3617 (1949) (Chromatographic separation of N-nitroso-N-ethylamine and of some nitramines from propellants) e)S.I.Burmistrov, ZhAnalKhim **5**,119–22 (1950) & CA **44**,4828–9(1950) (Identification of primary nitramines. Spot test colorations with various reagents are given for 45 nitramines) (Translation RJ–44 of the Associated Technical Services, PO Box 271, East Orance, NJ) f)E.W. Malmberg et al, Anal Chem **25**,901–7(1953) & CA **47**,12095(1953) (Detn of nitramine impurities in RDX by a chromatographic method)

Amine Nitrates are compds of the general formulae $R \cdot NH_2 \cdot HNO_3$, $R_2NH \cdot HNO_3$ or $R_3N \cdot HNO_3$, where R, R_1 and R_2 may be hydrocarbon-, alcohol, or other radicals. Those of them which are expl are listed here individually, such as aminomethane-, aminoethane-, aminoethanol- nitrates, etc *Refs:* 1)A.Franchimont, Rec **2**,329(1883) 2)P. van Romburgh, Rec **5**,246(1886) 3)J.B.Willis, TrFaradSoc **43**,97(1947) & CA **41**,5008(1947) 5)T.L.Cottrell & J.E.Gill, JCS **1951**,1798–1800 & CA **45**,10028(1951)

Amine Nitrites are compds of general formulae $R \cdot NH_2 \cdot HNO_2$, etc *Refs:* 1)J.K.Wolfe & K.L.Temple, JACS **70**, 1414(1948) 2)Ibid,USP 2,635,116(1953) & CA **48**, 7048(1954)

Amine Peroxides as Explosives. Some amines may yield expl peroxides. For instance, hexamethylenetetramine when treated with hydrogen peroxide in the presence of an organic acid(citric) which combines with the liberated NH_3, forms *hexamethylenetriperoxidediamine*(qv) (Ref 1). Schiff's bases, ammoniacal aldehydes or their

derivs form peroxides in the presence of an oxidizing agent. Thus, tricrotonylidenetetramine treated with H_2O_2 in the presence of malonic (or lactic) acid and mineral or organic salts, yields *tricrotonylidenetriperoxide tetramine*, $H_2N \cdot R \cdot CH \cdot R \cdot NH \cdot R \cdot NH_2$ where R= MeCH:CH·O_2·CH= This compd may be used in detonators and primers (Ref 2)

Refs: 1)Davis(1943),451 2)SFMCTG, FrP893,942 (1944) & CA **47**,8373(1953)

Amine Picrates. Molecular combinations of picric acid with various amines were prepd and examined before WW II in Rumania. Some of these complexes are explosive and are described under individual amines

Refs: 1)R.Rascanu, AnnSciUnivJassy **25** (,395 423(1939) (In French) (58 refs) 2)Ibid **26** (,3–40 (1940) & CA **34**,394 & 4385(1940)

Amines as Rocket Fuels. See Refs 6 & 8 and Addnl Refs e,g,i,k & l under Amines and Imines

Aminized Cellulose. See Aminated Cellulose

Aminoacetic Acid; Aminoethanoic Acid; Glycine or Glycocoll (Gelatin Sugar or Glycocin) (Leim zucker or Leimsüss in Ger and Sucre de gélatine, in Fr) $H_2N \cdot CH_2 \cdot COOH$, mw 75.07, N 18.66%. Col crysts, mp 232–6° (decomp), d 1.161. Very sol in w, sl sol in alc, insol in eth. Was obtained in 1820 by Braconnot by treating isinglass (fish glue) with H_2SO_4(Ref 2). Can also be prepd by the action of concd NH_4OH on monochloroacetic acid as well as by alkaline hydrolysis of gelatine, etc

Was used by Burstenberger(Ref 3,p 89) for the prepn of an expl compn in which cellulose, dried mushrooms, etc were treated with glycine and then impregnated with NG

Refs: 1)Beil **4**,333,(462) & [771] 2)E.N.Hornsford, Ann **60**,1–57(1846) (Glycocoll and some derivatives) 3)Daniel(1902),89 4)T.Cocking, IndChemist **13**,137–8(1937) (Manuf, props and methods of identification of glycine)

Aminoacetic Acid Nitrate or Glycine Nitrate, $H_2N \cdot CH_2 \cdot COOH + HNO_3$, mw 138.08, N 20.29%, OB to CO_2 –23.2%, OB to CO 0%. Rhombic plates or ndls, mp ca 145° with evolution of gas(Ref 3). Was prepd in 1820 by Braconnot by the action of nitric acid on glycine (Ref 2)

According to Thorpe(Ref 4), glycine nitrate is a powerful expl resembling PA in its props. On deton, it evolves a considerable amt of toxic carbon monoxide, as is shown by the equation: $H_2N \cdot CH_2 \cdot COOH \cdot HNO_3 = 2CO + 3H_2O + N_2$ It has been used with good results in some propellant compns

Refs: 1)Beil **4**,340 2)E.N.Hornsford, Ann **60**, 26(1846) 3)A.P.N.Franchimont, Rec **2**,339 (1883) 4)Thorpe **2**(old edit 1917),467

Nitraminoacetic Acid or Nitroglycine, $O_2N \cdot HN \cdot CH_2 \cdot COOH$, mw 120.07, N 23.33%. Ndls, mp 103–4° (decomp). Sol in alc, eth & acet; diff sol in chlf, benz, ligroin and cold w. Was obtained from its ammonium salt, as described in Ref 2. Some of its salts are expl, eg $Ag_2C_2H_2N_2O_4$

Refs: 1)Beil **4**,575 2)A.Hantzsch & W.Metcalf, Ber **29**,1684–5(1896)

AMINOACETOPHENONES AND DERIVATIVES

Aminoacetophenones; Acetylphenylamines or Aminophenacyls, $H_2N.C_6H_4.CO.CH_3$. Three isomers are described in Beil **14**, 41,45,46, (364,365,366) & [28,30]

Aminoacetophenones, Azido Derivatives $C_8H_8N_4O$, mw 176.18, N 31.80%. One isomer *o-aminoazido acetophenone or o-aminophenacylazide*, $H_2N.C_6H_4.CO.CH_2.N_3$, crysts, mp 66-70 is described by J.H.Bayer & D.Straw, JACS **75**, 1642 & 2684-5(1953). Diazotization of this comp and treatment with $NaNO_2$ in dil acid gave expl *o-azidophenacyl azide*

Aminoacetophenone, Diazido Derivative, $C_8H_7N_7O$ was not found in Beil or CA through 1956

Mononitraminoacetophenones, $C_8H_8N_2O_3$. The following isomer is described in Beil:

2-Nitraminoacetophenone or 2-Acetylphenylnitramine, $(O_2N)HN.C_6H_4.CO.CH_3$, crysts, mp 103-4°; was prepd from methylanthranil, Na nitrite and HCl

Its *silver salt*, $(O_2N)AgN \cdot C_6H_4 \cdot CO \cdot CH_3$, wh voluminous ppt, deflagrates suddenly on heating with evoln of yel vapor

Refs: Beil **16**,(401) 2)E.Bamberger, Ber **48**, 548 & 557–8(1915) & CA **9**, 1780(1915)

Dinitroaminoacetophenone and higher nitrated derivs were not found described in Beil or CA through 1956

Aminoacridines, $C_{13}H_{10}N_2$, mw 194.23, N 14.48%. Three isomers are described in the literature. No azido- or diazido-compds were found in Beil or CA through 1956. Mononitro-, dinitro- and trinitro-acridines are described in the literature but none of them are reported to be expl. If *tetranitroamino-acridine* was known it very likely would be an expl because its NO_2 nitrogen content is ca 15%. This compd however, is not listed in Beil or CA through 1956

Following are some refs on amino-acridine, aminoacridine nitrate and mono-, di- & trinitro-aminoacridines

Refs: 1)Beil **21**,[280, 282, 283] 2)Beil **22**,462–3,(643) & [376–8] 3)OrgSynth **22**(1942),6 4)J.B. Willis, TrFaradSoc **43**,97–102(1947) 5)A.Hampton & D.Magrath, JCS **1949**,1008–11 & CA **44**,633 (1950)

Aminoalcohols (Hydroxyamines; Alcamines or Alkanolamines) contain both the amino- and the hydroxyl groups attached to different C atoms, usually adjacent, such as: monoethanolamine or 2-aminoethanol $HOCH_2CH_2NH_2$, diethanolamine or 2,2'-iminodiethanol $(HOCH_2CH_2)_2NH$ and triethanolamine $(HOCH_2CH_2)_3N$. Many amino alcohols and their derivs are important products of commerce and some of them serve for the prepn of expls

Aminoalcohols, Nitrated and Nitrited. Similar to the amines, aminoalcohols may be converted into various types of derivatives contg oxynitro groups. Many of these comps are expl, as for instance, diethanolnitraminedinitrate $(O_2NO \cdot CH_2 \cdot CH_2)_2 : N \cdot NO_2$; diethanolnitrosaminedinitrate $(O_2NO \cdot CH_2 \cdot CH_2)_2 : N \cdot NO$; nitroxyethyleneaminenitrate, $O_2NO \cdot CH_2 \cdot CH_2 \cdot NH_2 \cdot HNO_3$; 1,9-dinitroxypentamethylene-2,4,6,8-tetranitramine, $O_2NO \cdot CH_2 \cdot N(NO_2) \cdot CH_2 \cdot N(NO_2)_2 \cdot CH_2 \cdot N(NO_2) \cdot CH_2 \cdot N(NO_2) \cdot CH_2 \cdot ONO_2$. The individual nitrated derivs are described under the parent aminoalcohol

Refs: 1)Sidgwick(1942),41–3 2)Kirk & Othmer **1**(1947),729 3)Hickinbottom (1948),158–60

Addnl Refs: a)Dynamit A–G, GerP 514,955(1929) & CA **25**,2739(1931) (Prepn of monoethanolamine dinitrate from monoethanolamine or its mononitrate is described) b)Ibid, GerP 516,284(1929) & CA **25**,3362(1931) (Modification of the above patent) c)Ibid, BritP 350,293(1929) & CA **26**,5423(1932) (Prepn of mono- and dinitrates of monoethanolamine. Mention is also made of triethanolamine tetranitrate) d)Ibid, BritP 357,581(1930) & CA **26**,6141(1932) (Prepn of diethanolamine dinitrate) e)Ibid, BritP 358,157 & CA **26**,6141(1932) [Nitration of aliphatic amino alcohols for use as or in expls. For instance, dihydroxypropylamine, dipropanolamine, n-butylmono-ethanolamine, etc were nitrated by the method described in BritP 357,581(1930) & CA **26**,6141(1932)] f)J.Barbière, BullFr[5], **7**,621–6(1940) & CA **36**,1913 (1942) (Nitration of aromatic alkylaminoalcohols) g)R.C.Elderfield, OSRD Rept 158(1941) (Expls from hydroxy- and aminocompds) h)A.T.Blomquist, OSRD Rept 4134(1944) (Nitramine-nitrates from aminoalcohols) i)J.Barbière, BullFr[5], **11**,470–80 (1944) & CA **40**,2110(1946) (Nitric acid esterification and nitration of aminoalcohols) j)B.L.Zenitz & W.H.Hartung, JOC **11**,444–53(1946) & CA **41**, 420(1947) (Aminoalcohols) k)P.Fréon & S.Ser, CR **226**,1098–9(1948) & CA **42**,6770(1948) (Prepn of aminoalcohols with a tertiary alcohol group) l)A.T.Blomquist & F.T.Fiedorek, USP 2,481,283 (1949) & 2,485,855(1949); CA **44**,3516 & 4925–6 (1950) (Prepn and props of some nitrated amines and aminoalcohols suitable for use in expls and as plasticizers for NC in propellants) m)V.E. Haudry, USP 2,497,548(1950) & CA **44**,4494(1950) (Aminoalcohols) n)S.A.Ballard & B.P.Geyer, USP 2,513,132(1950) & CA **44**,10732(1950) (Amino-alcohols) o)R.E.Holmen & D.D.Carroll, JACS **73**, 1859–60(1951) & CA **46**,428–9(1952) (Synthesis of some aminoalcohols) p)A.W.D.Avison, JApplChem (London) **1**,469–72(1951) & CA **46**,11109–10(1952) (Application of $LiAlH_4$ to the prepn of some amino-alcohols) r)A.T.Blomquist & F.T.Fiedorek, USP 2,678,946(1954) & CA **49**,4704(1955) (Nitroxyl-alkylnitramines) s)J.R.Johnson, A.T.Blomquist & F.T.Fiedorek, USP 2,683,165(1954) & CA **49**,7590 (1955) (Hydroxyalkyl, alkylenedinitramines and

their nitrate esters). Some nitrate esters are suitable as nonvolatile NC gelatinizers

Aminoalcohols, Aliphatic, Nitrated Derivatives, investigated during WW II under the direction of Prof A.T.Blomquist included: a)*N-(β-Nitroxyethyl)nitramine*, designated as *NENA* b)*N-(β-Nitroxyethyl)methylnitramine*, designated as *MeNENA* c)*N-(β-Nitroxyethyl) ethylnitramine*, designated as *EtNENA* d)*N-(β-Nitroxypropyl)methylnitramine*, designated as *Me_2NENA* e)*N-(β-Nitroxypropyl)nitramine*, designated as *IsoMeNENA* f)*Dinitraminoisopropyl Nitrate* g)*N,N-Bis(β-nitroxyethyl)nitramine*, designated as *DINA* h)*N,N-Bis(β-nitroxyethyl) ethylenedinitramine* and i)*N,N-Bis(β-nitroxypropyl) nitramine*

All of these compds, with the exception of a), e) and f), were prepd by the catalyzed Bamberger reaction. This reaction, which appears to be generally applicable to the prepn of secondary nitramines, is as follows:

$$R_2NH_2^+NO_3^- \xrightarrow[Cl]{AC_2O} R_2NNO_2 + H_2O$$

Ref: A.T.Blomquist & F.T.Fiedorek, OSRD Rept 4134(PB 18867)(1944),pp 28–9 (Several refs are given in the report together with the description of each of the above compds)

Aminoalkylguanidines and Alkylaminoguanidines. Aminoalkylguanidines are compds in which an amino-group has replaced a H atom of the alkyl group of an alkylguanidine(eg, aminomethylguanidine $H_2NC(:NH)NH\cdot CH_2NH_2$, aminoethylguanidine, etc), whereas alkylaminoguanidines are compds in which one or more alkyl-groups have replaced a H atom of the amine group of an aminoguanidine(eg, methylaminoguanidine, $[H_2N\cdot C(:NH)NHCH_3]$

Since both types of substituted guanidines are described together in many papers, the refs given in this work in most cases include both types

It should be noted that derivs contg one or two alkyls of low molecular weight(such as methyl-, ethyl-, propyl-, etc) are compds of high nitrogen content and may be of interest as components of propellants and expls. Their nitrated and/or nitrited derivs would be of even more interest. The individual explosive derivs are described under the parent compd

Below are some refs to alkylamino- and aminoalkyl-guanidines:

Refs: 1)E.Strack,ZPhysiolChem **180**,198(1929) & CA **23**,1880(1929) (Prepn of 1-propyl-3-aminoguanidine) 2)G.W.Kirsten & G.B.L.Smith,JACS **58**,800–1(1936) & CA **30**,4820(1936) (Prepn of salts of 1-methyl-3-aminoguanidine, 1-ethyl-3-aminoguanidine and 1-n-butyl-3-aminoguanidine) 3)J.J.Pitha et al,JACS **70**,2823(1948) & CA **42**, 8165(1948) (Prepn of diacid salts of 1-methyl-3-aminoguanidine) 4)A.H.Greer & G.B.L.Smith, JACS **72**,874–5(1950) & CA **45**,1958(1951) (Prepn and props of 1-methyl-1-aminoguanidine and its salts) 5)R.A.Henry & G.B.L.Smith,JACS **73**, 1858–9(1951) & CA **46**,2502(1952) (Prepn of 1-methyl-3-aminoguanidine) 6)W.H.Finnegan et al, JACS **74**,2981–3(1952) & CA **48**,9329(1954) (Prepn of 1-methylamino-guanidine and its salts)

Nitrated Derivatives of Aminoalkyguanidines and of Alkylaminoguanidines. Following are some refs to compds of this group contg alkyl groups of low molecular weight:

Refs: 1)R.A.Henry & G.B.L.Smith,JACS **73**, 1858–9(1951) & CA **46**,2502(1952) [From the reaction betw methylamine and nitroguanidine the above authors isolated, among other products, 1–methyl–2–amino–3–nitroguanidine. Its isomer 1-methyl-1-amino-3-nitroguanidine was prepd by the interaction of methylhydrazine and 1-methyl-1-nitroso-3-nitroguanidine, using the method described in JACS **69**,3028(1947) & **71**,1968(1949)] 2)J.E.DeVries & E.St.Clair Gantz,JACS **76**,1009 (1954) & CA **48**,7995(1954) (Dissociation constants of 1-methyl-1-amino-3-nitroguanidine) 3)L.A. Burkardt,AnalChem **28**,323(1956) & CA **50**,7540 (1956) (X-ray diffraction spectra of 1-amino-1-methyl-2-nitroguanidine) (This compd was apparently prepd at the US NavOrd Test Sta,China Lake, Calif but no ref to its method of prepn is given in this paper)

Aminoalkyltetrazoles and Alkylaminotetrazoles. Aminoalkyltetrazoles are compds in which an amino-group has replaced a H atom of the alkyl group of an alkyltetrazole (eg, aminomethyltetrazole,

$H_2N \cdot H_2C$–C–NH–N , whereas alkylaminotet-
‖ ‖
N — N
razoles are compds in which one or more alkyl-groups have replaced a H atom of the amino group or an aminotetrazole(eg, methylaminotetrazole
N=CH–N·NH·CH_3 ·
| |
N=====N

Inasmuch as both types of tetrazole derivs(when they contain one or two alkyls of low mw) are high nitrogen compds, they may be of interest as components of explosives and propellants

Below are some of the references to alkylamino- and aminoalkyl-tetrazoles:

1)J.Thiele & H.Ingle,Ann **287**,249–53(1895) (Some alkyl derivs of aminotetrazole) 2)J.von Braun & W.Keller, Ber **65**,1677–80(1932) & CA **27**,723(1923) (Synthesis of tetrazole compds from acid nitriles) 3)R.M.Herbst et al, JOC,**16**,139–49(1951) & CA **45**, 6629–31(1951) (18 refs) (Prepn of various 1–alkyl–5–aminotetrazoles by the interaction of alkylcyanides with hydrazoic acid in benzene soln in the presence of a concd acid; some props of alkylamino- and arylaminotetrazoles) 4)L.A. Burkardt & D.W. Moore, AnalChem **24**,1579–85 (1952) (X-ray diffraction patterns of some tetrazole derivs) 5)W.G.Finnegan et al, JOC **18**, 779–91(1953) & CA **48**,7006–8(1954) (Prepn and isomerization of alkylaminotetrazoles) 6)W.L. Garbrecht & R.M.Herbst, JOC **18**,1003–13(1953) (21 refs) & CA **48**,8224–6(1954) (Prepn and props of some mono- and dialkyl- aminotetrazoles) 7)R.M.Herbst & D.F.Percival, JOC **19**,439–40 (1954) & CA **49**,4636(1955) (Structure of some alkylaminotetrazoles) 8)R.A.Henry et al, JACS **76**,88–93(1954) & CA **49**,2427(1955) (Thermal isomerization of substituted aminotetrazoles) 9)R.A.Henry & W.G.Finnegan,JACS **76**,923–6 (1954) & CA **49**,10939(1955) (Monoalkylation of sodium 5-aminotetrazole in aq medium) 10) Ibid,JACS **76**,926–8(1954) & CA **49**,10940(1955) (Prepn of some alkyl- and aryl- aminotetrazoles) 11)R.A.Henry et al,JACS **76**,2894–8(1954) & CA **49**,10274–6(1955) (1,3-and 1,4-dialkyl-5-aminotetrazoles) 12)K.Hattori, E.Lieber & J.P.Horwitz,JACS **78**,411–15(1956) & CA **50**,12993–4 (1956) (Prepn of some alkylaminotetrazoles)

(See also Aminomethyltetrazole, Aminoethyltetrazole, etc)

Aminoalkyltriazoles and Alkylaminotriazoles. Aminoalkyltriazoles are compds in which an amino-group has replaces replaced a H atom of the alkyl group of an alkyltriazole(eg, aminoethyltriazole), whereas alkylaminotriazoles are compds in which one or more alkyl-groups have replaced a H atom of the amino group of an aminotriazole(eg, methylaminotriazole)

Inasmuch as both types of triazole derivs(when contg one or two alkyls of low mw) are high-nitrogen compds, they may be of interest as components of expls and propellants

Below are some of the references to alkylamino- and aminoalkyl-triazoles:

1)J.C.Sheehan & C.A.Robinson,JACS **71**,1437 (1949) & CA **43**,6620(1949) (Synthesis of some triazole compds) 2)C.Ainsworth & R.G.Jones, JACS **76**,5651–4(1954) & CA **49**,1378–80(1955) (Prepn of nine new 3-aminoalkyl-1,2,4-triazoles) 3)C.Ainsworth & R.G.Jones,JACS **77**,621–4 (1955) & CA **50**,1785(1956) (Prepn of some aminotriazoles) 4)J.H.Boyer & F.C.Canter,JACS **77**, 1280–1(1955) & CA **50**,1786(1956) (Prepn of some alkyloxatriazoles) 5)R.G.Jones & C.Ainsworth, JACS **77**,1538–40(1955) & CA **56**,1784(1956) (Prepn of some triazole derivs) 6)R.G.Jones & C.Ainsworth, USP 2,710,296(1955) & CA **50**,5768 (1956((Substituted triazoles)

(See also Aminoethyltriazole, Aminomethyltriazole and Aminodimethyltriazole)

1-Amino-5-allylamino-α-tetrazole (Called Amino-1-allylamino-5-tetrazol by Stollé), CH_2:CH·CH_2·NH–C·N(NH_2)·N, mw 140.15, N 59.97%. Lt yel ndls,
‖ ‖
N——N
mp 94°. Was obtained in poor yield by Stollé et al starting from allylthiosemicarbizide, NaN_3 and PbO_2(Ref 2,p 220). Although the compd is not an expl, yet as a high nitrogen compd it may prove to be useful as an ingredient of propellants

Refs: 1)Beil–not found 2)R.Stollé & E.Gaertner, JPrChem **132**,212 & 220(1932) 3)F.R.Benson, ChemRevs **41**,16(1947)

Note: No azido- or nitrated derivs of 1-allylamino-α-tetrazole were found in Beil or CA through 1956

Aminoaminotetrazine. Same as Diaminotetrazine

Aminoaminotriazine. Same as Diaminotriazine

Aminoaminotriazole. Same as Diaminotriazole

Aminoaniline. Same as Phenylenediamine, also called Diaminobenzene

AMINOANISOLES AND DERIVATIVES

Aminoanisoles(Anisidines, Methoxyaminobenzenes or Aminophenolmethyl Ethers), $H_2N \cdot C_6H_4 \cdot OCH_3$, mw 123–15, N 11.37%. Three isomers, o-, p- and m- exist and they are described in:
Beil **13**, 358, 404, 435,(108, 129, 145) & [165, 211 & 223]

Aminoanisoles, Azido Derivatives, $C_7H_6N_4O$ – were not found in Beil or CA through 1956

Aminoanisoles, Diazido Derivatives, $C_7H_7N_7O$ – were not found in Beil or CA through 1956

Mononitroaminoanisoles, $H_2N(O_2N) \cdot C_6H_3 \cdot OCH_3$, mw 168.15, N 16.66%. Ten isomers are described in Beil **13**,388–90, 421–2, 520–1,(121, 136–7, 186) & [191–2, 194–5, 215–16; 284 & 286] (See also CA **42**,148i, 3968c, 7053d, 8175h & 8790a)

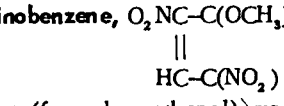
Nitraminoanisoles, $(O_2NHN)C_6H_4 \cdot OCH_3$ – not found in Beil

Dinitroaminoanisoles, $H_2N(O_2N)_2 \cdot C_6H_2 \cdot OCH_3$, mw 213.15, N 19.72%. Ten isomers are described in:
Beil **13**,393–5, 423–4, 525, 527–9,(122, 137, 188–90, 193) & [196, 290, 293]

Dinitronitraminoanisoles, $(O_2NHN)(O_2N)_2 \cdot C_6H_2 \cdot OCH_3$ – Not found in Beil or CA through 1956

Trinitroaminoanisoles; Methoxy-trinitroaminobenzenes or Trinitroanisidines, $H_2N(O_2N)_3 \cdot C_6H \cdot OCH_3$, mw 258.15, N 21.71%, OB to CO_2 –62.0%, OB to CO –18.6%. The following isomers are described in the literature:

2,4,6-Trinitro-3-aminoanisole or 3-Methoxy-2,4,6-trinitroaminobenzene,



Lt yel crysts(from abs methanol), mp 131°; explodes at 238 or 254° (Note). Can be prepd either by boiling tetranitroaniline with methanol or by boiling trinitro-m-dichlorobenzene first with Na or K alcoholate, then with ammonia. Impact sensitivity with the Kast app, max fall for no detonation using a 2kg wt > 60cm vs 49cm for tetryl and using 10kg wt > 24cm vs 14cm for tetryl(Ref 4, pp174–5); Trauzl test 250–260cc and thermal stability at 95° – no change in 30 8-hr days

Note: The expln temp of 238° was detnd by heating the sample at the rate of 5°/min, while a temp of 254° was obtained on heating at the rate of 20°/min(Ref 4)

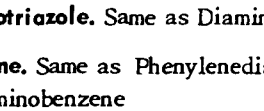
Refs: 1)Beil **13**,(140) & [217] 2)B.J.Flürscheim, BritP 18,777(1911) & CA **7**,1100(1913) 3)C. Claessen,GerP 288,655(1913) & CA **10**,3162(1916) 4)C.F.van Duin & B.C.R.van Lennep, Rec **39**,150, 170 & 174–5(1920) & CA **14**,2708(1920) 5)A.H. Blatt,OSRD 2014(1944)

2,3,5-Trinitro-4-aminoanisole or 4-Methoxy-2,3,6-trinitroaminobenzene,

Red crysts(from alc or water), mp 114–120° or 126–128°, when finely pulverized. Easily sol in acet & NB, sol in benz & eth. Can be prepd by saponifying 2,3,5-trinitro-4-chloroacetaminoanisole or by several other methods listed in Ref 1. Its expl props were not detd

Refs: 1)Beil **13**,532,(195) & [294] 2)R.Reverdin, Helv **6**,92(1923) 3)Ibid **9**,796(1926) 4)H.F.J. Lorang,Rec **46**,638(1927) & CA **22**, 230(1928)

2,3,6-Trinitro-4-aminoanisole or 4-Methoxy-2,3,5-trinitroaminobenzene,



Red crysts(from alc), mp 138–139°. Can be prepd by saponifying 2,3,6-trinitro-4-acetaminoanisole with concd H_2SO_4 at 105°. Its expl props were not reported

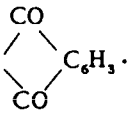
Refs: 1)Beil **13**,(197) 2)R.Meldola & H.Kuntzen, JCS **97**,456(1910) & CA **4**,2108(1910)

Trinitronitraminoanisoles–not found in Beil or CA through 1956

Aminoanisole, Analytical Procedures are discussed in OrgAnalysis **3**(1956),184

Aminoanthracene. Same as Anthramine

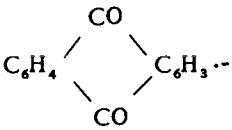
AMINOANTHRAQUINONES AND DERIVATIVES

Aminoanthraquinones,  mw 223.22, N 6.28%, are described in Beil **14**, 177, 191,(436,449) & [99, 107]

Aminoanthraquinones, Azido Derivatives, $C_{14}H_6N_4O_2$ – were not found in Beil or CA through 1956

Aminoanthraquinones, Diazido Derivatives, $C_{14}H_7N_7O_2$ – were not found in Beil or CA through 1956

Mononitroaminoanthraquinones, $C_{14}H_8N_2O_4$, mw 268.22, N 10.45%, are described in Beil **14**,187–9,195–6,(447–8,458–9) & [105–6, 117]

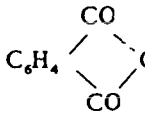
Nitraminoanthraquinones,  mw 268.22, N 10.45%, are listed in Beil **16**,671,(401) & [348]

Note: One of the compds, *2-nitraminoanthraquinone*, patented by the ChemFabrik Griesheim-Elektron, SwissP 62,348(1912) & CA **8**,2263 (1914), was reported to expl at 206°. It is a yel powder obtained by treating anthraquinone-2-isodiazotate with an oxidizing agent

Dinitroaminoanthraquinones, $C_{14}H_7N_3O_6$, mw 313.22, N 13.42% are listed in Beil **14**,190, 197 & [106]

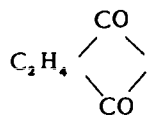
Nitronitraminoanthraquinones, $C_{14}H_7N_3O_6$, mw 313.22, N 13.42%. The following isomers are listed in the literature:

2(?)-N-Nitro-1-nitraminoanthraquinone,

 obtd by treating its Na salt(see below) with dil HCl; expl at 149–50° on rapid heating and 10–15° lower if slowly heated. Its *Na salt* was obtained by slowly adding 10 g aminoanthraquinone to 100 cc HNO_3(d 1.50) at –10°, stirring for 1½ hrs and treating the mixt with 20% CH_3COONa. Yel-brn crysts of the Na salt separate out

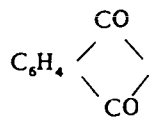
Refs: 1)Beil **16**,[348] 2)E.Terres, Monatsh **41**,603–12(1921) & CA **15**,3835(1921)

4-Nitro-1-nitraminoanthraquinone,

 explg ca 117°; insol in w, sol in aq NaOH soln & concd H_2SO_4. Can be prepd by treating 1-nitraminoanthraquinone with HNO_3(d 1.50)

Refs: 1)Beil **16**,679 2)Höchster Farbw, ChemZtr **1905** I, 313

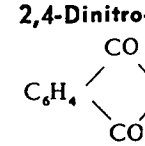
3-Nitro-2-nitraminoanthraquinone,

 explg at 180–291°, depending on the rate of heating; sl sol in acet & NB, sol in concd H_2SO_4 & in alkalies, insol in other org solvents. Can be prepd by treating 2-aminoanthraquinone with HNO_3(d 1.50) plus some urea at below –10°. Its Na salt expl when heated over an open flame

Refs: Beil **16**,679 & (401) 2)R.Scholl et al, Ber **37**,4431–5(1904)

Dinitronitraminoanthraquinones, $C_{14}H_6N_4O_8$, mw 358.22, N 15.64%, OB to CO_2 –102.7%. The following isomers are described in the literature:

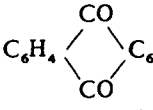
2,4-Dinitro-1-nitramino-anthraquinone,



crysts, mp – expl at 137–142° on rapid heating, decomp ca 100° on slow heating, sometimes expl when treated with a small amt of concd H_2SO_4; diff sol in org solvents of low bp. Was prepd by treating its Na salt with dil HCl. The *Na salt* was obtained in the same manner as the Na salt of 2(?)-N-nitro-1-nitraminoanthraquinone(above) by prolonging the reaction at –10° to 2 hrs and then continuing for 15 hrs at 0°

Refs: 1)Beil **16**,[348] 2)E.Terres, Montash **41**,603–12(1921) & CA **15**,3835(1921)

1,3-Dinitro-2-nitramino-anthraquinone,



$C_6H_4(CO)_2C_6H(NO_2)_2NHNO_2$. Lt yel ndls, explg ca 99°; sol in acet and some other org solvents. Can be prepd by treating 2-aminoanthraquinone with HNO_3(d 1.52) at 35–40°

Refs: 1)Beil **16**,679 2)R.Scholl et al, Ber **37**,4436–7(1904)

Trinitroaminoanthraquinones, $C_{14}H_6N_4O_8$, mw 358.22, N 15.64% and higher nitrated derivs were not found in Beil or CA through 1956

Aminoarylguanidines; Arylaminoguanidines; Aminoaryltetrazoles; Arylaminotetrazoles; Aminoaryltriazoles and Arylaminotriazoles are compds similar to aminoalkylguanidines, alkylaminoguanidines, etc except that they contain aryl-groups instead of alkyl-groups

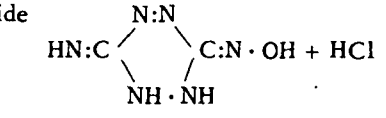
There are also alkylaryl derivatives of amino-guanidines, tetrazoles and triazoles

Some of these compds are described individually, such as aminobenzohydroxytriazole, aminobenzotriazole, aminobenzyltetrazole, aminoethoxyphenyltetrazole, aminomethoxyphenyltetrazole and aminophenyltetrazole.

Refs – see under the individual compounds mentioned above

Aminoazaurolic Acid(Amidoazaurolsäure, in Ger), $H_2N \cdot C(NO):N \cdot NH \cdot C(:NOH) \cdot NH_2$, mw 146.12, N 57.52%. Orange-red ndls with a bluish surface luster, mp – decomp explosively ca 184°; sl sol in cold w, sol in hot w, nearly insol in usual org solvents. Can be prepd by treating an aq soln of dihydroxyquanidine hydrobromide, $HO \cdot N:C(NH_2) \cdot NH \cdot OH + HBr$, under strong cooling with an aq soln of NaOH

On heating with dil HCl, aminoazaurolic acid partly decomp and also forms a deriv of tetrazine, isonitrosoaminodihydrotetrazine hydrochloride



Refs: 1)Beil **3**,121 2)H.Wieland & H.Bauer, Ber **40**,1683–7(1907)

AMINOAZOBENZENES AND DERIVATIVES

Aminoazobenzenes; Phenylazoanilines or Benzeneazoanilines,

$C_6H_5 \cdot N:N \cdot C_6H_4 \cdot NH_2$, mw 197.23, N 21.31%; exist as three isomers of which the para(4) – isomer is of interest because it forms salts some of which, such as the *picrate*(Ref 1, p 311) and the *perchlorate*(Ref 3) are explosive

4-(or p-) Aminoazobenzene exists as yel monocl crysts, mp 125–127°, v sl sol in hot w; more sol in eth and hot alc. Many methods for its prepn are listed in Ref 2. Its Q_c^v was reported as 7983.7 cal/g(Ref 4) and as 1573.7 kcal/mol(Ref 2 p [150]). Fire hazard and toxicity are unknown. When heated to decompn, it emits toxic fumes(Ref 5)

Refs: 1)Beil **16**,303–11,(308–9) & 147–8 2)Beil **16**,307(310) & [149] 3)C.J.S.Lundsgaard, BritP 163,946(1920) & CA **16**,165(1922) 4)Swientoslawski & M.Popoff, JChimPhys **22**, 395(1925) & CA **20**,326(1926) 5)Sax(1957), 267

Note: R.L.Datta & N.R.Chatterjee, JCS **115**, 1008(1919) reported that aminoazobenzene exploded at 598°

Aminoazobenzenes, Azido Derivatives, $C_{12}H_{10}N_6$ – were not found in Beil or CA through 1956

Aminoazobenzenes, Diazido Derivatives, $C_{12}H_9N_9$ – were not found in Beil or CA through 1956

Mononitroaminoazobenzenes, $C_{12}H_{10}N_4O_2$, mw 242.23, N 23.13%. Five isomers are listed in Beil **16**,311,(310) & [151,178]; without indicating whether they are explosive or not

Nitrontraminoazobenzenes, $C_{12}H_9N_5O_4$, mw 287.23, N 24.38%, not found in Beil or CA through 1956

Dinitroaminoazobenzenes, $C_{12}H_9N_5O_4$, mw 287.23, N 24.38%. The following isomers are listed in Beil **16**,342 & (309), without describing their expl props:

2,3'-Dinitro-4-aminoazobenzene, $O_2N \cdot C_6H_4 -N{:}N-C_6H_3(NO_2)NH_2$, yel pdr; mp 175–6° (decomp)

4,6-Dinitro-3-aminoazobenzene, $C_6H_5-N{:}N-C_6H_3(NO_2)_2NH_2$, red ndls, mp 200°

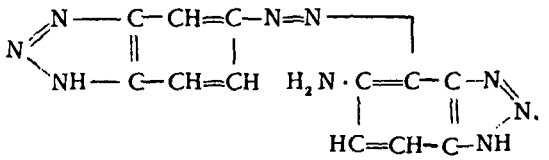
Dinitronitraminoazobenzenes, $C_{12}H_8N_6O_6$, mw 332.23, N 25.30%; *Trinitroaminoazobenzenes*, $C_{12}H_8N_6O_6$, mw 332.23, N 25.30% and *Tetranitroaminoazobenzenes*, $C_{12}H_7N_7O_8$, mw 377.24, N 26.0% were not found in Beil or CA through 1956

Aminoazobenzoditriazole. See Aminoazobenzotriazole

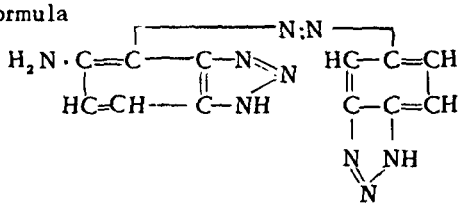
AMINOAZOBENZOTRIAZOLE AND DERIVATIVES

Aminoazobenzotriazoles or Aminoditriazoleazobenzenes are compds of the general formula $TrC_6H_3-N{=}N-C_6H_2(NH_2)Tr$, where Tr is a triazole radical

A high-nitrogen compd with the empirical formula $C_{12}H_9N_9$, mw 279.26, N 45.15%, is listed in Beil as *5-Amino-(4,5'-azobenztriazole)* and its formula is given as:



In this formula, the connections of the azo-groups are made at positions 4 and 5' and not at positions 1 and 1', as is customary. By placing the azo nitrogens in positions 1,1' and rewriting the above formula as is proposed in our nomenclature, we obtain the formula



and the name *2'Amino-4,5:5',6'-ditriazole-azobenzene*, yel-brn leaflets, mp > 300°. Can be prepd by treating an aq soln of 5-aminobenztriazole hydrochloride with $NaNO_2$

Refs: 1)Beil **26**,342 2)R.Nietzki & R.Prinz, Ber **26**,2958(1893) 3)K.Fries & J.Empson, Ann **389**,349–50(1912)

Aminoazobenzotriazoles, Azido Derivatives, $C_{12}H_8N_{12}$, – were not found in Beil or CA through 1956

Aminoazobenzotriazoles, Diazido Derivatives, $C_{12}H_7N_{15}$ – were not found in Beil or CA through 1956

Nitraminoazobenzotriazole, $C_{12}H_8N_{10}O_2$, and higher nitrated derivatives were not found in the literature through 1956

AMINOAZOXYBENZENES AND DERIVATIVES

Aminoazoxybenzenes, $C_6H_5 \cdot (N_2O) \cdot C_6H_4 \cdot NH_2$, mw 213.23, N 19.71%. Several isomers are described in Beil **16**,654 & [338–9]

Aminoazoxybenzenes, Azido Derivatives, $C_{12}H_{10}N_6O$ – were not found in Beil or CA through 1956

Aminoazoxybenzenes, Diazido Derivatives, $C_{12}H_9N_9O$ – were not found in Beil or CA through 1956

3'-Nitro-3-amino-azoxybenzene, $O_2N.C_6H_4\cdot(N_2O)\cdot C_6H_4\cdot NH_2$, mw 258.23, N 21.70%. Was obtained by reduction of 3,3'-dinitroazoxybenzene by hydrogen in the presence of Pt black in ether(Ref 2). Its expl props were not described

Refs: 1)Beil **16**,[338] 2)G.Cusmano, Ann-ChimAppl **12**,129(1919) & CA **14**,1315(1920)

Note: No higher nitrated derivs of aminoazoxybenzenes were found in Beil or CA through 1956

AMINOBENZALDEHYDES AND DERIVATIVES

Aminobenzaldehydes, $H_2N\cdot C_6H_4\cdot CHO$, mw 121.13, N 11.56%. Three isomers, o-, m-, and p-, exist and are described in Beil **14**, 21, 28, 29,(356,359) and [21–2]

Note: Prepn of o-aminobenzaldehyde is described in OSRD Rept 739(1942)

Aminobenzaldehydes, Azido Derivatives, $C_7H_6N_4O$ – were not found in Beil or CA through 1956

Aminobenzaldehydes, Diazido Derivatives, $C_7H_5N_7O$ — were not found in Beil or CA through 1956

Mononitroaminobenzaldehydes, $H_2N(O_2N)\cdot C_6H_3\cdot CHO$, mw 166.13, N 16.86%. Several isomers æe described in Beil **14**,28–9,39, (364) & [21,27]

Nitraminobenzaldehyde, $(O_2N\cdot HN)C_6H_4\cdot CHO$, mw 166.13, N 16.88%. Its isomer, *2-Nitraminobenzaldehyde* is described in Beil **16**,(400)

Dinitroaminobenzaldehydes, $H_2N(O_2N)_2\cdot C_6H_2\cdot CHO$, mw 211.13, N 19.90%. Following isomer is described in Beil:

3,5-Dinitro-4-aminobenzaldehyde. Yel crysts (from alc), mp 171°; easily sol in chlf, bz, AcOH and et acet; diff sol in eth and pet eth. Can be prepd by treating 3,5-dinitroanisaldehyde with alcoholic NH_3 or by nitrating (4-aminobenzal)aniline with mixed HNO_3-H_2SO_4 at not higher than 5–6°. Its expl props were not investigated.

Refs: 1)Beil **14**,40 & [28] 2)M.P.DeLange, Rec **45**,48(1926)

Dinitronitraminobenzaldehyde, $(O_2N\cdot HN)\cdot(O_2N)_2C_6H_2\cdot CHO$ and higher nitrated derivatives were not found in Beil or in CA through 1956

AMINOBENZAMIDES AND DERIVATIVES

Aminobenzamides (Aminobenzosäure-amid, in Ger), $H_2N\cdot C_6H_4\cdot CO\cdot NH_2$. Several isomers are described in Beil **14**,320. 390,425,(531, 559) & [210]

Aminobenzamides, Azido Derivatives, $C_7H_7N_5O$ – were not found in Beil or CA through 1956

Aminobenzamides, Diazido Derivatives, $C_7H_6N_8O$ – were not found in Beil or CA through 1956

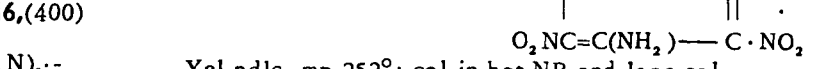
Nitraminobenzamide, $O_2NHN\cdot C_6H_4\cdot CONH_2$ – not found in Beil

Nitroaminobenzamide, $H_2N\cdot C_6H_3(NO_2)\cdot CONH_2$. Several isomers are described in Beil **14**, 376,415,441 & [272]

Nitronitraminobenzamide, $O_2NHN\cdot C_6H_3(NO_2)\cdot CONH_2$, mw 226.15, N 24.78%. Not found in Beil

Dinitroaminobenzamide, $H_2N\cdot C_6H_2(NO_2)_2\cdot CONH_2$, mw 226.15, N 24.78%. The following isomers are described in the literature:

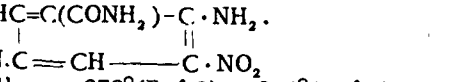
3,5-Dinitro-4-aminobenzamide,



Yel ndls, mp 252°; sol in hot NB and less sol in alc. Was prepd by warming 4-chloro-3,5-dinitrobenzoyl chloride with concd NH_4OH(Ref 2) or by bubbling a current of dry NH_3 through an etheral soln of 4-fluoro-3,5-dinitrobenzoyl chloride(Ref 3). Its expl props were not investigated

Refs: 1)Beil **14**[273] 2)H.Lindemann & W. Wessel,Ber **58**,1224(1925) & CA **19**,2824(1925) 3)H.Goldstein & A.Giddey, Helv **37**,2087(1954) & CA **50**,241(1956)

3,5-Dinitro-2-aminobenzamide,



Yel ndls, mp 278°(Ref 3) or 284°(Ref 2); sol in acet & AcOH, sl sol in alc, eth, benz & w. Can be prepd either by warming 2-chloro-3,5-dinitrobenzoyl chloride with concd NH_4OH(Ref 2) or by bubbling a current of dry NH_3 through an ethereal soln of 2-fluoro-3,5-dinitrobenzoyl chloride(Ref 3). Its expl props were not investigated

Refs: 1)Beil – not found 2)J.Blanksma & G.Verberg, Rec **53**,994–5(1934) & CA **29**,462 (1935) 3)H.Goldstein & A.Giddey, Helv **37**, 1126(1954) & CA **49**,10231(1955)

Dinitronitraminobenzamide, $O_2N \cdot NH-C_6H_2(NO_2)_2 \cdot CONH_2$, mw 271.15, N 25.83%, and higher nitrated derivs were not found in Beil or in CA through 1956

Aminobenzazides and Nitrated Derivatives. See under Aminobenzoic Acids and Derivatives

6-Amino-benzazimidole or 2'-Amino-1-hydroxy-1H-benzo-triazole. See (2'-Aminobenzo)-5',6':4.5'-(1-hydroxy)-vic-triazole

Aminobenzene. See Aniline

Aminobenzenearsonic Acid. Same as Aminophenylarsonic Acid

1-Aminobenzene-4-diazonium Hydroxide or Anilino-4-diazonium Hydroxide, $H_2N \cdot C_6H_4 \cdot N_2 \cdot OH$, mw 137.14, N 30.64%. It is known in the form of salts of which the *hydrochloride*, $H_2N \cdot C_6H_4 \cdot N_2 \cdot Cl + H_2O$, may serve as a starting material for the prepn of other salts, some of which are explosive. *Chromate*, $H_2N \cdot C_6H_4 \cdot N_2 \cdot O \cdot CrO_3H$, N 17.7%. Red crysts (from water) explodes violently ca 160°; insol in alc and sol with darkening in glacial AcOH. Can be prepd either by adding chromic acid to an aq soln of the hydrochloride or by treating p-phenylenediamine(suspended in dil H_2SO_4) with the calcd amt of $NaNO_2$ followed by adding concd $Na_2Cr_2O_7$ soln. *Picrate*, $H_2N \cdot C_6H_4 \cdot N_2 \cdot O \cdot C_6H_2(NO_2)_3$, N 24.1%. Glistening plates(from alc), exploded at 160° or burned rapidly but quietly on a heated spatula. It is sparingly sol in boiling alc or acet. Can be prepd by mixing an aq soln of the hydrochloride with PA

Refs: 1)Beil **16**,602 & [306] 2)R.Meldola & L.Eynon,JCS **87**,2–3(1905) 3)W.H.Gray, JCS **1926**,3178–9

Aminobenzenephosphonic Acid. Same as Aminophenylphosphonic Acid

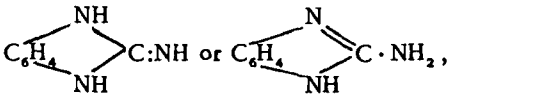
Aminobenzenyloxytetrazotic Acid Methyl Ether, $H_2N \cdot C_6H_4 \cdot C{:}N_4 \cdot O \cdot CH_3$, mw 191.19, N 36.63%. Ndls(from w), mp 110° and deflagrates at higher temps. Very sol in alc & eth. Was prepd by reduction of methyl ether of nitrobenzenyloxytetrazotic acid with $SnCl_2$ in HCl

Refs: 1)Beil **9**,332 2)W.Lossen & F.Fuchs, Ann **298**,66(1897) & JCS **74**,83(1898)

Note: The position of NH_2 was not indicated in the above refs and no later ref was found

AMINOBENZIMIDAZOLES AND DERIVATIVES

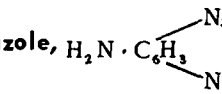
2-Aminobenzimidazole(Benzimidazolonimid or N,N-o-Phenylenguanidin in Ger)



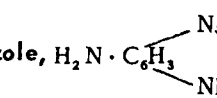
mw 133.15, N 31.56%. Ndls(from w), mp 222–224°; v sol in alc & acet, sol in w, diff sol in eth & benz. Can be prepd by prolonged treatment of o-phenylenediamine with cyanogen bromide in water or by other methods. Some of its salts are explosive: *nitrate* $C_7H_7N_3 \cdot HNO_3$, mw 196.17, N 28.56% ndls(from w), mp–expl ca 225°; *picrate*, $C_7H_7N_3 + C_6H_3N_3O_7$, mw 326.26, N 23.20%, mp–decomp explosively ca 270°

Refs: 1)Beil **24**,116 & (240) 2)P.Pierron, Ann Chim(Paris)[8]15,189 & 193(1908) & CR **151**,1365(1910) 3)G.Pellizzari & A. Gaiter, Gazz **48** II,173(1918) & CA **13**,1584 (1919) 4)IG Farbenind, FrP 773,944(1934) & CA **29**,2177(1935). GerP 617,544(1935) & CA **30**,734(1936) (Prepn of 2-amino-benzimidazole) 5)G.Bruto Crippa et al, Gazz **65**, **38**(1935) & CA **29**,4007(1935) (Discussion on

methods of prepn of 2-aminobenzimidazoles. The methods of Pierron and Pellizzari & Gaiter are considered best

4(or 6)-Aminobenzimidazole or 4-Amino-1,3-benzodiazole,  . Crysts, mp 120–121°. Was prepd by condensing 1,2,3-triaminobenzene with formic acid, similar to the method Woolley used for the prepn of 5(6)-aminobenzimidazole(qv). Its *picrate* $C_7H_7N_3 \cdot C_6H_3N_3O_7$, obtained by crystsn from w as orange colored needles, decomp ca 250°

Refs: 1)Beil & not found 2)G.M.van der Want,Rec **67**,45–51(1948) & CA **42**,5020(1948)

5(or 6)-Aminobenzimidazole or 5-Amino-1,3-benzodiazole,  . Crysts, mp 108.5–109°. Was first obtained by Woolley (Ref 1) by the condensation of 1,2,4-triaminobenzene with formic acid. He reported the mp of the resulting compd as 105–106°. Van der Want(Ref 2) prepd the same compd and obtained, by crystn from water, light-reddish-brown crysts, mp 108.5–109° with decompn. Its *picrate* melts ca 205.5° with decompn

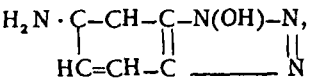
Refs: 1)Beil – not found 2) D.W.Woolley, J BiolChem **152**,225(1944) 3)G.M. van der Want,Rec **67**,45–51(1948) & CA **42**,5020(1948)

Aminobenzimidazoles, Azido Derivatives, $C_7H_6N_6$ – were not found in Beil or CA through 1956

Aminobenzimidazoles, Diazido Derivatives, $C_7H_5N_9$ – were not found in Beil or CA through 1956

4(or 7)-Amino-6(or 5)-nitrobenzimidazole, $C_7H_6N_4O_2$, mw 178.15, N 31.45%. Yel crysts, mp 240–1°(dec). It was prepd by refluxing 5-nitro-1,2,3-triaminobenzene and formic acid in aq HCl, as described in Ref 2

Refs: 1)Beil – not found 2)C.T.Bahner et al, JACS **74**,3689(1952) & CA **48**,5183(1954)

(2'-Aminobenzo)-5',6':4,5-(1-hydroxy)-victriazole; 6-Amino-benzazimidole or 2'-Amino-1-hydroxy-1H-benzotriazole (1-Oxy-6-amino-benztriazol in Beil),  mw 150.14,N 37.32%. Solid which decomps on heating or on standing. Can be prepd by the reduction of 6-nitro-benzazimidol(See under Benzazimidol) with tin and concd HCl. It forms salts, some of which are explosive, eg the *acetate* $C_6H_6N_4O + CH_3COOH$, reddish prisms which explode at 235–36° without melting

Refs: 1)Beil **26**,326 2)T.Curtius & M.Mayer, JPrChem **76**,395(1907) & JCS **941**,53–4(1908)

AMINOBENZOIC ACIDS AND DERIVATIVES

Aminobenzoic Acids or Anthranilic Acids, $H_2N \cdot C_6H_4 \cdot COOH$, mw 137.13, N 10.21%. All three existing isomers, 2(ortho)-, 3(meta)- and 4(para)- are described in Beil **14**,310, 383, 418,(529,558,565) & 205,237,246

Aminobenzoic Acid,Azido Derivatives; Anthranilic Acid Azides; Aminobenzazides or Aminobenzoylazides, $H_2N \cdot C_6H_4 \cdot CO \cdot N_3$, mw 162.15, N 34.56%. The following isomers are described in the literature:

2-Aminobenzazide, lt yel ndls(from benz + petr eth), mp 82–83°(decomp); deflgr on rapid heating; insol in w & ligroin; sol in many org solvents. Can be prepd from the hydrazide of anthranilic acid as described in Ref 2

Refs: 1)Beil **14**,[212] 2)G.Heller & A. Siller, JPrChem **116**,13–14(1927)

3-Aminobenzazide, lt yel ndls(from dil alc), mp 85°; easily sol in eth. Was prepd by interaction of 3-aminobenzohydrazide and benzenediazonium sulfate in aq soln. Its expl props were not investigated

Refs: 1)Beil **14**,391 2)A.Struve & R.Radenhausen, JPrChem **52**,241(1895) & JCS **70**, 36(1896)

4-Aminobenzazide or Anthranoylazide, yel ndls, darkening rapidly in air; mp 85–90° (decomp), defgr on rapid heating; insol in w; diff sol in benz, sol in alc, v sol in eth and sol in many other org solvents. Was prepd by interaction of 4-aminobenzohydrazide and $NaNO_2$ in AcOH or HCl soln

Refs: 1)Beil **14**,(571) & [259] 2)G.Heller & A.Siller, JPrChem **116**,16(1927) & CA **21**, 2697(1927)

Aminobenzoic Acid, Diazido Derivatives, $C_7H_5N_7O_2$ – were not found in Beil or CA through 1956

2-Nitro-4-aminobenzazide, $H_2N \cdot C_6H_3(NO_2) \cdot CO \cdot N_3$, mw 207.15, N 33.81%. Red floc ppt; defgrg on heating; insol in w, alc or eth. Was prepd by treating 4-aminobenzohydrazide in AcOH with $NaNO_2$ in w

Refs: 1)Beil **14**,440 2)T.Curtius & F.Bollenbach, JPrChem **76**,296(1907) & JCS **92** i, 1078(1907)
Note: No other nitrated aminobenzazides were found in Beil or CA through 1956

Mononitroaminobenzoic Acids, $H_2N \cdot C_6H_3(NO_2)COOH$, mw 182.13, N 15.38%. Various isomers are described in Beil **14**,373,374, 375,378,414,415,417,439,440,(555,556,557, 565,583) & [233,234,245]

Nitronitraminobenzoic Acids, $O_2N \cdot HN \cdot C_6H_3(NO_2)COOH$, mw 227.13, N 18.50%. Two isomers: *2-nitro-6-nitramino-* and *4-nitro-2-nitraminobenzoic acids* are listed in Gazz **55**, 1632(1955) by G.Berti, S.Carboni & A.Da Settimo[See also CA **50**,10041a(1956)]

Dinitroaminobenzoic Acids or Dinitroanthranilic Acids, $H_2N \cdot C_6H_2(NO_2)_2 \cdot COOH$, mw 227.13, N 18.50%. The isomers *2-amino-3,5-dinitro-*, *3-amino-2,4-dinitro-* and *4-amino-3,5-dinitro-* are described in Beil **14**,379,445, (557,565) & [236,273]

The isomer *3-amino-4,6-dinitro-* is described by H.Goldstein & R.Stamm Helv **35**,1472(1952), the *2-amino-3,5-dinitro-* by H.Goldstein & A. Giddey in Helv **37**,1121 & 1124(1954) and the *4-amino-3,5-trinitro-benzoic acid* by H.Goldstein & A.Giddey in Helv **37**,2084 & 2087(1954)

Dinitronitraminobenzoic Acids, $O_2N \cdot HN-C_6H_2(NO_2)_2 \cdot COOH$, mw 272.13, N 20.59%. The following isomers are described in the literature:

3,4-Dinitro-2-nitraminobenzoic Acid or 3,4-Dinitro-N-nitroanthranilic Acid. Yel, unstable solid, which may be stored for some time in the dark. No mp given. Sol in alc & eth; less sol in w; nearly insol in chlf & benz. Was prepd by adding HNO_3(d 1.48) dropwise to a soln of 4-nitroaminobenzoic acid, while maintaining the temp below –5°. Its expl props were not investigated

Refs: Beil – not found 2)G.Berti, S. Carboni & A.Da Settimo, Gazz **85**,1637, 1640–1 (1955) & CA **50**,10041(1956)

3,5-Dinitro-4-nitraminobenzoic Acid. Its monohydrate, $C_7H_4N_4O_8+H_2O$, exists as yel plates which lose H_2O at ca 95°, decomp ca 135–136° and explodes when heated on a Pt foil. Very sol in alc, eth & AcOH; sol in warm w, diff sol in chlf; nearly insol in benz. Was prepd by nitration of 3,5-dinitro-4-aminobenzoic acid with HNO_3(d 1.48) at 0°

Refs: 1)Beil **16**,[350] 2)L.Elion, Rec **42**, 175–6(1923) & CA **17**,3489(1923)

Trinitroaminobenzoic Acid, $H_2N \cdot C_6H(NO_2)_3 \cdot COOH$ and higher nitrated aminobenzoic acids were not found in Beil or CA through 1956

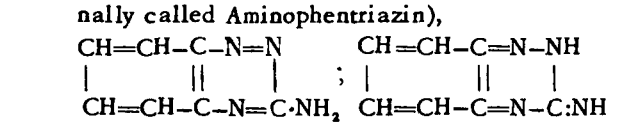
Aminobenzophenones, $C_6H_5 \cdot CO \cdot C_6H_4 \cdot NH_2$. Several isomers and some nitrated compds, none of them expl, are listed in Beil **14**. No azido- or diazido- derivs were found in Beil or CA through 1956

AMINOBENZOTRIAZINES AND DERIVATIVES

Aminobenzotriazines, $C_7H_6N_4$, mw 146.15,

N 38.34%. The following isomers are listed in the literature:

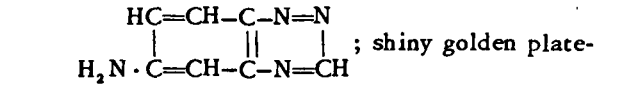
3-Amino-asym-benzotriazine or 3-Imino-2,3(or 3,4)-dihydro-1,2,4-benzotriazine(originally called Aminophentriazin),



Yel ndls, mp 207°. Sol in hot alc and less in ether. May be prepd by oxidation of 3-amino-1,2-dihydro-1,2,4-benzotriazine with K ferricyanide(Ref 2) or by the method described in Ref 3. The compd was investigated by Merck Co(Ref 4). It is described here because it has fairly high N content

Refs: 1)Beil **26**,(44) & [90] 2)F.Arndt, Ber **46**,3528(1913) 3)F.Arndt & B.Eistert, Ber **60**,2602(1927) & CA **22**,1162(1928) 4) F.J.Wolf et al, JACS **76**,3551-2(1954) & CA **49**,12494(1955)

3'-Aminobenzo-asym-triazine(Called in JCS 6-Aminobenzo-1,2,4-triazine),



; shiny golden platelets, mp 298-9°(dec). Was obtained by hydrogenation of N-2,4-dinitrophenyl-N'-formylhydrazine, $2,4\text{-}(O_2N)_2C_6H_3\cdot NH\cdot NH\cdot CHO$, in hot ethanol in the presence of palladium-charcoal. It is described here for the same reason as the above compd

Refs: 1)Beil – not found 2)R.A.Abramovitch & K.Schofield,JCS **1955**,2333 & CA **15**,12077(1956)

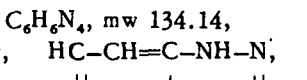
Aminobenzotriazines, Azido Derivatives, $C_7H_5N_7$ – were not found in Beil or CA through 1956

Aminobenzotriazines, Diazido Derivatives, $C_7H_4N_{10}$ – were not found in Beil or CA through 1956

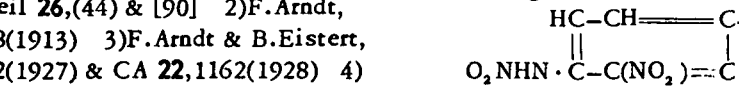
Mononitroaminobenzotriazines, $C_7H_5N_5O_2$, and higher nitrated and nitrited, derivs were found in Beil or CA through 1956

AMINOBENZOTRIAZOLES AND DERIVATIVES

Aminobenzotriazoles, $C_6H_6N_4$, mw 134.14, N 41.77%. One isomer,



described in Ref 1 as **5(or 6)-Aminobenzotriazole.** It may also be called **(3'-Aminobenzo)-5',6':4,5-(α-vic-triazole. or (3'-Aminobenzo)-1H-1,2,3-triazole).**It forms several salts and a dinitroderiv which contains a phenyl group in the triazole ring. This deriv is called in Ref 2: **1-Phenyl-4-nitro-5-nitraminobenzotriazole** and may also be called: **[(3'-Nitramino-4'-nitro)benzo]-5',6':4,5-(1-phenyl-α-vic-triazole.** It defgrs at ca 175° and its probable formula is

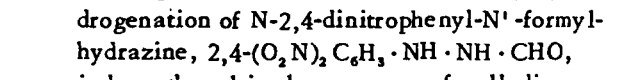


, mw 300.23 N 27.99%. Lt yel ndls obtnd by treating 1-phenyl-5-acetaminobenzotriazole with nitric acid(d 1.52)(Refs 2 & 3)

Another isomer of aminobenzotriazole,

, listed in Ref 1 as:

4(or 7)-Aminobenzotriazole, exists in the form of the nitroderiv,



mw 179.14, N 39.10%, dk yel ndls. Can be obtained from 5-nitro-1,2,3-triaminobenzene through a series of operations described in Ref 3. We propose naming the nitrocompound **[2'-Nitro-4'-amino)benzo]-5',6':4,5-(α-vic-triazole).** It is probably expl, judging by the fact that a similar nitrated aminobenzotriazole deriv with a lower nitrogen content is expl.

Refs: 1)Beil **26**,323 2)Beil **26**,(107) 3)R. Nietzki & H.Hagenbach,Ber **30**,544(1897) 4)K.Fries & J.Empson, Ann **389**,354(1912)

Aminobenzotriazoles, Azido Derivatives, $C_6H_5N_7$ – were not found in Beil or CA through 1956

Aminobenzotriazoles, Diazido Derivatives, $C_6H_4N_{10}$ – were not found in Beil or CA through 1956

Aminobenzoylazides or Aminobenzazides. See under Aminobenzoic Acids and Derivatives

5-Amino-1-benzyl-vic-tetrazole or 1-Benzyl-5-amino-vic-tetrazole, [1-Benzyl-tetrazolon-(5)-imid, in Ger], $H_2N \cdot C{-}N(C_6H_5CH_2){-}N$ (with N bridging the two C atoms) or $HN{:}C{-}N(C_6H_5CH_2){-}N$ (with HN bridging the two C atoms), mw 175–19, N 39.98%. Fine ndls(from hot w); mp 187° (Ref 2); 191°(Ref 1). Methods of prepn are indicated in Refs 1,2 & 3. It is a high nitrogen compd and probably can be nitrated to form expls

Hydrogenative fission of aminobenzyl-tetrazole gave aminotetrazole(Ref 4)

Refs; 1)Beil **26,**[249] 2)Knoll A–G Chemische Fabriken, GerP 540,409(1926) & CA **26,**3263 (1932) 3)J.von Braun & W.Keller, Ber **65B,** 1679(1932) & CA **27,**723(1933) 4)L.Birkofer, Ber **75B,**433(1942) & CA **37,**3067(1943)

AMINOBIPHENYLS AND DERIVATIVES

Aminobiphenyls; Biphenylamines; Aminodiphenyls or Phenylanilines, $C_6H_5 \cdot C_6H_4 \cdot NH_2$, mw 169.22, N 8.28%. Three isomers, o-(or 2-), m-(or 3-) and p-(or 4-) are known and described in Beil **12,**1317–18(546) & [747,751 & 753]

The most important of these is **4-(or p-) Aminobiphenyl,** leaflets(from alc), mp 53–55°, bp 302°; diff sol in cold w; sol in alc, eth, chlf & hot w. Was first prepd by Hofmann (Ref 2) from the high boiling residue obtained in the manuf of aniline and named *"Xenylamin"*. It can also be prepd by the reduction of 4-nitrobiphenyl or by other methods(Ref 1)

When a salt of 4-aminobiphenyl, such as the hydrochloride, is treated with $NaNO_2$+acid, diazotization takes place. If diazotization of aminobiphenyl is followed by treatment with perchloric acid the resulting compd is an explosive, **biphenyldiazonium perchlorate,** $C_6H_5 \cdot C_6H_4 \cdot N_2 \cdot ClO_4$, which was found to be suitable for charging detonators(Ref 3)

Refs: 1)Beil **12,**1318 & [753] 2)A.W.Hofmann,CR **55,**901(1862) 3)T.L.Davis & F.H. Huntress,USP 1,828,960(1932) & CA **26,**849 (1932)

Aminobiphenyls, Azido Derivatives, $C_{12}H_{10}N_4$ – not found in Beil or CA through 1956

Aminobiphenyls, Diazido Derivatives, $C_{12}H_9N_7$ – not found in Beil or CA through 1956

Mononitroaminobiphenyls, $C_6H_5 \cdot C_6H_3(NO_2)NH_2$ or $(O_2N)C_6H_4 \cdot C_6H_4 \cdot NH_2$, mw 214.22, N 13.08%. Several isomers are described in Beil **12,**1320–1,(547) & [750–3, 760–1]

Dinitroaminobiphenyls, $C_{12}H_9N_3O_4$, mw 259.22, N 16.21%. Several isomers are described in Beil **12,**1321(546) & [750, 762–3]

Dinitronitraminobiphenyls, $C_{12}H_8N_4O_6$ – not found in Beil or CA through 1956

Trinitroaminobiphenyls, $C_{12}H_8N_4O_6$, mw 304.22, N 18.42%, OB to CO_2 –115.7%, OB to CO –52.6%. Following isomers are described in the literature:

3,5,4'–Trinitro-2-aminobiphenyl, $(O_2N)C_6H_4 \cdot C_6H_2 \cdot NH_2(NO_2)_2$. Prisms(from pyridine) mp 239°. Can be prepd by treating 3,5,4'-trinitro-2-p-toluensulfonylaminobiphenyl, $O_2N \cdot C_6H_4 \cdot C_6H_2(NO_2)_2 \cdot NH \cdot SO_2 \cdot C_6H_4 \cdot CH_3$, with H_2SO_4. Its expl props were not investigated

Refs: 1)Beil **12,**[751] 2)F.Bell,JCS **1928,** 2775 & **1930,**1075

3,2',4'-Trinitro-4-aminobiphenyl, $(O_2N)_2C_6H_3 \cdot C_6H_3(NO_2)NH_2$. Orange-yel needles(from glac AcOH), mp 192–3°. Was prepd in small quantity by heating 4'-bromo-2,4,3'-trinitrobiphenyl with satd alcoholic NH_3 in a sealed tube at 150° for 8–10 hrs. Its expl props were not investigated

Refs: 1)Beil **12,**[764] 2)J.W.Le Fèvre et al,JCS **1927,**2337

Trinitronitraminobiphenyl, $C_{12}H_7N_5O_8$; *Tetranitroaminobiphenyl*, $C_{12}H_7N_5O_9$ and higher nitrated derivs were not found in Beil or CA through 1956

Aminobiuret. Same as Allophanylhydrazide

Amino-Boranes are complexes of boranes (BH_3) with amines. Most of the secondary and tertiary amines form these complexes. The following amine boranes are manufd by the Callery Chemical Co: a)Dimethylamine-borane, $(CH_3)_2NH \cdot BH_3$, wh solid b)Trimethylamine-borane, $(CH_3)_3N \cdot BH_3$ wh solid c)Pyridine-borane, $C_5H_5N \cdot BH_3$, col liquid

These amine-boranes are relatively stable complexes and are of interest because they act as selective reducing agents, polymerization catalysts, anti-oxidants and stabilizing agents. They may also be used for the prepn of diborane and as petroleum additives. Further information may be obtained from Tech Bull C-200(Ref 2)

Refs: 1)Advertisements of the Callery Chemical Co in C&EN **36**,p 97(May 26, 1958) and p 15(June 23,1958) 2)Technical Bulletin, Callery Chemical Co, Pittsburgh, Pa

Aminoboronhydride Compounds. Many aminoboronhydride compds and their derivatives are volatile and self-inflammable. A series of papers by H.I.Schlesinger et al published in JACS on boron hydrides(qv) include the prepn and props of the following aminoboronhydrides and their derivatives: borineamine, $H_3N \cdot BH_3$ or BH_6N; dimethylaminoborine(or dimethylaminoboric acid) $(CH_3)_2BNH_2$ dimethylaminodiborane$(CH_3)_2NB_2H_5$; borinetriamine $B_3N_3H_6$; borinetrimethylene $(CH_3)_3NBH_3$; aminodiborane $H_2N \cdot B_2H_5$ or B_2H_7N; methylaminodiborane $CH_3HN \cdot B_2H_5$; dimethylaminodiborane$(CH_3)_2NB_2H_5$ and the very volatile and self-inflaming chloroderivative of dimethylaminodiborane, $(CH_3)_2NB_2H_4Cl$
(See also under Boron)

Refs: 1)H.I.Schlesinger et al, JACS **58**, 409-14(1936) 2)A.Burg & H.I.Schlesinger, Ibid **59**,780 & 785-6(1937) 3)S.H.Bauer, Ibid **60**,524-30(1938) 4)H.I.Schlesinger et al, Ibid **60**,1296-1300 & 2297-2300(1938) 5)H.I.Schlesinger et al, Ibid **61**,1078-83(1939) 6)A.Burg & C.L.Randolph,Ibid **71**,3451-55(1949) (See also German refs listed in these papers)

Aminoboron-Silicon Compounds. See Silylaminoboron Compounds

Aminobutane. Same as Butylamine

2-Amino-1-butanol or 1-Butanol-2-Amine, $CH_3 \cdot CH_2 \cdot CH(NH_2) \cdot CH_2OH$, is the parent compd of the following derivative:

2-Nitramino-1-butanol Nitrate or 1-Nitroxy-2-nitramino-butane, $CH_3 \cdot CH_2 \cdot CH(NH \cdot NO_2) \cdot CH_2 \cdot ONO_2$, mw 179.14, N 23.46%, OB to CO_2 -67.0%, OB to CO -22.3%. Properties - not found in Beil or CA through 1956

According to Ref 2, the prepn is in agreement with the following scheme:

$CH_3 \cdot CH_2 \cdot CH(NH_2) \cdot CH_2OH$

↓ Add $ClCOO \cdot C_2H_5$, followed by aq NaOH

$CH_3 \cdot CH_2 \cdot CH(NH \cdot COO \cdot C_2H_5) \cdot CH_2OH$

↓ Add the above mixt with stirring to 98% nitric acid at 10°

$CH_3 \cdot CH_2 \cdot CH(N(NO_2) \cdot COO \cdot C_2H_5) \cdot CH_2 \cdot ONO_2$

↓ Add anhyd NH_3 to the ethereal soln of the above nitrate(ammonolysis)

$CH_3 \cdot CH_2 \cdot CH(NH_2^+) \cdot CH_2 \cdot ONO_2$ NO_3^-

↓ Add concd hydrochloric acid

$CH_3 \cdot CH_2 \cdot CH(NH \cdot NO_2) \cdot CH_2 \cdot ONO_2$

Details of this method of prepn are given in Ref 2,pp 122–3 and in the patent(Ref 3, p 15), but no props are described. This nitrate was suggested as a possible gelatinizer for NC

Refs: 1)Beil – not found 2)A.T.Blomquist & F.T.Fiedorek,OSRD Rept 4134(PB Rept 18867)(1944),pp 122–3 3)Ibid, USP 2,485,855(1949),p 15

AMINOCARBAZOLES AND DERIVATIVES

Aminocarbazoles, C_6H_4——$C_6H_3 . NH_2$, (NH)

mw 182.22, N 15.38%. Its 1-amino, 2-amino- and 3-amino-isomers are described in Beil **22,**460(642) [370-1] and 1-aminocarbazole is also described by H. Lindemann & F.Werther in Ber **57,** 1316(1924)

Note: There is also an *N-aminocarbazole* or *N,N-diphenylenehydrazine,* C_6H_4——C_6H_4, $N . NH_2$

described in Beil **20,** (166)

Aminocarbazoles, Azido Derivatives, $C_{12}H_9N_5$ –not found in Beil or CA through 1956

Aminocarbazoles Diazido Derivatives, $C_{12}H_8N_8$ –not found in Beil or CA through 1956

Mononitroaminocarbazoles, $C_{12}H_9N_3O_2$, mw 227.22, N 18.49%. The 2-nitro-3-amino- and 4-nitro-3-amino isomers are listed Beil **22,** 373-4

Note: No higher nitrated aminocarbazoles were found in Beil or CA through 1956. It is suggested that these compds may have some value in expl compns or fuse powders as flash reducing agents or burning rate modifiers

Aminocarbonylaminosuccinyl Diazide or **Aminocarbonyliminosuccinyl Diazide** (Amidocarbon-iminobernsteinsäure-diazid in Ger),

$H_2N . CO . N$(CH . CO . N_3)(CH . CO . N_3)

mw 224.15, N 50.0%. Oil, expl violently on heating. Can be prepd by adding dropwise the calcd amt of concd aq $NaNO_2$ to aminocarbonyliminosuccinyl dihydrazide (in HCl and covered with a layer of ether), while maintaining the temp at –10° or below

Refs: 1)Beil–not found 2)T.Curtius & W.Dörr, JPrChem **125,** 442-3(1930) & CA **24,** 3214(1930)

Aminocellulose. See Aminated Cellulose

1-Amino-5-(o-chlorophenyl)-α-tetrazole or 1-Amino-5-(2'-chlorophenyl)-1H-tetrazole, (o-Cl . C_6H_4) . C(=N)–N(=NH_2)–N

mw 195.62, N 35.80%. Leaflets (from alc), mp 173°, decomp at higher temps with vigorous evoln of gas; sl sol in eth, benz, chlf & hot w, nearly insol in cold w, fairly sol in hot alc & toluene, sol in hot acet. Was obtained by heating 1-(o-chlorobenzalamino)-5-phenyl-α-tetrazole(sl wet with alc) with concd HCl for 10 hrs. Serves for the prepn of other tetrazoles, some of them expl, eg 1-dichloroamino-5-(o-chlorophenyl) -α-tetrazole(qv)

Refs: 1)Beil–not found 2)R.Stollé et al, JPrChem **138,** 2 & 9-10(1933)

Aminochrysammic or Aminochrysamminic Acid. See 2,4,5,7-Tetranitro-8-amino-1-hydroxy-anthraquinone, described under Aminohydroxyanthraquinone and Derivatives

Aminocompounds are described individually, such as aminoacetic acid, aminobenzoic acid, aminocarbazole, aminotetrazole, aniline, etc

AMINOCRESOLS AND DERIVATIVES

Aminocresols; Aminohydroxytoluenes; Methylaminophenols or Aminohydroxymethylbenzenes, $H_2N . C_6H_3(CH_3)OH$. All possible isomers are described in Beil **13,** 572, 574, 576, 579, 589, 590, 593, 598, 601, (212, 216, 222, 226, 227) & [319, 324, 326, 330, 337, 338]

Aminocresols, Azido Derivatives, $C_7H_8N_4O$-not found in Beil or CA through 1956

Aminocresols, Diazido Derivatives, $C_7H_7N_7O$ not found in Beil or CA through 1956

Mononitraminocresols, $O_2N.HN.C_6H_3(CH_3)OH$ were not found in Beil or CA through 1956

Mononitroaminocresols, $H_2N.C_6H_2(CH_3)(NO_2)OH$. All possible isomers are described in Beil **13**, 574, 578, 595, 605, (213, 228) & [319, 345, 346]

Nitronitraminocresols, $C_7H_7N_3O_5$ were not found in Beil or CA through 1956

Dinitroaminocresols, $C_7H_7N_3O_5$, mw 213.5, N 19.72%. The following isomers are described in the literature:

2,4-Dinitro-6-amino-m-cresol; 2,6-Dinitro-4-amino-3-hydroxy-toluene or 2,6-Dinitro-4-amino-3-hydroxy-1-methyl-benzene, $H_2N.C_6H(CH_3)(NO_2)_2OH$. Yel crysts (from alc), mp 151°(Ref 3), 156°(Ref 4); decomp at sl higher temp; insol in cold w; sol in alc, very sol in eth. Can be prepd by treating 2,4,6-trinitro-3-hydroxytoluene with ammonium sulfide, as indicated in Ref 5

On treating dinitroaminocresol with nitrous acid, the diazocompd, $C_6H(CH_3)(NO_2)_2(OH).N=N.NH.C_6H(CH_3)(NO_2)_2OH$, is obtained as golden yel leaflets which expl violently on heating to ca 160°

Refs: 1)Beil **13**, 591 & [327] 2)W. Kellner & F.Beilstein, Ann **128**, 166-7(1863) 3)C.L.Liebermann & W.A.von Dorp, Ann **163**, 104-5(1872) 4)O.Emmerling & A.Oppenheim, Ber **9**, 1094(1876) 5)R.B.Drew, JCS **117**, 1617(1920)

2,6-Dinitro-4-amino-m-cresol; 2,4-Dinitro-6-amino-3-hydroxy-toluene or 2,4-Dinitro-6-amino-3-hydroxy-1-methyl-benzene, $H_2N.C_6H(CH_3)(NO_2)_2OH$. Ruby-red ndls(from aq alc); mp 160°(Ref 2), 166.5-167.5°(Ref 3); diff sol in w. Can be prepd by heating 2,4-dinitro-6-acetamino-3-hydroxytoluene with HCl(Ref 3)

Refs: 1)Beil **13**, 595 & [335] 2)R.Nietzki & F.Ruppert, Ber **23**, 3479-80(1890) 3)M.T. Bogert & G.H.Connitt, JACS **51**, 907(1929) & CA **23**, 1888(1929)

2,6-Dinitro-3-amino-p-cresol; 3,5-Dinitro-2-amino-4-hydroxy-toluene or 3,5-Dinitro-2-amino-4-hydroxy-1-methyl-benzene, $H_2N.C_6H(CH_3)(NO_2)_2OH$. Brn-red ndls (from alc), mp 141-142°. Can be prepd by briefly heating 3,5-dinitro-2-amino-4-methylamino-toluene with aq NaOH

Refs: 1)Beil **13**, 601 2)A.Sommer, JPrChem **67**, 551(1903) & JCS **84**, 656(1903)

4,6-Dinitro-3-amino-o-cresol; 3,5-Dinitro-6-amino-2-hydroxy-toluene or 3,5-Dinitro-6-amino-2-hydroxy-1-methyl-benzene, $H_2N.C_6H(CH_3)(NO_2)_2OH$. Yel ndls (from w). Can be prepd by treating 3,5-dinitro-2-azido-1-methyl-benzene with concd H_2SO_4

Note: The identity of this compd was not definitely established. It might be *2,6-Dinitro-3-amino-p-cresol*

Refs: 1)Beil **13**, 614 2)P.Drost, Ann **313**, 315(1900)

eso-Dinitro-eso-amino-cresol or x,x-Dinitro-x-amino-x-hydroxy-methyl-benzene, $H_2N.C_6H(CH_3)(NO_2)_2OH$. Red ndls (from w); mp ca 172° with decompn. Can be prepd by treating 3,5-dinitro-4-azido-1-methyl-benzene with concd H_2SO_4

Refs: 1)Beil **13**, 614 2)P.Drost, Ann **313**, 314-15(1900)

Dinitronitraminocresols, $O_2NHN.C_6H(CH_3)(NO_2)_2OH$; *Trinitroaminocresols*, $H_2N.C_6(CH_3)(NO_2)_3OH$ and higher nitrated derivs of aminocresols were not found in Beil or CA through 1956

Aminodiazacycloalkenes Nitrated Derivatives. Several nitrated products of 2-amino-1,3-diazacyclo-2-hexenes and 2-amino-1,3-diazacyclo-2-pentenes are described in the literature. Inasmuch as some of these compds contain more than 40% nitrogen, they may be of interest as components of propellants. Some of these compds are described here individually, as well as in the following refs: 1)Beil–not found 2)A.F.McKay & G.F. Wright, JACS **70**, 3990(1948) & CA **43**, 2203(1949)(The nitration products of 2-nitramino-Δ^2-1,3-diazacycloalkenes) 3) A.F.McKay & D.F.Manchester, JACS **71**, 1972(1949) & CA **43**, 9065(1949)(The nitration products of some substituted 2-nitramino-1,3-diazacycloalkenes) 4)L.

Fishbein & J.A.Gallaghan, JACS **76**, 3218 (1954) & CA **49**, 8991(1955)(Prepn & props of 2-amino-1-nitro-1,3-diazacyclo-2-hexene; 2-amino-1-nitro-1,3-diazacyclo-2-hexene nitrate; 2-amino-1,3-dinitrocyclo-2-hexanone and other derivs)

Aminodiazacyclohexanone. See Aminotetrahydropyrimidone

Aminodiazacyclohexene. See Aminotetrahydropyrimidol

Aminodiazacyclopentane. See Aminoimidazoline

Aminodiazacyclopentanone. See Aminoimidazolidone

Aminodiazacyclopentene. See Aminoimidazoline

4-Amino-diazoaminobenzene, $C_6H_5.N{:}N.NH.C_6H_4.NH_2$, mw 212.25, N 26.40%. Brn-yel ndls (from dil alc), dec at 157° and expl when heated in a tube; sol in alc diff sol in ether & insol in w, or petr ether. Was prepd by treating 4-acetamino-diazoaminobenzene with Na ethylate

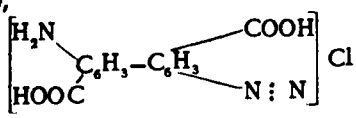
Refs: 1)Beil **16**, 732 2)R.Willstätter & M.Benz, Ber **39**, 3491(1906) & CA **1**, 302 (1907)

2-Amino-7-diazonaphthalene Bromide Hydrobromide, $[H_2N.C_{10}H_6.N:N]Br.HBr$, mw 331.03, N 12.7%. Yel ndls, mp- expl violently. Prepd by diazotizing 2,7-naphthalenediamine in alcoholic soln with hydrobromic acid and amyl nitrite. The compd expl also on contact with concd HNO_3.

Refs: 1)Beil **16**, 610 2)F.Kaufler & U. Karrer, Ber **40**, 3262(1907) & JCS **92**, 795 (1907)

Aminodibenzofuranes. See Aminodiphenyleneoxides

6-Amino-2,2'-dicarboxybiphenyl-6'-diazonium Chloride,



mw 319.70, N 13.14%. Red-brn prisms, mp-defgr ca 100°; stable in storage and insensitive to impact. Was prepd by diazotization of diaminodiphenic acid

Ref: 1)Beil–not found 2)J.Schmidt & R. Schall, Ber **40**, 3003(1907)(footnote 1)

Note: Beil **16**, 612 gives the name and structural formula for the 4' diazonium salt but the props are those given in Ber for the 6' diazonium compd. No litr was found for the 4'-compd

Aminodiethanol or Dihydroxyethylamine. Same as Diethanolamine

Aminodimethyldiazacyclopentene. See Aminodimethylimidazoline

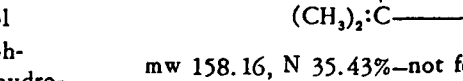
AMINODIMETHYLIMIDAZOLINE AND DERIVATIVES

Aminodimethylimidazoline or Aminodimethyldiazacyclopentane, $C_5H_{11}N_3$, mw 113.16, N 37.14%, may be considered as the parent compd of the following derivs:

Aminodimethylimidazoline, Azido Derivative, $C_5H_{10}N_6$–not found in Beil or CA through 1956

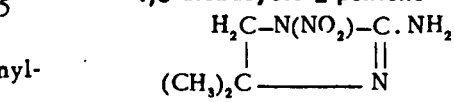
Aminodimethylimidazoline, Diazido Derivative, $C_5H_9N_9$–not found in Beil or CA through 1956

2-Amino-4,4-dimethyl-1-nitro-Δ^2-imidazole or 2-Amino-4,4-dimethyl-1-nitro-1,3-diazacyclo-2-pentene,



mw 158.16, N 35.43%–not found in Beil or CA through 1956

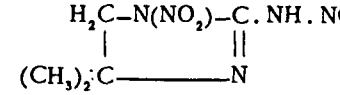
2-Amino-4,4-dimethyl-1-nitro-Δ^2-imidazole Nitrate or 2-Amino-4,4-dimethyl-1-nitro-1,3-diazacyclo-2-pentene Nitrate,



mw 221.18, N 31.67%. Crysts (from abs alc), mp 179-181°. Was prepd by refluxing 1-(β-nitroxy-tert-butyl)-3-nitroguanidine with some n-butanol for 30 mins followed by evaporation of the soln

Refs: 1)Beil–not found 2)L.Fishbein & J.A.Gallaghan, JACS **76**, 3219(1954) & CA **49**, 8991(1955)

2-Nitramino-4,4-dimethyl-1-nitro-Δ^2-imidazole or *2-Nitramino-4,4-dimethyl-1-nitro-1,3-diazacyclo-2-pentene,*

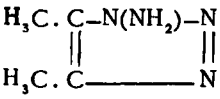


mw 203.16, N 34.48%—not found in Beil or CA through 1956

AMINODIMETHYLTRIAZOLES AND DERIVATIVES

Aminodimethyltriazoles, $C_4H_8N_4$, mw 112.14, N 49.97%. The following isomers are listed in the literature:

1-Amino-4,5-dimethyl-α-vic(1H-1,2,3)-triazole,



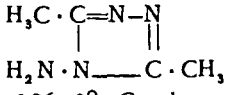
Leaflets, mp 95°, defg on rapid heating. Was prepd in 1900 by von Pechmann & Bauer by heating 1-benzamino-4,5-dimethyl-1,2,3-triazole in a sealed tube at 95-100° and believed to have the structure of "Dimethylosotriazin" (Ref 2). The correct structure was established in 1909(Ref 3)

Its *silver nitrate-double salt,* $(C_4H_8N_4)_2 \cdot AgNO_3$, melts with decompn at 188° or expl with the formation of flame when heated rapidly

Refs: 1)Beil **26**,28 2)H.von Pechmann & W.Bauer, Ber **33**, 645(1900) 3)Ibid, Ber **42**, 665–7(1909)

1-Amino-3,5-dimethyl-1,2,4-triazole, listed as such in CA **28**,2679(1936), seems to be identical with 4-amino-3,5-dimethyl-4,1,2-triazole, listed in CA Formula Index 1920–1946,p 61, bottom of right-hand column

4-Amino-3,5-dimethyl-4H-1,2,4-triazole or **3,5-Dimethyl-4-amino-4,1,2-triazole,**



Prisms, mp 196–9°. Can be prepd by treating an alc soln of acetonitrile with hydrazine hydrate in air(Ref 2) or by other methods listed in Refs 1,3 & 4

Note: Although this compd is not listed in the literature as an expl, it is included here because it is isomeric with 1-amino-4,5-dimethyl-α-vic-triazole, which is an expl

Refs: 1)Beil **26**,29–30 2)K.A.Hofmann & O.Ehrhart, Ber **45**,2732(1912) 3)W.Oberhummer, Monatsh **63**,285(1933) & CA **28**, 2679(1934) 4)H.Aspelung & A.M.Augustson,Acta Acad Aboensis Math Phys **7**,No 10, 1–7(1933) & CA **29**,5088(1935)

Aminodimethyltriazoles, Azido Derivatives, $C_4H_7N_7$ – not found in Beil or CA through 1956

Aminodimethyltriazoles, Diazido Derivatives, $C_4H_6N_{10}$, – not found in Beil or CA through 1956

Nitrated and/or Nitrited Aminodimethyltriazoles were not found in Beil or CA through 1956

4-Amino-3,5-dioxo-1,2,4-triazolidine o. Urazine, 4-amino-[1H-1,2,4-triazole-3,5 (2H,4H)-dione]. See 4-Aminourazole

Aminodiphenyl. See Aminobiphenyl

AMINODIPHENYLAMINES AND DERIVATIVES

Aminodiphenylamines, $H_2N \cdot C_6H_4 \cdot NH \cdot C_6H_5$. The isomers 2-, 3- & 4-aminodiphenylamines are described in Beil **13**,16,76,(623) & [13,26,40]

Aminodiphenylamines, Azido Derivatives, $C_{12}H_{11}N_5$, – not found in Beil or CA through 1956

Aminodiphenylamines, Diazido Derivatives, $C_{12}H_{10}N_8$ – not found in Beil or CA through 1956

Mononitroaminodiphenylamines, $H_2N \cdot C_6H_4–NH–C_6H_4 \cdot NO_2$, mw 229.23, N 18.33%. The isomers:2'-nitro-2-amino, 4'-nitro-2-amino-, 2'-nitro-3-amino, 4'-nitro-3-amino-, 2'-nitro-4-amino- and 4'-nitro-4-aminodiphenylamine are described in Beil **13**, 17,41,78–9

Mononitroaminodiphenylamines, $H_2N(O_2N)\cdot C_6H_3-NH-C_6H_5$, mw 229.23, N 18.33%. The isomers 4-nitro-2-amino- and 6-nitro-2-amino-diphenylamine, are described in Beil **13**,29,(10) & [21]

Nitronitraminodiphenylamines, $C_{12}H_{10}N_4O_4$, mw 274.23, N 20.43%, were not found in Beil or CA through 1956

Dinitroaminodiphenylamines, $H_2N\cdot HN.C_6H_3(NO_2)_2$, mw 274.23, N 20.43%. The isomers 2',4'-dinitro-2-amino-; 2',4'-dinitro-3-amino- and 2',4'-dinitro-4-amino-diphenylamine are described in Beil **13**,41 & (7)

Dinitroaminodiphenylamines, $H_2N(O_2N)C_6H_3\cdot NH\cdot C_6H_4\cdot NO_2$, mw 274.23, N 20.43%. The isomer 4,3'-dinitro-2-aminodiphenylamine is described in Beil **13**,(10)

Dinitroaminodiphenylamines, $H_2N(O_2N)_2C_6H_2\cdot -NH-C_6H_5$, mw 274.23, N 20.43%. The isomers 4,6-dinitro-3-amino- and 2,6-dinitro-4-amino-diphenyl-amine are described in Beil **13**, & [32,60]

Dinitronitraminodiphenylamines, $C_{12}H_9N_5O_6$, mw 319.23, N 21.94%. Not found in Beil or CA through 1956

Trinitroaminodiphenylamines, $C_{12}H_9N_5O_6$, mw 319.23, N 21.94%, OB to CO_2 −112.8%. The following isomers are described in the literature:

2',4',6'-Trinitro-2-amino-diphenylamine or **N-Picryl-o-phenylenediamine,** $H_2N\cdot C_6H_4\cdot NH-C_6H_2(NO_2)_3$. Red crysts(from xylene), mp 177–8°(decomp with frothing), expl on rapid heating; nearly insol in w, diff sol in eth & alc, easily sol in acet, NB & xylene. Can be prepd by heating an alc mixt of o-phenylenediamine, picryl acetate and K acetate at 50°, followed by washing the resulting crysts with warm w, warm alc and then recrystallizing from boiling xylene

Refs: 1)Beil **13**,17 2)H.Leemann & E. Grandmoujin, Ber **41**,1308(1908)

2',4',6'-Trinitro-3-amino-diphenylamine or **N-Picryl-m-phenylenediamine,** $H_2N\cdot C_6H_4\cdot NH-C_6H_2(NO_2)_3$. Red crysts(from acet), mp 206–7°, expl on rapid heating; easily sol in acet, diff sol in alc & AcOH. Can be prepd by a 2-hr heating of picryl chloride with equivalent quantities of m-phenylenediamine-hydrochloride and Na acetate in alc

Refs: 1)Beil **13**,41 & [26] 2)G.Jaubert, Ber **31**,1181(1898)

2,4,6-Trinitro-3-aminodiphenylamine, $H_2N\cdot C_6H(NO_2)_3-NH-C_6H_5$. Red ndls(from acet by pptg with alc), mp 191°. Can be prepd by heating a mixt of aniline and 2,3,4,6-tetranitroaniline in benz, or by other methods. Its expl props were not examined

Refs: 1)Beil **13**,61 & (17) 2)C.F.vanDuin, Rec **38**,94(1919)

2',4',6'-Trinitro-4-amino-diphenylamine or **N-Picryl-p-phenylenediamine,** $H_2N\cdot C_6H_4-NH-C_6H_2(NO_2)_3$. Dk red(almost black) crysts (from et acet), mp 185–7°, expl on rapid heating; sol in et acet & amyl alc, v sol in boiling AcOH or chlf. Can be prepd by treating picryl chloride with p-phenylenediamine in alc, or by other methods

Refs: 1)Beil **79** 2)E.Wedekind, Ber **33**,435 (1900) 3)R.Ciusa & C.Agostinelli, Atti AccadLin (5)**15II**,240(1906) 4)G.T.Morgan & M.G.Micklethwait, JCS **93**,608(1908) 5)T.C. Jones et al, JCS **117**,1278(1920)

3,2',4'-Trinitro-4-aminodiphenylamine, $H_2N\cdot C_6H_3(NO_2)-NH-C_6H_3(NO_2)_2$. Red-brn ndls (from AcOH), mp 226°. Can be prepd from 2-nitro-1,4-phenylenediamine and 4-chloro-1,3-dinitrobenzene. Its expl props were not investigated

Refs: 1)Beil **13**,121 2)Höchster Farbwerke, GerP 110,360(1899) & ChemZtr **1900** II,301 3)F.Reverdin & E.Delétra, Ber **37**,1727(1904)

Trinitronitraminodiphenylamines, $C_{12}H_8N_6O_8$, mw 364.23, N 23.08% – were not found in Beil or CA through 1956

Tetranitroaminodiphenylamines, $C_{12}H_8N_6O_8$, mw 364.23, N 23.08%, OB to CO_2 –87.86%. The following isomer is listed in Beil:

2,4,6,3'-Tetranitro-3-amino-diphenylamine {Called in Beil N-[3-Nitro-phenyl]-2,4,6-trinitrophenylendiamin-(1,3)}, $H_2N(O_2N)_3C_6H-NH-C_6H_4 \cdot NO_2$. Crysts(from et acet), mp 272°(decomp). Can be prepd either by heating 2,3,4,6-tetranitroaniline with 3-nitrobenzene in benzene or by fusing N-nitro-N-methyl-2,4,6-trinitro-1,3-phenylenediamine with 3-nitroaniline at 110–120°. Its expl props were not reported

Refs: 1)Beil **13**,(17) 2)C.F.van Duin, Rec **38**,95(1919)

Tetranitronitraminodiphenylamine, $C_{12}H_7N_7O_{10}$, mw 409.23, N 23.96% and *Pentanitroaminodiphenylamine,* $C_{12}H_7N_7O_{10}$, mw 409.23, N 23.96% were not found in Beil or CA through 1956

AMINODIPHENYLENEOXIDES AND DERIVATIVES

Aminodiphenyleneoxides or **Aminodibenzofuranes,** $C_{12}H_9NO$, mw 183.20, N 7.65%, may be considered as parent compds of derivs listed below. The isomers 2-amino- and 3-aminodiphenyleneoxide are known

Refs: 1)Beil **18**,587(557) & [422–3] 2)F. Brumberg, Doctoral Dissertation, Göttingen (1925) 3)N.M.Cullinane, JCS **1930**,2268 4)Ibid, **1932**,2367 5)H.Gilman et al, JACS **56**,2475(1934)

Aminodiphenyleneoxides, Azido Derivatives, $C_{12}H_8N_4O$ – not found in Beil or CA through 1956

Aminodiphenyleneoxides, Diazido Derivatives, $C_{12}H_7N_7O$ – not found in Beil or CA through 1956

Mononitroaminodiphenyleneoxides, $C_{12}H_8N_2O_3$, mw 228.20, N 12.28%. The following isomers are described in the literature: 3-nitro-2-aminodiphenyleneoxide, mp 222°(Refs 1 & 2), 2-nitro-3-aminodiphenyleneoxide, mp 238–9° (Refs 1 & 2) and 6-nitro 2-aminodiphenyleneoxide, mp 268°(Ref 3)

Refs: 1)Beil **18**,[422–3] 2)F.Brumberg, Doctoral Dissertation, Göttingen(1925), pp 12,22 & 27 3)N.M.Cullinane, JCS **1932**,2367

Dinitrodiphenyleneoxides, $C_{12}H_7N_3O_5$, mw 273.20, N 15.38%, – not found in Beil or CA through 1956

Trinitrodiphenyleneoxides, $C_{12}H_6N_4O_7$, mw 318.20, N 17.61% – not found in Beil or CA through 1956

Tetranitrodiphenyleneoxides, $C_{12}H_5N_5O_9$, mw 363.20, N 19.28%. The following isomer is described in Beil:

x-Tetranitro-3-aminodiphenyleneoxide, dk red ndls decompg above 280°; diff sol in alc, acet, chlf & NB, more sol in AcOH. Was prepd by heating 3-bromo-x-tetranitrodiphenyleneoxide with alc NH_3 in a sealed tube at 150°. This compd is undoubtedly a mild expl, but its expl props were not examined

Refs: 1)Beil **18**,[423] 2)F.Brumberg, Doctoral Dissertation, Göttingen(1925),30

Pentanitrodiphenyleneoxides, $C_{12}H_4N_6O_{11}$, and higher nitrated derivs were not found in Beil or CA through 1956

AMINODIPHENYLETHERS AND DERIVATIVES

Aminodiphenylethers or Aminophenolphenylethers, $H_2N-C_6H_4-O-C_6H_5$, mw 185.22, N 7.56%. Several isomers are described in Beil **13**,359,404,438,(109,147) & [167,227]

Aminodiphenylethers, Azido Derivatives, $C_{12}H_{10}N_4O$ – not found in Beil or CA through 1956

Aminodiphenylethers, Diazido Derivatives, $C_{12}H_9N_7O$ – not found in Beil or CA through 1956

Mononitroaminodiphenylethers, $C_{12}H_{10}N_2O_3$,

mw 230.22, N 12.17%. Several isomers are described in Beil **13**,(121) & [285–6]

Nitraminodiphenylether, $C_{12}H_{10}N_2O_3$ – not found in Beil or CA through 1956

Nitronitraminodiphenylether, $C_{12}H_9N_3O_5$ – not found in Beil or CA through 1956

Dinitroaminodiphenylethers, $C_{12}H_9N_3O_5$, mw 275.22, N 15.27%. One isomer, 2'4'-dinitro-4-amino-diphenylether, $H_2N \cdot C_6H_4-O-C_6H_3(NO_2)_2$, is listed in Beil **13**,438

Dinitronitraminodiphenylethers, $C_{12}H_8N_4O_7$ – not found in Beil or CA through 1956

Trinitroaminodiphenylethers, $C_{12}H_8N_4O_7$, mw 320.22, N 17.50%. The following isomer is described in the literature:

3,2',4'-Trinitro-4-aminodiphenylether, $H_2N(O_2N)C_6H_3-O-C_6H_3(NO_2)_2$. Grn-yel leaflets(from alc), mp 188°. Was obtained from 3-nitro-4-aminophenol and 4-chloro-1,3-dinitrobenzene

Refs: 1)Beil **13**,521 2)F.Reverdin & A. Dresel, Ber **38**,1595(1905)

Trinitronitraminodiphenylethers, $C_{12}H_7N_5O_9$, mw 365.22, N 19.18% – not found in Beil or CA through 1956

Tetranitroaminodiphenylethers, $C_{12}H_7N_5O_9$, mw 365.22, N 19.18%. The following isomer is described in the literature:

3,5,2',4'-Tetranitro-4-aminodiphenylether, $H_2N(O_2N)_2C_6H_2 \cdot O \cdot C_6H_3(NO_2)_2$. Lemon-yel ndls(from AcOH or acet), mp 225–6°; insol in w or in aq soda soln; diff sol in alc, benz, chlf & ligroin. Can be prepd by heating 3,5-dinitro-4-aminophenol with alc soln of 4-chloro-1,3-dinitrophenol. Its expl props were not investigated, although it is probably an expl

Refs: 1)Beil **13**,529 2)F.Reverdin & A. Dresel, Ber **38**,1594(1905)

Tetranitronitraminodiphenylethers, $C_{12}H_6N_6O_{11}$, mw 410.22, N 20.49% – not found in Beil or CA through 1956

AMINOETHANE AND DERIVATIVES

Aminoethane or **Ethylamine**, $CH_3 \cdot CH_2 \cdot NH_2$, mw 45.08, N 31.07%. Col liq, mp –80.6°, bp 16.6°, d 0.689 at 15°/15°; easily inflammable. Forms numerous salts and other derivs. Prepn and props are given in Beil **4**,87–94,(342–5) & [586–9]

Aminoethane, Azido Derivative, called **β-Azidoaminoethane, β-Azidoethylamine or β-Triazoethylamine**, $N_3 \cdot CH_2 \cdot CH_2 \cdot NH_2$, mw 84.08, N 66.64%. Liq, bp 47° at 16.5 mm, d 1.0429 at 25/4°, N_D 1.4635 at 25°; decomp vigorously on contact with H_2SO_4. This high nitrogen compd was prepd and investigated as described in the refs

Refs: 1)Beil **4**,(360) 2)M.O.Forster & H.S.Newman, JCS **99**,1278(1911) 3)Th.Curtius et al, Ber **45**,1086(1912) 4)J.C.Philip, JCS **101**,1868(1912)

Mononitroaminoethane, $O_2N \cdot C_2H_4 \cdot NH_2$, mw 90.08, N 31.10% – not found in Beil

Nitraminoethane, N-Nitroethylamine or **Ethylnitramine**, $CH_3 \cdot CH_2 \cdot NHNO_2$, mw 90.08, N 31.10%. Col, non-volatile liq with acidic reaction, mp +6°, d 1.1675 at 15°, Q_c^v 372.82 kcal/mol and Q_f 23.1 kcal/mol(Ref 4). Was prepd in 1888 from ethylester of N-ethylcarbamic acid and nitric acid(Ref 3). Other methods of prepn are given in Refs 3 & 5.

Nitraminoethane forms numerous salts, of which the following are explosive: $Ba(C_2H_5N_2O_2)_2$, mp 228°, expl at higher temps; $Cu(C_2H_5N_2O_2)_2 + 2H_2O(?)$ – expl on rapid heating; and $Hg(C_2H_5N_2O_2)_2$ – expl on heating Ref 2)

Refs: 1)Beil **4**,569(568) & [968] 2)A.P.N. Franchimont & E.A.Klobbie, Rec **7**,355–6 (1888) 3)H.Umgrove & A.P.N.Franchimont, Rec **16**,388–93(1896) 4)F.Swarts, Rec **32**, 78(1913) 5)L.C.E.Kniphorst, Rec **44**,697 & 702(1925) 6)G.Kortum & B.Finkh, ZPhysChem B**48**,32(1940)(Ultraviolet absorption spectra)

Nitronitraminoethane, $O_2N \cdot C_2H_4 \cdot NHNO_2$, mw 135.08, N 31.11%, was not found in Beil or in CA through 1956

1-Nitramino-2-nitroxyethane, known as NENA, is a deriv of aminoethanol(qv)

Aminoethanecarboxylic Acid. See Aminopropanoic or Aminopropionic Acid

Aminoethanediperoxide or Ethylaminoperoxide, $H_2N \cdot C_2H_5 \cdot 2H_2O_2$, oil; obtained by treating a concd ethereal soln of aminoethane with a concd ethereal soln of H_2O_2. Its expl props have not been examined

Refs: 1)Beil – not found 2)G.L.Matheson & O.Maas,JACS **51**,680–1(1929)

Aminoethanoic Acid. Same as Aminoacetic Acid or Glycine

AMINOETHANOL(ETHANOLAMINE) AND DERIVATIVES

Aminoethanol; Monoethanolamine; Aminoethyl Alcohol or β-Hydroxyethylamine (Aminooxyäthan in Ger), $H_2N \cdot CH_2 \cdot CH_2 \cdot OH$, mw 61.08, N 22.93%. Prepn and props in Beil **4**,274(424) & [717]

Note: Aminoethanol(monoethanolamine) intended for use by the US Ordnance Corps must comply with the requirements of Specification MIL– M–2776

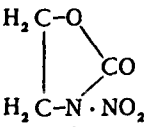
Aminoethanol, Azido Derivative, $C_2H_6N_4O$ – not found in Beil or CA through 1956

Aminoethanol, Diazido Derivative, $C_2H_5N_7O$ – not found in Beil or CA through 1956

Aminoethanol-bis[copper(II) diazide], Ethanolamine di(cupric azide) or Monoethanolaminotetrazido-copper, $[(N_3)_2 \cdot Cu – H_2N \cdot CH_2 \cdot CH_2 \cdot OH–Cu(N_3)_2]$, dk grn crysts explg ca 186° or when thrown on a preheated metal block. It was obtained in a impure state and in small yield from Cu diazide and aminoethanol)

Refs: 1)Beil – not found 2)M.Straumanis & A.Cirulis,ZAnorgChem **251**, 352-3(1943) & CA **37**,6574(1943)

1-Nitramino-2-ethanol or β-Nitraminoethyl Alcohol (1-Hydroxy-2-nitramino-ethane) (α-Oxy-β-nitramino-äthan, in Ger), $(O_2N.HN)CH_2 –CH_2 \cdot OH$, mw 106.08, N 26.41%. Thick, col syrup, misc with w. Was prepd by boiling 3-nitroöxoöxazoletetrahydride,



(called in Ref 2 μ-céto-N-nitrotetrahydro-oxazol), with water

Its *silver salt,* $AgC_2H_5N_2O_3$, a col or sl greyish powd, detonated on heating but not as violently as the *mercuric salt,* $Hg(C_2H_5N_2O_3)_2$, white, fine ndls; very sl sol in w

Refs: 1)Beil **4**,573–4 2)A.Franchimont & A.Lublin,Rec **21**,50–4(1902) 3)J.Vaughan, JCS **1950**,748–9 & CA **44**,6818(1950) {Prepn of 2-nitraminoethanol by hydrolysis of $[CON(NO_2)CH_2CH_2ONO_2]_2$}

Aminoethanol(Ethanolamine) Dinitrate; Nitroxyaminoethane Nitrate or Nitroxyethylammonium Nitrate [β-Nitroxyethylamine Nitrate, Nitroxyethanolamine Nitrate(Called in CA Formula Index vol **50**,p 12F, 2-Aminoethanol Nitrate *Nitrate)* or Nitroxyethylammonium Nitrate], $O_2NO \cdot CH_2 \cdot CH_2 \cdot NH_3 \cdot NO_3$ or $(NO_3)(H_3N \cdot CH_2 \cdot CH_2 \cdot ONO_2)$, mw 169.10, N 24.85%, OB to CO_2 –14.2%. Wh crysts, mp 103°, d 1.53(cast). Can be prepd by the nitration of aminoethanol with concd HNO_3 or mixed $HNO_3–H_2SO_4$(Refs 2 & 3). It is a powerful expl, with a Trauzl value of 78.5% of NG, or 93% of TNT, but it is unstable, acidic and hygroscopic. Although it was patented in Germany for use in expls (Ref 2), Médard(Ref 5) does not consider it suitable for that purpose on account of its extreme hygroscopicity(See also Ref 4)

Refs: 1)Beil – not found 2)Dynamit A–G, GerP 500,407(1929) & CA **24**,4397(1930); GerP 514,955(1929) & CA **25**,2739(1931); GerP 516,284(1929) & CA **25**,3362(1931) 3) Aubry,MP **25**,189–191(1932–3) 4)Blatt, OSRD Rept 2014(1944) 5)L.Médard, MP **36**, 93(1954) & CA **50** 6795(1956)

1-Nitramino-2-ethanol Nitrate; N-(β-Nitroxyethyl) nitramine or 1-Nitramino-2-nitroxy-ethane or NENA(called in Ref 4 **1-Nitroxy-3-nitro-2-aza-propane**), $(O_2N \cdot HN) \cdot CH_2 \cdot CH_2 \cdot ONO_2$, mw 151.08, N 27.81%, OB to CO_2 −15.9%, OB to CO +5.3%. Yel oil, fr p 15°; volatizes rapidly at 360° without explg; but it expld when struck with a 2 kg hammer falling from a height ca 107 cm(50% pt) vs 33 cm for RDX. When heated at 135°, it became acidic in 75 mins. It is nearly insol in w. Power by ballistic mortar test 133.9%(TNT = 100%). Its UV absorption spectra are discussed in Ref 4

NENA can be prepd from aminoethanol and ethylchlorocarbonate by the following series of reactions:

a) $H_2N \cdot CH_2 \cdot CH_2 \cdot OH + C_2H_5 \cdot COOCl +$ aqNaOH ⟶ $C_2H_5 \cdot COO \cdot HN \cdot CH_2 \cdot CH_2 \cdot OH$

b) Add the reaction mixt dropwise with stirring to 98% HNO_3 at 10°: $C_2H_5 \cdot COO \cdot HN \cdot CH_2 \cdot CH_2 \cdot OH \xrightarrow{HNO_3} C_2H_5 \cdot COO \cdot N(NO_2) \cdot CH_2 \cdot CH_2 \cdot ONO_2$

c) Add NH_3 to the ethereal soln of the above nitrate(ammonolysis) and then add HCl immediately

$C_2H_5 \cdot COO \cdot N(NO_2) \cdot CH_2 \cdot CH_2 \cdot ONO_2 \xrightarrow[\text{followed by HCl}]{NH_3} O_2N \cdot HN \cdot CH_2 \cdot CH_2 \cdot ONO_2$

NENA is of interest from the standpoint of expl props because it possesses a structure intermediate betn ethyleneglycoldinitrate (EGDN) and ethylenedinitramine(EDNA). It was proposed as a gelatinizer of NC for use in propellants and also as an ingredient of some expl compns

NENA being sl acidic(pH 2.7), can form salts, some of them expl: *silver salt*, $C_2H_4N_3O_5Ag$, wh solid darkening on exposure to light and decompg ca 120°(Ref 1, p 72)

Refs: 1)Beil – not found 2)A.T.Blomquist & F.T.Fiedorek, OSRD Rept 4134 & PB Rept 18867(1944), 30–31 3)Ibid,USP 2,485,855(1949) & CA **44**,3516–17(1950) 4)R.N.Jones & G.D.Thorn, CanJRes **27B**, 829 & 838–9(1949)

2-Amino-2,2-dinitroethanol or 2,2-Dinitromonoethanolamine, $H_2N \cdot C(NO_2)_2 \cdot CH_2OH$, mw 151.08, N 27.81% – not found in Beil or CA through 1956. It may be considered as the parent compd of its K salt although the salt was not prepd from it

Its *potassium salt*, $H_2N \cdot C(NO_2)_2 \cdot CH_2OK$, yel pdr, prepd from K dinitroethanolate and NH_3, as described in patents listed as Ref 2 It was proposed as a potential ingredient of expl and propellent compns

Refs: 1)Beil – not found 2)F.R.Schenck & G.A.Wetterholm, SwedP 148,217(1954); BritP 129,469(1955); USP 2,731,460(1956) (Example 7) & CA **50**,1893,7125(1956)

Aminoethanol, Alkylderivatives of. N-alkyl derivatives of aminoethanol and of aminodiethanol were patented in Germany for use as/or in explosives.

Refs: 1)Beil – not found 2)Dynamit A–G, GerP 513,653(1930) & CA **25**,1675(1931)

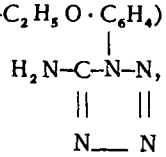
Aminoethanol Derivatives, Proposed by E. von Herz for use as/or in expls included among other compds the acyl- and sulfonyl-derivs of nitrated aminoethanol. As an example of an *acyl derivative* may be cited the compd, $[O_2NO \cdot CH_2 \cdot CH_2 \cdot N(NO_2)CO]_2$, mp 88°, prepd by the condensation of $HO \cdot CH_2 \cdot CH_2 \cdot NH_2$ with $(COOH)_2$, followed by nitration. As an example of a *sulfonyl (sulfuryl) derivative* may be cited $[O_2NO \cdot CH_2 \cdot CH_2 \cdot N(NO_2)]SO_2$, prepd by condensing $HO \cdot CH_2 \cdot CH_2 \cdot NH_2$ with SO_2Cl_2 followed by nitration(Ref 3). The same inventor proposed another deriv of amino ethanol, $C_6H_2(NO_2)_3 \cdot N(NO_2) \cdot CH_2 \cdot CH_2 \cdot ONO_2$, which may be called , *1-nitroxy-N-nitro-N-(2',4',6'-trinitrophenyl)-aminoethane* or *trinitrophenylethanolnitramine nitrate*(Ref 2)

Refs: 1)Beil – not found 2)E.von Herz, GerP 530,704(1930) & CA **26**,309(1932) 3)E.von Herz, GerP 543,174(1930) & CA **26**, 2598(1932)

AMINOETHOXYPHENYLTETRAZOLES AND DERIVATIVES

Aminoethoxyphenyltetrazoles, $C_9H_{11}N_5O$, mw 205.22, N 34.13%. The following isomer is described in the literature:

5-Amino-1-p-ethoxyphenyl-α-vic-tetrazole or *1-p-Ethoxyphenyl-5-amino-1H-1,2,3,4-tetrazole,*



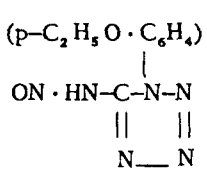
wh ndls, mp 197°(with previous shrinkage); fairly sol in hot w, nearly insol in cold w & eth, sol in hot alc & sl sol in cold alc. Can be prepd by passing a stream of CO_2 gas through a boiling mixt of p-ethoxyphenylthiourea, $PbCO_3$ and NaN_3 in alcohol. Its nitroso compd is expl(see below)

Refs: 1)Beil – not found 2)R.Stollé et al, JPrChem **134**,282–3 & 301(1932)

Azidoaminoethoxyphenyltetrazoles, $C_9H_{10}N_8O$, – not found in Beil or CA through 1956

Diazidoaminoethoxyphenyltetrazoles, $C_9H_9N_{11}O$ – not found in Beil or CA through 1956

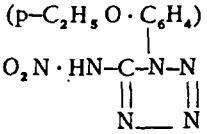
5-Nitrosamino-1-p-ethoxyphenyl-α-vic-tetrazole,



mw 234.22, N 35.88). Wh ndls(from acet), mp defgr ca 117°; insol in w, sl sol in hot benz, fairly sol in eth, sol in alc & acet. Can be prepd by adding dropwise to a soln of 5-amino-1-p-ethoxy-phenyl-α-vic-tetrazole in dil HCl a calcd amt of aq $NaNO_2$. The resulting ppt is treated with dil soda soln and dil HCl is added to the dissolved portion. The portn undissolved in soda soln is unreacted aminoethoxyphenyltetrazole

Refs: 1)Beil – not found 2)R.Stollé et al, JPrChem **134**,282–3 & 302(1932)

5-Nitramino-1-p-ethoxyphenyl-α-vic-tetrazole,



mw 250.22, N 33.59% – not found in Beil or CA through 1956

Note: No higher nitrated derivs were found in the literature

Aminoethylamine. See Ethylenediamine

Aminoethylation. In connection with its general research activities in the field of solid propellants, the Interior Ballistics Laboratory of the BRL(Ballistics Research Laboratory) of Aberdeen Proving Ground studied the polyaminoethylation of cellulose in an exploratory program. The product of this reaction process was called AEC, *aminoethylcellulose.* It was formed by the graft polymerization of *ethyleneimine* onto cellulose and could be perchlorated to yield a reasonably stable product AECP, *aminoethylcellulose perchlorate.* The subsequent successful course of the preparative and burning-characteristic studies led to an expanded program of research and development in this area. Under a contract with the Department of the Army, Ordnance Corps, the Wyandotte Chemicals Corporation, Wyandotte, Mich, undertook an investigation comprising research and development work on AEC, AECP and other fast burning propellants

By March 1, 1957, the aminoethylation of the following materials and compds had been achieved by the Wyandotte Chemicals Corp: cellulose, cellulose derivatives, regenerated cellulose, 2-hydroxymethyl-2-nitro-1,3-propanediol, nylon, polyurethane, polyvinyl alcohol, polyvinylchloride, protein(wool), starch and toluene diisocyanate

Refs: 1)T.S.Gardner, JPolSci **1**,289(1946) 2)L.M.Soffer et al,BRL Memo Rept No 674, Apr 1953 3)L.M.Soffer et al,TextileJRes **24**, 847(1954) and Refs 4)C.T.Lenk et al of Wyandotte Chemicals Corp,"Studies on the

Preparation of Aminoethylcellulose Perchlorate and Other Fast-Burning Propellants'', Summary Reports: No 1(June 1955), No 2 (May 1956) and No 3(June 1957); Contract No DA-20-018-ORD-13364, Project No TB 3-0230, Wyandotte, Mich

Aminoethylation of Cellulose Derivatives and of Regenerated Cellulose. Cellulose obtained by regeneration from a cuprammonium soln of cotton(in the manner described in Rept No 2,p 14) from the Wyandotte Chem Corp gave an amorphous product contg ca 23.5% N when heated with ethyleneimine in a sealed tube at 120° in the presence of toluene. The cellulose derivatives *carboxymethylcellulose* and *hydroxyethylcellulose* (prepd in the manner described in Rept No 3, pp 14-18) gave solid products with nitrogen contents of 24.8 and 28.1% respectively, when heated with ethyleneimine in a sealed tube, in the manner described in Rept No 1, p 6

All the above products resembled aminoethylcellulose and could be perchlorated in the manner described under Aminoethylcellulose Perchlorate

Refs: C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast-Burning Propellants'', Wyandotte Chem Corp Summary Repts No 1(1955), No 2(1956) and No 3(1957)

Aminoethylation of N-(2-Hydroxypropyl)-ethylenediamine. "Monolene'', which is the trade name of the Wyandotte Chemicals Corp for *N-(2-hydroxypropyl)-ethylenediamine,*,

$$(CH_3 \cdot \underset{\displaystyle OH}{\underset{|}{CH}} \cdot CH_2) \cdot HN \cdot CH_2 \cdot CH_2 \cdot NH_2,$$

on treatment with ethyleneimine, in the manner described in Rept No 2,p 18, yielded a dark solid ppt contg 29.2% N

Ref: C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast-Burning Propellants'', Wyandotte Chemicals Corp Summary Rept No 2

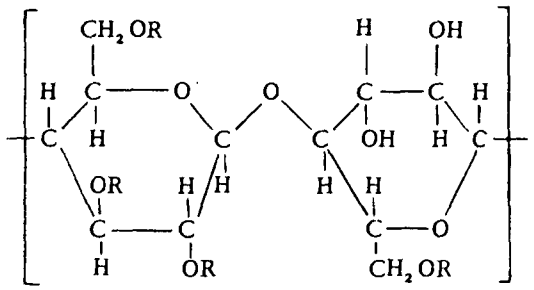
Aminoethylation of Toluene Diisocyanate (AETDI). Inasmuch as the aminoethylation of polyurethane had apparently degraded the polyurethane and yielded a methanol-insol product, it was of interest to det whether *toluene diisocyanate,* $CH_3 \cdot C_6H_3(NCO)_2$, a precurser constituent of polyurethane, could also be aminoethylated to yield an insol product

For aminoethylation, a soln of 1 g of toluene diisocyanate(80% 2,4-isomer and 20% 2,6-isomer) in 10 ml toluene was heated for 16 hrs at 100° in a sealed tube with 10 ml of ethyleneimine and 0.1 ml of benzyl chloride. The material insoluble in the cooled reaction mixt was washed with methanol and ether and dried. The yield was 8.6 g for a 98% conversion of ethyleneimine to AETDI. The product was a white rubbery solid which swelled strongly in methanol, gradually decompd at ca 150° and melted when placed on a block at 300°. When 3.0 g of this product was perchlorated with 70% perchloric acid in methanol, the AETDIP was obtained in a low yield(4.7 g instead of the calcd 7.0 g) but the perchlorate burned readily leaving only a small residue

Ref: Same as under Aminoethylvinyl Chloride, Rept No 3,p 29

Aminoethylcellulose(AEC) (Polyaminoethylated Cellulose). The aminoethylation of cellulose with ethyleneimine had been studied prior to WW II but not for the purpose of using the product as a propellant. Most of the aminoethylated celluloses prepd before work was undertaken by the BRL and the Wyandotte Chem Corp under contract with the Dept of the Army, Ordnance Corps(see Ref 1 under Aminoethylation Reactions) contained only a small amt of nitrogen. The material prepd at BRL contained up to 20% N(Refs 2 & 3) and the material prepd by the Wyandotte Corp contained 26+% N (Ref 4)

The structure and formula of AEC has not been clearly established but is assumed to be:



where R = $(CH_2CH_2NH)_nCH_2CH_2NH_2$ and n is an integer usually between 4 and 8

It seems that the ethyleneimine graft polymerizes on the hydroxy groups of cellulose. It is not known to what extent the three hydroxyls of the anhydroglucose are involved in the reaction but it is safe to assume that the more reactive OH group of the 6th carbon would be more aminoethylated than other OH groups

For discussion of the reaction of cellulose with ethyleneimine see Refs 1,2 & 3

For the prepn of AEC with a N content of ca 20%, BRL treated a small amt of cellulose with a large excess of ethyleneimine in a bomb reactor at 160–200° for 6–20 hrs and recycled the product 2 or 3 times under the same conditions to get a higher N content (Refs 2 & 3). Later work(Ref 4) raised the N content to a max of 28.8%. This material was used for the prepn of fast burning salts, such as the perchlorates and nitrate. A detailed description of the prepn of high nitrogen AEC is given in Ref 4, Rept No 1,p 6

Refs: Same as under Aminoethylation

Aminoethylcellulose Nitrate(AECN). This salt was prepd by adding 50 ml of 69% nitric acid to a vigorously stirred mixt of 10 g aminoethylcellulose (27.2% N) and 75 ml absolute methanol and then continuing to stir for 9 hours. After leaving the resulting slurry under refrigeration overnight, it was filtered and the ppt washed with methanol, then with ether, and dried over P_2O_5 in vacuo. Analysis by the gravimetric nitron method showed ca 53% HNO_3, indicating an approx 90% conversion to AECN. The material burned slowly, leaving considerable residue

Ref: C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast Burning Propellants", Wyandotte Chem Corp, Summary Rept No 3(1 June 1957),18

Aminoethylcellulose Perchlorate(AECP) or Polyaminoethylcellulose Perchlorate. This salt was prepd by adding 70% perchloric acid to a vigorously stirred AEC(aminoethylcellulose)methanol mixt cooled with an ice bath. A series of methanol washes removed excess acid from the salt(Ref 1 and Ref 2,No 2,p 5). Yields of 80–85% conversion were obtained with the N content of the product 10–11% and the Cl content 20–21%. The N content of AEC used for preparing the perchlorate was 26+%

Following are some properties of AECP: *explosion temp*(PA method) 305–10°(5 sec); *impact test* with 2kg wt–detonated at 12"; *hygroscopicity*(% gain in wt at RT and 77% RH) 23% after 6 days and 22.3% after 13 days; *thermal stability*–relatively stable at 85° for long periods of time but decomp extensively at 125° within a week; *tensile strength*–decreases with increase in perchlorate content; *solubility*–insol in common solvents, sometimes dissolved at elevated temps with decompn, swelled in some polar liquids; dissociated to some extent in H_2O; *compatibility with NC*–incompatible

It seems that this substance is not as suitable for use in propellants as the polyethyleneimine perchlorate(qv)

Refs: 1)L.M.Soffer et al, BRL Memo Rept No 674,Apr(1953) 2)C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast Burning Propellants", Wyandotte Chem Corp, Summary Repts: No 1(1955), No 2(1956) and No 3(1957)

AMINOETHYLGUANIDINE AND DERIVATIVES

Aminoethylguanidine, $H_2N \cdot CH_2 \cdot CH_2 \cdot NH \cdot C(:NH) \cdot NH_2$, may be considered as the parent compd of the following derivs:

Azidoaminoethylguanidine, $C_3H_9N_7$ – not found in Beil or CA through 1956

Diazidoaminoethylguanidine, $C_3H_8N_{10}$ – not found in Beil or CA through 1956

1-β-Nitraminoethyl-1-nitroso-2-nitroguanidine, $O_2N \cdot HN \cdot CH_2 \cdot CH_2 \cdot N(NO) \cdot C(:N \cdot NO_2) \cdot NH_2$, mw 221.14, N 44.34%. Yel crysts(from abs methanol), mp 113° with decompn. Was obtained by treating 1-nitro-2-amino-2-nitraminoimidazolidine, $CH_2-N(NO_2)-C(NH_2)(NH \cdot NO_2)$ with CH_2 ——— NH with $NaNO_2$ and aq HNO_3. Its expl props were not examined. Being a high nitrogen compd, it might prove to be useful as a component of propellants

Refs: 1)Beil – not found 2)R.H.Hall, A.F. McKay & G.F.Wright, JACS **73**,2207(1951) & CA **46**,1988(1952)

Note: No higher nitrated or nitrited derivs were found in Beil or CA through 1956

β-Aminoethylnitramine. See under Ethylenediamine

Aminoethylnylon(AEN) and Its Perchlorate (AENP). A product(AEN) contg 29.2 to 29.8% N was obtained by heating a small amt of nylon with ethyleneimine in a sealed tube. The procedure was the same as for aminoethylation of cellulose(see Rept No 1,p 6). AENP was prepd by adding, with stirring, a soln of 50 ml of 70% perchloric acid in 200 ml ethanol to 4 g AEN(28.8% N) in 100 ml ethanol. After standing for several hours the AENP was filtered off, washed by decantation with methanol and ether and dried. The product contained ca 20.2% Cl and burned readily but with more smoke than aminoethylcellulose perchlorate (See also Aminoethylation)

Ref: C.T.Lenk, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast-Burning Propellants", Wyandotte Chemicals Corp, Summary Repts: No 1(1955), p 6 and No 3(1957),pp 23–26

Aminoethylpolyurethane(AEPU). A white, rubbery solid contg ca 30.6% N was obtained by treating 1 g of polyurethane(dissolved in 25 ml of toluene) with 10 ml ethyleneimine and 0.1 ml benzyl chloride in a sealed tube at 100° for 43 hours. Material remaining insol in the cooled reaction mixt was washed with methanol and ether and then dried. Aminoethylation of polyurethane was accompanied by degradation. The dried product should be suitable for perchloration(See also Aminoethylation of Toluene Diisocyanate)

Ref: Same as under Aminoethylpolyvinyl Chloride

Aminoethylpolyvinyl Alcohol(AEPVA) and Its Perchlorate(AEPVAP). Treatment of PVA("Elvanol") with ethyleneimine in a bomb reactor at 100° as indicated in Rept No 2,p 37, produced AEPVA with as high as 26.8% N. Treatment of the dry product with 70% perchloric acid in the manner used for aminoethylcellulose perchlorate(AECP) yielded the perchlorate(AEPVAP) with 20.3% Cl and 10.8% N(See also Aminoethylation)

Ref: C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose and Other Fast-Burning Propellants", Wyandotte Chemicals Corp, Summary Repts: No 2(1956), pp 34–38 and No 3(1957),p 19

Aminoethylpolyvinyl Chloride(AEPVC). A deep red, gummy product, difficult to work with and contg 25.6% N, was obtained by treating one gram of PVC(in 10 ml toluene) with 10 ml of ethyleneimine and 0.1 ml benzyl chloride in a sealed tube at 100° for 43 hours. Material remaining insol in the cooled reaction mixt was washed with methanol and ether and finally dried(See also Aminoethylation)

Ref: C. T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and

Other Fast-Burning Propellants'', Wyandotte Chemicals Corp, Summary Rept No 3(1957), pp 27–29

Aminoethylprotein(AEP). A spongy tan gum, contg ca 27.8% N, was prepd by treating wool fabric with ethyleneimine in the manner described under Aminoethylvinylchloride. The aminoethylated wool fabric became a gum when soaked in methanol(See also Aminoethylation Reactions)

Ref: Same as under Aminoethylpolyvinyl Chloride

AMINOETHYLPROPYLUREAS AND DERIVATIVES

Aminoethylpropylureas, $C_6H_{15}N_3O$ may be considered as the parent compds of the following derivs:

Azidoaminoethylpropylureas, $C_6H_{14}N_6O$ – not found in Beil or CA through 1956

Diazidoaminoethylpropylureas, $C_6H_{13}N_9O$ – not found in Beil or CA through 1956

N-(β-Nitraminoethyl)-N'-propyl-urea), $O_2N \cdot HN \cdot CH_2 \cdot CH_2 - HN \cdot CO \cdot NH \cdot C_3H_7$, mw 190.20, N 29.46%, crysts, mp 78.9–80.5°. It was prepd by Hall & Wright as described in Ref 2

Refs: 1)Beil – not found 2)R.H.Hall & G.F.Wright, JACS **73**,2212(1951)

N-(β-Nitraminoethyl)-N-propyl-N'-nitro-urea, $O_2N \cdot HN \cdot CH_2 \cdot CH_2 - N(C_3H_7) \cdot CO \cdot NH \cdot NO_2$, mw 235.20, N 29.78%, crysts, mp 130.2–130.5%. It was prepd by Hall & Wright as described in Ref 2(see above)

Refs – same as above

Aminoethylstarch(AES) and Its Perchlorate. A gummy substance contg ca 28.3% N was prepd by heating anhydrous soluble starch with ethyleneimine in toluene in the presence of a small amt of ethylenechlorohydrin in a sealed tube at 100° for 48 hours and then repeating the procedure. Details of the method are given in Rept No 2,p 39. A product contg ca 26% N was also prepd at atmospheric pressure as indicated in Rept No 2,pp 39–40

Treatment of AES with perchloric acid in the manner described under aminoethylcellulose perchlorate gave *aminoethylstarch perchlorate* (AESP) with a N content ca 14% and Cl 16.8 to 20%

Since the AES prepd from sol starch was not stable at elevated temps, other starches, such as rice, corn, tapioca and potato were investigated(Rept No 3,p 20). None of them can be recommended because agglomeration occurred and workup was difficult

After this, a modified sealed tube procedure was used for soluble starch which gave AES with 28.4% N and the AESP with 21.9% Cl. Details of the procedure are given in Rept No 3,p 20(See also Aminoethylation)

Ref: C.T.Lenk et al, "Studies on the Preparation of Aminoethylcellulose Perchlorate and Other Fast-Burning Propellants'', Wyandotte Chemicals Corp, Summary Repts: No 2(1956), pp 39–40 and No 3(1957),p 20

AMINOETHYLTETRAZOLES AND DERIVATIVES

Aminoethyltetrazoles and Ethylaminotetrazoles, $C_3H_7N_5$, mw 113.13, N 61.91%. The following isomers are described in the literature:

5-Amino-1-ethyl-α-tetrazole or 5-Amino-1-ethyl-1H-tetrazole, $H_2N \cdot C{-}N(C_2H_5){-}N$ (N=C–N=N). Crysts (from w), mp 147–148.5°. It was prepd by Herbst et al (Ref 3) by ethylation of 5-aminotetrazole using the procedure of von Braun & Keller (Ref 2), which is also described in Ref 3, pp 140–1(see also Ref 5)

Refs: 1)Beil – not found 2)J.von Braun & W.Keller, Ber **65**,1677(1932) 3)R.M.Herbst, C.W.Roberts & E.J.Harvill, JOC **16**,140–2 & 146(1951), CA **45**,6630(1951) 4)L.A. Burkardt & D.W.Moore, Anal Chem **24**,1582–3

(1952) (X-ray diffraction pattern) 5)W.G. Finnegan, R.A.Henry & E.Lieber, JOC **18**, 788(1953) & CA **48**,7006(1954) 6)R.A. Henry, W.G.Finnegan & E.Lieber, JACS **76**, 89(1954) & CA **49**,2427(1955) (Thermal isomerization of 5-amino-1-ethyltetrazole)

5-Ethylamino-α-tetrazole or 5-Ethylamino-1H-tetrazole, $(C_2H_5)HN \cdot C(=N-NH-N=)$, crysts, mp 175–6°(Ref 4). This is one of the 5-alkylaminotetrazoles prepd and studied after WW II at the US Naval Ordnance Test Station, China Lake, Calif. It is the product of so-called thermal isomerization of 5-amino-1-ethyl-tetrazole. When 5-amino-1-ethyl-tetrazole is kept in the molten state at ca 200°, about 4% of it is isomerized to 5-ethylaminotetrazole and the following equilibrium is established according to the equation

$$H_2N \cdot C(=N-N(C_2H_5)-N=) \rightleftharpoons (C_2H_5)HN \cdot C(=N-NH-N=)$$ (Refs 3 & 4)

Since the mp of the isomerized product(5-ethylaminotetrazole) is higher than that of 5-amino-1-ethyltetrazole, the equilibrium may be continuously displaced toward the isomerized product by cooling the melt to below the mp of the isomerized product(Ref 3)

The isomerized product can also be prepd directly, similarly to one of the methods of prepn of 5-methylaminotetrazole described in Ref 2,p 785.

Refs: 1)Beil–not found 2)W.G.Finnegan et al, JOC **18**, 780 & 785(1953) & CA **48**, 7006 (1954) 3)R.A.Henry et al, JACS **76**,89(1954) 4)A.G.Whittaker & D.W.Moore, JChemPhys **25**, 366-7(1956) & CA **50**, 15229(1956) (Observation of thermal isomerization of 5-ethylaminotetrazole by nuclear magnetic resonance spectroscopy)

5-β-Aminoethyl-1H-tetrazole, $H_2N \cdot C_2H_4 \cdot C(=N-NH-N=)$. Its hydrochloride, $C_3H_7N_5 \cdot HCl$, crysts, mp 128–9°(from eth–alc), was obtained by refluxing 5-β-benzamidoethyltetrazole suspended in dil hydrochloric acid.

Refs: 1)Beil – not found 2)C.Ainsworth, JACS **75**,5728–9(1953) & CA **49**,6928(1955)

Azidoaminoethyltetrazoles, $C_3H_6N_8$ – not found in Beil or CA through 1956

Diazidoaminoethyltetrazoles, $C_3H_5N_{11}$ – not found in Beil or CA through 1956

Aminoethyltetrazoles, Nitrated and/or Nitrited Derivatives were not found in Beil or CA through 1956

AMINOETHYLTRIAZOLES AND DERIVATIVES

Aminoethyltriazoles, $C_4H_8N_4$, mw 112.14, N 49.97%. The following isomers of this high-nitrogen compd are described in the literature:

3-Amino-5-ethyl-α-sym-triazole or 5-Amino-3-ethyl-1H-1,2,4-triazole [Called in Ger 3-Äthyl-1.2.4-triazolon-(5)-imid],

$$C_2H_5 \cdot C(=N-NH-N=)C \cdot NH_2$$

or

$$H_2N \cdot C(=N-NH-N=)C \cdot C_2H_5$$

or

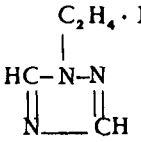
$$HN{:}C(-NH-N=)C \cdot C_2H_5$$

Crysts(from ethyl acetate), mp 152°. Can be prepd from aminoguanidine nitrate and propionic acid. Its *nitrate,* $C_4H_8N_4 \cdot HNO_3$, mw 175.15, N 39.99%, crysts(from et acet + alc), mp 167°, is a mild explosive which is sol in hot w and alc

Refs: 1)Beil **26,** [79] 2)J.Reilly & D.Madden

JCS **1929**,816 & CA **23**,3470(1929) 3)E. Lieber & G.B.L.Smith,ChemRevs **25**,255 (1939)

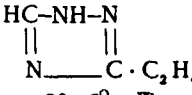
1-(β-Aminoethyl)-α-sym-triazole or 1-(2'-Aminoethyl-1H-1,2,4-triazole,



Its *dihydrochloride*, $C_4H_8N_4 \cdot 2HCl$, N 30.28%, crysts, mp 182–3°, was prepd by hydrolyzing 1β-phthalimidoethyl 1,2,4-triazole with 6 normal HCl(Ref 2)

Refs: 1)Beil – not found 2)C.Ainsworth & R.G.Jones,JACS **77**,621 & 623(1955) & CA **50**,1785(1956)

3-(β-Aminoethyl)-α-sym-triazole or 3-(2'-Aminoethyl-1H-1,2,4-triazole,

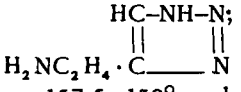


crysts, mp 83–5° . Was obtained by treating 3-β-aminoethyl-1,2,4-triazole dihydrochloride in abs alc with Na methylate(Ref 2)

Its *dipicrate*, $C_{16}H_{14}N_{10}O_{14}$, N 23.97%, crystallized from alc as yell cubes, mp 190°

Refs: 1)Beil – not found 2)C.Ainsworth & R.G.Jones,JACS **75**,4917(1953) 3)Ibid, JACS **76**,5651–4(1954)(Some pharmaceutical props) 4)R.G.Jones & C.Ainsworth,USP 2,710,296(1955) & CA **50**,5768(1956)

4-(β-Aminoethyl)-α-vic-triazole or 1H-1,2,3-Triazole-4-ethylamine,



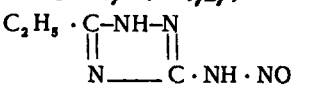
crysts, mp 157.5–159°; sol in w & in hot alc; nearly insol in eth,acet, ethyl acetate & chlf. Was prepd by treating 1, 2,3-triazole-4-ethylamine hydrochloride with Na ethoxide in ethanol, whereas the hydrochloride was obtained from 1,2,3-carboxaldehyde by a series of reactions described in Ref 2

Refs: 1)Beil – not found 2)J.C.Sheehan & C.A.Robinson,JACS **71**,1436 & 1439(1949) 3)C.Ainsworth,JACS **75**,5728(1953)(Pharmaceutical props of some triazoles and tetrazoles)

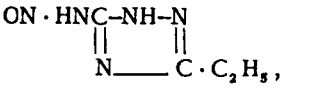
Azidoaminoethyltriazoles, $C_4H_7N_7$ – not found in Beil or CA through 1956

Azidoaminoethyltriazoles, $C_4H_6N_{10}$ – not found in Beil or CA through 1956

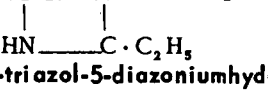
3-Nitrosamino-5-ethyl-α-sym-triazole or 5-Nitrosamino-3-ethyl-1H-1,2,4-triazole,



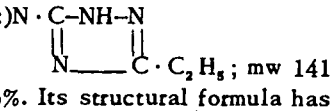
or



also called **5-Nitrosimino-3-ethyl-1H-1,2,4-triazole,** ONN:C–NH–N or



3-Ethyl-1,2,4-triazol-5-diazoniumhydroxide,



; mw 141.14, N 49.63%. Its structural formula has not been definitely established and no methods of prepn or props are given

Refs: 1)Beil **26**[80] 2)J.Reilly & D.Madden, JCS **1929**,816(give only the chloroauric salt of the above compd, under the name of 5-diazo-3-ethyl-1,2,4-chloroaurate, $2C_4H_6N_5Cl_3Au,H_2O$)

Nitraminoethyltriazole, $C_4H_7N_5O_2$ – not found in Beil or CA through 1956

Nitronitraminoethyltriazole, $C_4H_6N_6O_4$ – not found in Beil or CA through 1956

3-(β-Ethylaminoethyl)-α-sym-triazole Dipicrate, $C_{18}H_{18}N_{10}O_{14}$, mw 598.40, N 23.41%, ndls, mp 161°. Was prepd by interaction of 3-(β-chloroethyl)-1,2,4-triazole hydrochloride and ethylamine, followed by treatment of the reaction product with picric acid

Refs: 1)Beil – not found 2)C.Ainsworth

& R. G. Jones, JACS **76**, 5654(1954) & CA **49**, 13980(1955)

3-(β-Diethylaminoethyl)-α-sym-triazole Dipicrate, $C_{20}H_{22}N_{10}O_{14}$, mw 626.46, N 22.36%, prisms, mp 160°. Was obtained from 3-(β-chloroethyl)-1,2,4-triazole hydrochloride and diethylamine, followed by treatment of the reaction product with picric acid

Refs: 1)Beil – not found 2)C. Ainsworth & R.G. Jones, JACS **76**, 5654(1954) & CA **49**, 13980(1955)

AMINOETHYLUREAS AND DERIVATIVES

2-Aminoethylurea (β-Amino-äthyl-harnstoff in Ger), $H_2N–CH_2–CH_2–NH–C(:O)–NH_2$, mw 103.13, N 40.75%. Prepn and props are given in Beil **4**, [693]

Azidoaminoethylureas, $C_3H_8N_6O$ – not found in Beil or CA through 1956

Diazidoaminoethylureas, $C_3H_7N_9O$ – not found in Beil or CA through 1956

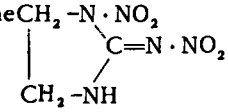
3-(2-Aminoethyl)-1-nitrourea or 3β-Aminoethylnitrourea, $H_2N \cdot CH_2–CH_2–NH–C(:O)–NH \cdot NO_2$, mw 148.13, N 37.83%. Crysts, mp 136–7°(decomp)

According to CA **46**, 1988a(1952), the compd prepd by S.S. Barton, R.H. Hall & G.F. Wright, JACS **73**, 2205(1951), from 2-nitrimino-2-imidazolidone and called by them 2-hydroxy-2-nitraminoimidazoline, might be the above nitrourea

The same compd was prepd by M.W. Kirkwood & G.F. Wright, JACS **76**, 1838(1954), who named it *3β-Aminoethylnitrourea* and was characterized by its X-ray diffraction pattern

The kinetics of the alkaline hydrolysis of 3β-aminoethylnitrourea has been studied at 25° by M.A. Weinberger & A.F. McKay, JACS **77**, 1321–4(1955); CA **49**, 10272 (1955)

3-(2-Nitraminoethyl)-1-nitrourea, 3-(β-Nitraminoethyl)-1-nitrourea, or (2-Nitraminoethyl)-nitrourea, (Called 1β-Nitraminoethyl-3-nitrourea by McKay et al), $O_2N \cdot HN–CH_2–CH_2–NH–C(:O)–NH \cdot NO_2$, mw 193.13, N 36.24%. Wh crysts (from et acet or hot w), mp 104–105°. Was prepd from 1-nitro-2-nitriminoimidazolidine



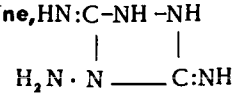
and Na_2CO_3 or NaOH in H_2O.

Refs: 1)Beil – not found 2)M.W. Kirkwood & G.F. Wright, JOC **18**, 638(1953) & CA **48**, 6968(1954) 3)A.F. McKay et al, JACS **76**, 6372–3(1954) & CA **49**, 15861(1955)

Aminoformaldehyde and Derivatives. See Formamide and Derivatives

AMINOGUANAZOLE AND DERIVATIVES

4-Aminoguanazole; 4-Amino-3,5-diimino-1,2,4-triazolidine; 3,4,5-Triamino-α-sym-triazole or Guanazine,



or $H_2N \cdot C(=N–NH_2)–NH–C(=NH)–NH_2$, mw 126.13, N 66.64%.

Col crysts (from w or alc), mp 255–7°(decomp), easily sol in w, diff sol in alc and insol in eth, benz and ligroin, reacts strongly alkaline. Was prepd by heating its hydrobromide with Pb hydroxide for several hours in w at 100°. The hydrobromide was obtd by treating either hydrazine or N,N'-diaminoguanidine hydrobromide with cyanogenbromide (Refs 2,3 & 4). Its constitution was established by Stollé (Refs 4 & 5)

It forms salts, some of which are explosive, eg, the *nitrate,* $C_2H_6N_6 \cdot HNO_3$, crysts, mp 210°, expl when heated on a Pt foil (Ref 3)

Refs: 1)Beil **26**, 206 & (61) 2)G. Pellizzari & C. Cantoni, Ber **38**, 283(1905) 3)Ibid, Gazz **35** I, 300 & 302(1905) 4)G. Pellizzari & A. Repetto, Gazz **37** II, 319(1907) 5)R. Stollé, JPrChem **75**, 423(1907) 6)R. Stollé &

W.Dietrich, J Pr Chem **139**, 209(1934) & CA **28**, 2714(1934)

Azidoaminoguanazoles, $C_2H_5N_9$ – not found in Beil or CA through 1956

Diazidoaminoguanazoles, $C_2H_4N_{12}$ – not found in Beil or CA through 1956

Nitrated and/or Nitrited Derivatives of 4-Aminoguanazole were not found in Beil or CA through 1956

AMINOGUANIDINE AND DERIVATIVES

Aminoguanidine or Guanylhydrazine(Kohlen säure-amidin-hydrazid or Hydrazinmonocarbon-säure-amidin in Ger) (Was called Amido-guanidin by Thiele), abbr as AGu, $H_2N \cdot C(:NH) \cdot NH \cdot NH_2$, mw 74.09, N 75.63%, OB to CO_2 –108%. Crysts, mp–decomp, sol in w, insol in alc. Can be prepd by the reduction of nitroguanidine by Zn dust in dil AcOH or by other methods.

Aminoguanidine Nitrate(AGuN), $CH_6N_4 \cdot HNO_3$, mw 137.11, N 51.08%, OB to CO_2 –17.5%, OB to CO –5.85%. Col crysts, mp 144–145°; expl at higher temp. Sol in w and alc. Its soly in w at various temps is given in Ref 3. Can be prepd in nearly theoretical yi eld by treating 1 mol of aminoguanidine bicarbonate with 1 mol of dilute (1:1) nitric acid(Ref 3). When an aq soln of the nitrate was heated with $NaNO_2$ and AcOH, as described in Ref 2,p 48, a yel amorphous substance, $C_2H_8N_{10}O$, was obtained. It expld on heating

Due to the high mp of AGuN it cannot be used alone for cast loading projectiles. Its mp may be somewhat reduced by incorporating either AN or GuN. For instance, the mp of a 50/50 mixt of AGuN/AN is 109° and of an 85/15 mixt of AGuN/GuN is 126°(Ref 3)

Refs: 1)Beil **3**, 117 2)J.Thiele, Ann **270**, 25–6 & 48(1892) 3)J.Barlot & S.Marsaule, MP **35**, 357–61(1953)

Aminoguanidine Picrate(AGuP) (Called Amido-guanidinpikrat by Thiele), $CH_6N_4 + C_6H_3N_3O_7$, mw 303.20, N 32.34%. Yel crysts(from w); mp–expl. Can be prepd by the action of PA or a picrate on a salt of aminoguanidine in aq soln

Refs: 1)Beil **6**, 279 2)J.Thiele, Ann **270**, 27(1892)

Azidoaminoguanidine, CH_5N_7 – not found in Beil or CA through 1956

Diazidoaminoguanidine, CH_4N_{10} – not found in Beil or CA through 1956

Nitrosaminoguanidine, $ON \cdot HN \cdot C(:NH) \cdot NH \cdot NH_2$, mw 88.07, N 63.62%. This compd is listed in CA 47, 1044 g(1953) as being prepd by C.Holstead & A.H.Lamberton, JCS **1952**, 1889. It is evidently an error because the compd described is *nitraminoguanidine*(see below)

AGu forms salts, nitrated and other derivs, some of them expl. Several AGu salts were studied in France(Ref 6)

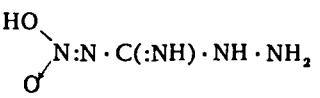
Refs: 1)Beil **3**, 117(57) & [95] 2)J.Thiele, Ann **270**, 23(1892) & **302**, 332(1898) 3)J.A. Wyler, USP 2,123,032(1938) & CA **32**, 6674 (1938) 4)E.Lieber & G.B.L.Smith, Chem Revs **25**, 213–71(1939) (Chemistry of aminoguanidine) (179 refs) 5)Degering(1950), 470 6)J.Barlot & S.Marsaule, MP **35**, 349–64(1953) 7)Wm.G.Finnegan, R.A.Henry & E.Lieber, JOC **18**, 783 & 786–7(1953); CA **48**, 7006(1954) (Substituted aminoguanidines) (See also Refs under Nitroaminoguanidine)

Aminoguanidinediazonium Hydroxide(Amido-guanidindiazohydroxyd, in Ger) are the names (misnomers) first given by K.A.Hofmann & R.Roth, Ber **43**, 682–4(1910) to a compd of the formula $C_2H_7N_{10}OH$, which is now known as *guanylnitrosaminoguanyltetrazene*, also called *tetracene*

Nitraminoguanidine(NAGu) Aminonitroguanidine (N'-Nitro-N-aminoguanidine or 1-Amino-3-nitroguanidine), $CH_5N_5O_2$, mw 119.09, N 58.82%, OB to CO_2 –33.6%

Note: NAGu, possessing a labile H atom, is

capable of existing in two forms, one of which has acid characteristics and consequently is able to form metallic derivatives. The structure of the *normal* form is assumed to be $O_2N \cdot NH \cdot C(:NH) \cdot NH \cdot NH_2$ and of the *pseudo-acid* form



NAGu consists of wh monocl crysts(from w), apparent d 0.22 g/cc, mp 184°(decomp); expl ca 190°. When heated on a metallic spatula near a flame, each particle of NAGu expl as it is ignited by the flame. It may be ground in a mortar without producing an expln. One of the methods for its prepn is the interaction of nitroguanidine with hydrazine sulfate in ammonia. This and other methods are described in Refs 1,2,3,4a,6, 7,8 and 9

The following properties of NAGu were detnd at Picatinny Arsenal: *brisance*(by sand test) 39.8g(TNT 43.0g); *minimum chge* for detonating 0.4g of NAGu in sand test, 0.27g of MF & 0.20g tetryl; *power*(by ballistic mortar test, deflection with 10g sample) 14° (TNT 12°25'); *impact test* with 2kg wt 22cm(tetryl 30cm); *friction pendulum test* (with steel shoe) snaps but does not burn or detonate; *ignitability* – not ignited by black powder fuse; *explosion temp*(5 secs) 190°; *hygroscopicity* at 30° and 90% RH,0.72% in 96 hrs; *100° heat test*,% loss in 1st 48 hrs 0.12, 2nd 48 hrs 0.15 and no explosion in 100 hrs; *120° vacuum stability test*,11+cc in 16 hrs; *solubility* at 30° in g/100 cc of water 1.07(0.32 at 10°), ethanol 0.59, methanol 0.55, acetone 3.3 and ethylenedichloride 0.32(See also Ref 7a); Q_c^v 270.14 kcal/mol and Q_f^v +5.30 kcal/mol(Refs 4a and 11)

NAGu reduces Fehling's soln with the formation of an expl copper salt, it also reduces an ammoniacal $AgNO_3$ soln with the formation of an expl silver salt and some gases(See under Nitraminoguanidine Salts)

Refs: 1)Beil **3**,[101] 2)R.Phillips & J.W. Williams, JACS **50**,2465–70(1928) (Prepn and props of NAGu) 3)E.R.Riegel & K.W.Buchwald, JACS **51**,492(1929) (Ultraviolet absorption of NAGu,etc) 4)E.Lieber & G.B.L.Smith, ChemRevs **25**,225(1939) 4a)A.J.Phillips, PATR 1104(1941) and PB Rept 3054(1941) 5)T.E.O'Connor, G.Fleming & J.Reilly, JSCI **68**,309–10(1949) (Diazotization of NAGu yielded NGu and nitroguanylazide) 6)Thorpe **4**(1949),148 (Prepn and props of NAGu) 7)Navord Rept 1158,NOTS 214, Inyokern,Calif,May 1949(C) (Prepn of NAG from NGu and hydrazine hydrate according to a conf procedure of NOTS 7a)Wm. McBride et al, JACS **71**,2937–8(1949) (Solubility of NAGu in H_2O) 8)R.A.Henry et al,JACS **72**(1950) (NAGu was obtained together with AGu and diaminoguanidine by hydrazinolysis of NGu in aq soln) 9)R.A. Henry et al,JACS **73**,474(1951) & CA **46**, 1986(1952) (Prepn of NAGu by adding hydrazine hydrate dropwise and with stirring, to NGu. Analysis for detn of purity is described) 10)E.Lieber et al,JACS **73**,2327–29(1951) & CA **46**,1987(1952) (Study of the interaction of nitrous acid and NAGu resulted in the isolation of several expl products, eg, 5-nitroaminotetrazole, some salts of guanidinium-5-nitraminotetrazole and of nitroguanyl azide) 11)W.S.McEwan & M.W. Rigg,JACS **73**,4726(1951) (Heats of combus and formn of NAGu and other compds) 12)R.A.Henry,USP 2,617,826(1952) & CA **47**,9352(1953) (Prepn of NAGu in 50–60% yields by treating $N_2H_4 \cdot H_2O$ with $H_2NC(:NH) \cdot NHNO_2$ in an unbuffered H_2O-system, and neutralizing prior to isolating the NAGu) 12a)C.Holstead & A.H.Lamberton,JCS **1952**, 1889(Note 1) (Prepn of NAGu) 13)F.L. Scott et al,JApplChem **2**,370(1952) & CA **48**, 3354(1954) (Prepn of NAGu by adding an aq soln of $N_2H_4 \cdot H_2O$ to an aq soln of azidonitroamidine, $N_3-C(:NH)-NH \cdot NO_2$) 14)W.D. Kumler & P.T.Sah,JOC **18**,669–72(1953) & CA **48**,6969(1954) (The structure of NGu and NAGu)

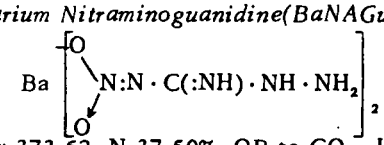
Addnl Refs on NAGu: a)R.A.Henry &

G.B.L.Smith, JACS **71**,1872–3(1943) & CA **43**,6983–4(1949) (Rearrangement of NAGu in a soln of ammonium carbonate) b)R.A. Henry & G.B.L.Smith, JACS **73**,1858–9(1951) & CA **46**,2502(1952) (Some reactions of NAGu with methylamine) c)E.Lieber et al, AnalChem **23**,1594–1604(1951) & CA **46**, 3857(1952) (Infrared absorption spectra of compds of high-nitrogen content, among them NAGu) d)E.Lieber et al, JOC **18**,218–28 (1953) & CA **48**,1343–4(1954) (Acetylation and ring closure in reduction of NGu and NAGu) e)W.D.Kumler, JACS **76**,815(1954) & CA **48**,8051(1954) (Infrared spectra of NAGu and related compds) f)J.E.De Vries & E.St.Clair Gantz, JACS **76**,1009(1954) & CA **48**,7995(1954) (Spectrophotometric studies of dissociation constants of NAGu and related compds) g)L.M.Hall et al, JACS **77**,6507–8(1955) & CA **50**,5376(1956) (Basic.equilibr ium constants of NGu and NAGu) h)L.A.Burkardt, AnalChem **28**,323–4(1956) & CA **50**,7540(1956) (X-ray diffraction patterns of NAGu and of some other guanidine derivs)

Nitroaminoguanidine(NAGu) Salts. As mentioned under nitroaminoguanidine, this compd exists in two forms: the normal and the pseudo-acid. The existence of a pseudo-acid form explains the possibility of formation of metallic salts

The following heavy metal salts, which may be considered as derived from the pseudo-acid form of nitroaminoguanidine are explosive:

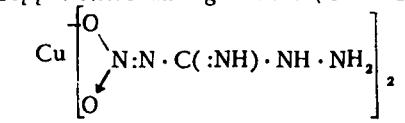
Barium Nitraminoguanidine(BaNAGu),



mw 373.52, N 37.50%, OB to CO_2, H_2O & BaO −21.4%. Crysts, mp 187°, expl at higher temp. Can be prepd by treating an aq soln of NAGu at ca 85° with a hot slurry of $Ba(OH)_2$, filtering and cooling to obtain crysts(Ref 2). A detailed description of the method used at PicArsn is given in Ref 3. BaNAGu was patented for use in priming compositions and for other purposes(Ref 4)

Refs: 1)Beil – not found 2)K.D.Ashley, USP 2,251,101(1941) & USP 2,286,327(1942) 3)A.J.Phillips, PATR 1183(1942),8 4)L.R.V. Clark, USP 2,325,742(1943) & CA **38**,489–90 (1944)

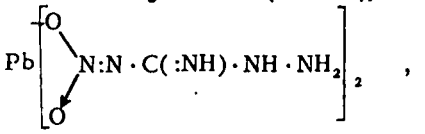
Copper Nitroaminoguanidine(CuNAGu),



mw 299.73, N 46.73%, OB to CO_2, H_2O & CuO −26.7%. Crysts, mp–expl. Can be prepd by treating an aq soln of NAGu with Cu hydrate(Ref 2). A detailed description of the method of prepn from NAGu, Cu hydrate and Cu acetate is given in Ref 3. CuNAGu was patented for use in priming and other explosive compositions.

Refs: 1)Beil – not found 2)K.D.Ashley, USP 2,251,101(1941) & CA **35**,7195(1941) 3)A.J.Phillips, PATR 1183(1942),8 4)L.R.V. Clark, USP 2,325,742(1943) & CA **38**,489–90 (1944)

Lead Nitroaminoguanidine (LNAGu),



mw 443.37, N 31.59%, OB to CO_2, H_2O & PbO −18.0%. It is the most important of the NAGu salts. Pale yell crysts, apparent d 0.092–0.096 g/cc, loading d 1.47, mp 182°; insol in org solvents, hydrolyzed by boiling water. Can be prepd by treating an aq soln of NAGu with Pb hydrate(Refs 2 & 3). In Ref 4 is given a detailed description of the lab method of prepn used at PicArsn: NAGu (1g) was dissolved in 25 cc H_2O and brought to 65°. Pb hydrate(1g), contg 85% H_2O, was slurried in 15 cc of H_2O to which was added dextrin or urea in the amt of 0.5 to 1% of the wt of NAGu. The slurry was poured into the agitated NAGu soln during a period of 3 min and the resulting ppt of LNAGu filtered off, rinsed with H_2O and dried

The following props of LNAGu were

determined at PicArsn: *brisance*(by sand test, when initiated by MF) 29.9g(TNT 43.0g); *explosion temp*(5 secs) 208°; *impact sensitivity* with 2 kg wt 9cm(tetryl 30cm); *hygroscopicity* at 30° and 95% RH 1.91% in 96 hrs; *100° heat test*, % loss of wt 1st 48 hrs 0.40, 2nd 48 hrs 0.77 and no explosion in 100 hrs

LNAGu was patented for use in priming and initiating compositions(Refs 5,6,7, & 8)

Tests conducted at Pic Arsn(Ref 4) have shown that a compn consisting of LNAGu 33.6, $KClO_3$ 14.3, Sb_2S_3 21.5 and glass 30.6%, loaded wet with 2% of shellac, proved to be suitable for caps in lieu of MF or LA compns. The impact test of the above mixt with a 2 kg wt was 2.5 cm, and the delay assembly of a M-48 fuse contg that mixt gave no failures in 30 tests

Refs: 1)Beil – not found 2)A.J.Phillips, PATR 1104(1941) & PB Rept 3054(1941) 3)K.D.Ashley,USP 2,251,101(1941) & CA & **35**,7195(1941) (LNAGu and a process of making it) 4)A.J.Phillips,PATR 1183(1942) p 8 4a)K.D.Ashley,USP 2,286,327(1942) (Ba salt of pseudo-acid of NAGu) 5)L.R.V. Clark,USP 2,325,742 & 2,326,008(1943) & CA **38**,488-90)(1944) (Use of heavy metals salts such as Pb in initiating expls) 6) L.R.V.Clark,CanP 435,873(1946) and USP 2,405,189(1946) & CA **40**,6818(1946) (Electric blasting cap contg a base chge of PETN and a superimposed initiating chge comprised of a mixture of LNAGu 80 and $KClO_3$ 20%. LNAGu can be rendered more stable for storage by incorporating 0.1 to 5% of an anhydrous salt, such as $CuSO_4$, capable of taking up H_2O and NH_3; this prevents catalytic decomps of LNAGu) 7)American Cyanamide Corp,BritP 593,878 (1947) & CA **42**,7046(1948) (Improvement of the method of prepn described in USP 2,251,101 for the purpose of increasing the loading density of crysts from 0.4 to 0.6 or even 1.0. In the new method, pptn from the aq soln of NAGu and lead hydroxide is made in the presence of more than 0.001% of a hydrophilic colloid based on the wt of the slurry. Suitable colloids include Me cellulose, animal glue, gelatin, agar-agar and pepsin. Examples of various mixts suitable for blasting caps are given in the patent) 8)L.R.V. Clark,USP 2,456,583(1948) & CA **43**,3200 (1949) (Same as previous patent)

Nickel Nitroaminoguanidine(An additive product), $2CH_5O_2N_5 + NiO$, mw 312.87, N 44.77%, OB to CO_2 −20.4%. Brownish solid; mp–expld with a flash on heating in a flame, but did not expl on heating in a test tube to 220°. Expld mildly on impact. Insol in w and in most org solvents; dissolves in caustics with a blue coloration; decomp by H_2SO_4 with the formation of poisonous gases. Can be prepd by boiling a dil soln of ammoniacal $NiSO_4$ with a 1% soln of NAGu in the presence of some metallic Ni for 20 min

An intense deep-blue coloration develops when a trace of a Ni salt is added to an aq soln of NAGu contg some caustic alkali. The coloration lasts 15–20 min and as little as 0.0002 mg may be detected by this method. Cobalt and a number of other metals failed to give this test(Ref 2,p 2467)

Refs: 1)Beil **3**,[101] 2)R.Phillips & J.F. Williams,JACS **50**,2467–9(1928)

Silver Nitroaminoguanidine(SNAGu). An explosive silver compd was obtained by the interaction of aq NAGu and ammoniacal silver nitrate in the cold. The composition of this product was not determined(Ref 2)

Refs: 1)Beil – not found 2)R.Phillips & J.F.Williams,JACS **50**,2467–9(1928)

Nitronitrosaminoguanidine, $O_2N \cdot NH \cdot C(:NH) \cdot NH \cdot NH \cdot NO$, mw 148.09, N 56.72%, OB to CO_2 −10.8%. Solid, explg at 210°, Trauzl test value 70% PA, impact sensitivity expressed as FI(figure of insensitivity) 93% PA. No method of prepn is given in the literature and in Ref 2

Refs: 1)Beil – not found 2)A.H.Blatt, OSRD Rept 2044(1944)

Aminoguanidine and Derivatives, Analytical Procedures. Some info on this subject may be found in the following papers: 1)R.P. Zimmerman & E.Lieber, AnalChem **22**,1151–5(1950) (21 refs) & CA **45**,499(1951) (Behavior of some compds contg–NH·NO_2 groups, such as NAGu, in attempts to reduce them with titanous chloride) 2)R.A.Henry et al, JACS **73**,474(1951) & CA **46**,1986(1951) (Detn of purity of NAGu by the modified Jamieson method using K iodate for titration 3)J.E.De Vries & E.St.Clair Gantz, AnalChem **25**,1020–22(1953) (10 refs) & CA **47**,1131–2 (1953) (Spectrophotometric detn of NAGu etc) 4)Wm.R.McBride et al,AnalChem **25**,1042–6 (1953) & CA **47**,9862(1953) (Potentiometric titrations of organic derivs of hydrazine. This includes some derivs of aminoguanidine) 5)P.D.Sternglanz et al,AnalChem **25**,1111–13 (1953) & CA **47**,9864(1953) (10 refs) (Reduction of NAGu by titanous chloride) (See also addnl refs c,e,f & h under Nitraminoguanidine)

Aminoguanidinium-3,5-bis(nitramino)-asym-triazole or Aminoguanidinium-3,5-di(nitramino)-1,2,4-triazole. See under Diaminotriazoles

AMINOGUANYLBIGUANIDINE AND DERIVATIVES

2-(Aminoguanyl)-biguanidine,

$$H_2N\cdot C(:NH)\cdot NH\cdot NH\cdot \underset{\displaystyle N\cdot C(:NH)\cdot NH\cdot NH_2}{\overset{\displaystyle NH_2}{\overset{|}{\underset{\|}{C}}}},$$

may be considered as the parent compd of the derivs described below

Azidoaminoguanylbiguanidine, $C_3H_{10}N_{12}$ and *Diazidoaminoguanylbiguanidine*, $C_3H_9N_{15}$ – were not found in Beil or CA through 1956

Nitroaminoguanylbiguanidine and *Nitraminoguanylbiguanidine*, $C_3H_{10}N_{10}O_2$ – were not found in Beil or CA through 1956

1,6-Dinitro-2-(aminoguanyl)-biguanidine,

$$O_2N\cdot HN\cdot C(:NH)\cdot NH\cdot NH\cdot \underset{\displaystyle N\cdot C(:NH)\cdot NH\cdot NH_2}{\overset{\displaystyle NH\cdot NO_2}{\overset{|}{\underset{\|}{C}}}},$$

mw 263.19, N 58.54%. Rosettes or col ndls (from w), decomg vigorously at 182°. Impure product expld at 180–2°. Was prepd from ammonium-1,6-dinitro-2(aminoguanyl)-biguanide in cold H_2O acidified with conc HNO_3 to pH 4.5. X-ray data is given

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **75**,95 & 96(1953); CA **48**,2052(1954)

1,6-Dinitro-2-(aminoguanyl)-biguanidine, Ammonium Salt, $C_3H_{14}N_{12}O_5$, mw 298.24, N 56.36%. Crysts, mp 178–9°. Was prepd by boiling an aq soln of bis-(aminoguanidinium)-1,6-dinitrobiguanide and NH_4Cl for 10 min

Refs: 1)Beil – not found 2)R.Henry et al, JACS **75**,957 & 959–60(1953); CA **48**,2051(1954)

1,6-Dinitro-2-(aminoguanyl)-biguanidine Nitrate, $C_3H_9N_{11}O_4\cdot HNO_3$, mw 326.21, N 51.53%. Wh crysts, mp 115–115.5°(decompn). Was prepd by adding concd HNO_3 to bis-(aminoguanidinium)-1,6-dinitro-2-(aminoguanyl)-biguanidine dropwise in cold H_2O to a pH of 2.0, followed by filtration and chilling the soln overnight at 0°

Refs: 1)Beil – not found 2)R.Henry et al, JACS **75**,957 & 960(1953); CA **48**,2050 et seq(1954)

Bis-(aminoguanidinium)-1,6-dinitrobiguanidine or *Di-(aminoguanidinium)-1,6-dinitrobiguanidine*, $O_2N\cdot HN\cdot C(:NH)\cdot NH\cdot NH\cdot C(:NH)\cdot NH\cdot NO_2 + 2CH_6N_4$, mw 354.32, N 63.26%. Yel crysts, mp 166–7°(dec); sol in w. Was obtained by refluxing nitroguanidine and hydrazine hydrate in MeOH

Note: CH_6N_4 = aminoguanidine

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **75**,957–8(1953) & CA **48**,2052(1954)

Bis-(aminoguanidinium)-1,6-dinitro-2-(aminoguanyl)-biguanidine or *Di-(aminoguanidinium) 1,6-dinitro-2-aminoguanyl)-biguanidine,*

$O_2N \cdot HN \cdot C(:NH) \cdot NH \cdot NH \cdot C(=N \cdot C(:NH) \cdot NH \cdot NH_2) \cdot NHNO_2 + 2CH_6N_4$

mw 411.37, N 64.70%. Rosettes or yel-orange ndls, mp 147–8 (decompn); readily sol in cold H_2O, sparingly in boiling MeOH with slow decompn. Was prepd by refluxing nitroguanidine and hydrazine hydrate in abs alc

Note: CH_6N_4 = aminoguanidine

Refs: 1)Beil – not found 2)R.Henry et al, JACS **75**,957 & 959–60(1953); CA **48**,2050 (1954)

2-(Aminoguanyl)-1,6-dinitro-biguanidine-benzalhydrazone, $O_2N \cdot HN \cdot C(:NH) \cdot NH \cdot NH \cdot C(=NC(:NH) \cdot NH \cdot N:CH \cdot C_6H_5) \cdot NH \cdot NO_2$,

mw 351.29, N 43.86%. Wh ndls, mp 178–80°(or 181–3°) with decompn. Was prepd by shaking together the ammonium salt of 1,6-dinitro-2-(aminoguanyl)-biguanide, few drops of concd HNO_3 and benzaldehyde. X-ray data are given

Refs: 1)Beil – not found 2)R.Henry et al, JACS **75**,957 & 961(1953); CA **48**,2052(1954)

Benzalaminoguanidinium-1,6-dinitro-2-(aminoguanyl)-biguanidine benzalhydrazone, $O_2N \cdot HN \cdot C(:NH) \cdot NH \cdot NH \cdot C(=NC(:NH) \cdot NH \cdot N:CH \cdot C_6H_5) \cdot NH \cdot NO_2 + C_8H_{10}N_4$

mw 513.48, N 40.92%. Wh platelets, mp 193°(dec). Was prepd by refluxing bis-(benzalaminoguanidinium)-1,6-dinitrobiguanidine and benzaldehyde in MeOH or by refluxing 1,6-dinitrobiguanide with benzalaminoguanidine in MeOH. X-ray data are given

Note: $C_8H_{10}N_4$ = benzalaminoguanidine

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **75**,959(1953) & CA **48**,2051(1954)

Bis-(benzalaminoguanidinium)-1,6-dinitrobiguanidine or *Di-(benzalaminoguanidinium)-1,6-dinitrobiguanide,* $O_2N \cdot NH \cdot C(:NH) \cdot NH \cdot NH \cdot C(:NH) \cdot NH \cdot NO_2 + 2C_8H_{10}N_4$,

mw 530.52, N 42.28%. Orange platelets(from alc), mp 176–7 ; sparingly sol in alc & in ether, sol in hot w with decompn; dec on prolonged heating in alc. Was prepd by shaking benzaldehyde in alc with bis-(aminoguanidinium)-1,6-dinitro-2-aminoguanyl-biguanidine in w. X-ray data are given

Note: $C_8H_{10}N_4$ = benzalaminoguanidine

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **75**,957 & 960(1953); CA **48**,2052(1954)

Aminoguanylaminoguanyl-tetrazene. Same as Bis-(aminoguanyl)-tetrazene

Aminoguanylnitraminoguanyl-tetrazene. See under Bis-(aminoguanyl)-tetrazene

Aminoguanylnitrosaminoguanyl-tetrazene, designated as **Tetracene.** See under Bis-(aminoguanyl)-tetrazene

Aminohemimellitene; 5-Amino-1,2,3-trimethylbenzene or **3,4,5-Trimethylaniline** (Aminohemellitol in Ger), $H_2N \cdot C_6H_2(CH_3)_3$. Ndls, mp ca 75°. Can be prepd by heating sym-m-xylidene hydrochloride with methanol, or by other methods

Refs: 1)Beil **12**,1150 & (498) 2)L.Limpach, Ber **21**,643(1888)

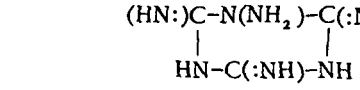
Note: No azido-, nitroamino-, nitramino-derivs were found in Beil or CA through 1956. (See also Aminomesitylene and Aminopseudocumene)

Aminoheptanes or Heptylamines, $C_7H_{17}N$. Several isomers are described in Beil **4**,193 (385) & [652]. The most common isomer is *1-Aminoheptane* or *n-Heptylamine,* $CH_3 \cdot (CH_2)_5 \cdot CH_2 \cdot NH_2$. Its *perchlorate* was investigated by R.L.Datta & N.R.Chatterjee, JCS **115**, 1008(1918) and found to explode at 265°

Aminohexanes or Hexylamines, $C_6H_{15}N$. Several isomers are described in Beil **4**,188, (384) & [649–50]. The most common isomer is *1-Aminohexane* or *n-Hexylamine,* $CH_3 \cdot (CH_2)_4 \cdot CH_2 \cdot NH_2$. Its *perchlorate* was

investigated by R.L.Datta & N.R.Chatterjee, JCS **115**,1008(1918) and found to explode at 278°.

1-Aminohexahydro-2,4,6-triimino-sym-triazine or **Aminoisomelamine**, called in CA **Hexahydro-2,4,6-triimino-s-triazine**,



mw 141.14, N 69.47%. Solid, mp–decomp ca 242°. Can be prepd by treating its hydrochloride with 10% NaOH soln. The hydrochloride was prepd by heating a mixt of K dicyanoguanidine with hydrazine dichloride in water on a steam bath for 3 hrs, followed by filtration

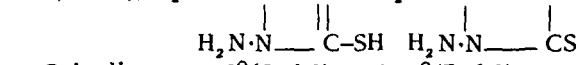
Aminoisomelamine was patented for use in resin formulations, ion exchange resins, pharmaceuticals, etc(Ref 2). It forms salts, such as *picrate*, mp 233°

Note: Aminoisomelamine is included because of its high N content as a possible component of propellants

Refs: 1)Beil – not found 2)J.J.Roemer & D.W.Kaiser,USP 2,729,639(1956) & CA **50**,14004(1956)

Aminohydrazinomercaptotriazoles or **Aminohydrazinotriazolethiones**, $C_2H_6N_6S$, mw 146.12, N 26.41%. The following isomer is described in the literature:

4-Amino-5-hydrazino-3-mercapto-4H-s-triazole, or **4-Amino-5-hydrazino-4H-1,2,4-triazole-3-thione** (Called in Beil *4-Amino-5-hydrazono-3-thion-1,2,4-triazolidin*) (Called *4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole* in Refs 3,4 & 5),

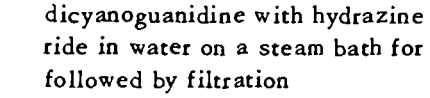


Col ndls, mp 228°(Ref 2), 230–1°(Ref 3), 228°(Ref 4), sl sol in w, insol in alc & eth, easily sol in dil acids & alkalies. Can be prepd from 2,5-dimercapto-1,3,4-thiadiazole or by other methods(Refs 2–5)

Refs: 1)Beil **26**,217 2)R.Stollé & P.E. Bowles,Ber **41**,1101(1908) 3)E.Hoggarth, JCS **1952**,4817 & 4820 & CA **48**,5181(1954) 4)H.Beyer et al,Ber **87**,1404(1954) & CA **49**, 15869(1955) (Obtained in small quantity during the prepn of thiocarbohydrazide by the method of P.C.Guha & S.C.De,JCS **1924**, 1215–8) 5)L.F.Audrieth et al,JOC **19**,741 (1954) & CA **49**,10857(1955)

Aminohydrazinotetrazoles, CH_5N_7, mw115.11, N 85.19%. The following isomer is listed in the literature:

1-Amino-5-hydrazino-vic(1,2,3,4)-tetrazole (Amino-1-hydrazino-5-tetrazol, in Ger),



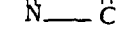
Stollé and Gaertner (Ref 2,p 213), stated that this compd can be prepd by treating thiocarbohydrazide with PbO and NaN_3, but they did not describe the method. They did, however, give the method of prepn of *1-amino-5-hydrazino-vic-tetrazole hydrochloride*, $CH_5N_7 \cdot HCl$, lt yel crys which vigorously decompd with evoln of gas ca 171°; easily sol in w, giving a strongly acidic soln. Its method of prepn consisted of heating 1-dibenzalamino-5-hydrazinotetrazole with 20% hydrochloric acid

When an aq soln of aminohydrazinotetrazole hydrochloride was treated with $NaNO_2$ and a part of the reaction mixt evaporated to dryness, the resulting crysts(contaminated with NaCl) expld vigorously when heated in a flame. A still stronger expln took place when a benzene extract of the above reaction mixt was evapd and the resulting crysts heated in a flame

Refs: 1)Beil – not found 2)R.Stollé & E.Gaertner,JPrChem **132**,213 & 222–3(1931); & CA **26**,1607(1932)

Aminohydrazinotriazoles, $C_2H_6N_6$, mw 114.12, N 73.65%. The following deriv is listed in the literature:

3-Amino-5-hydrazino-1,2,4-triazole Dihydrochloride, $H_2N \cdot HN \cdot C(NH \cdot N) \cdot NH \cdot C(NH_2) \cdot N + 2HCl$,



N 44.9%, crysts, mp 217° with vigorous decompn; dissolves in w, giving a strongly acidic soln, nearly insol in alc and insol in eth. Was prepd by adding the dinitroso deriv of guanazole to a soln of $SnCl_2$ in HCl

Refs: 1)Beil – not found 2)R.Stollé & W.Dietrich, J PrChem **139**, 199(1934) & CA **28**, 2714(1934)

AMINOHYDROXYANTHRAQUINONES AND DERIVATIVES

Aminohydroxyanthraquinones (Oxyaminoanthrachinone, in Ger), $C_{14}H_9NO_3$. Various isomers are described in Beil **14**, 267–8, 272–3, 275, (502–3, 510, 512) & [167–8, 172–4]

Azidoaminohydroxyanthraquinones, $C_{14}H_8N_4O_3$ – not found in Beil or CA through 1956

Diazidoaminohydroxyanthraquinones, $C_{14}H_7N_7O_3$ – not found in Beil or CA through 1956

Mononitroaminohydroxyanthraquinones, $C_{14}H_8N_2O_5$ – not found in Beil

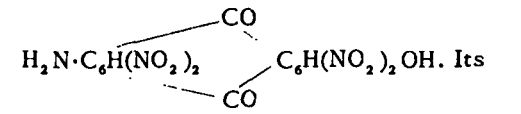
Nitraminohydroxyanthraquinone, $C_{14}H_8N_2O_5$ – not found in Beil

Nitronitraninohydroxyanthraquinones, $C_{14}H_7N_3O_7$. The isomer 3-nitro-2-nitramino-1-hydroxyanthraquinone is described in Beil **16**, 681

Dinitroamino-, Dinitronitramino-, Trinitroamino- and Trinitronitramino- hydroxyanthraquinones were not found in Beil or CA through 1956

Tetranitroaminohydroxyanthraquinones, $C_{14}H_5N_5O_{11}$, mw 419.22, N 16.71%, OB to CO_2 –74.42%. The following isomer is listed in the literature:

2,4,5,7-Tetranitro-8-amino-1-hydroxy-anthraquinone (*Aminochrysammic Acid or Chrysammidic Acid*), (2.4.5.7-Tetranitro-8-amino-1-oxy-anthrachinon or Chrysammidsäure in Ger) (Called by Schunck Amidochrysamminsäure),



$H_2N{\cdot}C_6H(NO_2)_2$ (CO) (CO) $C_6H(NO_2)_2OH$. Its *ammonium salt*, dk olive-grn ndls, was obtained by heating 2,4,5,7-tetranitro-1,8-dihydroxyanthraquinone(chrysammic acid) with NH_3. This salt, as well as those of *potassium* and *barium* deton violently on heating

Refs: 1)Beil **14**, 274 2)E.Schunck, Ann **65**, 236–8(1848)

Note: No higher nitrated derivs of aminohydroxyanthraquinones were found in Beil of CA through 1956

Aminohydroxyazobenzenes, $HO{\cdot}C_6H_4{-}N{:}N{-}C_6H_4{\cdot}NH_2$ or $C_6H_5{-}N{:}N{-}C_6H_3(OH){\cdot}NH_2$. Various isomers are described in Beil **16**, 304, 322, (338) & [148, 159]

Note: No azido- or diazido-derivs were found in Beil or CA through 1956. Only one mononitro- and one dinitro-deriv are listed in Beil **16**, 402 & [149] but neither is expl. No higher nitrated derivs were found in Beil or CA through 1956

Aminohydroxybenzene. Same as Aminophenol

AMINOHYDROXYBENZOIC ACIDS AND DERIVATIVES

Aminohydroxybenzoic Acids (Amino-oxybenzosauren, in Ger), $H_2N{\cdot}C_6H_3(OH){\cdot}COOH$, are described in Beil **14**, 577, 579, 587, 589, 592–3, (649–50) & [350, 352, 355–7, 359–60]

Azidoaminohydroxybenzoic Acids, $C_7H_6N_4O_3$ – not found in Beil or CA through 1956

Diazidoaminohydroxybenzoic Acids, $C_7H_5N_7O_3$ – not found in Beil or CA through 1956

Nitroaminohydroxybenzoic Acids, $C_7H_6N_2O_5$, mw 198.13, N 14.14%. The following isomers are described in the literature: 3-amino-5-nitro-4-hydroxy- and 3-amino-6-nitro-4-hydroxybenzoic acids.

Refs: 1)Beil **14**, 598 2)J.Nevole, Ber **77B**,

61(1944) & CA **39**,288(1945) (3-amino-4-hydroxy-5-nitro-benzoic acid)

Nitraminohydroxybenzoic Acid and *Nitronitraminobenzoic Acid* were not found in Beil or CA through 1956

Dinitroaminohydroxybenzoic Acids, $C_7H_5N_3O_7$, mw 243.13, N 17.28%, are not described in Beil, but the following nitrile is known:

2,6-Dinitro-4-amino-3-hydroxybenzonitrile or **2.4-Dinitro-6-amino-3-cyanphenol,** $H_2N{\cdot}C_6H(NO_2)_2(OH){\cdot}CN$, mw 224.13, N 25.00%. Red-yel pdr, mp–expl. Was prepd by treating its K salt with HCl, whereas the K salt was obtained by warming picramic acid with KCN. Some of its salts are expl (Refs 1 & 2)

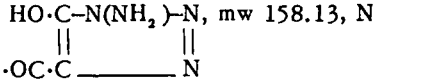
Refs: 1)Beil **14**,590 2)W.Borsche & A. Heyde, Ber **38**,3941–2(1905)

Dinitronitramino- and *Trinitroaminohydroxybenzoic Acids* were not found in Beil or CA through 1956

Amino- and **Hydroxy-methylnitramines.** See Hydroxy- and Amino-methylnitramines

2-Amino-2-hydroxymethyl-1,3-propanediol Trinitrate. See Tris(hydroxymethyl)-aminomethane Trinitrate

1-Amino-5-hydroxy-1,2,3-triazole-4-carboxylic acid hydrazide or **1-Amino-4-hydrazidocarbon-5-hydroxytriazole,**(Called 1-Amino-4-hydrazidocarbon-5-oxytriazol in Ref 2),



mw 158.13, N 53.15%. Crysts, mp–decomp. Was prepd by heating 1-α-naphthalene-sulfone-4-carbethoxy-s-hydroxytriazole with an excess of hydrazine hydrate in a sealed tube for 4 hrs at 95°

Its *diammonium salt*, $C_3H_{10}N_6O_2$, N 58.9%, crysts, mp 210° accompanied by expln, was obtained from the mother liquor of the previous operation

Refs: 1)Beil - not found 2)H.Bottler & G.Hasse, J PrChem **125**,377–8(1930) & CA **24**,3230(1930)

Aminohydroxytriazolopyrimidine. Same as Aminotriazolopyrimidinol, p A271

AMINOIMIDAZOLES AND DERIVATIVES

2-Aminoimidazole; 2-Amino-1,3-diazole or **2-Aminoglyoxaline**[Imidazolon-(2)-imid, in Ger],



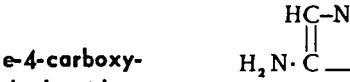
mw 83.09, N 50.57%. Prepn and props are in Refs 1 & 2

This high nitrogen compd forms salts, eg *nitrate*, $C_3H_5N_3{\cdot}HNO_3$, plates mp 135–6° and *picrate*, $C_3H_5N_3{\cdot}C_6H_3N_3O_7$, ndls, mp 236°

Refs: 1)Beil **24**,(188) & [7] 2)R.G.Fargher & F.L.Pyman, JCS **115**,246–7(1919) 3)K. Hoffmann, "Imidazole and Its Derivatives", Interscience, NY(1953),141–2 (One of the monographs on the Chemistry of Heterocyclic Compounds, edited by A.Weissberger)

Note: No azido-, diazido-, nitro-, nitramino-, nitroso- or nitronitrosamino-derivs were found in Beil or CA through 1956

4(5)-Aminoimidazole or **4(5)-Aminoglyoxaline**[Imidazolon-(4 or 5)-imid in Ger],



mw 83.09, N 50.57%. Prepn and props are in Refs 1, 2 & 3

4(5)-Aminoimidazole Dipicrate, $C_3H_5N_3 + 2C_6H_3N_3O_7$, ndls, darkening at 200° and melting ca 234°. A small quantity was prepd by Fargher by treating 4(or 5)-aminoimidazole with picric acid in w. Its expl props were not determined(see Refs 2,p 673)

Refs: 1)Beil **24**,[8] 2)R.G.Fargher, JCS **117**,672–3(1920) & CA **14**,3219(1920) 3)G. Hunter & J.A.Nelson, Can J Res **19B**,296–304 (1941) 4)K.Hofmann, "Imidazole and Its Derivatives", Interscience, NY(1953),142–3 (One of the monographs of "The Chemistry of Heterocyclic Compounds", edited by A.Weissberger)

4(5)-Amino-5(4)-imidazolecarboxamidine, $C_4H_7N_5$, mw 125.14, N 55.97%, was reported as the *dihydrochloride*, $C_4H_7N_5 \cdot 2HCl$, N 35.3%. It was prepd by heating adenine sulfate with HCl in a sealed tube at 150° ± 2° for 2 hrs

Refs: 1)Beil – not found 2)L.F.Cavalieri et al, JACS **71**,3976(1949) & CA **45**,121(1951)

Note: No azido-, diazido-, nitro-, nitramino-, nitroso- or nitronitrosamino-derivs were found in Beil or CA through 1956

AMINOIMIDAZOLIDINES AND DERIVATIVES

2-Aminoimidazolidine or *2-Aminotetrahydroimidazole*, $H_2C(-NH-CH(-NH_2)-)_2NH$. This substance may be considered the parent compd of:

1-Nitro-2-amino-2-nitraminoimidazolidine, $H_2C(-N(NO_2)-C(NH_2)(-NH-NO_2)-)_2NH$, mw 192.15, N 43.7%. Solid, mp 184.8–185.3°. Was obtained by treating 1-nitro-2-nitramino-Δ^2-imidazoline, $H_2C(-N(NO_2)-C(-NH-NO_2)=)_2N$, with ammonia followed by acidification to a pH of 1 with aq HCl(Ref 2,p 3993). Its explosive props were not reported in Refs 2,3 or 4

Refs: 1)Beil – not found 2)A.F.McKay & G.F.Wright, JACS **70**,3991–3(1948) & CA **43**, 2203(1949) 3)R.H.Hall, A.F.McKay & G.F. Wright, JACS **73**,2205 & 2207(1951) & CA **46**, 1988(1952) 4)M.W.Kirkwood & G.F.Wright, J OC **18**,634 & 640–1(1953) & CA **48**,6968 (1954)

Note: No azido- and diazido-derivs were found in Beil or CA through 1956

AMINOIMIDAZOLINES AND DERIVATIVES

2-Amino-Δ^2-imidazoline; 2-Amino-Δ^2-1,3-diazacyclopentene; 2-Amino-4,5-dihydro-2-imidazole or **N,N'-Ethyleneguanidine,** [Imidazolidon-(2)-imid, in Ger] $H_2C(-NH-C(-NH_2)=)_2N$ or $H_2C(-NH-C(=NH)-)_2NH$, mw 85.11, N 49.38%. Its prepn and props are given in Beil **24**,p 3

It forms salts and yields nitrated and nitrited derives, but no azido- or diazo-derivs were found in Beil or CA through 1956

2-Nitramino-Δ^2-imidazoline or 2-Nitramino-Δ^2-1,3-diazacyclopentene $H_2C(-NH-C(-NH-NO_2)=)_2N$ mw 130.11, N 43.07%. Solid, mp 220–1° with decomp. Can be prepd by treating an aq alkaline soln of nitroguanidine with diaminedihydrochloride. Its expl props were not examined

Refs: 1)Beil **24** – not found 2)A.F.McKay & G.F.Wright, JACS **70**,430–1 & 3991–2(1948) & CA **42**,2253(1948) & **43**,2203(1949) 3)Ibid, USP 2,525,927(1950) & CA **45**,2513(1951) 4)A.F.McKay, J.P.Picard & P.E.Brunet, Can J Chem **29**,751(1951) & CA **46**,2501–2(1952) (Structure of 2-nitramino-Δ^2-imidazoline by UV absorption spectra)

2-Amino-1-nitro-Δ^2-imidazoline, $H_2C(-N(NO_2)-C(-NH_2)=)_2N$, mw 130.11, N 43.07%. Crysts, mp 133.5°. Can be prepd by treating 2-amino-1-nitro-Δ^2-imidazoline hydrochloride with NH_4OH. Prepn of the hydrochloride is described in Ref 2,p 1618

Its *nitrate*, $C_3H_7N_5O_5$, mw 193.12, N 36.26%, mp 161°(with decomp), was prepd by treating N-β-nitroxyethyl-N'-nitroguanidine, $O_2NO \cdot CH_2-CH_2-NH-C(NH)-NH \cdot NO_2$, as indicated in Ref 2,p 1619. Its *picrate*, $C_9H_9N_7O_9$, mw 359.22, N 27.30%, crysts, mp 189.6°, was prepd by treating 2-amino-1-nitro-Δ^2-imidazoline hydrochloride with a saturated picric acid soln

Refs: 1)Beil **24**– not found 2)A.F.McKay & J.E.M.Milks, JACS **72**,1618–19(1950) &

CA **44**,10661(1950) 3)A.F.McKay, JACS **77**, 1058(1955) & CA **49**,8929(1955)

1-Nitroso-2-nitramino-Δ^2-imidazoline,

$H_2C-N(NO)-C-NH\cdot NO_2$, mw 159.11, N 44.02%.
H_2C ——— N

Crysts, mp 141.2° with decompn. Can be prepd by treating a soln of 2-nitramino-Δ^2-imidazoline in 70% nitric acid with $NaNO_2$. Treating 1-nitroso-2-nitramino-Δ^2-imidazoline with aromatic amines in aq ethanol at 30° gave 1-substituted-2-nitramino-Δ^2-imidazolines and 1,2-disubstituted-3-nitroguanidines

Refs: 1)Beil **24** – not found 2)A.F,McKay, W.R.R.Park & S.J.Viron, JACS **72**,3659-60 (1950) & CA **45**,2887–8(1951) 3)A.F.McKay, JOC **16**,1395–1404(1951) & CA **46**,5583–4 (1952) (Reaction of primary amines with 1-nitroso-2-nitramino-Δ^2-imidazoline) 4)A.F. McKay, JOC **16**,1846–50(1951) & CA **46**, 9096–7(1952) (Reactions of ethylamine and p-aminoacetanilide with 1-nitroso-2-nitramino-Δ^2-imidazoline) 5)A.F.McKay, Can J Chem **31**,284–6(1953) & CA **48**,153(1954) (Reaction of methylamine with 1-nitroso-2-nitramino-Δ^2-imidazoline)

1-Nitro-2-nitramino-Δ^2-imidazoline or **1-Nitro-2-nitramino-Δ^2-1,3-diazacyclopentene,**

$H_2C-N(NO_2)-C-NH\cdot NO_2$, mw 175.11, N 40.00%.
H_2C ——— N

Wh crysts(from dioxane %, mp 151.5–152.5° with decompn. Can be prepd by nitrating 2-nitramino-Δ^2-imidazoline as described in Refs 2 & 3

Although this compd is a powerful expl (1.3 × TNT in the ballistic mortar and 1.6 × TNT in the Trauzl block at d 0.8), it cannot be considered suitable for military purposes because of its low stability and high sensitivity to impact and friction(Ref 2)

Refs: 1)Beil **24** – not found 2)A.F.McKay & G.F.Wright, JACS **70**,3990–2(1948) & CA **43**,2203(1949) 3)A.F.McKay & W.G.Hatton, JACS **75**,964(1953) & CA **48**,2049(1954) 4)W.D.Kumler, JOC **18**,676–9(1953) & CA **48**, 6969(1954)

AMINOIMIDAZOLINE AND IMIDAZOLIDINE SUBSTITUTED DERIVATIVES

a)1-Methyl-2-nitramino-Δ^2-imidazoline,

$H_2C-N(CH_3)-C-NH\cdot NO_2$ mw 144.14, N 38.87%.
H_2C ——— N

Crysts, mp 115–116°. Methods of prepn are described in Refs 6,p 386 and 10,p 965

b)1-Ethyl-2-nitramino-Δ^2-imidazoline, $C_5H_{10}N_4O_2$, mw 158.16, N 35.43%. Crysts, mp 86.5°. Its prepn is described in Ref 6, p 386

c)1-(2-Hydroxyethyl)-2-nitramino-Δ^2-imidazoline or *1-β-Hydroxyethyl-2-nitramino-Δ^2-imidazoline,* $C_5H_{10}N_4O_3$, mw 174.16, N 32.17%. Crysts, mp 131.5–132°. Its prepn is described in Ref 6,p 386

d)1-(2-Nitroxyethyl)-2-nitramino-Δ^2-imidazoline or *1-β-Nitroxyethyl-2-nitramino-Δ^2-imidazoline,* $C_5H_9N_5O_5$, mw 219.16, N 31.96%. Crysts, mp 114.8–115.2°. Its prepn by nitration of compd c)with mixed HNO_3–H_2SO_4 is described in Ref 6,p 387

e)1,2-Bis(2-nitramino-2-imidazolin-1-yl)-ethane or *Bis-1-(2-nitramino-2-imidazolinyl)-ethane,* $C_8H_{14}N_8O_4$, mw 286.26, N 39.14%. Crysts, mp 300–301°(decomp). Its prepn is described in Ref 6,p 387

f)1-(2-Nitroxyethyl)-2-nitrimino-3-nitroimidazolidine or *1-β-Nitroxyethyl-2-nitrimino-3-nitroimidazolidine,* $C_5H_8N_6O_7$, mw 264.16, N 31.82%. Crysts, mp 115–116° with decomp. Its prepn by nitration of comp c)with HNO_3 in Ac_2O medium is described in Ref 6,p 387. It combines violently with phenylhydrazine giving a partially charred mass

g)1-(2-Nitroxyethyl)-3-nitro-2-imidazolidone or *1-β-Nitroxyethyl-3-nitro-2-imidazolidone,* $C_5H_8N_4O_6$, mw 220.15, N 25.45%. Crysts, mp 101–2 . Its prepn by hydrolysis of f)is described in Ref 6,p 388

h)1,2-Bis(2-nitrimino-3-nitro-1-imidazolidyl)-ethane or *Bis-1-(2-nitrimino-3-nitroimidazolidinyl)-ethane,* $C_8H_{12}N_{10}O_8$, mw 376.26, N 37.23%, mp 180–1° with decompn. Its prepn

by nitrating compd e) in Ac_2O–HNO_3 is described in Ref 6,p 388

i)1,2-Bis(3-nitro-2-oxo-1-imidazolidyl)ethane or *Bis-1-(3-nitro-2-imidazolidonyl)ethane*, $C_8H_{12}N_6O_6$, mw 288.22, N 29.16%. Crysts, mp 242–3° with decompn. Its prepn by hydrolysis of compd h) in boiling water is described in Ref 6,p 388

j)1-Methyl-2-nitrimino-3-nitroimidazolidine, $C_4H_7N_5O_4$, mw 189.14, N 37.03%. Crysts, mp 169–70° with decompn. It's prepn by nitration of 1-methyl-2-nitramino-2-imidazoline is described in Ref 6,p 388

k)1-Ethyl-2-nitrimino-3-nitroimidazolidine, $C_5H_9N_5O_4$, mw 203.16, N 34.48%. Crysts, mp 137–8° with decompn. Its prepn by nitration of 1-ethyl-2-nitramino-2-imidazoline is described in Ref 6,p 389

l)Guanidinoethylaminoimidazoline, Nitrated and Nitrited Derivatives are described under Aminoimidazolin-1-yl-ethylguanidine

m)1-Nitro-2-amino-4-methyl-Δ^2-imidazoline Nitrate, $C_4H_9N_5O_5$, mw 207.15, N 33.81%. Crysts, mp 150°. Was obtained in 43% yield by treating 1-nitro-2-amino-4-methyl-Δ^2-imidazoline hydrochloride with aq alc $AgNO_3$ soln(Ref 7,p 2207)

n)1-Nitro-2-nitramino-4-methyl-Δ^2-imidazoline or *1-Nitro-2-nitramino-4-methyl-Δ^2-1,3-diazacyclopentene*, $C_4H_7N_5O_4$ mw 189.14, N 37.03%. Crysts, mp 121.6–123°. Was obtained by treating 2-nitramino-2-imidazoline with HNO_3 + Ac_2O(Rev 3,pp 1971–2 & Ref 4, p 3965). The 2-nitramino-4-methyl-2-imidazoline, crysts, mp 170.5°, was prepd according to the method described in Ref 5

o)1-Nitro-2-ethoxy-2-nitramino-imidazolidine, $C_5H_{11}N_5O_5$, mw 221.18, N 31.67%. Crysts, mp 133.6–134°. Its prepn is described in Ref 2, p 3993

p)1-Nitro-2-n-propoxy-2-nitraminoimidazolidine, $C_6N_{13}N_5O_5$, mw 235.20, N 29.78%. Crysts, mp 124.4–125.5°. Its prepn is described in Ref 2,p 3993 and Ref 9,p 2215

q)1-Nitro-2-propylamino-2-nitraminoimidazolidine, $C_6H_{14}N_6O_4$, mw 234–22, N 35.88%. Crysts, mp 124.8–125.7°. Its prepn is described in Ref 2,pp 3993–4 and its reactions are given in Ref 8

r)1-Nitro-2-propylamino-Δ^2-imidazoline, $C_6H_{12}N_4O_2$, exists in the form of its hydrate, $C_6H_{14}N_4O_3$, and salts, such as the *nitrate*, $C_6H_{13}N_5O_5$, mw 235.20, N 29.78%, mp 148.8–149° and the *styphnate*, $C_{12}H_{15}N_7O_{10}$, mw 417.30, N 23.50%, mp 163–163.5° with decompn(Ref 8,p 2212)

s)2-Nitrimino-3-propylimidazolid-2-one, $C_6H_{12}N_4O_2$, mw 172.19, N 32.54%, mp 104–104.2° and *1-Nitro-2-nitrimino-3-propylimidazolid-2-one*, $C_6H_{11}N_5O_4$, mw 217.19, N 32.25%, mp 124.5–125.0°, are described in Ref 8,p 2212

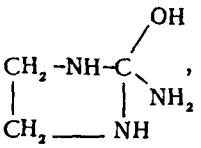
t)1-Nitro-2-methylamino-Δ^2-imidazoline, $C_4H_8N_4O_2$, mw 144.14, N 38.87%. Crysts, mp 76.5–77°. Its prepn is described in Ref 10, p 965

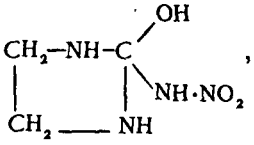
u)1-Nitro-2-methylamino-Δ^2-imidazolinium Nitrate, $C_4N_9N_5O_5$, mw 207.15, N 33.81%. Crysts, mp 172° with decompn. Its prepn is described in Ref 10, p 965

v)1-Nitro-2-ethylamino-Δ^2-imidazolinium Nitrate, $C_5H_{11}N_5O_5$, mw 221.18, N 31.67%. Crysts, mp 157.5° with decompn. Its prepn is described Ref 10,p 965

Refs: 1)Beil – not found 2)A.F.McKay & G.F.Wright, JACS **70**,3993–4(1948) & CA **43**, 2203(1949) 3)A.F.McKay & D.F.Manchester, JACS **71**,1971–2(1949) & CA **43**,9065(1949) 4)A.F.McKay & S.J.Viron, JACS **72**,3965(1950) & CA **45**,2936(1951) 5)A.F.McKay & G.F. Wright,USP 2,525,927(1950) & CA **45**,2512 (1951) 6)A.F.McKay & J.R.G.Bryce & D.E. Rivington,CanJChem **29**,382–90(1951) & CA **46**,7094–5(1952) 7)R.H.Hall,A.F.McKay & G.F.Wright,JACS **73**,2205–8(1951) & CA **46**, 1988(1952) 8)R.H.Hall & G.F.Wright,JACS 2208–13(1951) & CA **46**,1988–9(1952) 9) Ibid,JACS **73**,2213–16(1951) & CA **46**,1989 (1952) 10)A.F.McKay & W.G.Hatton,JACS **75**,963–5(1953) & CA **48**,2049(1954)

AMINOIMIDAZOLINOL AND DERIVATIVES

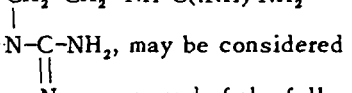
2-Amino-2-imidazolinol or **2-Hydroxy-2-aminoimidazolidine,**  , mw 103.13, N 40.75%. May be considered the parent compd of:

2-Nitramino-2-imidazolinol or **2-Hydroxy-2-nitraminoimidazolidine,**  , mw 148.13, N 37.83%. Crysts, mp 136–7° (decomp). One of the hydration products obtained by Barton et al(Ref 2) from 2-nitriminoimidazolidine(see also Ref 3). Its explosive props were not detd

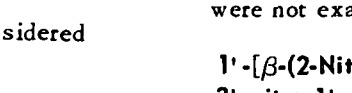
Refs: 1)Beil – not found 2)S.S.Barton, R.H.Hall & G.F.Wright, JACS **73**, 2204–5(1951) & CA **46**, 1987(1952) 3)M.W.Kirkwood & G.F. Wright, JACS **76**, 1836(1954) & CA **49**, 6927 (1955)

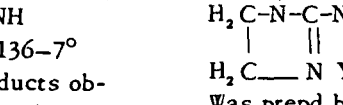
Note: No higher nitrated- or nitrited-derivs as well as azido- and diazido-derivs were found in Beil or CA through 1956

AMINOIMIDAZOLIN-1-YL AND DERIVATIVES

Aminoimidazolin-1-yl-ethylguanidine,  , may be considered a parent compd of the following substances:

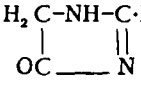
1-[2-(2-Nitramino-Δ²-imidazolin-1-yl)ethyl]-3-nitro-guanidine; 1-[2-(3-Nitroguanidino)-ethyl]-2-nitramino-Δ²-imidazoline or **1-β-Nitroguanylaminoethyl-2-nitramino-Δ²-imidazoline,**  , mw 260.22, N 43.07%. Crysts(from H_2O), mp 197° with decompn. Was prepd by adding 1,5-diamino-3-azapentane, $NH(CH_2CH_2NH_2)_2$, to methylnitrosonitroguanidine, $CH_3N(NO)C(:NH)NHNO_2$, as described in Ref 2,p 387. Its expl props were not examined

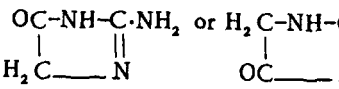
1'-[β-(2-Nitramino-Δ²-imidazolin-1-yl)ethyl]-3'-nitro-1'-nitroso-guanidine; 1-[β-(3'-Nitro-1'-nitrosoguanidino)ethyl]-2-nitramino-Δ²-imidazoline or **1-(N-Nitroguanyl-N'-nitroso-β-aminoethyl)-2-nitramino-Δ²-imidazoline,**  , mw 289.22, N 43.56%. Yel crysts, mp 176° with decompn. Was prepd by treating a soln of the previous compd in dil nitric acid with Na nitrite, as described in Ref 2,pp 389–90. Its expl props were not examined

1'-[β-(2-Nitrimino-3-nitro-Δ²-imidazolin-1-yl-ethyl]-1',3'-dinitro-guanidine; 1-[β-(1',3'-Dinitroguanidino)ethyl]-2-nitrimino-3-nitro-Δ²-imidazolidine or **1-(N-Nitroguanyl-N'-nitro-β-aminoethyl)-2-nitrimino-3-nitroimidazolidine,**  . Crysts, mp 161–2° with decompn. Was prepd by treating 1-[2-(2-nitramino-2-imidazolin-1-yl)ethyl]-3-nitroguanidine with nitric acid and acetic anhydride as described in Ref 2,p 389. Its expl props were not examined

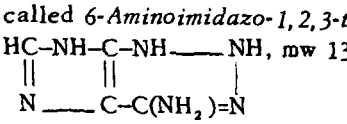
Refs: 1)Beil – not found 2)A.F.McKay, et al, Can J Chem **29**, 384, 387 & 389–90(1951) & CA **46**, 7094–5(1952)

Note: Azido- and diazido-derivs of aminoimidazolinol were not found in Beil or CA through 1956

2-Amino-4(or 5)-Δ²-imidazolone [Glykocyamidin, 4-Oxo-2-imino-imidazolidin or Hydantoinimid-(2), *in* Ger], 


OC–NH–C·NH$_2$ or H$_2$C–NH–C:NH, mw 99.09
H$_2$C—N OC—NH
N 42.41%. Prepn and props are in Beil **24**,244 & **25**,451

Note: No azido-, diazido-, nitrated and/or nitrited derivs of aminoimidazolone were found in Beil or CA through 1956

4-Amino-7H-imidazo-[4,5-d]-triazine, also called *6-Aminoimidazo-1,2,3-triazine,*

HC–NH–C–NH—NH, mw 136.12, N 61.75%.
N—C–C(NH$_2$)=N
This high nitrogen compd was obtained by Woolley et al on treating 4-amino-5-imidazole-carboxamidine-di-HCl with $NaNO_2$. No props are given in CA

Refs: 1)Beil – not found 2)D.W.Woolley et al, JBiolChem **189**,401(1951) & CA **45**, 5764(1951)

AMINOIMIDOETHANE AND DERIVATIVES

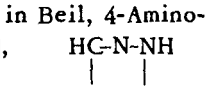
α-Amino-α-imidoethane; Acetamidine; Ethaneamidine or **Ethenylamidine,** CH_3·C(:NH)·NH_2, mw 58.08, N 48.23%. This high nitrogen compd is known only in the form of salts, such as *nitrate,* $C_2H_6N_2$·HNO_3, ndls, mp 189°, obtained by electrolysis of ammoniacal-alcoholic soln of ammonium carbonate(Ref 2) and the *picrate,* $C_2H_6N_2$·$C_6H_3N_3O_7$, orange prisms, mp 252°(Ref 3)

Refs: 1)Beil **2**,185(85) & [183–4] 2)F. Fichter et al, ZElectrochem **18**,651(1913); ChemZtr **1913 I**,1271 & CA **7**,3967(1913) 3)R.G.Fargher, JCS **117**,674(1920) 4)K. Takeda & K.Tokuyama, JPharmSocJapan **75**, 957–9(1955) & CA **50**,4980(1956)

Note: Azido-, diazido-, nitrated and/or nitrited derivs were not found in Beil or CA through 1956

AMINOIMINODIHYDROTRIAZOLE AND DERIVATIVES

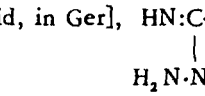
Aminoiminodihydrotriazoles, $C_2H_5N_5$, mw 99.10, N 70.68%. The following isomer is listed in the literature:

4-Amino-3-imino-2,5-dihydro-α-sym-triazole or **4-Amino-3-imino-2,5-dihydro-2H-1,2,4-triazole** [Called in Beil, 4-Amino-1.2.4-triazolon-(3)-imid], 
HC–N–NH
H$_2$N·N—C:NH
. Yel ndls (from alc), mp 208°; very sol in w, sol in alc. Was prepd by treating its hydrobromide with moist Ag_2O. The hydrobromide was obtained by heating N,N'-diaminoguanidine hydrobromide with formic acid on a water bath. The aq soln of the above triazole gives with aq $AgNO_3$ a wh ppt sol in NH_3 or in HNO_3. When treated with HNO_3 the triazole gives *nitrate,* $C_2H_5N_5$ + HNO_3, crysts, mp 194°, and when treated with picric acid *picrate,* crysts, mp 192°. Expl props of these compds were not examined

Refs: 1)Beil **26**,(39) 2)A.Gaiter, Gazz **45I**, 457(1915) & CA **10**,603(1916)

Note: Azido-, nitrated and/or nitrited derivs of aminoiminodihydrotriazole were not found in Beil or CA through 1956

AMINOIMINOMETHYLDIHYDROTRIAZOLE AND DERIVATIVES

4-Amino-5-imino-3-methyldihydro-α-sym-triazole or **4-Amino-5-imino-3-methyldihydro-1H-1,2,4-triazole** [4-Amino-3-methyl-1.2.4-triazolon-(5)-imid, in Ger], 
HN:C–NH–N
H$_2$N·N—C·CH$_3$
, mw 113.13, N 61.91%. Ndls(from alc). Was prepd by treating its nitrate with aq $Ba(OH)_2$ soln. The *nitrate,* $C_3H_7N_5$·HNO_3, wh ndls, mp 184°, was obtained by heating N,N'-diaminoguanidine nitrate with AcOH on a water bath in the presence of a small amt of concd HNO_3. Treatment of an aq soln of the nitrate with a concd aq soln of picric acid yielded the *picrate,* $C_3H_7N_5$·$C_6H_2(NO_2)_3OH$, small yel crysts decompg ca 189°. Expl props of these compds were not examined

Refs: 1)Beil **26**,(40) 2)A.Gaiter,Gazz **45** I, 460(1915) & CA **10**,603(1916)

Note: Azido-, nitro- and nitroso- derivs of aminoiminomethyldihydrotriazole were not found in Beil or CA through 1956

AMINOINDAZOLES AND DERIVATIVES

Aminoindazole, Benzodiazole or *Benzopyrazoles*, $C_7H_7N_3$. Several isomers are described in Beil **24**,112 & 59 and **25**,317–18 & 308. Some isomers described in Beil are also called *Indazolonimide*

Azidoaminoindazole, $C_7H_6N_6$ and *Diazidoaminoindazole*, $C_7H_5N_9$ – were not found in Beil or CA through 1956

Mononitroaminoindazole, $(O_2N)C_7H_6N_3$ – not found in Beil or in CA through 1956

5,7-Dinitro-6-amino-indazole, $(O_2N)_2C_7H_3N_2 \cdot NH_2$, mw 223.15, N 31.39%. Brn-yel scales, mp–melted with decompn on heating on Pt foil in flame; very diff sol in w, easier in boiling AcOH. Was prepd by heating 5,7-dinitro-6-indazolesulfonic acid with ammonia

Refs: 1)Beil **25**,318 2)T.Zincke & A. Kuchenbecker,Ann **339**,240(1905)

5,7-Dinitro-6-nitramino-indazole, $(O_2N)_2C_7H_3N_2 \cdot NH(NO_2)$, mw 268.15, N 31.34%. Refs to this compd and higher nitrated derivs, which undoubtedly would be expl were not found in Beil or CA through 1956

Aminoisomelamine. See 1-Aminohexahydro-2,4,6-triimino-sym-triazine

5-Amino-2-mercapto-1,3,4-thiodiazole; [5-Imino-2-thion-1.3.4-thiodiazolidin or 5-Amino-1.3.4-thiodiazolthion-(2) in Ger], $H_2N{\cdot}C(=N{-}N){-}S{-}C{\cdot}SH$, $HN{:}C(-S-C){:}S$ or $H_2N{\cdot}C(=N{-}NH){-}S{-}C{:}S$, mw 133.07, N 31.58%, OB to CO_2 & SO_2 –114.2%. OB to CO & SO_2 –90.2%. According to Beil(Ref 1), there are two forms of this compd, one, called *endo-iminothiobiazol*, which melts at 245°(with decompn) and the other called *imino-thiol-dihydro-thiobiazol*, which melts at 234°. Their prepn is described in Beil and in refs listed in it. The yields were low(ca 25%)

Audrieth & Scott(Ref 2), in the course of their work on high-nitrogen compds under contracts with US Ordnance Corps, prepd a compd which they named *3-amino,5-thiol-1,2,4-thiadiazole*; they assigned to it the structures $HS{\cdot}C{=}N{-}N$ / $S{-}C{\cdot}NH_2$ and $S{:}C{-}NH{-}NH$ / $S{-}C{:}NH$. Crysts, mp 232–237°; very sl sol in w; dissolves in bases from which solns it may be repptd by addition of acid. It was obt in yields up to 60% by deamination of bis-thiocarbamyl-hydrazide, $H_2N{\cdot}CS{\cdot}NH{\cdot}NH{\cdot}CS{\cdot}NH_2$, in the presence of polyphosphoric acid contg 82–84% P_2O_5 as the deaminating and cyclizing agent. This compd seems to be identical with the 234° mp form described in Beil

Because of the fairly high nitrogen content of aminothiodiazole, its low soly in H_2O and its compatibility with NC, Audrieth & Scott suggested its possible use as a flash-reducing agent in gun propellants to replace K_2SO_4 currently used

Refs: 1)Beil **27**,674,(600) & [757](Numerous refs) 2)L.F.Audrieth & E.S.Scott, "Compounds of High Nitrogen Content", Univ of Illinois Fourth Quarterly Progress Report, Urbana, Ill, October 1, 1951

AMINOMESITYLENES AND DERIVATIVES

Aminomesitylenes or **Amino-1,3,5-trimethylbenzenes**(Trimethylanilines or Mesidines), $C_9H_{13}N$, mw 132.20, N 10.36%. Several isomers are listed in Beil **12**,1160, 1163,(503) & [631]

Azidoaminomesitylenes, $C_9H_{12}N_4$ – not found in Beil or CA through 1956

2-Amino-4,6-diazido-mesitylene; Bistriazomesidine

or **Aminomesitylenebisazoimide**, $(CH_3)_3C_6(N_3)_2 \cdot NH_2$, mw 217.23, N 45.14%. Col crysts, mp 68°; swells if heated rapidly; decomp vigorously with evoln of gas by concd H_2SO_4. Was prepd by treating hydrochloric acid soln of triaminomesitylene trihydrochloride successively with $NaNO_2$ and NaN_3 at −5°, as described in Ref 2

Refs: 1)Beil **12**,[633] 2)G.T.Morgan & G.R.Davies, JCS **123**,237(1923) & CA **17**,1633 (1923)

Mononitroaminomesitylenes, $(CH_3)_3 \cdot C_6H(NO_2) \cdot NH_2$, mw 180.20, N 15.55%. One isomer, *4-nitro-2-amino-mesitylene* is described in Beil **12**,1162 & [632]

Nitronitraminomesitylene, $(CH_3)_3C_6H(NO_2) \cdot NHNO_2$, not found in Beil or CA through 1956

Dinitroaminomesitylenes, $(CH_3)_3C_6(NO_2)_2 \cdot NH_2$, mw 225.20, N 18.66%. One isomer, *4,6-dinitro-2-aminomesitylene*, yel ndls, mp 193–5°, is listed in Beil **12**,1163

Dinitronitraminomesitylene, $(CH_3)_3C_6(NO_2)_2 \cdot (NHNO_2)$ – not found in Beil or CA through 1956

6-Nitro-4-azido-2-aminomesitylene (Triazonitroaminomesitylene or Nitromesidineazoimide), $(CH_3)_3C_6NH_2(NO_2)N_3$, mw 221.22, N 31.66%, Lt yel crysts (from petr eth), mp 83–84°. Was prepd by adding NaN_3 to a soln of nitroaminomesitylenediazonium chloride

Refs: 1)Beil **12**,[633] 2)G.T.Morgan & G.R. Davies, JCS **123**,235(1923) & CA **17**,1633(1923)

2-Nitramino-4-azido-6-nitro-mesitylene or Nitraminomesitylenеazoimide, $(CH_3)_3C_6(N_3)NO_2 \cdot NHNO_2$, mw 266.22, N 31,57% was not found in Beil or CA through 1956

2-Nitroamino-4,6-diazido-mesitylene or Nitraminomesitylenebisazoimide, $(CH_3)_3C_6(N_3)_2 \cdot NH \cdot NO_2$, mw 262.23, N 42.74% was not found in Beil or CA through 1956

Aminomesitylenebisdiazonium Chloroaurate Hydrochloride. Diazotization of triaminomesitylene trihydrochloride, followed by treatment with a soln of chloroauric acid, gave two expls, $C_9H_{11}N_5Cl_8Au_2$ and $C_9H_{12}N_5Cl_9Au_2$, both solid

Refs: 1)Beil – not found 2)G.T.Morgan & G.R.Davies, JCS **123**,236–7(1923) & CA **17**, 1633(1923)

1-Amino-mesitylene; 1-Amino-1,3,5-trimethyl-benzene; 3,5-Dimethylbenzylamine or **Mesitylamine**, $H_2N \cdot CH_2 \cdot C_6H_3(CH_3)_2$ is described in Beil **12**,1163

AMINOMETHANE AND DERIVATIVES

Aminomethane or **Methylamine** (Monomethylamine or Carbinamine), CH_3NH_2, mw 31.06, N 45.1%, OB to CO_2 −231.8%, OB to CO −180.3%. Col, inflammable gas, fr p −93.5°, bp −7.69°, d 0.662 at 20/4° or 0.769 at −10/4°, fl p (closed cup) 0°F, autoignition point 806°F, expl range with air 4.95–20.75%(Ref 8). Sol in w, alc & eth. Can be prepd by several methods which are described in Refs 1,2,3,4,5 & 6. A large scale, inexpensive method of prepn was used during WW II at Dyhernfurth, near Breslau, Germany, and produced the following compds in one operation: monomethylamine (used for the manuf of Man-Salz), dimethylamine [used for the manuf of the war gas Tabun, described in PATR 2510 (1958) under Trilon] and trimethylamine (used for the manuf of Tetra-Salz). The method of manuf consisted essentially of passing a mixt of ammonia and methanol, in the absence of air, through a pressure reactor. The pressure was 200–300 psi and the temp 180–200°. Alumina (Al_2O_3) was used as the catalyst(Ref 9)

As a fire hazard, aminomethane is dangerous when exposed to flame; as an explosion hazard, it is moderate when exposed to sparks or flame. It can react vigorously with oxidizing materials(Ref 8)

Being a weak base, aminomethane forms salts with acids, some of which are expl. It

also forms some expl addn compds. Aminomethane is used in the synthesis of wetting agents(such as Igepon T), drugs, photographic developers, dye intermediates, solvents, etc. It has also been used for the prepn of tetryl. In this synthesis aminomethane is treated with dinitrochlorobenzene to form N-methyl-2,4-dinitroaniline, which on nitration yields N-methyl-N,2,4,6-tetranitroaniline(tetryl) (Refs 4,5 & 7). During WW II millions of pounds of tetryl were manufd by this process

Refs: 1)Beil **4**,32,(316), & [546] 2)E.Briner & J.Gandillon,Helv **14**,1283(1931) & CA **26**, 1575(1932) 3)E.B.Punnet,USP 2,113,241 (1938) & CA **32**,4175(1938) 4)Kirk & Othmer **9** (1952),64–5 5)Rohm & Haas, "The Methylamines", Philadelphia(1954) (84 pages) 6) M.Ishidate et al,JPharmSoc,Japan **74**,882–3 (1954) & CA **49**,9486(1955) (Method of synthesis of CH_3NH_2 by heating a mixt of hexamethylenetetramine,$(CH_2)_6N_4$, formamide, $HCONH_2$, and Raney nickel in an autoclave in an atm of hydrogen under pressure) 7)R. Williams,Jr et al, C&EN **33**,3982–3(1955) & CA **49**,14634(1955) (Production, consumption, use, market, etc, of methylamine) 8)Sax (1957),920(Monomethylamine) 9)H.Walter, PicArsn,Dover,NJ, private communication (1958)

Azidoaminomethane, $N_3 \cdot CH_2 \cdot NH_2$ – not found in Beil or CA through 1956

Aminomethane Chlorite, $CH_3 \cdot NH_2 \cdot HClO_2$, mw 99.53, N 14.07%, OB to CO_2 −48.2%. Syrup. Its aq soln was prepd by Levi by neutralizing a 30% soln of aminomethane with 2N H_2SO_4 and treating the resulting mixt with $Ba(ClO_2)_2$, followed by filtration. The filtrate was evapd in vacuo over $CaCl_2$ to a dense, syrupy liq contg about 66% of $CH_3NH_2 \cdot HClO_2$, but no crysts were formed. When this syrup was poured onto a cold iron plate, a slight expln occurred.

Refs: 1)Beil **4**,[549] 2)G.R.Levi,Gazz **52 I**, 207–9(1922) & CA **16**,2474(1922)

Aminomethane Nitrate or **Methylamine Nitrate** (Man-Salz, in Ger), $CH_3 \cdot NH_2 \cdot HNO_3$, mw 94.07, N 29.78%, OB to CO_2 −34.0%. Prism crysts, mp 109–11°, sol in w and very hygroscopic. Can be prepd by neutralizing aminomethane with nitric acid or by reacting methylnitrate with ammonia

This salt was thoroughly investigated in Germany before and during WW II. A detailed description of the method for its prepn and its explosive props are given in PATR 2510(1958), under Man-Salz

Addnl info not included in the German Section(PATR 2510) follows: Aminomethylnitrate is of satisfactory thermal stability and is more powerful than TNT(the Trauzl test gives 325 cc expansion vs 290 cc for TNT, which is about 112% TNT). Its vel of deton is ca 6600 m/sec at a d not specified; Q_c^v 215.4 kcal/mol and Q_f^v 81.6 kcal/mol(Ref 5). It has been used in some cast expl mixts, such as with dinitrodimethylsulfamide(Ref 2)

Dynamit A–G(Ref 3) proposed expl compns prepd by melting together AN with nitrates of simple or multiple aliphatic amines, such as aminomethane nitrate or diaminoethylene dinitrate. SECI(Ref 4) proposed an expl compn consisting of aminomethane nitrate and AN prepd by heating a mixt of formaldyhyde, or its polymers or higher homologs, in the presence of H_2O with AN in excess of the amt theoretically required for the prepn of aminomethane nitrate. The dried product could be waterproofed by mixing or coating with molten paraffin

Aminomethane nitrate was used during WW II in Germany in admixt with $NaNO_3$, $Ca(NO_3)_3$ and RDX(10–15%) for bomb and shell loading(Ref 6). Some of these mixts melted at ca 76° and could be easily cast-loaded

Refs: 1)Beil **4**,36 & [318] 2)P.Naoúm,GerP 499,403(1928) & CA **24**,4160(1930) 3)Dynamit A–G,FrP 742,312(1933) & BritP 384,966(1932), CA **27**,3612,5981(1933) 4)SECI,FrP 815,880

(1937) & CA **32**,1934(1938) 5)L.Médard & M.Thomas,MP **35**,172(1953) 6)H.Walter, PicArsn,Dover,NJ; private communication (1958)

Nitroaminomethane or *C-Nitromethylamine*, $O_2N{\cdot}CH_2{\cdot}NH_2$ – not found in Beil or CA through 1956

Nitraminomethane, Methylnitramine or **N-Nitromethylamine**, $CH_3{\cdot}NH{\cdot}NO_2$, mw 76.06, N 36.84%, OB to CO_2 -42.1%. Ndls(from ether), mp 38°, d 1.2433 at 48.6°, n_D 1.46162 at 48.6°(Ref 2); expl violently when heated in a capillary(Ref 5). Easily sol in w, alc, chlf & benz; less sol in eth; very sl sol in petr eth. Was first prepd by Franchimont & Klobbie(Ref 2) by nitration and hydrolysis of methyl-N-methyl-carbamate, $CH_3CO_2{\cdot}NH{\cdot}CH_3$. Other methods are known (Refs 1 & 4). Johnson(Ref 5) prepd it in 66% yield by nitration and hydrolysis of ethyl-N-methyl-carbamate. Detailed description of procedure is given. It is a powerful expl, with a Trauzl test value of 144%PA. Its toxicity is probably similar to that of aminomethane(qv)

Nitraminomethane was patented for use as an additive to Diesel fuels(Ref 10). There is no info at our disposal about its uses in expls or prplnts

Its Raman spectra are discussed in Ref 7 and UV spectra in Refs 8 & 9

Many salts of nitraminomethane are known, some of them more or less explosive, eg, ammonium, barium, cobalt, cadmium, copper, nickel, potassium, silver, sodium and zinc (Refs 1,2 & 3)

Refs: 1)Beil **4**,567,(568) & [968] 2)A.P.N. Franchimont & E.A.Klobbie,Rec **7**,354(1888) & **8**,295–7(1889) 3)A.P.N.Franchimont, Rec **13**,308–30(1894) 4)O.Diels & M.Paquin, Ber **46**,2013(1913) 5)J.R.Johnson,OSRD Rept 915(1942),28–9 6)A.H.Blatt,OSRD 2014(1944) 7)K.W.Kohlrausch & H.Wittek, ActaPhysAustriaca **1**,292(1948) & CA **42**, 6665(1948) 8)M.Karmack & J.J.Leavitt, JACS **71**,1222(1949) 9)R.N.Jones & G.D. Thorn,CanJRes **27B**,828(1949) 10)J.B. Hinkamp & R.Sugimoto,USP 2,595,789(1952) & CA **46**,8362(1952)

Aminomethane Nitroform or **Methylaminonitroform**, $CH_3NH_2{\cdot}CH(NO_2)_3$, mw 182.10, N 30.77%, OB to CO_2 -8.8%. Powerful expl (Trauzl test 168% PA), but unstable and hygroscopic

Ref: A.H.Blatt et al, OSRD Repot 2014(1944)

Aminomethane Perchlorate, $CH_3NH_2{\cdot}HClO_4$, mw 131.52, N 10.65%; OB to CO_2, H_2O, N_2O & Cl_2 -18.2%; OB to CO_2, H_2O, N_2 & Cl_2 -12.2%. Crysts, d 1.68(cast), mp 210°(242°, Ref 5); expl violently ca 338°; sol in w(110 g in 100 ml at 15°). Can be prepd by neutralizing aminomethane with perchloric acid

Aminomethane perchlorate is a more powerful explosive than TNT(Trauzl test value 160% of TNT) but much more sensitive to impact, comparable to LA. Its vel of deton is 7540 m/sec at d 1.68 and 6600 at d 1.565. Corresponding pressures of gases developed at expln are 1000 and 750 kg/cm²

As this expl has a high mp and is highly sensitive to shock, additives are mixed with it to lower its mp and sensitivity. It is suitable, when in such mixts, for loading in STET projectiles. It also can be used in AN mining expls as well as in plastic expls (Refs 4,5 % 6)

[See also Man-Salz Perchlorate in PATR 2510(1958)]

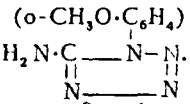
Refs: 1)Beil **4**,(318) 2)K.A.Hofmann et al, Ann **386**,306(1912) 3)B.L.Datta & N.N. Chatterjee,JCS **115**,1008(1919) 4)G.Lundsgaart & K.Herbst,BritP 168,333(1921) & CA **16**,344(1922) 5)J.F.Roth,SS **28**,42–6(1933) & CA **27**,2579–80(1933) 6)SAEH,BelgP 425,369(1938) & CA **33**,2719(1939)

Nitraminomethanecarboxylic Acid. Same as Nitraminoacetic or Nitraminoethanoic Acid

AMINOMETHOXYPHENYLTETRAZOLES AND DERIVATIVES

Aminomethoxyphenyltetrazoles; Anisylamino-tetrazoles or Anisidinotetrazoles, $C_8H_9N_5O$, mw 191.19, N 36.63%. The following isomers are described in the literature:

5-Amino-1-(o-methoxyphenyl)-α(or 1H)-tetrazole or 1-(2-Anisyl)-5-amino-tetrazole

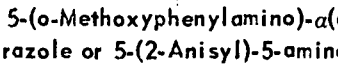


(o-CH_3O·C_6H_4)

H_2N·C(=N–N=N)–N. Fine ndls (from alc), mp 172°(Ref 2); crysts(from et acetate), mp 174(Ref 3); sl sol in hot w, easily sol in hot w. Was first prepd by Stollé (Ref 2) by prolonged heating of o-methoxyphenylthiourea with $PbCO_3$ and NaN_3 while passing in CO_2 gas. Henry et al (Ref 3) prepd it by cyclization of o-anisylguanylazide

Refs: 1)Beil – not found 2)R.Stollé et al, JPraktChem **134**,282–3 & 300(1932) 3)R.A.Henry et al, JACS **76**,92(1954)

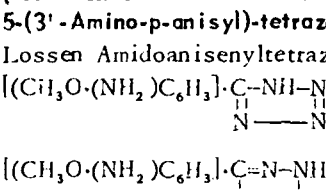
5-(o-Methoxyphenylamino)-α(or 1H)-tetrazole or 5-(2-Anisyl)-5-aminotetrazole,



(o-CH_3O·C_6H_4)·HN·C(=N–NH–N=N). Crysts(from 95% alc), mp 213–14°. Was obtained by isomerization of previous compd

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **76**,92(1954) 3)Ibid,**77**,2264–70(1955) (Isomerization of substituted 5-aminotetrazoles)

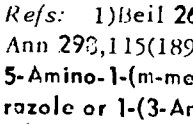
5-(3'-Amino-p-anisyl)-tetrazole, called by Lossen Amidoanisenyltetrazotsäure,



[(CH_3O·(NH_2)C_6H_3]·C(=N–NH–N=N) or [(CH_3O·(NH_2)C_6H_3]·C(=N–NH=N). Brn ndls of the monohydrate(from w), mp 223°; insol in eth; easily sol in hot w & alc. Was prepd by heating 5-(3-nitro-4-methoxyphenyl)-tetrazole with $SnCl_2$ in HCl of d 1.17

Refs: 1)Beil **26**,586 2)W.Lossen et al, Ann **298**,115(1897)

5-Amino-1-(m-methoxyphenyl)-α(or 1H)-tetrazole or 1-(3-Anisyl)-5-aminotetrazole



(m-CH_3O·C_6H_4)

H_2N·C(=N–N=N). Crysts(from 95% alc), mp 140.5–141.5°. Was prepd by cyclization of m-anisylguanylazide, H_2N·C(N_3):N(m-CH_3O·C_6H_4)

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **76**,92(1954)

5-Amino-1-(p-methoxyphenyl-α(or 1H)-tetrazole or 1-(4-Anisyl)-5-aminotetrazole,

(p-CH_3O·C_6H_4)

H_2N·C(=N–N=N)–N. Crysts(from 95% alc), mp 209–10°. Can be prepd by cyclization of p-anisylguanylazide

Refs: 1)Beil – not found 2)W.G.Finnegan et al, JOC **18**,788(1953) 3)R.A.Henry et al, JACS **76**,92(1954)

5-(p-Methoxyphenylamino)-α(or 1H)-tetrazole or 5-(4-Anisyl)-5-aminotetrazole,

(p-CH_3O·C_6H_4)·HN·C(=NH–N=N). Crysts(from 40% alc), mp 200–2°. Was prepd by catalytic dibenzylation of 5-(benzyl-4-anisyl)-5-aminotetrazole over palladium in AcOH

Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **76**,92(1954)

Aminomethoxyphenyltetrazoles, Thermal Isomerization. See R.A.Henry et al, JACS **76**,88(1954)

Aminomethoxyphenyltetrazoles, Identification by Means of IR Spectroscopy. See W.G.Finnegan et al, JACS **77**,4420(1955)

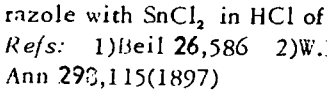
Aminomethoxyphenyltetrazoles, Azido–$C_8H_8N_8O$ and *Diazido*–, $C_8H_7N_{11}O$ *Derivatives* were not found in Beil or CA through 1956

Aminomethoxyphenyltetrazoles, Nitrated and/or Nitrited Derivatives were not found in Beil or CA through 1956

AMINOMETHYLAMINOIMIDAZOLIDINES AND DERIVATIVES

Aminomethylaminoimidazolidines, $C_4H_{12}N_4$, mw 116.17, N 48.23%. The isomer *2-amino-2-(methylamino-imidazolidine* may be considered the parent compd of the following derivs:

2-Amino-2-(methylnitramino)-1-nitro-imidazolidine,



H_2C–N(NO_2)–C(NH_2)–N(CH_3)·NO_2 / H_2C–NH, mw 206.17, N 40.77%. Crysts, mp 103–103.7°. This high nitrogen compd was obt in small yield in the course of the prepn of methylnitraminoethylnitroguanidine(Ref 2,pp 639–40)

Refs: 1)Beil – not found 2)M.W.Kirkwood & G.F.Wright, JOC **18**,634 & 639–40(1953); CA **48**, 6968(1954)

Aminomethylaminoimidazoline, Azido– $C_4H_{11}N_7$ and *Diazido–*$C_4H_{10}N_{10}$ *Derivatives*–not found in Beil or CA through 1956

Aminomethylanilines. Same as Methylphenylenediamines

Aminomethylbenzene. Same as Aminotoluene

1'-Amino-1-methylbenzene or **ω-Aminotoluene.** Same as Benzylamine

AMINOMETHYLBIPHENYLS AND DERIVATIVES

Aminomethylbiphenyls or *Methylbiphenylamines,* $C_{13}H_{13}N$, exist in several isometric forms, such as $H_2N{\cdot}C_6H_4{-}C_6H_4{\cdot}CH_3$; H_2N (attached to one ring) $C_6H_3{-}C_6H_5$; $H_3C{\cdot}HN{\cdot}C_6H_4{-}C_6H_5$ and $H_2N{\cdot}H_2C{\cdot}C_6H_4{-}C_6H_5$. There is also the isomer aminobiphenylmethane, $H_2N{\cdot}C_6H_4{-}CH_2{-}C_6H_5$. They are described in Beil **12**,1326 & [770–1]

Azidoaminomethylbiphenyls, $C_{13}H_{12}N_4$ and *Diazidoaminobiphenyls,* $C_{13}H_{11}N_7$ – were not found in Beil or CA through 1956

Mononitroaminomethylbiphenyls, $C_{13}H_{12}N_2O_2$. The isomer 3-nitro-4-methylamino-biphenyl is described in Beil **12**,[760] and the isomer 2-nitro-2-amino-biphenylmethane in JCS **1948**,299

Nitraminomethylbiphenyls, $C_{13}H_{12}N_2O_2$ and *Nitronitraminomethylbiphenyls,* $C_{13}H_{11}N_3O_4$ – were not found in Beil or CA through 1956

Dinitroaminomethylbiphenyls, $C_{13}H_{11}N_3O_4$. The isomer 3,5-dinitro-4-methylamino-biphenyl is described in Beil **12**,[762] and the isomer 3,4'-dinitro-4-amino-biphenyl-methane in JCS **1933**,1064

Dinitronitrosaminomethylbiphenyls, $C_{13}H_{10}N_4O_5$. The isomer 3,5-dinitro-4-methylnitroso-biphenyl is described in Beil **12**,[762]

Dinitronitraminomethylbiphenyls, $C_{13}H_{10}N_4O_6$ – not found in Beil or CA through 1956

Trinitroaminomethylbiphenyls, $C_{13}H_{10}N_4O_6$, mw 318.24, N 17.61%. The isomer N-(2,4,6-trinitro-benzyl)-aniline, or phenyl-(2,4,6-trinitrobenzyl)-amine is described in Beil **12**, (468); the isomer N-benzyl-4-nitro-aniline or picrylbenzylamine is in Beil **12**,[549] and the isomer x,x,x-trinitro-2-aminodiphenylmethane is in Beil **12**,(547). None of these isomers seem to be explosive

Tetranitroaminomethylbiphenyls, $C_{13}H_9N_5O_8$, mw 363.24, N 19.28%. The following isomer is described in the literature:

N-(2,4,6-Trinitrobenzyl)-3'-nitroaniline or *(3'-Nitrophenyl)-(2,4,6-trinitrobenzyl)-amine,* $(O_2N)_3C_6H_2{\cdot}CH_2{\cdot}NH{\cdot}C_6H_4NO_2$. Red ndls, mp 153°. Was prepd from 2,4,6-benzylbromide and 3-nitroaniline in boiling benz. Its expl props were not examined

Refs: 1)Beil **12**,(468) 2)S.Reich,O.Wetter & M.Widmer,Ber **45**,3059(1912) & CA **7**,1011 (1913)

Note: No later refs were found in CA through 1956

Pentanitrophenylbenzylamine, $C_{13}H_8N_6O_6$, mw 408.24, N 20.59%. Yel ndls(from acet), mp 274°(dec); sol in acet, AcOH & hot alc; v sl sol in chlf & eth. Was prepd by treating phenylbenzylurethane with fuming HNO_3 + H_2SO_4 at a temp somewhat higher than ~10°. Its expl props were not investigated

Refs: 1)Beil **12**,[566] 2)H.Ryan & J. O'Donnovan,SciProcRoyDublinSoc **17**,134 (1923) & CA **17**,1792(1923)

Note: No higher nitrated and/or nitrited derivs were found in Beil or CA through 1956

Aminomethyldiazacyclopentene. Same as Aminomethylimidazoline

Aminomethyldiphenyl. See Aminomethylbiphenyl

AMINOMETHYLDIPHENYLAMINES AND DERIVATIVES

Aminomethyldiphenylamines, $C_{13}H_{14}N_2$, mw 198.26, N 14.13%. Following isomers are listed in Beil **13**,18,42,80–1,130,154–5 & [13,42,61]: N-tolylphenylenediamine or

aminotoluinobenzene, $H_2N{\cdot}C_6H_4{-}NH{-}C_6H_4{\cdot}CH_3$, and N-phenyltoluenediamine or aminoanilinotoluene, H_2N (attached to C_6H_3 with $NH{-}C_6H_5$ and H_3C)

Azidoaminomethyldiphenylamines, $C_{13}H_{13}N_5$ – not found in Beil or CA through 1956

Diazidoaminomethyldiphenylamines, $C_{13}H_{12}N_8$ – not found in Beil or CA through 1956

Mononitroaminomethyldiphenylamines, $C_{13}H_{13}N_3O_2$, mw 243.26, N 17.28%. Several isomers described in Beil **13**,30,130 & [21,66]

Nitronitraminomethyldiphenylamines, $C_{13}H_{12}N_4O_4$ – not found in Beil or CA through 1956

Dinitroaminomethyldiphenylamines, $C_{13}H_{12}N_4O_4$, mw 288.26, N 19.44%. Several isomers are described in Beil **13**,42,79,81, 131,155 & [13,14,26,42]

Dinitronitraminomethyldiphenylamines, $C_{13}H_{11}N_5O_6$ – not found in Beil or CA through 1956

Trinitroaminomethyldiphenylamines, $C_{13}H_{11}N_5O_6$, mw 333.26, N 21.02%. Several isomers, none of them expl, are described in Beil **13**,30,61,79 & 81

Trinitronitraminomethyldiphenylamine, $C_{13}H_{10}N_6O_8$, mw 378.26, N 22.22%, OB to CO_2 –97.3%. The following isomer is described in the literature:

2,4,6-Trinitro-3-methylnitramino-diphenylamine, also called in Beil *N-Nitro-N-methyl-N'-phenyl-2.4.6-trinitrophenylendiamin-(1.3)* or *Methyl-[2.4.6-trinitro-3-anilino-phenyl]-nitramin,* $C_6H_5{\cdot}NH{\cdot}C_6H(NO_2)_3{\cdot}N(NO_2){\cdot}CH_3$. Yel crysts, mp 183°; easily sol in boiling acet, diff sol in alc. Was prepd by treating aniline with methyl-(2,3,4,6-tetranitrophenyl)-nitramine in benz

Refs: 1)Beil **13**,(17) 2)P.van Romburgh & J.H.Scheppers, VerslagKAkadWettenschappen **22**,298(1913) & CA **8**,3656(1914)

Tetranitroaminomethyldiphenylamines, $C_{13}H_{10}N_6O_8$ – not found in Beil or CA through 1956

Tetranitronitraminomethyldiphenylamines, $C_{13}H_9N_7O_{10}$, mw 423.26, N 23.17%, OB to CO_2 –77.5%. The following isomers are described in the literature:

2,4,6,2'-Tetranitro-3-methylnitramino-diphenylamine, also called in Beil *N-Nitro-N-methyl-N'-[2-nitro-phenyl]-2.4.6-trinitrophenylendiamin-(1.3),* $O_2N{\cdot}C_6H_4{\cdot}NH{\cdot}C_6H(NO_2)_3{\cdot}N(NO_2){\cdot}CH_3$. Yel crysts (from AcOH), mp 200°(dec). Was prepd by treating 2,4,6-trinitro-3-methylnitramino-diphenylamine with HNO_3(d 1.49) at RT. Its expl props were not examined

Refs: 1)Beil **13**,(18) 2)C.F.van Duin & B.C.R.van Lennep, Rec **38**,366(1919)

2,4,6,3'-Tetranitro-3-methylnitramino-diphenylamine, also called in Beil *N'-Nitro-N'-methyl-N³-[3-nitro-phenyl]-2.4.6-trinitrophenylendiamin-(1.3),* $O_2N{\cdot}C_6H_4{\cdot}NH{\cdot}C_6H(NO_2)_3{\cdot}N(NO_2)\,CH_3$. Yel crysts (from AcOH), mp 206°. Was prepd by heating methyl-(2,3,4,6-tetranitrophenyl)-nitramine with 3-nitro-aniline in benz. Its expl props were not examined

Refs: 1)Beil **13**,[34] 2)C.F.van Duin & D.R. Koolhaas, Rec **46**,380(1927)

2,4,6,4'-Tetranitro-3-methylnitramino-diphenylamine, also called in Beil *N'-Nitro-N-methyl-N³-(4-nitro-phenyl)-2.4.6-trinitro-phenylendiamin-(1.3),* $O_2N{\cdot}C_6H_4{\cdot}NH{\cdot}C_6H(NO_2)_3{\cdot}N(NO_2){\cdot}CH_3$. Orange-yel crysts (from AcOH), mp 200°. Was prepd by treating methyl-(2,3,4,6-tetra-nitrophenyl)-nitramine with 4-nitroaniline in boiling benz. Its expl props were not examined

Refs: 1)Beil **13**,(18) & [34] 2)C.F.van Duin & B.C.R.van Lennep, Rec **38**,367(1919) 3)C.F. van Duin & D.R.Koolhaas, Rec **46**,478(1927)

Pentanitroaminomethyldiphenylamines, $C_{13}H_9N_7O_{10}$ – not found in Beil, or CA through 1956

Pentanitronitraminodiphenylamines, $C_{13}H_8N_8O_{12}$ mw 468.26, N 23.94%, OB to CO_2 -61.5%. The following isomer is described in the literature:

2,4,6,2',4'-Pentanitro-3-methylnitraminodiphenylamine, also called in Beil, *N-Nitro-N-methyl-N'-(2.4-dinitrophenyl)-2.4.6-trinitrophenylendiamin-(1.3),* $(O_2N)_2C_6H_3{\cdot}NH{\cdot}C_6H(NO_2)_3{\cdot}N(NO_2){\cdot}CH_3$. Crysts(from AcOH), mp 224–5 . Can be prepd by nitrating 2,4,6,2- or 2,4,6,4'-tetranitro-3-methylnitraminodiphenylamine with cold HNO_3 (d 1.52) or by nitrating 4,6-dinitro-3-methylnitraminodiphenylamine with mixed HNO_3–H_2SO_4 at RT

Refs: 1)Beil **13**,(18) 2)C.F.van Duin & B.C.R.van Lennep,Rec **38**,365–6(1919)

Note: No later refs and no higher nitrated and/or nitrited derivs were found in CA through 1956

AMINOMETHYLGUANIDINES AND DERIVATIVES

Aminomethylguanidines and *Methylaminoguanidines,* $C_2H_8N_4$, mw 88.12, N 73.59% are high nitrogen compds, which might be useful as components of prplnts or expls. None of these compds was found in Beil. The following isomers and derivs were either prepd or could be prepd if there was a need, or interest:

1-Amino-1-methylguanidine (using the numeration recommended by ACS), $H_2N{-}N(CH_3){-}C(:NH){-}NH_2$. Its nitrated deriv was described in Ref 3 as *1-Amino-1-methyl-3-nitroguanidine,* $H_2N{-}N(CH_3){-}C(:NH){-}NH{\cdot}NO_2$, mw 133.12, N 52.61%, rosettes or flat ndls(from alc), mp 170–1°. It was prepd by Henry & Smith(Ref 3) from methylhydrazine and 1-methyl-1-nitroso-3-nitroguanidine using a method developed by McKay & Wright (Ref 2)

Note: According to Dr. Henry(Ref 5) the above aminomethylnitroguanidine is the same as *1-Amino-1-methyl-2-nitroguanidine* mentioned by Burkardt(Ref 4) and its formula is $H_2N{-}N(CH_3){-}C(:N{\cdot}NO_2){-}NH_2$. The name *1-amino-1-methyl-2-nitroguanidine* is preferred because it uses the smallest possible numbers(Ref 5)

Refs: 1)Beil – not found 2)A.F.McKay & G.F.Wright, JACS **69**,3028(1947) 3)R.A. Henry & G.B.L.Smith,JACS **73**,1858–9(1951) & CA **46**,2502(1952) 4)L.Burkardt,AnalChem **28**,823(1956) (X-ray diffraction pattern of 1-amino-1-methyl-2-nitroguanidine, stated to have been prepd by R.A.Henry, W.G. Finnegan & J.Cohen. No reference, no method of prepn or no props are given) 5)Dr Ronald A.Henry,NOTS,China Lake,Calif; private communication 5056/RAH:ef,22 Dec 1958

2-Amino-1-methylguanidine, $(CH_3)HN{-}C(:NNH_2){-}NH_2$. Its nitrated deriv *2-amino-1-methyl-3-nitroguanidine* was mentioned in Ref 2 without giving its method of prepn or props. Its formula is presumed to be $(CH_3)HN{-}C(:NNH_2){-}NHO_2$

According to Dr Henry(Ref 3), "Actually this compound has never been isolated in the free form: only its benzal hydrazone has been described. At the time this work was done we could not think of a straightforward synthesis leading to the free compound. I believe that we could do it today if there was a need or interest"

Refs: 1)Beil – not found 2)R.A.Henry & G.B.L.Smith,JACS **73**,1858–9(1951) & CA **46**, 2502(1952) 3)Dr Ronald A.Henry,NOTS, China Lake,Calif; private communication 5056/RAH:ef 22 Dec 1958

1-Amino-2-methylguanidine, $H_2NHN{-}C(:NCH_3){-}NH_2$ (Ref 3). This compd was one of the products obtained by Henry and Smith(Ref 2) as result of the reaction between methylamine and nitroaminoguanidine. It was first named 3-amino-1-methylguanidine

According to Dr Henry(Ref 3), "Direct nitration of 1-amino-2-methylguanidine is not possible because the hydrazino group would be

oxidized. If the hydrazino group could be protected, one would probably get a mixture of nitramines, for example: 1-amino-2-methyl-3-nitroguanidine, $H_2NNH-C(:NCH_3)-NHNO_2$ and 1-amino-2-methyl-2-nitroguanidine, $H_2NHN-C(:NH)NNO_2$ (CH_3). This latter isomer is also described in the literature''

Note: The formula for 1-amino-2-methyl-2-nitroguanidine is given in Ref 3 and it does not seem to agree with the numeration proposed by the ACS. If the formula of the last compd is right, then its name should be *1-amino-3-methyl-3-nitroguanidine*

Refs: 1)Beil – not found 2)R.A.Henry & G.B.L.Smith, JACS **73**,1858–9(1951) & CA **46**, 2502(1952) 3)Dr Ronald A.Henry,NOTS, China Lake,Calif; private communication 5056/RAH:ef 22 Dec 1958

Methylaminoguanidine, $(CH_3)NH\cdot NH\cdot C(NH_2):NH$ or $(CH_3)HN\cdot N:C(NH_2)_2$, was prepd as the sulfate by heating on a water bath concd solns of methylhydrazine and S-methylisothiourea sulfate. It was patented in Germany

Refs: 1)Beil **4**,[959] 2)Schering-Kahlbaum, GerP 463,576 & ChemZtr **1928**,II,1846

Azidoaminomethylguanidine, $C_2H_7N_7$ – does not exist as such because it is assumed to isomerize at once to the corresponding tetrazyl-guanidine compd

Nitronitraminomethylguanidine, $C_2H_6N_6O_4$ and *Dinitraminomethylguanidine,* $C_2H_6N_6O_4$ – not found in Beil or CA through 1956

Aminomethylimidazoline and Derivatives. See under Aminoimidazozoline and Aminoimidazolidine Substituted Derivatives

Aminomethylnitramines and Hydroxymethylnitramines. See under Hydroxy- and Aminomethylnitramines

Aminomethylnitrosolic Acid, Nitrosoformamidoxime or Nitrosoaminoformadoxime(Called *Amido-methylnitrosolsäure* by Wieland), $ON\cdot C(:NOH)\cdot NH_2$, mw 89.00 N 35.90%. Green plates changing readily to a yel amorphous mass; is decompd violently by inorganic acids. Can be prepd by the action of methylalcoholic KOH on dihydroxyguanidine-hydrobromide

The *potassium salt,* $KCH_2N_3O_2$, crystallizes from 80% alc in brilliant, steel-blue ndls, which dec at 21 ° or expl at 220°(Refs 1 & 2). Its aq soln treated with aq $AgNO_3$ and HNO_3 yields the very explosive silver fulminate

Refs: 1)Beil **3**,97 2)H.Wieland,Ber **38**, 1456–61(1905) & JCS **88**,421(1905) 3)H. Wieland,Ber **42**,820(1909)

AMINOMETHYLPROPANEDIOLS AND DERIVATIVES

2-Amino-2-methyl-1,3-propanediol; 2-Amino-2-methyl-1,3-dihydroxy-propane or *Bis(hydroxymethyl)methyl-aminomethane*(called in Beil β-Amino-β-methyl-trimethylenglykol), $HO\cdot CH_2-C(NH_2)(CH_3)-CH_2\cdot OH$, is described in Beil **4**,303. It may be considered the parent compd of the following derivs:

2-Amino-2-methyl-1,3-propanediol Dinitrate or **2-Amino-2-methyl-1,3-dinitroxypropane,** also called *Bis(nitroxymethyl)methylaminomethane* or *β-Amino-β-methyl-trimethyleneglycol Dinitrate,* $O_2NO\cdot CH_2-C(NH_2)(CH_3)-CH_2\cdot ONO_2$, mw 195.14, N 21.54%, OB to CO_2 –69.7%. Col liq, d 1.368 at 20/20°, n_D 1.4759 at 20°, expl on heating, unstable even at RT. Was prepd at PicArsn by nitration of 2-methyl-2-amino-1,3-propanediol, drowning the spent acid in w and making the soln alkaline. It was considered unsuitable for military purposes

Refs: 1)Beil – not found 2)H.Aaronson, PATR 1412(1944)

Note: Description of this compd was not

found in CA through 1956

2-Nitramino-2-methyl-1,3-propanediol Dinitrate, $O_2NO{\cdot}CH_2{-}C(NH{\cdot}NO_2){-}CH_2{\cdot}ONO_2$ (CH_3)

not found in Beil or CA through 1956

Azidoaminomethylpropanediol, $C_4H_{10}N_4O_2$, *Diazidoaminomethylpropanediol,* $C_4H_9N_7O_2$ and *Azidoaminomethylpropanediol Dinitrate,* $C_4H_8N_6O_4$ – were not found in Beil or CA through 1956

Note: R.C.Elderfield et al [OSRD Rept 158 (1941), 7–8] prepd by condensing 2-amino-2-methyl-1,3-propanediol with 2,4-dinitrochlorobenzene the product identified as *2,4-dinitrophenyl-bis(hydroxymethyl)-methylamine,* mp 162–3°, which on nitration with mixed nitricsulfuric acid gave *2,4,6-trinitrophenyl-(bis-hydroxy)-tert-butylnitramine dinitrate,* mp 159°,(See also under Anilinomethylpropanediol

AMINOMETHYLPROPANOLS AND DERIVATIVES

2-Amino-2-methyl-1-propanol, 2-Amino-2-methyl-1-hydroxypropane or **Hydroxyamino-methylpropane,** $CH_3{\cdot}C(NH_2)(CH_3){\cdot}CH_2OH$. May be considered the parent compd of the following derivs:

2-Nitramino-2-methyl-1-propanol Nitrate, 1-Nitroxy-2-nitramino-2-methyl-propane or **1-Nitroxy-2-nitramino-2,2-dimethylethane,** $CH_3{\cdot}C(NH{\cdot}NO_2)(CH_3){\cdot}CH_2{-}ONO_2$, mw 179.14, N 23.46%; crysts, mp–not given.

Can be prepd from 2-amino-2-methyl-1-propanol according to the reactions given in Ref 2, as follows:

$$CH_3{\cdot}C(NH_2)(CH_3){\cdot}CH_2OH \xrightarrow[\text{by aq NaOH}]{ClCOOC_2H_5\ \text{followed}} CH_3{\cdot}C(NH{\cdot}COOC_2H_5)(CH_3){\cdot}CH_2OH \xrightarrow{HNO_3\ \text{at}\ 10^\circ} CH_3{\cdot}C[N(NO_2)COOC_2H_5](CH_3){\cdot}CH_2OH \xrightarrow[\text{by HCl}]{NH_3\ \text{followed}} CH_3{\cdot}C(NH{\cdot}NO_2)(CH_3){\cdot}CH_2{\cdot}ONO_2$$

It was proposed as a possible gelatinizer for NC in propellants

Refs: 1)Beil – not found 2)A.T.Blomquist & F.T.Fiedorek, USP 2,485,855(1949),pp 6 & 15; CA **44**,3516–17(1950)

Azidoaminomethylpropanol, $C_4H_{10}N_4O$, *Diazidoaminomethylpropanol,* $C_4H_9N_7O$ and *Azidonitraminopropanol Nitrate,* $C_8H_8N_6O_5$ were not found in Beil or CA through 1956

Aminomethylpyridine. Same as Aminopicoline

AMINOMETHYL- AND METHYLAMINO-TETRAZOLES AND DERIVATIVES

Aminomethyltetrazoles and **Methylaminotetrazoles,** $C_2H_5N_5$, mw 99.10, N 70.68%. The following isomers were found in the literature:

5-Amino-1-methyl-α-tetrazole or **5-Amino-1-methyl-1H-tetrazole,** called in Beil *1-Methyl-5-amino-tetrazol* or *1-Methyl-tetrazolon-(5)-imid,* $H_2N{-}C(=N{-}N(CH_3){-}N=)$. Ndls or crysts, mp 218–223.5°

(Refs 2,3,4,5); mp 226–232°(Refs 7,8,9); decomp with evoln of gas. Easily sol in hot, diff sol in cold w, sol in alc, sl sol in eth. Was first prepd by Thiele & Ingle(Ref 2) and then by Oliveri-Mandalà(Ref 3). Stollé(Ref 4) prepd it by passing CO_2 gas through the stirred and heated mixt of monomethylthiourea, $PbCO_3$ and NaN_3 in alc. Other methods of prepn are in Refs 6,7 & 8. This compd was also prepd by Dr R.A.Henry(see footnote in Ref 8,p 1026)

X-ray diffraction spectra of 5-amino-1-methyl-α-tetrazole are discussed in Ref 6, its thermal isomerization in Ref 9, and its UV and IR absorption spectra in Ref 10

Refs: 1)Beil **26**,404 & [245] 2)J.Thiele & H.Ingle,Ann **287**,252(1895) 3)E.Oliveri-Mandalà,Gazz **52** I,103(1922) & CA **16**,2112 (1922) 4)R.Stollé et al, JPrChem **134**,282–3 & 285–6(1932), CA **26**,5565(1932) 5)R.M. Herbst et al,JOC **16**,142(1951) & CA **45**,6630 (1951) 6)L.Burkard & D.W.Moore,AnalChem **24**,1582(1952) & CA **47**,2010(1953) 7)W.G. Finnegan et al,JOC **18**,785,788 & 790(1953); CA **48**,7007(1954) 8)W.L.Garbrecht & R.M. Herbst,JOC **18**,1025–6(1953) & CA **48**,8225 (1954) 9)R.A.Henry et al,JACS **76**,88(1954) & CA **49**,2427(1955) 10)D.B.Murphy & J.P. Picard,JOC **19**,1808 & 1810(1954); CA **49**, 15879(1955)

5-Methylamino-α-tetrazole or **5-Methylamino-1H-tetrazole,** $CH_3{\cdot}HN{-}C{-}NH{-}N$ (‖ ‖ ‖ rings) $N\!-\!\!-\!\!-\!N$ crysts (from abs alc), mp 185°. Can be prepd by hydrogenation of 5-methylbenzylamino-tetrazole or by other methods(Refs 3 & 4). Its X-ray absorption spectra are discussed in Ref 2, thermal isomerization in Ref 5, UV and IR absorption spectra in Ref 6, and kinetics of thermal isomerization in Ref 7

Refs: 1)Beil – not found 2)L.Burkardt & D.W.Moore,AnalChem **24**,1582(1952) & CA **47**,2010(1953) 3)W.G.Finnegan et al,JOC **18**,785(1953) & CA **48**,7007(1954) 4)W.L. Garbrecht & R.M.Herbst,JOC **18**,1026–7 (1953) & CA **48**,8225(1954) (According to footnote,p 1026,Dr R.A.Henry prepd 5-methylaminotetrazole by a method essentially like that of the authors) 5)R.A.Henry et al,JACS **76**,88(1954) & CA **49**,2427(1955) 6)D.B.Murphy & J.P.Picard,JOC **19**,1808 & 1810(1954); CA **49**,15879(1955) 7)R.A.Henry et al,JACS **77**,2264(1955(& CA **50**,2557(1956)

5-Amino-2-methyl-β-tetrazole or **5-Amino-2-methyl-2H-tetrazole,** $H_2N{-}C{=}N{-}N{-}CH_3$ (| | | ring) $N{=\!=}N$

Crysts, mp 104.5–105.5°. Was obtained in 25–35% yield, together with 35–50% of the 1-isomer(see above), when an aq soln of Na 5-aminotetrazole(1 mol) was heated with dimethyl sulfate(0.5 mol)

Refs: 1)Beil – not found 2)J.H.Bryden, JACS **75**,4863(1953) & CA **49**,6242(1955)

5-Nitrosamino-1-methyl-α-tetrazole, $(ON)HN{-}C{-}N(CH_3){-}N$, mw 128.10, N 65.61%. (|| || rings) $N\!-\!\!-\!\!-\!N$

Leaflets(from w), mp defgr at ca 177°, easily sol in hot w, diff sol in cold w, very sol in hot, less in cold alc and v sl sol in eth. Was prepd by treating the above aminomethyltetrazole with $NaNO_2$ plus dil HCl in the cold. Its alc soln gives with alc $AgNO_3$ a wh flaky ppt of a silver deriv which is insol in ammonia

Refs: 1)Beil – not found 2)R.Stolle et al, JPrChem **134**,282–3 & 286(1932) & CA **26**, 5565(1932)

5-Nitrosamino-1-methyl-β-tetrazole, $(ON)HN{-}C{=}N{-}N{\cdot}CH_3$ was not found in Beil or CA through 1956

Note: No nitrated derivs or azido compds were found in Beil or CA through 1956

AMINOMETHYLTHIAZOLES AND DERIVATIVES

Aminomethylthiazoles and **Methylaminothiazoles,** $C_4H_6N_2S$, may be considered the parent

compds of the following derivs:

2-Amino-4-methyl-5-nitrothiazole,
$O_2N{\cdot}C(=S)-S-C(=N)NH_2$, mw 159.17, N 26.41%.
$H_3C{\cdot}C$ — N
Crysts(from alc), mp 220°. Can be prepd by hydrolysis of 2-acetamido-4-methyl -5-nitrothiazole(Ref 2), or by other methods(Refs 3,4 & 5)

Refs: 1)Beil – not found 2)K.Ganapathi & A.Venkataraman,ProcIndianAcadSci **22A**,343 (1945) & CA **40**,4058(1946) 3)I.V.Bellavita, AnnChimAppl **38**,449(1948) & CA **44**,154(1950) 4)J.B.Dickey & E.B.Towne,USP 2,659,719 (1953) & CA **49**,1336e(1955) 5)J.B.Dickey & E.B.Towne,USP 2,746,953(1956) & CA **50**, 15093(1956)

Azidoaminomethylthiazole, $C_4H_5N_5S$ – not found in Beil or CA through 1956

2-Nitramino-4-methylthiazole, $HC-S-C(=NH)NHNO_2$
$H_3C{\cdot}C$ — N
not found in Beil or CA thru 1956

2-Nitramino-4-methyl-5-nitrothiazole,
$O_2N{\cdot}C(=S)-S-C(=NH)NHNO_2$ or $O_2N{\cdot}C(=S)-S-C(=N{\cdot}NO_2)$;
$H_3C{\cdot}C$ — N $H_3C{\cdot}C$ — NH
mw 204.17, N 27.45%. Yel plates, mp 185° (Ref 2), 184–5°(decomp)(Ref 3). Was prepd by nitrating 2-amino-4-methylthiazole with mixed nitric-sulfuric acids, as indicated in Ref 3

Refs: 1)Beil – not found 2)E.Ochiai & H.I.Nagasawa,JPharmSocJapan **59**,43(1939) 3)S.J.Viron & A.Taurins,CanJChem **31**,887 & 891(1953) & CA **49**,2423(1955)

2-(N-Methylamino)-5-nitrothiazole,
$O_2N{\cdot}C(=S)-S-C(=NH{\cdot}CH_3)$
HC — N ; crysts (from 2:1 alc-water), mp 223.4–224.5°. Was prepd by heating 2-chloro-5-nitrothiazole with $MeNH_2$–HOAc and AcONa in AcOH

Refs: 1)Beil – not found 2)S.R.MBushby & E.C.Copp,JPharm and Pharmacol **7**,112 (1955) & CA **50**,964c(1956)

2-(N-Methylnitramino)thiazole, $HC-S-C(=N(CH_3)NO_2)$
HC — N
Crysts, mp 271.5–272°. Can be prepd by treating 2-nitraminothiazole either with dimethyl sulfate or ethereal diazomethane as described in Ref 2

Refs: 1)Beil – not found 2)J.B.Dickey, E.B.Towne & G.F.Wright,JOC **20**,505(1955) & CA **50**,4128b(1956)

2-(N-Methylnitramino)-5-nitrothiazole,
$O_2N{\cdot}C(=S)-S-C(=N(CH_3)NO_2)$, mw 204.17, N 27.45%.
HC — N
Crysts(from alc), mp 168.5–160°. Can be prepd either by nitration of methylnitraminothiazole with abs HNO_3 at –70° or by treating nitronitraminothiazole with methyl sulfate

Refs: 1)Beil – not found 2)J.B.Dickey, E.B. Towne & G.F.Wright, JOC **20**,505(1955) & CA **50**, 4127–8(1956)

3,5-Dinitro-4-methyl-2-nitrimino-Δ^4-thiazoline (listed in CA as **3,5-Dinitro-4-methyl-2-nitrimino-4-thiazoline**), $O_2N{\cdot}C(=S)-S-C(=N{\cdot}NO_2)$
$H_3C{\cdot}C$ — $N{\cdot}NO_2$
Wh solid, mp expl ca 98°. Was prepd by treating the previous compd with mixt of 99–100% nitric acid, acetic anhydride and glac acetic acid(Ref 2)

When the nitro compd was warmed with solvents, such as benz & acet or glacial AcOH, brown fumes of nitrogen dioxide were liberated. (See also Aminothiazoles)

Refs: 1)Beil – not found 2)S.J.Viron & A. Taurins,CanJChem **31**,891(1953) & CA **49**, 2423(1955)

AMINOMETHYLTRIAZOLES AND DERIVATIVES

Aminomethyltriazoles, $C_3H_6N_4$, mw 98.11, N 57.11%, OB to CO_2 –146.8%, OB to CO –97.8%.

These high nitrogen compds might be of use in expl & propellent compns. The following isomers and their derivs are described in the literature:

1-Amino-5-methyl-α-vic-triazole or **1-Amino-5-methyl-1H-1,2,3-triazole,** $H_3C{\cdot}C{-}N(NH_2){-}N$, $HC{=}N$

col lfts, mp 70°; easily sol in chlf, alc & w, more diff sol in ether. Can be prepd by heating 1-benzalamino-5-methyl-victriazole with 5% HCl or by other methods. It reacts neutral in water and forms salts with some acids

Refs: 1)Beil **26**,23 & [9] 2)L.Wolf & A.A. Hall, Ber **36**, 3617–18(1903) 3)R.Stollé, Ber **59B**, 1745(1926)

3-Amino-5-methyl-α-sym-triazole or **3-Amino-5-methyl-1H-1,2,4-triazole,** $H_3C{\cdot}C{-}NH{-}N$, $N{-}C{\cdot}NH_2$

is isomeric with **5-Amino-3-methyl-β-sym-triazole** or **5-Amino-3-methyl-2H-2,4,1-triazole,** $H_2N{\cdot}C{=}N{-}NH$, $N{=}C{\cdot}CH_3$, also called **5-Amino-3-methyl-1,2,4-triazole**(called in Ref 2 *Amidomethyltriazol*). Wh ndls, mp 148°(Refs 1 & 2), mp 151–2°(Ref 4); very sol in w & alc, diff sol in acet and nearly insol in other organic solvents. Can be prepd from acetylaminoguanidine nitrate and soda by the procedure described in Ref 2. No methods of prepn are given in Refs 3 & 4

It forms salts, such as: a)*nitrate*, $C_3H_6N_4{\cdot}HNO_3$, mw 161.13, N 43.47%, OB to CO_2 −54.6%, OB to CO −24.8%, wh crysts, mp 176–7°(Ref 4), 171°(Refs 1 & 2), Q_c^v 466.68 kcal/mol and Q_f +54.59 kcal/mol (Ref 4) b)*picrate*, $C_3H_6N_4{\cdot}C_6H_3N_3O_7$, mw 327.22, N 29.97%, OB to CO_2 −75.8%, OB to CO −31.8%, yel ndls, mp 225°(decompn)

Refs: 1)Beil **26**,145,(39) & [77] 2)J.Thiele & K.Heidenreich, Ber **26**, 2599–2600(1893) 3)E.Lieber & G.B.L.Smith, ChemRevs **25**, 253 & 255(1939) 4)M.M.Williams et al, J PhChem **61**, 264 & 266(1957)

Azidoaminomethyltriazoles, $C_3H_5N_7$ – not found in Beil or CA through 1956

Nitrosaminomethyltriazoles, $C_3H_5N_5O$, mw 127.11, N 55.10%. The compd listed in Beil **26**, [78] as *5-Nitrosamino-3-methyl-1,2,4-triazol* seems to be the nitroso deriv of previous aminomethyltriazole. It can be called *5-Nitrosamino-3-methyl-β-sym-triazole,* $ON{\cdot}HN{\cdot}C{=}N{-}NH$, $N{=}C{\cdot}CH_3$, which is identical with *3-Nitrosamino-5-methyl-α-sym-triazole,* $H_3C{\cdot}HN{\cdot}C{-}NH{-}N$, $N{-}C{\cdot}NH{\cdot}NO$, also called *3-Nitrosamino-5-methyl-1H-1,2,4-triazole*

Nitraminomethyltriazoles, $C_3H_5N_5O_2$, mw 143.11, N 48.94%, OB to CO_2 −72.7%, OB to CO −39.2%. The compd listed by Henry(Ref 2) and by Lieber(Ref 3) as *3-Methyl-5-nitroamino-1,2,4-triazole* is identical with *3-Nitramino-5-methyl-1,2,4-triazole* of Williams(Ref 5). We call this compd *3-Nitramino-5-methyl-α-sym-triazole* which corresponds to the CA name *3-Nitramino-5-methyl-1H-1,2,4-triazole,* $H_3C{\cdot}C{-}NH{-}N$, $N{-}C{\cdot}NH{\cdot}NO_2$. It is identical with *5-Nitramino-3-methyl-β-sym-triazole* or *5-Nitramino-3-methyl-2H-2.4.1-triazole,* $O_2N{\cdot}HN{\cdot}C{=}N{-}NH$, $N{=}C{\cdot}CH_3$. DeVries(Ref 4) lists isomers 3-methyl-5-nitroamino-1,2,4-triazole and 3-nitroamino-5-methyl-1,2,4-triazole as two different compds, but it seems that they are identical

Nitraminomethyltriazole described in Ref 2 was obtained as col rosettes or ndls, mp 212–13° with decompn. Williams(Ref 5) gives mp 206–7°. Was prepd by Henry from 1-acetamido-3-nitroguanidine as described in Ref 2,p 5344. Spectrophotometric studies of dissociation constant are discussed in Ref 4 and IR absorption spectra in Ref 3a. Its Q_c^v is 465.67 kcal/mol and Q_f −12.72 kcal/mol(Ref 5)

Refs: 1)Beil – not found 2)R.H.Henry, JACS **72**,5344(1950) 3)E.Lieber et al,JACS **73**,1793(1951) 3a)E.Lieber et al,AnalChem **23**,1594(1951) 4)J.E.DeVries & E.St.Clair Gantz,JACS **76**,1009(1954) 5)M.M.Williams et al,JPhysChem **61**,264 & 266(1957)

AMINONAPHTHALENES AND DERIVATIVES

Aminonaphthalenes or *Naphthylamines*, $C_{10}H_7NH_2$, mw 143.18, N 9.78%, are described in Beil **12**,1212,1265,(519,532) & [675,710]

Azidoaminonaphthalenes, $C_{10}H_6{\cdot}N_3{\cdot}NH_2$ and *Diazidoaminonaphthalenes*, $C_{10}H_5(N_3)_2{\cdot}NH_2$ – were not found in Beil or CA through 1956

Nitroaminonaphthalenes, $O_2N{\cdot}C_{10}H_6{\cdot}NH_2$, mw 188.18, N 14.89%, are described in Beil **12**, 1258–61,1308,1313–15,(530,544) & [703–5, 731–3]

Nitraminonaphthalenes, $C_{10}H_7{\cdot}NHNO_2$, mw 188.18, N 14.89%. Two isomers are described in Beil **16**,675 & [346]

Dinitroaminonaphthalenes, $(O_2N)_2{\cdot}C_{10}H_5{\cdot}NH_2$, mw 233.18, N 18.02%. Several isomers are described in Beil **12**,1262–4,1315–16,(530) & [708,734–5]

Nitronitraminonaphthalenes, $O_2N{\cdot}C_{10}H_6{\cdot}NH{\cdot}NO_2$ and *Dinitronitraminoaphthalenes*, $(O_2N)_2{\cdot}C_6H_5{\cdot}NH{\cdot}NO_2$, were not found in Beil or CA through 1956

Trinitroaminonaphthalenes, $(O_2N)_3{\cdot}C_{10}H_4{\cdot}NH_2$, mw 278.18, N 20.14%. The following isomers are described in the literature:

2,4,5-Trinitro-1-aminonaphthalene or **2,4,5-Trinitro-α-naphthylamine**, yel ndls or microscopic prisms; mp ca 264° with darkening and defgr at higher temp(Ref 2); melts with decompn at 305°(Ref 3); decomp at 310° (Ref 4). Was first prepd by Staedel(Ref 2) by heating the ethyl ether of 2,4,5-trinitro-α-naphthylamine with alc NH_3 in a sealed tube at 50° for 2 hours. A simpler method is to pass NH_3 gas through an amyl alcohol soln of 4-chloro-1,3,8-trinitro-naphthalene (Ref 3). Other methods are described in Ref 4

Refs: 1)Beil **12**,1264,(532) & [709] 2)W. Staedel,Ann **217**,173(1883) 3)Max Ridl, JCS **103**,1915(1913) 4)H.W.Talen,Rec **47**, 355(1928)

2,4,8-Trinitro-1-aminonaphthalene or **2,4,8-Trinitro-1-naphthylamine**, orange-yel crysts, mp 189–90°. Was prepd by nitration of 8-nitro-1-toluene-p-sulphonamidonaphthalene in AcOH, followed by hydrolysis in sulfuric acid

Refs: 1)Beil – not found 2)E.R.Ward & L.A.Day,JCS **1951**,782–7 & CA **45**,9014(1951)

1,6,8-Trinitro-2-aminonaphthalene or **1,6,8-Trinitro-2-naphthylamine**. Yel ndls turning black at ca 266°, decomp ca 300°(Ref 3) and defgr at higher temps(Ref 2). Was first prepd by Staedel by heating the ethyl ether of 1,6,8-trinitro-β-naphthylamine with alc NH_3 in a sealed tube at 50°. Other methods of prepn are listed in Refs 1,3 & 4

Refs: 1)Beil **12**,1316 & [736] 2)W.Staedel, Ann **217**,174(1883) 3)E.J.van der Kam,Rec **45**,572 & 727(1927) 4)F.Bell,JCS **1929**,2786

Trinitronitraminonaphthalenes, $(O_2N)_3{\cdot}C_{10}H_4{\cdot}NH{\cdot}NO_2$ – were not found in Beil or CA through 1956

Tetranitroaminonaphthalenes, $(O_2N)_4C_{10}H_3{\cdot}NH_2$,

mw 323.18, N 21.67%. The following isomers are described in the literature:

2,4,5,7-Tetranitro-1-aminonaphthalene(Called by Merz, α-Tetranitro-1-naphthylamine), lt yel ndls, mp 194°. Was first prepd by Merz & Weith by treating 4-bromo-1,3,6,8-tetranitronaphthalene in benzene with NH_3(Ref 2). Another method is to heat the corresponding tetranitro-halogen compd with urea at atm pressure, using xylene as a diluent(Ref 3). This compd is expl

Refs: 1)Beil **12**,1264 & [710] 2)V.Merz & W.Weith,Ber **15**,2717–18(1882) 3)W.H.Bentley & W.Blythe & Co Ltd,BritP 263,552(1925) & CA **22**,92(1928);GerP 480,343(1926) & CA **23**,4950(1929)

2,4,5,8-Tetranitro-1-aminonaphthalene(Called by Merz, β-Tetranitro-naphthylamine). Yel ndls, mp 202°. Was obtained by Merz & Weith by moderately warming 4-bromo-1,3,5,8-tetranitronaphthalene(suspended in benzene) with NH_3(Ref 2). This compd is a mild expl

Refs: 1)Beil **12**,1264 2)V.Merz & W.Weith, Ber **15**,2720–1(1882)

Tetranitronitraminonaphthalenes, $(O_2N)_4C_{10}H_3 \cdot NHNO_2$, *Pentanitroaminonaphthalenes*, $(O_2N)_5C_{10}H_2 \cdot NH_2$ and higher nitrated derivatives were not found in Beil or CA through 1956

AMINONICOTINIC ACIDS AND DERIVATIVES

Aminonicotinic or *Amino-3-pyridinecarboxylic Acids*(Aminonicotinsäure or *Amino-pyridincarbonsäure* in Ger), $H_2N \cdot (C_5NH_3) \cdot COOH$. Aminonicotinic acids are aminopyridinecarboxylic acids in which the carboxyl group is attached to position 3(if it is attached to position 2 the compd is called aminopicolinic acid). Four isomers of aminonicotinic acid with the amino groups in 2,4,5 or 6 positions are known. There is also an *isonicotinic acid* in which the carboxyl group is in position 4 and the amino group in position 3 (See also Aminopicolinic Acid)

Ref: Beil **22**,542–3,(676) & [264]

Azidoaminonicotinic Acid, $C_6H_5N_5O_2$ and *Diazidoaminonicotinic Acid*, $C_6H_4N_8O_2$ – were not found in Beil or CA through 1956

Nitroaminonicotinic Acids, $H_2N \cdot (C_5NH_2) \begin{matrix} COOH \\ \diagup \\ \diagdown \\ NO_2 \end{matrix}$

mw 183.12, N 22.95%. The following isomers are described in the literature:

5-Nitro-2-aminonicotinic Acid. Col ndls, decompg on heating to ca 318°.

Refs: 1)Beil **22**,542 2)S.Carboni,Gazz **83**, 637(1953) & CA **49**,1039–40(1955)

5-Nitro-6-aminonicotinic Acid. Yel ndls melting above 300° with decompn.

Refs: 1)Beil **22**,542 &[465] 2)C.Räth & G.Prange,Ann **467**,8(1928)

Nitraminonicotinic Acids, $O_2N \cdot HN \cdot (C_5NH_3) \cdot COOH$, mw 183.12, N 22.95%. The following isomers are described in the literature:

2-Nitraminonicotinic Acid, yel crysts decompg violently ca 180°. Was prepd by nitrating 2-aminonicotinic acid

Refs: 1)Beil – not found 2)S.Carboni,Gazz **83**,637(1953) & CA **49**,1039–40(1955)

6-Nitraminonicotinic Acid [6-Nitramino-pyridin-carbonsäure-(3), in Ger], solid, decompg explosively ca 233°. Was prepd by nitrating 6-aminonicotinic acid with $HNO_3 + H_2SO_4$

Refs: 1)Beil **22**,[522] 2)C.Räth & G.Prange, Ann **467**,6–7(1928)

3-Nitramino-iso-nicotinic or **3-Nitramino-4-pyridinecarboxylic Acid**, $O_2NHN(C_5NH_3) \cdot COOH$, mw 183.12, N 22.95%. Crysts, mp–expl ca 188°. Was obtained by adding 2.5 g HNO_3(d 1.40) slowly to 3 g of 3-amino-4-pyridinecarboxylic acid in 30 cc concd H_2SO_4 while maintaining the temp below 0°, pouring the mixt onto 150 g of chopped ice, making alkaline with NH_4OH and bringing the mixt to a pH of 1 with HCl

Refs: 1)Beil – not found 2)S.Carboni & G.Berti,Gazz **84**,883(1954) & CA **50**,992(1956)

Note: No higher nitrated derivs of aminonicotinic and amino-iso-nicotinic acids were found in Beil

Aminonitroform, Aminotrinitromethane or **Trinitroaminomethane(TNAMe)**, $H_2N{\cdot}C(NO_2)_3$, mw 166.06, N 33.74%. Solid substance obtained before WW II from TeNMe(tetranitromethane) by Dr Hans Walter in the laboratory of Dr Friedrich L. Hahn at the University of Frankford a/Main

Dr W used the following series of reactions:

$$C(NO_2)_4 \xrightarrow[KOH]{HCHO} KC(NO_2)_3 \xrightarrow{chlorine}$$

$$ClC(NO_2)_3(oil) \xrightarrow{2NH_3} H_2N{\cdot}C(NO_2)_3 + NH_4Cl,$$

but he failed to publish the results of his work(Ref 3)

It should be noted that during WW II large quantities of TeNMe were obtained in Germany and other belligerents as a by-product in the manuf of TNT. Inasmuch as TeNMe is a very dangerous oxidizer and very toxic, attempts were made at the laboratory of the Keystone Ordnance Works, Meadville, Pennsylvania, to transform TeNMe into a less obnoxious substance. In addition to transforming it to nitroform through the reaction:

$$C(NO_2)_4 \xrightarrow{Na_2SO_3} C(NO_2)_3SO_3Na \xrightarrow{Acid}$$

$HC(NO_2)_3 + NaHSO_4$, attempts were made to reduce the TeNMe to TNAMe, $H_2N{\cdot}C(NO_2)_3$ with the view of nitrating this compd to $O_2N{\cdot}HN{-}C(NO_2)_3$. The work was not completed because the plant was closed at the termination of hostilities

Refs: 1)Beil – not found 2)CA through 1956 – not found 3)Dr Hans Walter & Dr B.T.Fedoroff, Picatinny Arsenal, Dover, NJ; private communications

Aminonitroguanidine. See under Aminoguanidine

Aminonitrosaminothiadiazole. See under Diaminothiadiazole

Aminonitrotetrazole. See under Aminotetrazoles

Aminonitrotoluenes. See under Aminotoluene

Aminonitrothiazole. See under Aminothiazole

Aminonitrotriazole. See under Aminotriazole

Aminonitroxylenes. See under Aminoxylenes

AMINOÖXAZOLINES AND DERIVATIVES

Aminoöxazoline, $C_3H_6N_2O$ may be considered a parent compd of the following derivs:

2-Nitraminoöxazoline, $CH_2{-}O{-}C{\cdot}NH{\cdot}NO_2$ (with $CH_2{-}N$), mw 131.09, N 32.06%. Crysts(from 95% ethanol), mp 111–113.5°. Can be prepd either from 1-β-chloroethyl-3-nitrourea, ethanol and KOH(Ref 2) or by treating the ring isomer of 3-β-aminoethylnitrourea(see formula II, p 1837 of Ref 3) with $NaNO_2$ and AcOH(Ref 3). Its expl props were not examined

2-Nitraminoöxazoline and *Nitric Acid.* A trace of an expl oily substance was obtained on treating 2-nitraminooxazoline with 100% HNO_3 (Ref 3)

2-Nitraminoöxazoline and *Diazomethane.* A trace of an expl oily subst was obtained on treating 2-nitraminoöxazoline with diazomethane (Ref 3)

Refs: 1)Beil – not found 2)R.H.Hall & G.F.Wright,JACS **73**,2214–16(1951) & CA **46**, 1989(1952) 3)M.W.Kirkwood & G.F.Wright, JACS **76**,1839(1954) & CA **49**,6927(1955)

Azidoaminoöxazoline, $C_3H_5N_5O$, *and Diazidoaminoöxazoline*, $C_3H_4N_8O$ were not found in Beil or CA through 1956

Aminoöxyäthan. Ger for Aminohydroxyethane (see Aminoethanol)

Aminoöxyanthrachinon. Ger for Aminohydroxyanthraquinone

AMINOÖXYTRIAZOLES AND DERIVATIVES

Aminoöxytriazoles, $C_2H_4N_4O$, mw 100.08, N 55.99%, are high nitrogen compds and might prove to be useful as ingredients of prplnts. The following isomers are described in the literature:

4-Amino-3-oxy-1,2,4-triazole or **4-Amino-3-hydroxy-1,2,4-triazole** [Called in Beil 4-Amino-1.2.4-triazolon-(5)], HC=N-NH or HC=N-N

$H_2N.N$—CO H_2N—C·OH

Crysts, mp 181°. Was prepd by heating carbohydrazide(Beil **3**,121) with the ethylester of orthoformic acid in a sealed tube at 100° (Ref 2). It forms salts, such as *silver aminoöxytriazole*, $AgC_2H_3N_4O$(Refs 2 & 3)

Note: This triazole was believed by Curtius to have the structure, OC–NH–NH and he

HN—N=CH

called it ''Methenylcarbohydrazid'' (Ref 2). The correct structure was established by Stollé(Ref 3)

Refs: 1)Beil **26**,142 2)T.Curtius & K. Heidenreich,Ber **27**,2685(1894) & JPrChem **52**,475(1895) 3)R.Stollé,JPrChem **75**,423 (1907)

5-Amino-3-oxy-1,2,4-triazole [Called in Beil 5-Oxo-3-imino-1.2.4-triazolidin, Urazolmonoimid, 5-Amino-1.2.4-triazolon-(3) or Imidurazol] (Called in JCS *Iminourazole*), H_2N–C–NH–NH or OC–NH–NH. Wh ndls,

N—CO HN—C:NH

mp 285°. Was obtained by heating aminoguanidinehydrochloride and urea at 150–160°

Refs: 1)Beil **26**,192 2)G.Pellizzari & C. Roncagliolo,Gazz **31**,I,487–8(1901) & JCS **80**I,773(1901)

Azidoaminoöxytriazole, $C_2H_3N_7O$ – not found in Beil or CA through 1956

Nitrated and/or *Nitrited Aminoöxytriazoles* were not found in Beil or in CA through 1956

Aminopentanes. Same as Amylamines

AMINOPHENETOLES AND DERIVATIVES

Aminophenetoles, Aminophenolethylethers or *Phenetidines* (Ethoxyaminobenzenes, Ethyloxyanilines or Ethoxyanilines), $H_2NC_6H_4·OC_2H_5$. The three known isomers are described in Beil **13**,359,404,436,(109,129, 146) & [166,211,224]

Azidoaminophenetoles, $C_8H_{10}N_4O$ and *Diazidoaminophenetoles*, $C_8H_9N_7O$ – were not found in Beil or CA through 1956

Mononitroaminophenetoles, $H_2N·C_6H_3(NO_2)·OC_2H_5$, mw 182.18, N 15.38%. Several isomers are described in Beil **13**,388–90, 422, 520–1(136–7,186) & [192,284,286]

Nitraminophenetoles, $O_2NHN·C_6H_4·OC_2H_5$ not found in Beil or CA through 1956

Dinitroaminophenetoles, $H_2N·C_6H_2(NO_2)_2·OC_2H_5$, mw 227.18, N 18.50%. Several isomers are described in Beil **13**,393,423,525, (138,188,190,193) & [292–3]

Nitronitraminophenetoles, $O_2NHN·C_6H_3(NO_2)·OC_2H_5$ and *Dinitronitraminophenetoles*, $O_2NHN·C_6H_2(NO_2)_2·OC_2H_5$ – not found in Beil or CA through 1956

Trinitroaminophenetoles, Trinitrophenetidines or **Ethoxytrinitroanilines**, $H_2N·C_6H(NO_2)_3·OC_2H_5$, mw 272.18, N 20.59%, OB to CO_2 –76.4%, OB to CO –29.4%. The following isomers are found in the literature:

2,4,6-Trinitro-3-aminophenetole, 2,4,6-Trinitro-m-phenetidine, 2,4,6-Trinitro-3-aminophenolether or **3-Ethoxy-2,4,6-trinitroaniline.** Lt yel crysts, mp 107–8°. Can be prepd by boiling pure 2,3,4,6-tetranitroaniline with abs alc and NaOAc. Another method consists of treating 2,4,6-trinitro-m-dichlorobenzene first with EtOH and then with NH_3(Refs 1,2,3). Its expln temp is 236° when heated at the rate of

5°/min and 257° when heated 20°/min; impact test values with a 10 kg wt >24 cm max fall for 0/6 shots vs 18–19 cm for tetryl; thermal stability at 95°–no change in 30 8-hour days(Ref 3)

Refs: 1)Beil **13**,(140) & [217] 2)B.J. Flürscheim,BritP 18,777(1911) & CA **7**,1100 (1913) 3)C.F.van Duin & B.C.R.van Lennep,Rec **39**,150–1,162–5 & 169–77(1920) 4)A.H.Blatt,OSRD Rept 2014(1944)

2,3,5-Trinitro-4-aminophenetole or **2,3,5-Trinitro-p-phenetidine** (called **4-Ethoxy-2,3,6-trinitroaniline** by Lorang). Red ndls with greenish sheen (from methanol), mp 125–7°; easily sol in acet, hot alc, AcOH and NB. Reacts in the same manner as the previous compd. Was prepd in an impure state in 1884 by Köhler, but he did not establish its structure (Ref 1,p 532). Much later it was prepd in a pure state by heating 2,3,5-trinitro-4-p-toluenesulfaminophenetole with concd H_2SO_4 at 70–80°(Ref 2). There is also another method of prepn(Ref 3). It is unquestionably an expl of superior stability against heat, but it was not investigated from this point of view

Refs: 1)Beil **13**,532(195) & [294] 2)F. Reverdin & L.Fürstenberg,BullFr [4] **13**,676 (1913) & JPrChem **88**,323(1913) 3)H.F. Lorang, Rec **46**,642–644(1927)

Note: No higher nitrated and/or nitrited derivs were found in Beil or CA through 1956

AMINOPHENOLS AND DERIVATIVES

Aminophenols or *Aminohydroxybenzenes* (called in JCS **119**,p 1310,*"Aminophenoxides"* Amino-oxy-benzol in Ger). Three isomers of $H_2N\text{-}C_6H_4\text{-}OH$, mw 109.12, N 12.84%, are known and described in Beil **13**,354,401,427, (108,128,143) & [164,209,220]

Azidoaminophenols, $C_6H_6N_4O$ and *Diazidoaminophenols* were not found in Beil or CA through 1956

Mononitroaminophenols or *Aminonitrophenols,* $H_2N\cdot C_6H_3(NO_2)\cdot OH$, mw 154.12, N 18.18%. Several isomers are described in Beil **13**, 388,390–1,421–2,520–1) & [121, 136, 185–6]

Nitraminophenols, $O_2NHN\cdot C_6H_4\cdot OH$ and *Nitronitraminophenols,* $O_2NHN\cdot C_6H_3(NO_2)OH$ not found in Beil or CA through 1956

Dinitroaminophenols or **Aminodinitrophenols,** $H_2N\cdot C_6H_2(NO_2)_2\cdot OH$, mw 199.12, N 21.10%, OB to CO_2 -84.3%, OB to CO -4.02%. As these compds and some of their derivs are mild expls, a brief description of each isomer is given

3,5-Dinitro-2-aminophenol or **2-Amino-3,5-dinitrophenol**, yel solid, mp 218°. It was prepd from o-aminophenol and p-toluene sulfochloride through a series of reactions described in Ref 2. Its *sodium salt* was also prepd

Refs: 1)Beil – not found 2)L.Horner, U. Schwenk & E.Junghanns,Ann **579**,226(1953) & CA **48**,2692(1954)

4,6-Dinitro-2-aminophenol; 2-Amino-4,6-dinitrophenol or **Picramic Acid(PAA)** (Dinitrophenamic Acid or 1-Hydroxy-2-amino-4,6-dinitrobenzene). Dark-red ndls (from alc) or prisms(from chlf), mp 169–170°; v sl sol in cold w, 10.14 g in 100 g at 22°, more sol in hot w, easily in alc, benz & AcOH; diff sol in eth & chlf. Was first prepd in 1853(Ref 2) by reducing PA with H_2S in alc NH_3. Other reducing agents such as Zn in NH_3, alc $(NH_4)_2S$, or aq Na_2S, can be used. Lyons & Smith(Ref 5) prepd PAA in good yields by reducing PA with iron turnings in a very dil soln of Fe or Na chloride at 80–85°. Other methods of prepn are listed in Ref 1 as well as in Refs 7 & 7a

According to Daniel(Ref 3), Turpin prepd PAA about 70 years ago in France on an industrial scale and used it, as well as its Na salt, in expl compns such as PAA 30 to 50% and KNO_3 70 to 50%

PAA is a mild explosive and its expl props were examined by Will(Ref 4). Its Q_c^v is 678.49 kcal/mol(Ref 6) or 677–74(Ref 8), and Q_c^p is 676.9(Ref 6) or 676.15(Ref 8); Q_f^v is +57.8(Ref 8) and Q_f^p +60.5(Ref 6) or +60.5(Ref 6) or 61.8 kcal/mol(Ref 8)

PAA forms salts, some of which are explosive(see below)

Refs: 1)Beil **13**,394,(123) & [196] 2)A. Girard,CR **36**,421(1853) & Ann **88**,281(1853) 3)Daniel(1902),615 4)W.Will,Chemische Industrie **26**,130(1902) 5)R.E.Lyons & L.T. Smith,Ber **60**,180(1927) 6)W.H.Rinkenbach, JACS **52**,1161(1930) 7)I.I.Vorontsov,Zh 7a)H.H.Hogson & E.R.Ward, JCS **1945**, 663 & CA **40**, 1149(1946) 8)L.Médard & M.Thomas,MP **31**, 196(1949)

Ammonium Picramate, $(NH_4)C_6H_4N_3O_5$, dk orange plates, mp 165° and dec at higher temp. Was prepd by Girard by neutralizing PAA with ammonia

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 281(1853)

Barium Picramate, $Ba(C_6H_4N_3O_5)_2$, red ndls with golden reflection, which can be safely heated to 200° but which detonate at a higher temp. It was prepd by Girard by mixing a hot aq soln of Amm picramate with an aq soln of Ba nitrate

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 281(1853)

Chromiumbexammine Picramate or *Hexam-minochromic Picramate,* $[Cr(NH_3)_6](C_6H_4O_5N_3)_3$, brn, amor ppt. Was prepd from hexammine-chromic hydroxide and picramic acid

Refs: 1)Beil – not found 2)H.J.S.King, JCS **125**,1335(1924)

Copper Picramate, $Cu(C_6H_4N_3O_5)_2$, yel grn amor ppt; insol in w & alc and sol in acids and NH_3; detonated mildly on heating. It was prepd by Girard by treating an aq soln of Amm picramate with a soln of a Cu salt

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 281(1853)

Lead Picramate, $[H_2N{\cdot}C_6H_2(NO_2)_2O]_2Pb$, mw 603.44, N 13.93%. Red-brn ndls, sl sol in w, sol in NH_3 and acids and insol in alc. Can be prepd by treating a soln of picramic acid with a sol lead salt. It expl on heating or on impact(Ref 2). Was proposed by Friederich(Ref 3) for use in primers, detonators and percussion caps.

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 281(1853) 3)W.Friederich,BritP 192,830 (1921) & CA **17**,3255(1923)

Potassium Picramate, $KC_6H_4N_3O_5$, red plates, decomp explosively at fairly high temp. Was prepd by treating hot soln of Amm picramate with KOH

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 282(1853)

Silver Picramate, $AgC_6H_4N_3O_5$, brick-red amor powder, mp 165° and burns without deton when placed on red-hot coal

Refs: 1)Beil **13**,395 2)A.Girard,Ann **88**, 283(1853)

Sodium Picramate, $NaC_6H_4N_3O_5$, dk red scales, sl sol in w. According to Dunn(Ref 3), Na picramate contg 15.5% H_2O was readily ignited by spark or flame and was only ignited and not detonated by a No 6 elec detonator; at 295–300° it exploded with considerable violence. After 72 hrs at 75°, it gave no evidence of decompn. It was first prepd by neutralizing picramic acid with NaOH. Hodgson & Ward(Ref 4) prepd it in almost quant yield by reducing Na picrate with Na sulfide

Na picramate was used in 1887–8 by Turpin in some expl compns, such as: a)Na picramate 20, Ba nitrate 60, nitrobenzene 10 & nitrophenol 10% b)Na picramate 25 to 53 & K nitrate 75 to 47%(Ref 2)

Refs: 1)Beil – not found 2)Daniel(1902), 615 3)B.W. Dunn, "Rept of Chief Inspector,

Bureau for Safe Transportation of Explosives and Other Dangerous Articles'', Mar 1, 1921 & CA **15**,2356(1921) 4)H.H.Hogson & E.R. Ward,JCS **1945**,664 & CA **40**,1149(1946)

Thalliumdiethyl Picramate (called by Goddard Thalliumdiethyl-4,6-dinitro -2-aminophenoxide), $(C_2H_5)_2Tl{\cdot}C_6H_4N_3O_5$, mw 432.57, N 9.71%. Small carmine-red plates with metallic lustre, mp 159° with decomp(darkening ca 140°); expl violently when moistened with fuming HNO_3. Moderately sol in alc, acet and pyridine, sl sol in chlf, eth or toluene, insol in CCl_4 & petr eth. Was prepd by Goddard by treating an aq soln of picramic acid with thalliumdiethyl bromide, $Tl(C_2H_5)_2Br$

Refs: 1)Beil **13**,[197] 2)A.E.Goddard,JCS **119**,1313(1921)

Thalliumdimethyl Picramate (called by Goddard Thalliumdimethyl-4,6-dinitro-2-aminophenoxide), $(CH_3)_2Tl{\cdot}C_6H_4N_3O_5$, N 9.72%. Small, deep red-violet plates darkening at 220° and melting with decompn at 236°. Easily sol in alc, acet and pyridine, moderately sol in eth, insol in chlf, CCl_4, toluene & petr eth. Was prepd by Goddard from picramic acid and thallium dimethyl iodide, $Tl(CH_3)_2I$. It probably expl, as does the diethyl salt, when moistened with fuming HNO_3

Refs: 1)Beil **13**,[197] 2)A.E.Goddard,JCS **119**,1313(1921)

2,6-Dinitro-3-aminophenol or **3-Amino-2,6-dinitrophenol.** Crysts(from chlf), mp 222–5°; diff sol in w or chlf, more readily sol in eth. Was prepd by Lipmann & Fleisser(Ref 2) by treating 2,4-dinitroaniline with alc KCN, filtering the pptd K salt, dissolving it in w and treating the soln with HCl to ppt the dinitroaminophenol

Note: Its structure as 2,6-dinitro-3-amino-isomer was established by Borsche & Feske (Ref 3). Prior to this, some investigators considered it as 2',4-dinitro-3-aminophenol [See Beil **13**,(137)]. The correct formula is given in Beil **13**,[216]

Refs: 1)Beil **13**,424,(137) & [216] 2)E. Lippmann & F.Fleissner, Monatsh **7**,95(1886) & JCS **50**,791(1886) 3)W.Borsche & E.Feske, Ber **61**,699(1928)

2,3-Dinitro-4-aminophenol or **4-Amino-2,3-dinitro-phenol.** Red cryst ppt. It was prepd in small quantity by heating 2,3-dinitro-4-acetaminophenol with concd H_2SO_4 for a few mins, as described in Ref 2. It was not possible to isolate the 2,3-DNAPh in a state suitable for analysis since it began to decomp with the evoln of gas as soon as it was freed from acid by washing on a filter. The mobile nitro-group in proximity to the amino-group is the determining cause of the instability

Refs: 1)Beil **13**,525 & (188) 2)R.Meldola & J.G.Hay,JCS **91**,1482(1907)

2,5-Dinitro-4-aminophenol or **4-Amino-2,5-dinitrophenol** (Called by Girard 3,6-Dinitro-4-aminophenol). Dk-violet ndls(from alc, w or benz); mp 166–167° with sublimation. Easily sol in alc, acet or AcOH, sol in w, diff sol in benz and insol in ligroin. Can be prepd by heating 2,5-dinitro-4-acetaminophenol with concd H_2SO_4(Ref 3) or by heating 2,5-dinitro-4-aminophenetol(Ref 2)

Refs: 1)Beil **13**,[292] 2)F.Reverdin & H.P.A.Roethlisberger,Helv **5**,304(1922) 3) A.Girard,BullFr [4] **35**,776(1924) & JCS **126** I, 959(1924)

2,6-Dinitro-4-aminophenol, 4-Amino-2,6-dinitro-phenol or **Isopicramic Acid** (Isopikraminsäure in Ger). Yel-brn ndls(from w), mp 170° with sl decompn. Sl sol in w(0.082 g/100 g at 22° and 0.812 g at 100°), very sol in alc and less sol in benz. It expl on heating above the mp. Was first prepd in 1883 by C.W.Dabney(Ref 1,p 528). Reverdin et al(Ref 2) prepd it by treating 2,6-dinitro-4-acetamidophenol, $CH_3CO{\cdot}NH{\cdot}C_6H_2(NO_2)_2OH$, with HCl. It forms salts, some of them expl, eg *potassium isopicramate*, $(O_2N)_2C_6H_2(NH_2)OK$, bluish-black ndls(from alc) v sol in w or alc (Ref 1,p 528)

Refs: 1)Beil **13,** 527, (190) & [293] 2) F.Reverdin et al Ber **37,** 4452(1905); **38,** 1598(1906) & **39,** 126(1907)

3,5-Dinitro-4-aminophenol or 4-Amino-3,5-dinitro-phenol. Red ndls with greenish luster; subl ca 150° and melts at 230-231°; easily sol in alc and hot w, sl sol in benz and nearly insol in ligroin. Can be prepd by heating 3,5-dinitro-4-(3-nitro-4-methylbenzene-sulfamino)-phenetole with concd H_2SO_4 on water bath (Ref 2) or by other methods (Refs 1 & 3)

Refs: 1)Beil **13,** 529,(193) & [293] 2)R. Reverdin & L.Fürstenberg Bull Fr [4] **13,** 673(1913) & JPrChem [2] **88,** 321(1913) 3) R.Reverdin, Helv **12,** 117 & 119(1929)

Dinitronitraminophenols, $O_2NHN \cdot C_6H_2(NO_2)_2 \cdot OH$, mw 244.12, N 22.95%. Not found in Beil or CA through 1956

Trinitroaminophenols, $H_2N \cdot C_6H(NO_2)_3OH$, mw 244.12, N 22.95%, OB to CO_2 -45.9%, OB to CO -6.55%. The following isomers were found in the literature:

3,5,6-Trinitro-2-aminophenol or 2-Amino-3,5,6-trinitrophenol, crysts, mp- expl ca 167°. Was prepd by Heller et al (Ref 2) by heating 2-acetamido-3,5,6-trinitrophenol with concd H_2SO_4 on a water bath, pouring the reaction mixt into water and recrystallizing from benzene

Its brominated product, yel ndls, structure not established, expld ca 180°

Refs: 1)Beil—not found 2)G.Heller et al, JPrChem **129,** 242-3(1931) & CA **25,** 2129 (1931)

2,4,6-Trinitro-3-aminophenol (TNAPh) or 3-Amino-2,4,6-trinitrophenol or 3-Amino-picric Acid. Yel-brn flat ndls (from alc), mp 178-180°(was reported by Blanksma as 218° and in Ref 5 as 222-3°); nearly insol in cold alc or w, sl sol in hot alc or benz. Several methods of prepn are described in the literature. C.F.van Duin et al (Ref 3) prepd it by treating an acetonic soln of tetranitroaniline with an aq soln of CH_3COONa at RT. Several other methods are given in Refs 1, 2 & 4

In a recent patent(Ref 5) is described the method of prepn of 3-amino-2,4,6-trinitro-phenol, mp 222-3°; from 3-chloropicric acid and gaseous NH_3

Expl props of TNAPh, as detnd by van Duin et al, are as follows: *explosion temp* -250°(when heated at the rate of 20°/min) and 231°(when heated at the rate of 5°/min) (corresponding temps for TNT are 321° & 304° and for tetryl 196° & 197°); *impact sensitivity* with Lenze-Kast app and 10kg wt—maxim fall for 0/6 shots 22-24cm (TNT >24 and tetryl 14cm), and minim fall for 6/6 shots > 24 cm (TNT & tetryl > 24 cm); *thermal stability at 95°—no change* in 30 8-hour days (Ref 3, pp 169-177 & Ref 5). These results show that as an explosive 2,4,6-trinitro-3-aminophenol lies between TNT and tetryl in sensitivity to impact and thermal stability

Refs: 1)Beil **13,** 425, (140) & [217] 2) J.J.Blanksma, Rec **21,** 259-61(1902) and Ber **47,** 687(1914) 3)C.F.van Duin et al, Rec **37,** 116(1918); 90-1(1919) & **39,** 149-50, 165 & 169-77(1920) 4)W.Borsche & E.Feske, Ber **61,**694-5(1928) 5)A.H.Blatt,OSRD Rept 2014(1944) 6)H.Feurer & A.A.Harban, USP 2,679,538(1954) & CA **49,**4715(1955)

2,3,6-Trinitro-4-aminophenol or 4-Amino-2,3,6-trinitrophenol. Red ndls(from AcOH), mp—decomp ca 145° and then expl. Can be prepd by heating 2,4,6-trinitroacetamidophenol, $CH_3CO \cdot NH \cdot C_6H(NO_2)_3OH$, for a short time in a water bath with concd H_2SO_4(Ref 2). Its constitution, previously reported as the 2,3,5-trinitro isomer, was established by Meldola & Reverdin as the 2,3,6-isomer(Ref 3)

In earlier work, Meldola and Hay(Ref 2, pp 1382-4) described the diazotization of the same TNAPh. One of the products obtained by them was the highly expl *trinitroquinonediazide*

Refs: 1)Beil **13,**533 & [197] 2)R.Meldola & J.G.Hay,JCS **95,**1381-4(1909) 3)R.Meldola & F.Reverdin,JCS **103,**1485(1913)

Trinitronitraminophenol, $O_2NHN \cdot C_6H(NO_2)_3OH$, not found in Beil or CA through 1956

Note: No higher nitrated derivs were found in Beil or CA through 1956

Aminophenols and Derivatives, Analytical Procedures are discussed in OrgAnalysis, Interscience, NY, 3(1956), 184

Aminophenolethylether. Same as Aminophentole

Aminophenolmethylether. Same as Aminoanisole

Aminophenylacetic Acid. See Anilinoacetic Acid

AMINOPHENYLARSONIC ACID AND DERIVATIVES

Aminophenylarsonic Acid or *Aminobenzenearsonic Acid*, $H_2N{\cdot}C_6H_4{\cdot}O{\cdot}As(OH)_2$. Its o-, m- and p-isomers are known; the para is of interest because its dinitro deriv is explosive

p-Aminophenylarsonic Acid or **p-Aminobenzenearsonic Acid**, also called *Arsanilic Acid; Atoxylic Acid; Arsenic Acid Anilide* or *m-Arsenious Acid Anilide*; col ndls(from w or alc); loses at 150° a mol of w to form the anhydride $H_2N{\cdot}C_6H_4{\cdot}AsO_2$; begins to brown at 280° but can be heated over 350° without melting or completely decompg; sparingly sol in cold w, AcOH or alc; easily sol in boiling w or alc and in MeOH; almost insol in eth, acet, benz & chlf. Was first prepd in 1863 by Bechamp by heating anilino-arsenic acid(see Beil **16**,878). Cheetham & Schmidt(Ref 3) prepd it by heating arsenic acid in a large excess of aniline. Detailed description of lab method of prepn of p-aminophenylarsonic acid is given in Ref 3

Refs: 1)Beil **16**,878(466) & [491] 2)H.C. Cheetham & J.H.Schmidt, JACS **42**,828(1920) 3)OrgSynthCollVol 1(1941),70

Azidoaminophenylarsonic Acid, $C_6H_7N_4O_3As$, and *Diazidoaminophenylarsonic Acid*, $C_6H_6N_7O_3As$, not found in Beil or CA through 1956

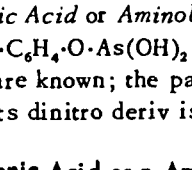
Nitroaminophenylarsonic Acid, $C_6H_7N_2O_5As$. The deriv *3-nitro-4-amino-phenylarsonic acid* is described in Beil **16**,881,(483) & [508] and *2-nitro-4-aminophenylarsonic acid* in Beil **16**, (484) & [510]

Nitroazidoaminophenylarsonic Acid, $C_6H_5N_5O_5As$ – not found in Beil or CA through 1956

Nitronitraminophenylarsonic Acid, $C_6H_6N_3O_7As$ – not found in Beil or CA through 1956

Dinitroaminophenylarsonic Acid, $C_6H_6N_3O_7As$, mw 307.05, N 13.69%. The following isomer is described in the literature

3,5-Dinitro-4-amino-phenylarsonic Acid or **2,6-Dinitro-1-amino-4-benzene-arsonic Acid**,



Brn-yel ndls or lflts(from 50%AcOH) (Ref 2); golden yel pdr, mp 285–95° (Ref 3); diff sol in alc, w and dil inorg acids; sol in alkalies & aq AcONa. Was first prepd by Benda(Ref 2) by nitrating p-aminophenylarsonic acid. DeLange (Ref 3) prepd it by treating 4-methoxy-3,5-dinitrophenylarsonic acid with alc ammonia, followed by acidification with HCl

This compd is probably expl, yet not reported as such. Its methyl deriv is expl although its NO_2-nitrogen content is smaller (See 3,5-Dinitro-4-methylaminophenylarsonic Acid under Methylaminophenyl-arsonic Acid)

Refs: 1)Beil **16**,(484) & [510] 2)L.Benda, Ber **45**,54(1912) 3)M.P.deLange, Rec **45**,51–3(1926)

Dinitronitraminophenylarsonic Acid, $C_6H_5N_4O_9As$ – not found in Beil or CA through 1956

AMINOPHENYLETHANOL AND DERIVATIVES

(See also Anilinoethanol)

β-Aminophenylethanol; 2-Aminophenyl-ethyl alcohol; β-Ethanolaniline or *β-Hydroxyethyl-aniline*, $H_2N{\cdot}C_6H_4{\cdot}CH_2{\cdot}CH_2OH$. Two isomers: β-(2-amino-phenyl)-ethanol and β-(4-amino-phenyl)-ethanol are described in Beil **13**,242 & [362] (See also under Anilinoethanols, $C_6H_5{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}OH$)

Azidoaminophenylethanol, $C_8H_{10}N_4O$ and *Diazidoaminophenylethanol*, $C_8H_9N_7O$ – were not found in Beil or CA through 1956

Nitroaminophenylethanol, $C_8H_{10}N_2O_3$; *Nitraminophenylethanol*, $C_8H_{10}N_2O_3$ and *Nitronitraminophenylethanol*, $C_8H_9N_3O_5$ – were not found in Beil or CA through 1956

Dinitroaminophenylethanol, $C_8H_9N_3O_5$, mw 227.18, N 18.50%. The following isomer is known:

2-(2',6'-Dinitro-4'-aminophenyl)-ethanol, $H_2N{\cdot}C_6H_2(NO_2)_2{\cdot}CH_2{\cdot}CH_2OH$. Yel ndls, mp 161–5°. Was prepd by reduction of 2-(2',4',6'-trinitrophenyl)-ethanol with hydrogen sulfide. Its expl props were not investigated

Refs: 1)Beil – not found 2)G.D.Parkes & A.C.Farthing, JCS **1948**, 1277–8 & CA **43**, 592(1949)

Dinitronitraminophenylethanol, $C_8H_8N_4O_7$ and *Trinitronitraminophenylethanol*, $C_8H_7N_5O_9$ were not found in Beil or CA through 1956

Trinitroaminophenylethanols, $H_2N{\cdot}C_6H(NO_2)_3CH_2{\cdot}CH_2{\cdot}OH$ – not found in Beil or CA through 1956. [Cf with β-(2,4,6-Trinitroanilino)ethanol described under Anilinoethanol]

Note: No other nitrated derivs of aminophenylethanols were found in Beil or CA through 1956 [Cf with β-(2,4,6-Trinitronitranilino)-ethanol Nitrate described under Anilinoethanol]

AMINOPHENYLPERIMIDINES AND DERIVATIVES

Aminophenylperimidines, $C_{17}H_{13}N_3$. Three isomers are described in Beil **25**, 369

o-Amidophenylperimidine Azoimide or 1,2,3-Benzotriazino[3,4-a]perimidine, $C_{17}H_{10}N_4$, mw 270.28, N 20.73%. Dk red solid, mp– expl suddenly at 140°; sol in alc eth, acet, MeOH, chlf, AcOH, benz, ethyl acetate and in hot dil sulfuric acid; insol in w. Was prepd by Sachs & Steiner from o-aminophenylperimidine and Na nitrite in AcOH with cooling

Refs: 1)Beil **26**, 382 2)F.Sachs & M. Steiner, Ber **42**, 3674(1909) 3)J.H.Erickson, et al, "The 1,2,3- and 1,2,4-Triazines, Tetrazines and Pentazines", Interscience, NY (1956), 41

AMINOPHENYLPHOSPHONIC ACIDS AND DERIVATIVES

Aminophenylphosphonic Acids or *Aminobenzenephosphonic Acids*, $C_6H_8NO_3P$. The meta-isomer is described in Beil **16**, 383 & [410] and the para-isomer in Beil **16**, [401]

Aminophenylphosphonic Acid, Azido–, $C_6H_7N_4O_3P$ and *Diazido–*, $C_6H_6N_7O_3P$ *Derivatives* were not found in Beil or CA through 1956

Nitroaminophenylphosphonic Acids, $C_6H_7N_2O_5P$. Two isomers are described in Beil **16**, [401], neither of them expl

Compound $C_6H_6N_3O_6P + 3H_2O$, called by Michaelis "Salpetersäure-Diazophosphenylsäure", prisms, losing $2H_2O$ at 130°, melting with decompn at 188° and explg violently at sl higher temp; very stable; sl sol in eth and easily sol in w & in alc. Was prepd by treating boiling nitric acid soln of m-aminophenylphosphonic acid with nitrous acid. Numerous salts are known

Refs: 1)Beil **16**, 823 2)A.Michaelis & E. Benziger, Ann **188**, 288–92(1877)

Note: No higher nitrated and/or nitrited derivs were found

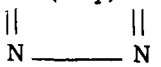
AMINOPHENYLTETRAZOLES AND DERIVATIVES

Aminophenyltetrazoles, $C_7H_7N_5$, mw 161.17, N 43.46%, OB to CO_2 –173.7%, OB to CO –104.2%, are compds with one amino- and one phenyl-group attached directly to the tetrazole ring or compds with the amino-group attached to a phenyl-group which, in turn, is attached to the tetrazole ring

Both types of tetrazoles are high-nitrogen compds and may be of interest as ingredients of prplnts and expls

The following aminophenyltetrazoles and their derivatives were found in the literature:

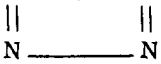
1-Amino-5-phenyl-α-tetrazole; 1-Amino-5-phenyl-1H-tetrazole or **5-Phenyl-1-aminotetrazole,** $C_6H_5 \cdot C{-}N(NH_2){-}N$. Crysts, mp



155°, expl on rapid heating; very sl sol in cold w, easily sol in alc and hot w, nearly insol in eth, insol in acids and alkalies. Was prepd by heating 1-benzalamino-5-phenyltetrazole with HCl(Ref 2). Another method of prepn is given in Ref 3. Some props are given in Refs 4 & 5

Refs: 1)Beil **26**,(113) & [216] 2)R.Stollé & F.Helwerth,Ber **47**,1140(1914) 3)R.Stollé et al,Ber **55**,1294–5 & 1302(1922) 4)F.R. Benson,ChemRevs **41**,16(1947) 5)Ph.Rochlin, D.B.Murphy & S.Helf,JACS **76**,1453(1954)

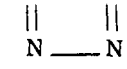
5-Amino-1-phenyl-α-tetrazole, 5-Amino-1-phenyl-1H-tetrazole or **1-Phenyl-5-aminotetrazole** [Called in Beil 1-Phenyl-tetrazolon-(5)-imin], $H_2N \cdot C{-}N(C_6H_5){-}N$. Scales(from



chlf), mp 158–160°; sol in alc & hot w, insol in eth & ligroin. Can be prepd by passing CO_2 through a heated mixt of monophenylthiourea, lead oxide and NaN_3 in alc(Refs 3 & 5). Other methods of prepn are given in Refs 2 & 4

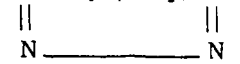
Its *hydrochloride* decomp on heating with evoln of gas

The compd prepd by Garbrecht & Herbst (Ref 6) melted at 163–163.5°, solidified at 165° and then remelted with decompn at 205–6°. When a suspension in xylene of the compd melting at 163–163.5° was refluxed for 2 hrs, the resulting col ndls, melting at 205–6°, proved to be *5-(phenylamino)-tetrazole,* $C_6H_5 \cdot NH \cdot C{-}NH{-}N$ (Ref 7)



Refs: 1)Beil **26**,(124) & [245] 2)E.Oliveri-Mandalà et al,Gazz **43** I,313(1913) 3)R. Stollé et al,Ber **55**,1294(1922) 4)R.Stollé et al,JPrChem **124**,268 & 293–4(1930) 5)Ibid **134**,282–3 & 288–9(1932) 6)W.L. Garbrecht & R.M.Herbst,JOC **18**,1019–20 (1953) & CA **48**,8224–5(1954) 6a)Ibid **18**, 1278(1953) & CA **48**,12092–93(1954) 7)D.B. Murphy & J.P.Picard,JOC **19**,1808,1810–11 (1954) & CA **49**,15879(1955)

1-(p-Aminophenyl)-α-tetrazole or **1-(p-Aminophenyl)-1H-tetrazole,** $HC{-}N(C_6H_4 \cdot NH_2){-}N$.



Ndls(from w), mp 155°. Can be prepd by the reduction of 1-[4'-nitrophenylene]-α-tetrazole with tin chloride and fuming HCl. Heating the product with K permanganate in dil H_2SO_4 gave the tetrazole

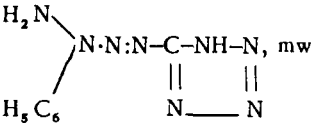
Refs: 1)Beil **26**,347 2)M.Freund & T. Paradies,Ber **34**,3121(1901)

5-Amino-(2-phenyl)-β-tetrazole or **2-Amino-(2-phenyl)-2H-tetrazole** [Called in Beil 2-Phenyl-tetrazolon-(5)-imid], $H_2N{-}C{=}N{-}N \cdot C_6H_5$.

Yellowish crysts(from w), mp 142°, decomp on further heating. Diff sol in cold & hot w, sol in eth, alc, AcOH and concd HCl. Can be prepd by heating 2-phenyl-5-carbethoxyaminotetrazole with concd HCl(at 200°) or concd H_2SO_4(at 105–110°). Its double salt with *silver nitrate* decomp ca 200° and expl mildly when heated in an open flame

Refs: 1)Beil **26**,[246] 2)R.Stollé & O.Orth, Ber **58B**,2103–4(1925)

N¹-Amino-N¹-phenyl-N³-(tetrazolyl-5)-triazene or Diazotetrazolephenylhydrazine, called in Beil 3-Phenyl-1-[tetrazolyl-(5)]-tetrazen-(1) or [Tetrazol-5-diazo]-[α-phenylhydrazid],



$N \cdot N{:}N{-}C{-}NH{-}N$, mw 204.20, N 54.88%,

OB to CO_2 −141.0%. Orange-yel crysts(from methanol+ethanol); decompg ca 139°; nearl

insol in w. Can be prepd by the interaction of phenylhydrazide with 5-diazotetrazole (Refs 1 & 2). It was proposed by Rathsburg for use in compositions for caps, detonators etc. Instead of mixing the ingredients by mechanical means R proposed the prepn of "mixed crystals" by the method described in his patent(Ref 3)

Some of its salts are explosive and may be used in priming and initiating compositions

Refs: 1)Beil **26**,(191) 2)K.A.Hofmann & H.Hock,Ber **44**,2592–3(1911) 3)H.Rathsburg, BritP 201,009(1923) & CA **18**,472(1924)

5-Nitrosamino-1-phenyl-α-tetrazole or **5-Nitrosamino-1-phenyl-1H-tetrazole** [Called in Beil 1-Phenyl-tetrazol-diazonium-hydroxyd-(5)], $(ON)HN{\cdot}C{-}N(C_6H_5){-}N$, mw 190.17, N 44.20%, OB to CO_2 −134.6%. Spongy mass, exploding mildly ca 108°. Easily sol in acet and alc(dec); insol in w, diss in aq Na_2CO_3 from which it may be recovered by acidifying with AcOH(but not with mineral acids). May be prepd by treating 5-amino-1-phenyl-α-tetrazole with an aq soln of $NaNO_2$+HCl

It forms salts, some of which are expl, eg, the *silver salt*, $AgC_7H_5N_6O$, mw 297.06. N 28.3%, wh voluminous ppt obtained by treating the nitroso compd with alc $AgNO_3$, decomp ca 224° and expl mildly when heated rapidly in a sealed glass tube above the mp

Refs: 1)Beil **26**,[350] 2)R.Stollé et al, Ber **55**,1295(1922) 3)R.Stollé et al,JPrChem **134**,291(1932)

1-Amino-5-(p-nitrophenyl)-α-tetrazole or **1-Amino-5-(4'-nitrophenyl)-1H-tetrazole,** $(O_2N{\cdot}H_4C_6){\cdot}C{-}N(NH_2){-}N$, mw 206.17, N 40.77%, OB to CO_2 −116.4%. Long col ndls, mp 154°; moderately sol in hot w & eth, easily sol in alc. Was prepd from p-nitrobenzal-amino-1-p-nitrophenyl-5-tetrazole and concd sulfuric acid as described in Ref 2. Its expl props were not investigated

Refs: 1)Beil – not found 2)R.Stollé et al, JPrChem **138**,2 & 13–14(1933) & CA **27**, 4798(1933)

Note: R.Stollé et al [JPrChem **137**,336(1933) & CA **27**,4233(1933)] obtained among the products of reaction between di-p-nitrobenzo-hydrazide chloride in alc and Na azide, a small quantity of a substance which deflagrated violently ca 149°. It was suggested that this compd was the *p-nitrobenzenyl azide* of *1-amino, 5-p-nitrophenyltetrazole* (See also under Di-p-nitrobenzohydrazide Azide)

5-Amino-1(p-nitrophenyl)-α-tetrazole, 5-Amino-1-(4-nitrophenyl)-1H-tetrazole or **1-(p-Nitrophenyl)-5-aminotetrazole,** $H_2N{\cdot}C{-}N(C_6H_4{\cdot}NO_2){-}N$. Pale yel plates, which on heating in a capillary tube, began to darken at ca 170°, shrank suddenly at ca 176° and melted with frothing at 221–3°. Can be prepd either by nitration of 5-amino-1-phenyltetrazole(Ref 2) or by treating p-nitrophenylcyanamide with hydrazoic acid (Ref 2 & 3)

Refluxing a xylenic suspension of 5-amino-1-(p-nitrophenyl)-tetrazole for 2 hours yielded pale yel ndls melting with decompn at 221–3°. The product proved to be *5-(p-nitrophenylamino) tetrazole,* $O_2N{\cdot}C_6H_4{\cdot}NH{\cdot}C{-}NH{-}N$ (Ref 3)

Refs: 1)Beil – not found 2)W.L.Garbrecht & R.M.Herbst,JOC **18**,1020(1953) & CA **48**,8225 (1954) 3)Ibid,JOC **18**,1280(1953) & CA **48**, 12092–93(1954) 4)D.B.Murphy & J.P.Picard, JOC **19**,1808 & 1810(1954) & CA **49**,15879(1955)

5-Amino-1-(m-nitrophenyl)-α-tetrazole, 5-Amino-1-(3'-nitrophenyl)-1H-tetrazole or **1-(m-Nitrophenyl)-5-aminotetrazole,** $H_2N{\cdot}C{-}N(C_6H_4{\cdot}NO_2){-}N$.

Fine yel ndls, which shrank at ca 170° and melted with decompn at 226.5–228°. Was obtained by treating m-nitrophenylcyanamide with hydrazoic acid(Ref 2)

Refluxing a xylenic suspension of the crystals for 2 hrs effected a thermal rearrangement. The resulting fine pale-yel ndls melted with decompn at 226° and proved to be *5-(m-nitrophenylamino)tetrazole*, $O_2N{\cdot}C_6H_4{\cdot}NH{\cdot}C(=N)-NH-N=N$ (Ref 2)

Refs: 1)Beil – not found 2)W.L.Garbrecht & R.M.Herbst, JOC **18**,1280(1953) & CA **48**, 12093(1954) 3)D.B.Murphy & J.P.Picard, JOC **19**,1808 & 1810(1954) & CA **49**,15879 (1955)

Aminophenyltriazoles, $C_8H_8N_4$, mw 160.18, N 34.98%, are described in Beil **26**, 135, 140 & [76] and in papers appearing after 1929. No expl derivs were found listed in the literature (See also Anilinotriazoles)

AMINOPICOLINES AND DERIVATIVES

Aminopicolines or *Aminomethylpyridines*, $C_6H_8N_2$ are described in Beil **22**,(633) & [342–3]

Azidoaminopicolines, $C_6H_7N_5$ and *Diazidoaminopicolines*, $C_6H_6N_8$ – not found in Beil and CA through 1956

Nitroaminopicolines or *Aminonitropicolines*, $H_3C(C_5N)H_2(NH_2)NO_2$, mw 153.14, N 27.44%. Several isomers are described in Beil **22**, (633) & [342–3] and in the following refs: a)E.D.Parker & W.Shive, JACS **69**,63–7(1947) & CA **41**,2044–5(1947) b)G.R.Lappin & F.B.Slezak, JACS **72**,2806–7(1950) & CA **44**,9966–7(1950) c)S.J.Childress & R.L. McKee, JACS **73**,3504(1951) & CA **46**,5583 (1952) d)H.E.Baumgarten & H.Chien-Fan Su, JACS **74**,3228-31(1952) & CA **47**,5958 (1953)

Nitraminopicolines, $H_3C(C_5N)H_3(NH{\cdot}NO_2)$, mw 153.14, N 27.44%. The following isomers are described in the literature:

α-Nitramino-β-picoline or *3-Nitro-3-methylpyridine*. Lt yel ndls, mp 159° with decompn. Was prepd by nitrating 2-amino-3-methylpyridine with HNO_3(d 1.4) in concd H_2SO_4 in the cold

Refs: 1)Beil **22**,[521] 2)O.Seide, Ber **57**, 1804–5(1924)

α-Nitramino-γ-picoline or *2-Nitramino-4-methyl-pyridine*. Lt yel prisms, mp 182° with decompn. Was prepd by nitrating 2-amino-4-methyl-pyridine with HNO_3(d 1.4) in concd H_2SO_4 in the cold

Refs: 1)Beil **22**,[521] 2)O.Seide, Ber **57**, 794(1924)

α-Nitramino-δ-picoline or *2-Nitramino-5-methyl-pyridine*. Lt yel ndls, mp 183–183.5° (decomp). Was prepd by nitrating 2-amino-5-methylpyridine with HNO_3 and H_2SO_4 (Refs 2 & 3)

Refs: 1)Beil – not found 2)S.J.Childress & R.L.McKee, JACS **73**,3504(1951) & CA **46**, 5583(1952) 3)L.A.R.Hall & C.A.van der Werf, JACS **73**,4466(1951) & CA **47**,136(1952)

Note: No higher nitrated aminopicolines were found in Beil or CA through 1956

AMINOPICOLINIC ACIDS AND DERIVATIVES

Aminopicolinic Acid or *Aminopyridinecarboxylic Acid*(Amino-pyridin-carbonsäure, in Ger), $H_2N(C_5NH_3)COOH$. Aminopicolinic acids are aminopyridine carboxylic acids in which the carboxyl group is attached in position 2 (next to the nuclear N). Two isomers, 3-amino- and 4-amino- are described in Beil **22**,541 & [463]. If the carboxyl is attached to position 3 of aminopyridinic acid, the compd is called *aminonicotinic acid*(qv)

Azidoaminopicolinic Acid, $C_6H_5N_5O_2$ and *Diazidoaminopicolinic Acids*, $C_6H_4N_8O_2$ – not found in Beil or CA through 1956

Nitroaminopicolinic Acid, $C_6H_5N_3O_4$ – not found in Beil or CA through 1956

3-Nitraminopicolinic Acid, $O_2N{\cdot}HN(C_5NH_3){\cdot}COOH$, mw 183.12, N 22.95%. Crysts, mp 178–80° with decompn. Was prepd by cautiously adding nitric acid(d 1.4) to 3-aminopicolinic acid in concd sulfuric acid at a temp not above 5°, allowing to stand for

20 mins and pouring the mixt onto ice. After neutralizing most of the acidity, the ppt was washed with w and dried

An aq NaOH soln of 3-nitraminopicolinic acid, acidified with AcOH precipitated the *sodium salt*, $C_6H_4N_3O_4Na$, which decomp explosively at 218°

Refs: 1)Beil – not found 2)S.Carboni & G.Berti,Gazz **84**,683(1954) & CA **50**,991(1956)

Nitronitraminopicolinic Acid, $C_6H_4N_4O_6$ and *Dinitroaminopicolinic Acid*, $C_6H_4N_4O_6$ – not found in Beil or CA through 1956

Aminopicric Acid. See 3-Amino-2,4,6-trinitro-phenol under Aminophenols

AMINOPROPANES AND DERIVATIVES

Aminopropanes or *Propylamines*, $C_3H_7{\cdot}NH_2$, mw 59.11, N 23.70% are described in Beil **4**,136,152,(360,368) & [619,629]

Aminopropane Salts. Some salts of aminopropane containing phosophoric groups are explosive, eg, the *perchlorate of 1-aminopropane*, $CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2{+}HClO_4$, solid, expl on heating to 290° in a reaction tube

Refs: 1)Beil **4**,360 2)R.L.Datta & N.R. Chatterjee,JCS **115**,1008(1919)

Azidoaminopropanes or **Azidopropylamines**, $C_3H_6N_4$, mw 100.13, N 55.96%. The following isomers are described in the literature:

2-Azido-1-aminopropane or **β-Azidopropylamine**, $CH_3{\cdot}CH(N_3){\cdot}CH_2{\cdot}NH_2$. Col liq, bp 44–6° at 16 mm. Was prepd by heating β-bromopropylaminohydrobromide with Na azide in w

Refs: 1)Beil 4,(368) 2)M.O.Forster & J.C.Withers,JCS **101**,493(1912)

3-Azido-1-aminopropane or **γ-Azidopropylamine**, $N_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2$. Col liq, bp 56–7° at 16 mm, d 1.0043 at 25/4°, n_D 1.4615 at 25°; miscible with w, alc or eth; volatile with steam. Was prepd by prolonged heating of γ-bromopropylamino-hydrobromide with Na azide in w. Its *picrate* melts at 96°

Refs: 1)Beil **4**,(368) 2)M.O.Forster & J.C.Withers,JCS **101**,491(1912)

Diazidoaminopropanes, $C_3H_7N_7$ – not found in Beil or CA through 1956

Mononitroaminopropanes, and *Nitraminopropanes*, $C_3H_8N_2O_2$, mw 104.11, N 26.91. The following isomers were found in the literature:

1-Nitraminopropane; Propylnitramine or **N-Nitropropylamine**, $H_3C{\cdot}CH_2{\cdot}CH_2{\cdot}NHNO_2$. Col liq, frp –21° to –23°, bp 128–9° at 40 mm, d 1.1046 at 15°, sl sol in w, misc with alc & eth. Prepn is described in Refs 1 & 2. Forms solid salts, some of which are explosive, eg, the *potassium* salt, $KC_3H_7N_2O_2$, scales expldg mildly on heating and the *silver* salt, $AgC_3H_7N_2O_2$, ndls, detonating strongly on heating

Refs: 1)Beil **4**,570 & (569) 2) Simon Thomas,Rec **9**,75–7(1890)

2-(or β-)Nitraminopropane; Isopropylnitramine or **N-Nitroisopropylamine**, $(CH_3)_2CH{\cdot}NH{\cdot}NO_2$, liq, frp –4°, bp 90–1° at 10 mm, d 1.098 at 15°. Forms salts, some of which are expl, eg, the *potassium* salt, $KC_3H_7N_2O_2$, ndls, expl on heating and the *silver* salt, $AgC_3H_7N_2O_2$, plates, detong on heating

Refs: 1)Beil **4**,571 2)Simon Thomas,Rec **9**, 77–9(1890)

Dinotroaminopropanes and **Nitronitraminopropanes**, $C_3H_7N_3O_4$, mw 149.11, N 28.18%, OB to CO_2 -59.0%. The following isomer was found in the literature:

1,1-Dinitro-2-aminopropane; α,α-Dinitro-β-aminopropane or **β,β-Dinitroisopropylamine**, $(O_2N)_2CH{\cdot}CH(CH_3){\cdot}NH_2$. Yel crysts, mp decomp ca 120°. Can be prepd from acetaldehyde-ammonia, $CH_3CH(OH)NH_2$ and dinitromethane

Refs: 1)Beil **4**,156 & [631] 2)P.Duden et al, Ber **38**,2038(1905)

Note: No higher nitrated aminopropanes were found in Beil or in CA through 1956

AMINOPROPANEDIOLS AND DERIVATIVES

Aminopropanediols, Aminopropyleneglycols (Aminodihydroxypropanes or Dihydroxypropylamines) (Called Aminodioxypropan or Dioxypropylamin, in Ger), $H_2N{\cdot}C_3H_5(OH)_2$, mw 91.11, N 15.37%. The following isomers are described in the literature:

1-Amino-2,3-propanediol or **3-Amino-1,2-propanediol** *(Glycerol-α-monoamine, 1-Amino-2,3-hydroxypropane, 1-Aminopropane-2,3-diol* or *γ-Aminopropylene-glycol)* (β,γ-Dioxypropylamin, in Ger), $H_2N{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2(OH)$. Viscous oil, d 1.1752 at 20°/4°, bp 264° at 739 mm(with sl decompn), n_D 1.49 at 10°; readily sol in w & alc, insol in eth or benz. Can be prepd by mixing glycidol, $CH_2{\cdot}O{\cdot}CH{\cdot}CH_2OH$, with NH_3(Ref 2) or by other methods (Refs 1 & 3)

Refs: 1)Beil **4**,301,(447) & [753] 2)L.Knorr & E.Knorr,Ber **32**,752–4(1899) 3)H.P.den Otter,Rec **57**,18–20(1938) & CA **32**,3354(1938)

2-Amino-1,3-propanediol *(Glycerol-β-monoamine, β-Aminotrimethyleneglycol, 2-Amino-1,2-dihydroxypropane* or *2-Aminopropane-1,3-diol)* (β,β'-Dioxyisopropylamin, in Ger), $HO{\cdot}CH_2{\cdot}CH(NH_2){\cdot}CH_2{\cdot}OH$. Extremely hygroscopic syrup. Can be prepd by the reduction of 2-nitro-1,3-propanediol(Ref 3) or by other methods

Refs: 1)Beil **4**,303 & (448) 2)E.Schmidt & G.Wilkendorf,Ber **52**,398(1919) 3)H.P. den Otter,Rec **57**,13–16(1938)

Azidoaminopropanediols, $C_3H_8N_4O_2$ and *Diazidoaminopropanediols,* $C_3H_7N_7O_2$ – not found in Beil or CA tnrough 1956

1-Amino-2,3-propanediol Dinitrate; 1-Amino-2,3-dinitroxypropane or **3-Amino-1,2-propanediol Dinitrate,** $H_2N{\cdot}CH_2{\cdot}CH(ONO_2){\cdot}CH_2(ONO_2)$, mw 181.11, N 23.20%. Its *nitrate salt,* $C_3H_7N_3O_6 + HNO_3$, crysts(from BuOH), mp 90°. Was prepd by Barbière (Ref 2) by nitrating the nitrate salt of 3-amino-1,2-propanediol with 97% HNO_3 at –5° removing the excess HNO_3 in vacuo, pptg the reaction product with ether at –10° and recrystg the final product from BuOH

Refs: 1)Beil – not found 2)J.Barbière, BullFr **11**,470(1944) & CA **40**,2111(1946)

Note: No other nitrated derivs were found in Beil or CA through 1956

Aminopropanediol Derivatives of den Otter. Several derivs were obtained by H.den Otter (See Ref 2 at the end of this section) by coupling aminopropanediols with aromatic nitro- or halogen-nitro compounds. On nitration of these derivs, several compds were obtained

Coupling reactions were achieved by boiling alcoholic solns of aminopropanediols under reflux with the necessary quantities of nitro/or halogen-nitro compds and CH_3COONa for several hrs on a water bath, filtering off the NaCl formed and evapg the soln obtained under vacuum(Ref 2,pp 15–16). Nitrations of the resulting compds were carried out by dissolving about 1g of the substance in 5–6 ml of fuming HNO_3(cooled in an ice-salt mixt) and allowing the soln to stand for 1hr. The container with the soln was then placed in an ice-salt mixt and water added dropwise in order to ppt the nitrated product. The resulting substance was purified either by recrystallizing it or by extracting it with a suitable solvent

Among the numerous products listed in Tables 1–4 of Ref 2, the following have expl properties comparable to those of a HE, such as tetryl:

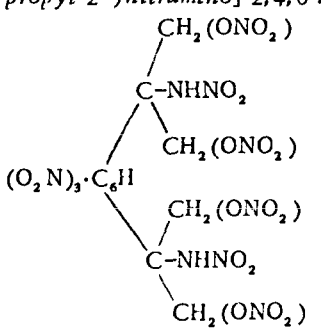
2-(2',4',6'-Trinitrophenyl-nitramino)-1,3-dinitroxy-propane or 2-(2',4',6'-Trinitrophenylnitramino)-1,3-propanediol Dinitrate, $(O_2NO)CH_2{\cdot}C[NH(NO_2)][C_6H_2(NO_2)_3]{\cdot}CH_2(ONO_2)$, mw 437.20, N 22.43%. Lt yel solid, mp 142–3° (with decompn); insol in w, alc & eth, sol in

acet, NB, AcOH, etc. Expl on heating or on impact(Ref 2, listed at the end of this section)

2-(2',4'-6'-Trinitro-3-chlorophenyl-nitramino)-1,3-dinitroxy-propane or 2-(2'-4'-6'-Trinitro-3-chlorophenylnitramino)-1,3-propanediol Dinitrate, $(O_2NO)CH_2 \cdot C[NH(NO_2)][C_6H(Cl)(NO_2)_3] \cdot CH_2(ONO_2)$, mw 471.65, N 20.79%. Lt yel sol, mp softens ca 40° becoming resinous; insol in w, alc & eth, sol in acet, NB, AcOH. Expl on heating or on impact(Ref 2)

2-(2',4'-Dinitronaphthylnitramino)-1,3-dinitroxypropane or 2-(2',4'-Dinitronaphthylnitramino)-1,3-propanediol Dinitrate, $(O_2NO)CH_2 \cdot C\,NH(NO_2)\,C_{10}H_5(NO_2)_2 \cdot CH_2(ONO_2)$, mw 442.26, N 19.00%. Yel crysts, mp 117° (softening at a lower temp); insol in w, eth & chlf, sol in alc, acet, AcOH & benz. Expl on heating or on impact(Ref 2)

1',3'-Bis-(1,3-Dinitroxy-2-nitramino-propane)-2',4' 6'-trinitrobenzene {called by den Otter, *Tetranitrate of 1,3-Bis-[(1',3'-dihydroxypropyl-2')nitramino]-2,4,6-trinitrobenzene*}



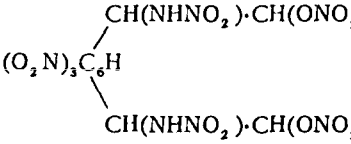
mw 66.30, N 23.30%. Lt yel crysts, mp–turns brn at 50–60° and then decomps; insol in w, chlf, benz, sl sol in alc & eth, sol in acet, methanol, AcOH, NB(Ref 2)

3-(2',4',6'-Trinitrophenyl-nitramino)-1,2-dinitroxy-propane or 3-(2',4',6'-Trinitrophenylnitramino)-1,2-propanediol Dinitrate, $(O_2NO)CH_2 \cdot CH(ONO_2) \cdot CH \cdot [NH(NO_2)][C_6H_2(NO_2)_3]$, mw 437.20, N 22.43%. Lt yel crysts, mp–softens ca 67° and decomps ca 80°, insol in w, pet eth & CCl_4, sol in alc, eth, acet, chlf, etc. Expl on heating or on impact(Ref 2)

3-(2',4',6'-Trinitro-3'-chlorophenyl-nitramino)-1,2-dinitroxy-propane or 3-(2',4',6'-Trinitro-3'-chlorophenyl-nitramino)-1,2-propanediol Dinitrate, $(O_2NO)CH_2 \cdot CH(ONO_2) \cdot CH[NH(NO_2)][C_6HCl(NO_2)_3]$, mw 471.65, N 20.79%. Lt yel visc fluid at 50°; insol in w, pet eth, chlf, CCl_4, sol in alc, acet, benz, NB and AcOH. Expl on heating or on impact (Ref 2)

3-(2',4'-Dinitronaphthyl-nitramino)-1,2-dinitroxypropane or 3-(2',4'-Dinitronaphthyl-nitramino-1,3-propanediol Dinitrate, $(O_2NO)CH_2 \cdot CH(ONO_2) \cdot CH[NH(NO_2)][C_{10}H_5(NO_2)_2]$, mw 442.26, N 19.00%. Lt yel crysts, changes to a viscous liq ca 73° and decompg ca 80°; insol in w, chlf, CCl_4, pet eth, sol in alc, eth, acet, benz, NB. Expl on heating or impact(Ref 2)

1',3'-Bis-(2,3-Dinitroxy-1-nitramino-propane)-2',4',6'-trinitrobenzene {called by den Otter, *Tetranitrate of 3-Bis[(2',3'-Dihydroxypropyl-1')-nitramino]-2,4,6-trinitrobenzene*},



mw 661.30, N 23.30%. Lt yel solid softening ca 60° and decompg ca 73°; insol in w, chlf, CCl_4; sol in alc, eth, acet, AcOH, benz & NB. Expl on heating or on impact(Ref 2)

Refs: 1)Beil - not found 2)H.P. den Otter, Rec **57**,23–24(1938) & CA **32**,3354(1938)

AMINOPROPANOIC ACIDS AND DERIVATIVES

Aminopropanoic Acids, Aminopropionic Acids or *Alanines*, $H_2N \cdot C_2H_4COOH$. Several isomers are described in Beil **4**,381,401 & [809,827]

Azidoaminopropanoic Acid, $C_3H_6N_4O_2$ and *Diazidoaminopropanoic Acid*, $C_3H_5N_7O_2$ – not found in Beil or CA through 1956

3-Nitraminopropanoic Acid, $O_2N \cdot HN \cdot CH_2 \cdot CH_2 \cdot COOH$, mw 134.09, N 20.89%. Ndls, mp 73°. Was obtained by treating its amide,

$O_2N{\cdot}HN{\cdot}CH_2{\cdot}CO{\cdot}NH_2$, with NaOH soln. Its *barium* and *silver* salts are expl

Refs: 1)Beil **4**,576 2)A.P.N.Franchimont & H.Friedmann,Rec **26**,220–2(1907)

Note: No higher nitrated aminopropanoic acids were found in Beil or in CA through 1956

AMINOPROPANOLS AND DERIVATIVES

(Propanolamines and Derivatives)

Aminopropanols or *Aminopropyl Alcohols* (Aminohydroxypropanes or Hydroxyaminopropanes), $C_3H_6(NH_2)OH$, mw 75.11, N 18.65%. Three isomers are described in Beil **4**,288, 289,(432,433,437) & [733,734,736]. Their picrates are listed under Picric Acid

Azidoaminopropanols, $C_3H_6N_4O$ and *Diazidoaminopropanols*, $C_3H_7N_7O$ – not found in Beil or CA through 1956

Aminopropanol Nitrate, $C_3H_8N_2O_3$ – not found in Beil or CA through 1956

Nitroaminopropanol, $C_3H_6N_2O_3$ and *Nitraminopropanol*, $C_3H_6N_2O_3$ – not found in Beil or CA through 1956

3-Amino-2-nitroxypropane Nitrate or **Aminoisopropanol Dinitrate**, $H_2N{\cdot}CH_2{\cdot}CH(ONO_2){\cdot}CH_3 + HNO_3$, mw 183.13, N 22.94%. Crysts from abs alc, mp 94°. Was prepd by Barbière (Ref 2) by treating 3-amino-2-propanol nitrate with concd HNO_3 at –5°, removing the excess HNO_3 in vacuo, pptg the reaction product with eth at –10° and recrystallizing it from abs alcohol

Refs: 1)Beil **4** – not found 2)J.Barbière, BullFr **11**,470–80(1944) & CA **40**,2110–11 (1946)

Nitroaminopropanol Nitrate, $C_3H_7N_3O_5$; *Nitraminopropanol Nitrate*, $C_3H_7N_3O_5$ and *Dinitroaminopropanol*, $C_3H_7N_3O_5$ – not found in Beil or CA through 1956

Nitraminopropanol Nitrate, 1-Nitramino-2-nitroxy-propane, 1-Nitramino-2-propanol Nitrate or **N-(β-Nitroxypropyl)nitramine** called by Blomquist & Fiedorek **Iso-Me-NENA**, $O_2N{\cdot}HN{\cdot}CH_2{\cdot}CH(ONO_2){\cdot}CH_3$, mw 165.11, N 25.45%, OB to CO_2 –43.6%, OB to CO –14.5%. Col crysts, mp 86–7°. Was prepd, starting with 1-amino-2-propanol and ethylchlorocarbonate, by the following series of reactions:

$$H_2N{\cdot}CH_2{\cdot}CH(OH)CH_3 \xrightarrow[\text{followed by aq NaOH}]{C_2H_5COOCl}$$

$(C_2H_5COO){\cdot}HN{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_3$
N–(2-propanol)urethane

$$\xrightarrow[\text{to 98\% } HNO_3 \text{ at } 10^\circ]{\text{Urethane added dropwise}} (C_2H_5COO)N(NO_2){\cdot}-CH_2{\cdot}CH(ONO_2){\cdot}CH_3$$

$$\xrightarrow[\text{soln of the nitrate}]{NH_3 \text{ added to the ethereal}} \begin{pmatrix} NH_2^+ \\ NO_3^- \end{pmatrix} {\cdot}CH_2{\cdot}-CH(ONO_2){\cdot}CH_3$$

$$\xrightarrow[\text{after } NH_3]{\text{HCl added immediately}} O_2N{\cdot}HN{\cdot}CH_2{\cdot}-CH(ONO_2){\cdot}CH_3$$

Details of the method of prepn are given in Refs 2 & 3

Iso-Me-NENA was proposed as a possible plasticizer for NC in prepn of prplnts and as an ingredient of expl compns

Refs: 1)Beil – not found 2)M.T.Blomquist & F.T.Fiedorek,OSRD Rept 4134 or PBRept 18867(1944),45–7 3)Ibid,USP 2,485,855(1949), 5–6 & 12–13; CA **44**,3516–17(1950)

AMINOPROPENES AND DERIVATIVES

3-Amino-1-propene, γ-Amino-α-propylene or **Allylamine**, $CH_2{:}CH{\cdot}CH_2{\cdot}NH_2$, mw 57.09, N 24.53%. Col liq, d 0.761 at 22°/4°, bp 56.5° at 756 mm; misc with w, alc & eth. Prepn in Ref 3 and props in Ref 1

Its *perchlorate* was reported to expl ca 262° and the *picrate* at 270° (Ref 2)

Refs: 1)Beil **4**,205,(389) & [662] 2)R.L. Datta & N.R.Chatterjee,JCS **115**,1007–8(1919) 3)OrgSynth,Coll Vol 2(1943),24

Note: R.Levy,MP **32**,309–12(1950) prepd several solid compds as possible stabilizers in smokeless propellants. One of these compds(p 312),$(C_6H_5)_2{:}N{\cdot}CO{-}NH{\cdot}CH_2{\cdot}CH{:}CH_2$, mp 82°, was prepd by refluxing for 6 hrs allylamine with $(CH_3)_2{:}N{\cdot}CO{\cdot}Cl$

Azidoaminopropene, $C_3H_6N_4$ – not found in Beil or CA through 1956

3-Nitramino-1-propene or *Allylnitramine*, CH_2:CH·CH_2·NH(NO_2), or other nitrated derivs of aminopropene were not found in Beil or CA through 1956

Note: E.E.Lewis & M.A.Naylor, JACS **69**, 1968 (1947) prepd *allylaminopicrate* as yel crysts, mp 141–141.5°. A.J.Restaino et al, JACS **78**, 2940(1956) prepd the compd with mp 144°. Both compds seem to be identical with the picrate listed on p A253(explodes at 270°)

Aminopropoxyaminoimidazolidine, Nitrated. See 1-Nitro-2-n-propoxy-2-nitraminoimidazolidine under Aminoimidazoline and Aminoimidazolidine, Substituted Derivatives

Aminopropylaminoimidazolidine, Nitrated. See 1-Nitro-2-propylamino-2-nitraminoimidazolidine and 1-Nitro-2-propylamino-2-imidazoline Nitrate under Aminoimidazoline and Aminoimidazolidine, Substituted Derivatives

γ-Aminopropyleneglycol. See 1-Amino-2,3-propanediol under Aminopropanediols

Aminopseudocumenes or Amino-1,2,4-trimethylbenzenes, $(CH_3)_3C_6H_2$·NH_2. Several isomers are known of which the *5-aminopseudocumene* is the most important. Its mono- and dinitro- compds are known but they are not expl. Azido- and diazido- derivs were not found in Beil or CA through 1956

Some of its salts are expl, for example, that of 2,4,6-trinitro-m-cresol, explg ca 477°(Ref 2)

Refs: 1)Beil **12**, 1150–3, 1159,(498–9,502) & [629–31] 2)R.L.Datta et al, JACS **45**, 2432(1923)

AMINOPURINES AND DERIVATIVES

Aminopurines or *Iminodihydropurines*, $C_5H_5N_5$, mw 135.13, N 51.83%, are compds of higher nitrogen content and may be of interest as gas producing components of expls and prplnts. The following isomers are described in Beil:

2-Aminopurine, 2-Imino-2,3-dihydropurine or **Isodenine**, $C_5H_5N_5 + H_2O$, ndls. Can be prepd by heating 2,4-diamino-5-formaminopyrimidine or by other methods

Refs: 1)Beil **26**,414 2)O.Isay, Ber **39**, 264 (1906)

6-Aminopurine, 6-imino-1,6-dihydropurine or **Adenine**, $C_5H_5N_5 + 3H_2O$, wh ndls or leaflets loosing w at 110°, mp 360.5° with decompn on rapid heating in a capillary tube. Can be derived from nucleic acid. Numerous methods for its prpn are listed in Ref 1. Its Q_c^v is 663.74 kcal/mol

Refs: 1) Beil **26**,420,(126) & [252] 2)P.A. Levene & L.W.Bass, "Nucleic Acids," NY (1931),95 3)J.Baddiley et al, JCS **1943**, 386–7 & CA **37**,6667(1943) (Synthesis of adenine) 4)CA – see under Adenine

Azidoaminopurine, $C_5H_4N_8$; *Nitroaminopurine*, $C_5H_4N_6O_2$ and *Nitraminopurine*, $C_5H_4N_6O_2$ – not found in Beil or CA through 1956

AMINOPYRIDINES AND DERIVATIVES

Aminopyridines or *Pyridylamines* (formerly called *Pyridonimides*), $C_5H_6N_2$, mw 94.11, N 29.77%. Several isomers are listed in Beil **22**,428,431,433,(629,633) & [322,339, 340]

Azidoaminopyridine, $C_5H_5N_5$ – not found in Beil or CA through 1956

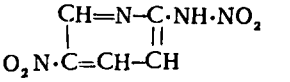
Note: E.Koenigs et al, Ber **57B**, 1172–8 & 1179–87 (1924) discussed diazotization and nitration of 4-aminopyridine, as well as the prepn of salts, such as *picrate*, yel ndls, mp 215–16°(p 1175)

Mononitroaminopyridines and **Nitraminopyridines**, $C_5H_5N_3O_2$, mw 139.11, N 30.21%, are listed in Beil **22**,(631,702) & [335–6, 465, 519–21]. Prepn of pure compds is described by L.N.Pino and W.S.Zehrung, JACS **77**, 3154–5(1955) & CA **50**, 3435(1956). Although these compds are not expl some of their salts and derivs are mild expls, eg, the *sodium* salt of **2-nitraminopyridine** was reported to defgr on heating. Methylation of 2-nitraminopyridine with Me_2SO_4 gave an expl compd, $C_6H_7N_3O_2$, mw 153.14, N 27.44%, darkens at 185°, melts at 189 and defgr at higher temps

Refs: 1)Beil – not found 2)A.E.Chichibabin & A.V.Kirsanov, Ber **60**, 2433–8(1927) & CA **22**, 961(1928)

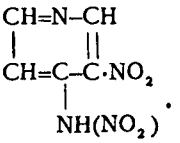
Nitronitraminopyridines, $C_5H_4N_4O_4$, mw 184.11, N 30.43%. The following derivs are described in the literature:

5-Nitro-2-nitraminopyridine,



Plates or ndls(from w), mp decomp on heating. Its *sodium* salt was reported to be a mild expl

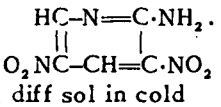
Refs: 1)Beil **22**,(703) & [522] 2)A.E.Chichibabin et al, JRusPhChemSoc **47**,1292(1915) Ibid **60**,978–80(1928) and Ber **58**,1708,1715 (1926) & **61**,1223,1232(1928)

3-Nitro-4-nitraminopyridine,  .

Dk yel ndls, mp–decomp at 202° and defgr on rapid heating. Can be prepd by nitration of 4-aminopyridine or 4-nitraminopyridine. Its *potassium* salt was reported to expl on heating, more violently than the basic compd

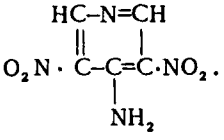
Refs: 1)Beil **22**,[521] 2)E.Koenigs et al, Ber **57**,1183–4(1924)

Dinitroaminopyridines, $C_5H_4N_4O_4$, mw 184.11, N 30.43%. The following isomers are listed in the litertaure:

3,5-Dinitro-2-aminopyridine,  .

Yel ndls(from w), mp 190.2°, diff sol in cold w, easily sol in inorg acids, insol in alkalies. Can be prepd by treating 5-nitro-2-aminopyridine or 3-nitro-2-aminopyridine with concd H_2SO_4

Refs: 1)Beil **22**(632) & [338] 2)A.E.Chichibabin et al, JRussPhChemSoc **47**,1293 (1915) & **60**,980(1928) 3)C.Räth,Ber **58B**, 346(1925) 4)A.E.Chichibabin et al,Ber **58B**, 1707–8(1925)

3,5-Dinitro-4-aminopyridine,  .

Yel ndls(from w), mp 170–1°, sl sol in alc & hot w, easily sol in inorg acids, dissolves in hot alkalies with decomp. Can be prepd by treating 4-aminopyridine with HNO_3+H_2SO_4 as indicated in Ref 2. Forms salts, some of which are expl, eg, the *picrate*

Refs: 1)Beil **22**,[342] 2)E.Koenigs et al, Ber **57**,1184(1924)

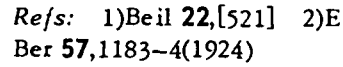
Note: No higher nitrated derivs of aminopyridine were found in Beil or CA through 1956

Aminopyridinecarboxylic Acid. See Aminonicotinic Acid

AMINOPYRIDYLPYRAZOLE AND DERIVATIVES

3-Amino-5-pyridyl-pyrazole or *3-Amino-5-pyridyl-1,2-diazole*, $C_8H_8N_4$, mw 160.18, N 34.98%, may be considered as the parent compd of the following:

Azidoaminopyridylpyrazole, $C_8H_7N_7$ – not found in Beil or CA through 1956

4-Nitro-3-amino-5-pyridyl-pyrazole,  , mw 205.18, N 34.12%.

Ndls. Was prepd by treating an alkaline soln of 3,4-dinitro-5-pyridyl -pyrazole with H_2S at 90°

When the nitroaminopyridylpyrazole was diazotized in 4 mols of 4N nitric acid without cooling, a clear soln was obtained which, on cooling in ice water, deposited crystals of the *diazonium nitrate*, $C_8H_5N_7O_5$, N 34.1%

The *diazonium perchlorate*, $C_8H_5N_6O_6Cl$, mw 316.63, N 26.54%, was obtained by diazotizing nitroaminopyridylpyrazole in 4N hydrochloric acid soln, filtering the lukewarm soln and adding 70% perchloric acid. The separated crysts of the perchlorate were dried and ground in a mortar without producing an expln. The substance, however, expl with great violence when struck with a hammer or when heated to 160°

Refs: 1)Beil – not found 2)H.Lund,JCS **1935**,418–19 & CA **29**,4359(1935)

Note: No other nitrated derivs of 3-amino-5-pyridyl-pyrazole were found in Beil or CA through 195

AMINOQUINOLINE AND DERIVATIVES

Aminoquinoline (Amino-chinolin, in Ger), $(C_9H_6N)NH_2$, mw 144.17, N 19.43%. Several isomers are described in Beil **22**,443–5, 447,450,(637–640) & [350,352–3,355–6]

Note: Nitration of 4-aminoquinoline was investigated by J.C.E.Simpson & P.H.Wright, JCS **1948**,2023–4 & CA **43**,3002(1949)

Azidoaminoquinoline, $C_9H_7N_5$ and *Diazidoaminoquinoline*, $C_9H_6N_8$ – not found in Beil or CA through 1956

Nitraminoquinolines, $(C_9H_6N)NH{\cdot}NO_2$, mw 189.17, N 22.21%. Two isomers are described in Beil **22**,593 & [522]

Nitroaminoquinolines, $(C_9H_5N)\begin{matrix}NH_2\\ \diagup\\ \diagdown\\ NO_2\end{matrix}$, mw 189.17, N 22.21%. Several isomers are described in Beil **22**,445,452,(637,639) & [352,354,357]

Nitronitraminoquinolines, $(C_9H_5N)\begin{matrix}NH{\cdot}NO_2\\ \diagup\\ \diagdown\\ NO_2\end{matrix}$, mw 234.17. N 23.93%. The following isomer is described in the literature:

6-Nitro-4-nitraminoquinoline. Golden yel ndls decompg ca 203°(Ref 2), orange-yel ndls decompg without melting at 216°(Ref 3). Was obtained by adding mixed $HNO_3 + H_2SO_4$ to 4-aminoquinoline dissolved in H_2SO_4 (Ref 2). Can also be obtained by adding concd HNO_3 to 6-nitro-4-aminoquinoline dissolved in concd H_2SO_4 at −15 to −20°

Refs: 1)Beil **22**,593 2)A.Claus & W. Frobenius, J PrChem **56**,197(1897) 3)J.C.E. Simpson & P.H.Wright, JCS **1948**,2024 & CA **43**,3002(1949)

Dinitroaminoquinolines, $(C_9H_4N)\begin{matrix}NH_2\\ \diagup\\ \diagdown\\ (NO_2)_2\end{matrix}$, mw 234.17, N 23.93%. The following isomers are described in the literature:

5,7-Dinitro-8-aminoquinoline. Lt yel ndls, mp 187–8°. Can be prepd by warming 5,7-dinitro-quinolinecarbamic acid with aq H_2SO_4 on a water bath, as described in Ref 2, or by other methods(Ref 1)

Refs: 1)Beil **22**,452,(640) & [358] 2)R.P. Dikshoorn, Rec **48**,525(1929)

6,8-Dinitro-5-aminoquinoline. Dark yel ndls decompg ca 275°. Can be prepd by heating a mixture of methyl-(or ethyl)-6,8-dinitroquinoline carbamate with concd H_2CO_4 at 105°(Refs 1 & 2)

Refs: 1)Beil **22**,[354] 2)R.P.Dikshoorn, Rec **48**,244(1929)

3(?),6-Dinitro-4-aminoquinoline. Pale yel ndls, mp 282–3°. Was prepd by adding HNO_3 (d 1.40) to a stirred soln of 4-aminoquinoline dissolved in concd H_2SO_4 at −7° to +2°

Refs: 1)Beil – not found 2)J.C.E.Simpson & P.H.Wright, JCS **1948**,2023–4

Note: No higher nitrated derivs of aminoquinoline were found in Beil or CA through 1956, but some of them probably can be prepd

Amino Resins and **Plastics.** The commercially important amino resins are the *urea-formaldehyde* and the *melamine-formaldehyde* condensates. Of lesser importance are the sulfonamide, aniline and thiourea resins

Ref: Kirk & Othmer **1**(1947),741–771(84 refs)

Note: Amino resins and plastics, as well as other resins and plastics, are finding more and more extensive use in ordnance

AMINORESORCINOLS AND DERIVATIVES

Aminorescorcinols, $H_2N{\cdot}C_6H_3(OH)_2$, mw 125.12, N 11.20%. Three isomers are described in Beil **13**,782–3,787 & [468–9]

Azidoaminoresorcinols, $C_6H_6N_4O_2$ and *Diazidoaminoresorcinols*, $C_6H_5N_7O_2$ – not found in Beil or CA through 1956

Mononitroaminoresorcinols, $H_2N{\cdot}C_6H_2(OH)_2NO_2$, mw 170.12, N 16.47%. Two isomers are described in Beil **13**,783,(315)

Nitraminoresorcinols, $O_2NHN{\cdot}C_6H_3(OH)_2$ – not found in Beil or CA through 1956

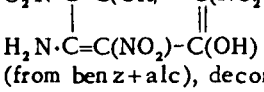
Dinitroaminoresorcinols, $H_2N{\cdot}C_6H(OH)_2(NO_2)_2$, mw 215.12, N 19.54%. One isomer, the

4,6-dinitro-2-aminoresorcinol, is described in Beil **13**,(783)

Dinitronitraminoresorcinols, $O_2NHN{\cdot}C_6H(OH)_2(NO_2)_2$ – were not found in Beil or CA through 1956

Trinitroaminoresorcinols, $H_2N{\cdot}C_6(OH)_2(NO_2)_3$, mw 260.12, N 21.54%, OB to CO_2 -36.9%, OB to CO 0.0%. The following isomer was found in the literature:

2,4,6-Trinitro-5-aminoresorcinol,



Dark yel crysts (from benz+alc), decompg at 236–7°. Was prepd by treating pentanitroaniline(free from benz) with CH_3COONa or Na_2CO_3 in aq acet. Its expl props were not investigated

Refs: 1)Beil **13**,[474] 2)B.Flürscheim & E.L.Holmes,JCS **1928**,3044 & CA **23**,823 (1929)

Note: No higher nitrated derivatives of aminoresorcinols were found in Beil or CA through 1956

AMINOSALICYLIC ACID AND DERIVATIVES

Aminosalicylic Acids or *Aminohydroxybenzoic Acids*, $C_7H_4O_2(OH){\cdot}NH_2$ are described in Beil **14**,577,579,(649,650) & [350,352]

Azidoaminosalicylic Acid, $C_7H_6N_4O_3$ and *Diazidoaminosalicylic Acid*, $C_7H_5N_7O_3$ – not found in Beil or CA through 1956

Nitroaminosalicylic Acids, $C_7H_6N_2O_5$. Two isomers are described in Beil **14**, 579,586, (649) & [350,354]

Note: No higher nitrated and/or nitrited derivs were found in Beil or CA through 1956

AMINOSTYRENES AND DERIVATIVES

Aminostyrenes, Vinylanilines or *Aminovinylbenzenes*, $CH_2{:}CH{\cdot}C_6H_4{\cdot}NH_2$, mw 119.16, N 11.76%. Three isomers, 2-, 3- and 4-, are described in Beil **12**,1187 & [648]

Azidoaminostyrene, $C_8H_8N_4$ and *Diazidoaminostyrene*, $C_8H_7N_7$ – not found in Beil or CA through 1956

ω-Aminostyrene or *Styrylamine*, $C_6H_5{\cdot}CH{:}CH{\cdot}NH_2$, is listed in Beil **12**,1188 & [648] but no description of its props and no method of prepn are given

4-Aminostyrene Polymer, $(C_8H_9N)_x$, is an amor substance which softens ca 76° and melts ca 81°. Can be prepd by heating 4-aminocinnamic acid(Ref 3) or by reduction of its ethylester with Sn+HCl(Ref 2)

Refs: 1)Beil **12**,1188 2)G.Bender,Ber **14**, 2359–61(1881) 3)A.Bernthsen & F.Bender, Ber **15**,1982(1882)

Mononitroaminostyrenes, $C_8H_8N_2O_2$, mw 164.16, N 17.07% – not found in Beil or CA through 1956

2,ω(or 3,ω)-Dinitro-4-amino-styrene or **3(or 2)-Nitro-4-(β-nitrovinyl)aniline** (Called by Friedländer "Nitroparamido-ω-nitrostyrol"), $O_2N{\cdot}CH{:}CH{\cdot}C_6H_3(NO_2){\cdot}NH_2$, mw 209.16, N 20.09%. Red-brn ndls(from alc), mp–not given. Was obtained by treating 4-aminocinnamic acid with mixed nitric-sulfuric acid in the cold. Its expl props were not investigated

Refs: 1)Beil **12**,1188 2)P.Friedländer & M.Lazarus,Ann **229**,247(1885)

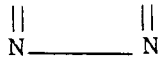
Note: No other nitrated and/or nitrited derivs were found in Beil or CA through 1956

AMINOTETRAZOLE AND DERIVATIVES

Aminotetrazoles, CH_3N_5, mw 85.06, N 82.34%, OB to CO_2 -65.8%, OB to CO -47.0%. The following isomers are theoretically possible: a)1-amino-*a*-tetrazole or 1-amino-1H-tetrazole b)2-amino-*β*-tetrazole or 2-amino-2H-tetrazole c)5-amino-*a*-tetrazole or 5-amino-1H-tetrazole and d)5-amino-*β*-tetrazole or 5-amino-2H-tetrazole. Of these the best known is item c

Items a and c exist only in the form of derivs, while item b or derivs were not found in the literature

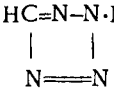
1-Amino-α-tetrazole or **1-Amino-1H-tetrazole**, HC–N(NH_2)–N, appears to be known in the



form of its derivs such as: a)*1-amino-5-phenyl-α-tetrazole* and its chlorophenyl-, nitro- anddichloroamino-derivs(see under Aminophenyltetrazoles) b)*1-amino-5-(p-tolyl)-α-tetrazole* and its dichloramino-deriv(see under Aminotolyltetrazoles) and c) *1-benzylidineamino-5-phenyl-α-tetrazole* (qv)

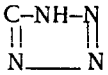
Refs: 1)Beil **26**,(113) & [216] 2)R.Stollé et al, JPrChem **138**,2–14(1933)

2-Amino-β-tetraozle or *2-Amino-2H-tetrazole*, HC=N–N·NH_2. This compd or its derivs



were not found in Beil or CA through 1956

5-AMINO-α-TETRAZOLE or 5-AMINO-1H-TETRAZOLE (One of the Ger names is *Tetrazolon-imid* and Thiele called it "Amidotetrazotsäure"). It is usually designated as **5-ATZ**, $H_2N{\cdot}C(=N{-}NH{-}N)$ or



HN:C–NH–N. It crystallizes from w in the



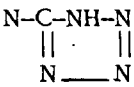
form of monohydrate, wh prisms or lflts, which lose $1H_2O$ above 100° and melts at 200–3°. It is diff sol in alc; insol in eth; sol in aq solns of bases and strong acids; sol in w to the extent of 117g per 100g H_2O at 18° and 7g at 100°(Ref 12,p 23). Its Q^v_c 246.2 kcal/mol and Q^v_f −49.7 kcal/mol (Refs 11 & 13)

5-ATZ is an acid, very hygroscopic and stable to heat. Its dissociation constant is 1×10^{-6}. In addn to the acidic props of the 1H-tetrazole ring, the 5-ATZ also has a basic amino-group. The compd is therefore amphoteric and behaves very much like an amino acid(Ref 12,p 19)

5-ATZ was first synthesized in 1892 by Thiele(Ref 2) by treating aminoguanidine with nitrous acid. The intermediate guanylazide was cyclized by ammonia to 5-ATZ:

$H_2N{-}C(:NH){-}NH{\cdot}NH_2 \xrightarrow{HONO}$

Aminoguanidine

$H_2N{\cdot}C(:NH){-}N_3 \xrightarrow{NH_4OH}$ 

Guanylazide

Dilute acids may be used in lieu of ammonia. Several other methods of prepn are described in the literature. In the method of Stollé(Ref 4) 5-ATZ is obtained by treating aminoguanidine with nitrous acid. Lieber & Levering(Ref 10), after modifying and working out Stollé's method in detail, obtained yields up to 74.5%. The same investigators prepd 5-ATZ in 70% yield by the reduction of tetrazolylazide(or its K salt) with H_2S. Garbrecht & Herbst(Ref 11b) prepd 5-ATZ by hydrogenolysis of 5-benzylaminotetrazole in abs alc in presence of Pd charcoal catalyst

Audrieth & Currier(Ref 12) investigated 5-ATZ as a high-nitrogen compd which might be of interest as a component of propints(or expls) or as a starting material for various derivs. According to these investigators the high chemical stability of the tetrazole ring plus the fact that substitutions in the ring are usually accomplished together with the synthesis of tetrazole itself, leaves only the amino group of 5-ATZ available for useful chemical reactions. Therefore, the reactions of 5-ATZ aside from salt formation deal principally with the reactions of this functional group. The amino group of 5-ATZ can be likened to that of aniline

X-ray diffraction spectra and IR absorption spectra of 5-ATZ are given in Ref 12, pp 81–9, IR absorption spectra in Ref 10a, X-ray diffraction patterns in Ref 11a, UV and IR absorption spectra in Ref 12a

5 ATZ forms metallic salts, some of them expl, eg, the *silver salt*, $AgCH_2N_5$. Wh amor ppt obtained by treating an aq soln of 5-ATZ with a $AgNO_3$ soln. It defgr on heating(Ref 2, p 59). The *mercury salt*, prepd in 1942 in Canada(Ref 6a) on treating 5-ATZ with $Hg(NO_3)_2$ expl on impact or heating

When 5-ATZ was treated with $NaNO_2$ + aq HCl under cooling, Thiele obtained a compd which was not analyzed because it exploded at ca 0°. Details of the procedure are given in Ref 2,p 62

Although not expl by itself, 5-ATZ may serve as the starting material for the prepn of expl derivs such as the nitrate, nitramino-compd, di(tetrazolyl-5):N¹,N⁶-hexazadiene, etc, some of which are described below

5-ATZ is a high nitrogen compd and can be used as a cooling agent in propellants. For instance, Hale & Audrieth(Ref 9) claimed that incorporation of up to 25% of 5-ATZ in smokeless propellants reduced the amt of flash and smoke without changing the ballistic potential or sensitivity to mechanical action

Refs: 1)Beil **26**,403(123) & [243] 2)J. Thiele,Ann **270**,54–61(1892) 3.A.Hantzsch & A.Vagt,Ann **314**,352–3,362–3(1901) 4)R.Stollé et al,Ber **62B**,1118-20(1929) 5)E.Lieber & G.B.L.Smith,ChemRevs **25**, 233 & 259(1939) 6)Davis(1943),448 6a)A. H.Blatt,OSRD Rept 2014(1944) (Under Tetrazoles) 7)F.R.Benson,ChemRevs **41**, 7(1947) 8)V.van Richter & R.Anschütz, "The Chemistry of the Carbon Compounds",Elsevier, NY,**4**(1947),172–3 9)G.C.Hale & L.F. Audrieth,USP 2,480,852(1949) & CA **44**,840 (1950) 10)E.Lieber & D.R.Levering,JACS **73**,1316(1951) 10a)E.Lieber et al,AnalChem **23**,1594(1951) 11)W.S.McEwan & M.W.Rigg, JACS **73**,4726(1951) 11a)L.A.Burkhardt & D.W.Moore,AnalChem **24**,1581–2(1952) 11b) W.L.Garbrecht & R.M.Herbst,JOC **18**,1028 1953 12)L.F.Audrieth & J.W.Currier, "Derivatives of 5-Aminotetrazole", Univ of Illinois Rept,Urbana, Ill, June 15, 1954(US Ordnance Corps Contract DA–11–022–ORD–33) 12a)D.B.Murphy & J.P.Picard, JOC **19**,1808 & 1810 (1954) 12b)P.Rochlin,D.B.Murphy & S.Helf, JACS **76**,1453(1954) (Relative basicities of 5-ATZ & derivs) 13)M.M.Williams et al, J PhysChem **61**,262(1957)

5-Amino-β(or 2H)-tetrazole, $H_2N{\cdot}C{=}N{-}NH$ (N=N). It seems to be known only in the form of its derivs, such as 2-phenyl-5-amino-β (or 2H)-tetrazole, $H_2N{\cdot}C{=}N{-}N{\cdot}C_6H_5$ (N=N), described in Beil 26,[243]

5-Amino-α-tetrazole Nitrate or **5-Amino-1H-tetrazole Nitrate** (called by Hantzsch et al, "Amidotetrazolnitrat"), $CH_3N_5 \cdot HNO_3$, mw 148.09, N 56.75%, OB to CO_2 –10.8%, OB to CO 0%. Crysts, mp-decompg explosively ca 174-5° (Refs 2&3). Its Q_c^v is 224.1 kcal/mol (Ref 4) and its X-ray diffraction spectra are given in Ref 5). The nitrate was prepd by dissolving 5-ATZ in the calcd amt of nitric acid

Refs: 1)Beil **26**,403 & [243] 2)A.Hantzsch & A.Vagt,Ann **314**,352–3(1901) 3)R.Stollé, et al,Ber **62B**,1120(1929) 4)W.S.McEwan & M.W.Rigg,JACS **73**,4726(1951) 5)L.A.Burkhardt & D.W.Moore,AnalChem **24**,1582(1952)

5-Nitraminotetrazole(5-NATZ) of O'Connor, $O_2N{\cdot}HN{\cdot}C{\cdot}NH{\cdot}N$ (N=N). mw 130.07, N 64.62%, OB to CO_2 –12.3%, OB to CO 0%. Col crysts, mp 195°, expl at sl higher temp, fairly sol in w; insol in alc, acet, eth, chlf & benz (Ref 2); Q_c^v 219.2 kcal/mol(Ref 3). Was prepd by treating nitroguanylazide with aq ammonia as described in Ref 2

Its *disodium salt*, $Na_2CN_6O_2$, mw 174.05, N 48.29%, OB to CO_2 -9.2%, OB to CO 0%. Yel ndls, mp–expl violently with a bright yel flash ca 207°. Very sol in w, sl sol in eth & benz; insol in acet, CS_2 & chlf. Was prepd by treating guanylazide in water with an aq Na acetate soln(Ref 2)

Refs: 1)Beil – not found 2)T.E.O'Connor et al,JSCI **68**,309(1949) & CA **44**,3443(1950) 3)W.S.McEwan & M.W.Rigg,JACS **73**,4726 (1951)

Note: Lieber et al(see next item) could not

obtain 5-NATZ by duplicating the reaction of O'Connor. Instead of obtaining the 5-NATZ they obtained its diammonium salt [See also F.L.Scott et al, J ApplChem **2**, 379(1952]

5-Nitroaminotetrazole(5-NATZ) of Lieber et al, $O_2N{\cdot}HN{-}\underset{\underset{N}{\|}}{C}{-}\underset{\underset{N}{\|}}{NH}{-}N$, called by us **5-Nitramino-*a*-tetrazole**, because NO_2 is attached to amino- group. Col crysts explg with an orange flash at ca 140°; extremely sensitive to shock and pressure. Its aq solns display strong acid props. Was prepd by dissolving with gentle heating potassium 5-ATZ in 1:1 hydrochloric acid, followed by cooling the soln and separating the resultant ppt by filtration. Purification was accomplished by dissolving the ppt in small amt of dioxane, adding a large amt of cold benz and then chilling the mixt to crystallize the pure 5-NATZ(Ref 2),p 2328)

Potassium-5-nitraminotetrazole, $KCHN_6O_2$, mw 168.17, N 49.98%, was obtained as almost colorless plates on treating nitraminoguanidine with K nitrite in water, as described in Ref 2,p 2328. The compd exploded with a purple flash when dropped on a hot bar at ca 220°

Diammonium-5-nitraminotetrazole, $(NH_4)_2{-}CN_6O_2$, mw 164.14, N 68.27%, col short ndls, mp 220–1°. Was obtained by heating 5-nitraminotetrazole with ammonia on a steam bath and then chilling the mixture. Another method of prepn consists in treating nitroguanylazide with ammonia(Ref 2, p 2327)

Guanidinium-5-nitraminotetrazole, $CH_5N_3 + CH_2N_6O_2$, mw 189.148, N 66.65% was obtained in the form of wh platelets(which melted after purification at 225–226°) from guanidine carbonate, AcOH and nitraminoguanidine as described in Ref 2,p 2328

Refs: 1)Beil – not found 2)E.Lieber et al, JACS **73**,2327–28(1951) & CA **46**,1987(1952) 3)E.Lieber et al, JACS **73**,2329–31(1952) (UV spectra of 5-NATZ and its salts)

Aldolcondensation Product of 5-Aminotetrazole, $CH_3{\cdot}CH(OH){\cdot}CH_2{\cdot}CH{:}N{-}\underset{\underset{N}{\|}}{C}{-}\underset{\underset{N}{\|}}{NH}{-}N$, mw 155.16, N 45.14%. Col lflts or prisms, mp ca 170°; fairly sol in hot w; sl sol in hot alc; insol in eth, acet & chlf. Was prepd by heating anhydrous 5-aminotetrazole with anhydrous freshly distilled(in an atm of CO_2) acetaldehyde for several hours on a water bath

Its *silver salt*, $AgC_5H_8N_5O$, wh expl ppt, was prepd by adding to an alc soln of the above aldol condensation product, the calcd amt of $AgNO_3$ in alc and some alc ammonia

When 5-ATZ was treated with freshly redistilled benzaldehyde(instead of acetaldehyde), the resulting mixt consisted of crysts explg at 183°, which proved to be *guanylaminotetrazole* together with some guanylaminotetrazole nitrate and some unreacted 5-aminotetrazole

Refs: 1)Beil – not found 2)R.Stollé & K.Heintz, J PrChem **148**,217–20(1937)

[N¹,N⁶-Bis(a-tetrazolyl-5)]-hexazadiene or **N¹,N⁶-(Ditetrazolyl-5)-hexazadiene.** Hofmann & Lieber called it *Bisdiazotetrazolylhydrazide.* Beil names it 1,6-Bis-[tetrazolinyliden-(5)]-hexazdien or 1,6-Di-[tetrazolyl-(5)]-hexazdien, $\underset{\underset{N}{\|}}{N}{-}\underset{\underset{N}{\|}}{NH}{-}C{-}N{:}N{\cdot}NH{\cdot}NH{\cdot}N{:}N{-}\underset{\underset{N}{\|}}{C}{-}\underset{\underset{N}{\|}}{NH}{-}N$ or $\underset{\underset{N}{\|}}{N}{-}\underset{\underset{N}{\|}}{NH}{-}C{=}N{\cdot}N{:}N{\cdot}N{:}N{\cdot}N{=}\underset{\underset{N}{|}}{C}{-}\underset{\underset{N}{\|}}{NH}{-}N$, mw 224.16, N 87.49%, OB to CO_2 -42.9%, OB to CO -28.6%. Lflts, mp–expl very violently ca 90°; also expl when rubbed with a glass rod or spatula. This substance, one of the richest in nitrogen, was prepd by diazotization of 5-ATZ, followed by treatment with an aq soln of hydrazine hydrochloride in the presence of Na acetate at low temp

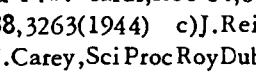
It forms salts, some of which are expl, eg, *sodium salt*, $NaC_2H_3N_{14}$, yel solid obtained by treating the tetrazolyl-hexazadiene with cold NaOH soln

Refs: 1)Beil **26**,(123–4) 2)K.A.Hofmann & H.Hock,Ber **44**,2953(1911) 3)E.Lieber & G.B.L.Smith,ChemRevs **25**,247–8(1939)

Addnl Refs on ATZ's, Their Salts and Nitrated Derivatives. a)L.Krauss, "Uber Amino-5-tetrazol", Heidelberg(1931) (38pp) b)H.Veldstra & P.W.Wiardi,Rec **61**,635–6 (1942) & CA **38**,3263(1944) c)J.Reilly, P. Teegan & M.F.Carey,Sci Proc Roy Dublin Soc **24**,349–53(1948) & **43**,1769–70(1949) (The diazo reaction in the tetrazole ring) d)E. Lieber et al,JACS **73**,2329–31(1951) & CA **45**,8887(1951) (UV spectra of 5-NAT and its NH_4 deriv) e)E.Lieber et al,AnalChem **23**,1594–6(1951) & CA **46**,3857(1952) (IR spectra of compds of high nitrogen content) f)W.S.McEwan & M.W.Rigg,JACS **73**,4725–7 (1951) & CA **46**, 4350(1952) (Heats of combustion of compds contg the tetrazole ring; included are 5-ATZ, 5-ATZ nitrate and 5-NATZ) g)F.L.Scott & J.Reilly, Chem & Ind **1952**,907–8 & CA **47**,6886(1953) (Prepn of 5-ATZ by acyl hydrazide transformations) h)L.A.Burkardt & D.W.Moore,AnalChem **24**, 1580–1(1952) & CA **47**,2010(1953) (X-ray diffraction patterns of 29 tetrazole derivs, among them 5-ATZ and its derivs) i)E. Lieber et al, JACS **74**,2684(1952) & CA **48**, 2693–4(1954) (Salts of 5-NAT prepd by the reaction of amines with nitroguanyl azide) j)R.A.Henry,JACS **74**,6303(1952) & CA **49**, 1023(1955) (Salts of 5-ATZ) k)R.M.Herbst & J.A.Garrison,JOC **18**,941–5(1953) & CA **48**,8779(1954) (The nitration of 5-ATZ) l)R.A. Henry & W.G.Finnegan,JACS **76**,290–1(1954) & CA **49**,12451 (An improved procedure for the deamination of 5-ATZ) m)Ibid,JACS **76**, 926–8(1954) & CA **49**,10940(1955) (Prepn and hydrogenation of azomethines derived from 5-ATZ) n)J.E.DeVries & E.St.Clair Gantz, JACS **76**,1009(1954) & CA **48**,7995(1954) (Spectrophotometric studies of dissociation constants of tetrazoles, triazoles and nitroguanidines)

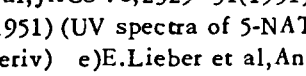
5-Aminotetrazole Derivatives Prepared and Examined under the Direction of Professor L.F.Audrieth. As part of the research on compds of high nitrogen content under contract DA–11–022–ORD–33 of the US Ordnance Corps, the following derivs of 5-ATZ were prepd and examd at the University of Illinois. Although these compds are not expl they may be of value as components of proplnt compns

a)N-(5-Tetrazolyl)-urethane,



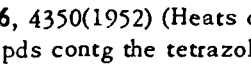
and its salts and derivs (Refs, pp 25-31)

b)4-(5-Tetrazolyl)-semicarbazide,



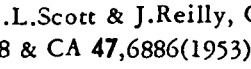
and its salts and derivs (Ref, pp 31-45 & 48-49)

c)N-(5-Tetrazolyl)-carbamyl Azide,



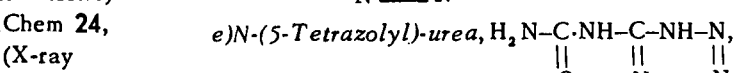
and its derivs(Ref,pp 46–8)

d)5-Tetrazolylammonium Cyanate,



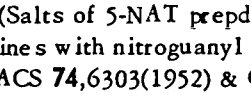
(Ref,pp 49–56)

e)N-(5-Tetrazolyl)-urea,



and its derivs(Ref,pp 57–67 & 68–71)

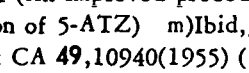
f)1-(5-Tetrazolyl)-semicarbazide,



and its derivs (Ref, pp 71-75)

g)N-Alkyl-N'-(5-tetrazolyl)ureas and *N,N-Alkyl-N'-(5-tetrazolyl)ureas* (Ref,pp 75–78)

h)1-Octadecyl-5-octadecyl-aminotetrazole,



(Ref,l pp 79–81)

Ref: L.F.Audrieth & J.W.Currier, "Derivatives of 5-Aminotetrazole", Rept of the Univ of Illinois, Urbana,Ill,June 15,1954

Aminotetrazole Substituted Derivatives. Many substituted alkyl-, dialkyl-, aryl-, alkylaryl-, etc compds are described in the literature, for instance, *methylaminotetrazole*, which is described as *5-amino-1-methyl-α-tetrazole*. Other substituted 5-aminotetrazoles,

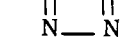
such as ethyl-, ethylmethyl-, propyl-, isopropyl-, dimethyl-, diethyl, etc are described by R.M.Herbst, C.W.Roberts & E.J. Harvill, JOC **16**,139–49(1951) & CA **45**, 6629(1951)

Addnl Refs on ATZ Substituted Compounds a)J.van Braun & W.Keller, Ber **65**,1677–80 (1932) (Syntheses of some derivs of tetrazole) b)W.G.Finnegan, R.A.Henry & E. Lieber, JOC **18**,779–91(1953) & CA **48**, 7005–7(1954) (Prepn and isomerization of 5-alkylaminotetrazoles) c)W.Garbrecht & R.M.Herbst, JOC **18**,1003–13(1953)(21 refs) & CA **48**,8224–5(1954) (Synthesis of certain 5-ATZ derivs) d)Ibid, JOC **18**,1014–21(1953) (21 refs) & CA **48**,8224(1954) (Synthesis of 1-substituted 5-ATZ's) e) Ibid, JOC **18**,1022–29(1953) (12 refs) & CA **48**,8225–6(1954) (Synthesis of 5-monoalkylaminotetrazoles) f)Ibid, JOC **18**,1269–82 (1953) (21 refs) & CA **48**,12092–3(1954) (Rearrangement of certain monosubstituted 5-ATZ derivs) g)Ibid, JOC **18**,1283–91 (13 refs) & CA **48**,12093(1954) (Acylation of some 5-ATZ derivs) h)R.A.Henry, W.G. Finnegan & E.Lieber, JACS **76**,88–93(1954) & CA **49**,2427(1955) (Thermal isomerization of substituted 5-ATZ's) i)Ibid **76**,923–6 (1954) & CA **49**,10939–40(1955) (Monoalkylation of sodium 5-ATZ in aq medium) j) Ibid, JACS **76**,2894–8(1954) & CA **49**,10274 (1955) (Dialkyl derivs of 5-ATZ) k)D.W. Moore & L.A. Burkardt, AnalChem **26**,1917–20(1954) & CA **49**,4364(1955) (X-ray powder diffraction patterns of some tetrazole derivs) l)P.Rochlin, D.B.Murphy & S.Helf, JACS **76**,1451–3(1954) & CA **49**,3950–1 (1955) (Some properties of 5-ATZ substituted derivs) m)D.B.Murphy & J.P.Picard, JOC **19**,1807–14(1954) & CA **49**,15879(1955) (Studies of tautomerism in 5-ATZ and its derivs by means of UV and IR absorption spectra) n)R.A.Henry, W.M.Finnegan & E. Lieber, JACS **77**,2264–70(1955) & CA **50**, 2556–8(1956) (Kinetics of the isomerization of substituted 5-ATZ's)

m-Aminotetryl or **2,4,6-Tinitro-3-methylnitramino-aniline.** See Methylaminoaniline and Derivatives

AMINOTHIADIAZOLE AND DERIVATIVES

2-Amino-1,3,4-thiadiazole or **2-Imino-1,3,4-thiadiazoline**, called by Freund "Imidothiobiazolin", HC–S–C·NH_2 or HC–S–C:NH.



Col crysts, mp 191–4°; easily sol in hot w & in alc; insol in eth, benz & chlf. Was prepd by treating with aq NaOH the hydrochloride of aminothiadiazole(ndls, mp 149–50°) which was prepd by prolonged treatment of 1-formyl-thio-semicarbazide with an excess of acetyl chloride at RT(Ref 2). If the last reaction was conducted under pressure, acetylaminothiadiazole was obtained

Refs: 1)Beil **27**,624 & [687] 2)M.Freund & C.Meinecke, Ber **29**,2515(1896) 3)R.Stollé & K.Fehrenbach, J PraktChem **122**,296–7(1929)

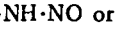
Note: Several later refs are given in CA

Azidoaminothiodiazole, $C_2H_2N_3S$ – not found in Beil or CA through 1956

2-Nitrosamino-1,3,4-thiadiazole, called by Freund "Nitrosoimidothiobiazolin", $C_2H_2N_4OS$, mw 130.13, N 43.06%. Yel crysts, mp 220°(dec). Was prepd by treating 2-amino-1,3,4-thiadiazole hydrochloride with aq Na nitrate while cooling

Refs: 1)Beil **27**,625 2)M.Freund & C.Meinecke, Ber **29**,2515(1896) 3)M.Kanaoka, J PharmSoc Japan **75**,1149(1954) & CA **50**,5647(1956)

5-Azido-2-nitrosamino-1,3,4-thiadiazole or **5-Azido-2-nitrosimino-1,3,4-thiadiazoline**, N_3C–S–C·NH·NO or N_3C–S–C:N·NO, mw 171.15,



N 57.29%. Dk brn ppt which expl violently ca 150°, sl sol in w, eth and hot alc. Was prepd by treating 5-hydrazino-2-amino-1,3,4-thiadiazole-dihydrochloride with aq Na nitrate sol. at 0°

Refs: 1)Beil **27**,[688] 2)R.Stollé & K.Fehrenbach, J PraktChem **122**,301(1929)

5-Nitro-2-Amino-1,3,4-thiadiazole, $C_2H_2N_4O_2S$, mw 146.13, N 38.34%. Crysts, mp 178°. Was

prepd by nitrating 2-amino-1,3,4-thiadiazole with fuming nitric acid at 40°

Refs: 1)Beil – not found 2)E.B.Towne & J.B.Dickey,USP 2,708,671(1955) & CA **49**, 15252(1955)

2-Nitramino-1,3,4-thiadiazole, $C_2H_4N_4O_2S$, crysts, mp 177°. Was prepd by nitrating with concd HNO_3 the 2-amino-1,3,4-thiadiazole dissolved in concd H_2SO_4

Refs: 1)Beil – not found 2)M.Kanaoka, JPharmSocJapan **75**,1149(1955) & CA **50**, 5647(1956)

Note: Apparently the last two derivs of 2-amino-1,3,4-thiadiazole are identical

AMINOTHIAZOLES AND DERIVATIVES

2-Aminothiazoles, called in Beil *Thiazolon-(2)-imid*, $HC{-}S{-}C{\cdot}NH_2$ or $HC{-}S{-}C{:}NH$, $HC{-}N$ $HC{-}NH$ mw 100.14, N 27.97%. Yel crysts, mp 90–92°; easily sol in hot w; diff sol in alc & eth. Can be prepd by refluxing aq suspension of thiourea and chloroacetaldehyde alcohol until the oily layer was no longer turbid (Refs 2 & 3)

Moran & Morrow obtained a col cryst *perchlorate* when they added aminothiazole to 20% perchloric acid. When its soln, cooled to 0°, was treated with ethyl nitrite in view to obtain diazonium perchlorate and then stirred, a sharp expln occurred and the cracking detonation was repeated at short intervals, even when the soln was allowed to remain unstirred in contact with fragments of ice. On account of these explns and because of its great soly, no attempts were made to isolate the diazonium perchlorate. When gold chloride was cautiously added to the above soln, an orange ppt was obtained. It was collected, washed with a little ice water and dried on a porous plate in a desiccator over KOH. The partly dried material was somewhat expl, owing either to co-pptd diazonium perchlorate of to adherent perchloric acid

Note: The prefixes Aminothia and Aminothio in the compds on pp 262-4 are interchangeable

Refs: 1)Beil **27**,(263) & [205] 2)V.Traumann,Ann **249**,35–6(1888) 3)G.T.Morgan & G.V.Morrow,JCS **107**,1293–5(1915)

Note: Later refs are given in CA's

Azidoaminothiazole, $C_3H_3N_5S$ – not found in Beil or CA through 1956

2-Amino-5-nitrothiazole or **Enheptin**, $O_2N{\cdot}C{-}S{-}C{\cdot}NH_2$, mw 145.14, N 28.95%. $CH{-}N$

Solid, mp 197–8°(decomp). Can be prepd by refluxing a soln of 2-acetamido-5-nitrothiazole in aq H_2SO_4 for 1 hr, followed by cooling and treatment with phosphoric acid(Refs 2 & 3) or by nitrating 2-aminothiazole with mixed nitric-sulfuric acids(Ref 4)

Refs: 1)Beil – not found 2)H.L.Hubbard, USP 2,753,641(1951) & CA **46**,3573(1952) 3)G.W.Steahly,USP's 2,573,656 & 2,573,657 (1951) & CA **46**,3573(1952) 4)J.B.Dickey, et al,JOC **20**,505(1955) & CA **50**,4128(1956)

2-Nitramino-5-nitrothiazole, $O_2N{\cdot}C{-}S{-}C{\cdot}NHNO_2$ $HC{-}N$ or $O_2N{\cdot}C{-}S{-}C{:}N{\cdot}NO_2$, mw 190.14, N 29.47%. $HC{-}NH$

Crysts, mp–explodes at 162–7°. Was prepd by treating a sulfuric acid soln of 2-aminothiazole with 90–95% nitric acid mixed with concd sulfuric acid. Abs alc was added dropwise to destroy the excess of nitric acid (Ref 3) (See also Ref 2)

Refs: 1)Beil – not found 2)H.von Babo & & B.Prijs,Helv **33**,306(1950) & CA **44**,5872 (1950) 3)S.J.Viron & A.Taurins,CanJChem **31**,887 & 890(1953) & CA **49**,2423(1955) 4)J.B.Dickey et al,JOC **20**,504–5(1955) & CA **50**,4128(1956)

2-Nitramino-dinitrothiazole, $C_3HN_5O_6S$ – was not found in Beil or in CA through 1956

2-Nitramino-3,4,5-trinitro-thiazolone or **2-Nitrimino-3,4,5-trinitro-4-thiazoline**, $O_2N{\cdot}C{-}S{-}C{:}N{\cdot}NO_2$, mw 280.14, N 30.03%. $O_2N{\cdot}C{-}N{\cdot}NO_2$

Wh crysts, mp 121–3° with violent decompn. Was prepd by nitrating 2-nitramino-5-nitrothiazole with 99–100% nitric acid in the presence of acetic anhydride and glacial acetic acid. The crude product must be well washed or it becomes gummy and explodes. (See also Aminomethylthiazole and Derivatives)

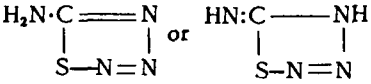
Refs: 1)Beil – not found 2)S.J.Viron & A.Taurins,CanJRes **31**,887(formula VI) & 890–1 & CA **49**,2423(1955)

Addnl Refs on Aminothiazoles and Derivs: a)K.Ganapathi & A.Venkataram,ProcIndian AcadSci **22A**,343–61(1946) & CA **40**,4056–59(1946) (Syntheses of 5-amino- and 4-aminothiazole derivs) b)I.V.Ballavita,AnnChimAppl **38**,449–55(1948) & CA **44**,154(1950) (Prepn of some aminothiazole derivs) c) J.B.Dickey & E.B.Towne,USP's 2,659,719 (1953) & 2,683,708–9(1954); CA **49**,1335 & 1336–7(1955) (2-amino-5-nitrothiazole dyes) d)S.R.M.Bushby & F.C.Copp,JPharm and Pharmacol **7**,112–17(1955) & CA **50**,964 (1956) (Some aminothiazols) e)J.B.Dickey, E.B.Towne & G.F.Wright,JOC **20**,499–510 (1955) & CA **50**,4128–9(1955) (Nitration of 2-aminothiazoles) f)J.B.Dickey & E.B.Towne, USP 2,730,523(1956) & CA **50**,7467(1956) {Derivs of 2-[p-(fluoralkylamino)phenylazo] -5-nitrothiazoles} g)J.B.Dickey & E.B.Towne, USP 2,746,953(1956) & CA **50**,15093(1956) (5-nitrothiazolylazoaniline compds)

5-Amino-1,3,4-thiodiazolthion-(2). See 5-Amino,2-mercapto-1,3,4-thiadiazole

3-Amino-5-thiol-1,2,4-thiadiazole. See 5-Amino,2-mercapto-1,3,4-thiadiazole

5-Amino-vic(or 1,2,3,4)-thiotriazole [1.2.3.4-Thiotriazolon-(5)-imid or 5-Imino-1.2.3.4-thiotriazolin, in Ger] (Called by Freund "Amido-Thiazsulfol")



mw 102.12, N 54.87%, OB to CO_2 and SO_2 –78.3%, OB to CO and SO_2 –62.7%. Ndls (from eth), prisms(from MeOH + eth), mp defgr at 128–130°. Sol in warm alc, AcOH, ethyl acet & aniline; more diff sol in chlf & CS_2; insol in benz; decomp by boiling w. Was prepd by treating thiosemicarbazide hydrochloride with an aq soln of NaOH in the cold

It forms salts, some of them expl, eg, $CH_2N_4S + HCl$, crysts, defgrg at 96°

Refs: 1)Beil **27**,781 2)M.Freund & A. Schander,Ber **29**,2502–2505(1896)

Note: This compd, known already for over 60 years, was recently described in the confidential Rept of the Naugatuck Chem Div, US Rubber Co, Progress Rept Mar 15–Apr 15,1949(Nord 10,121),788. This rept was not used here as a source of information

AMINOTHIATRIAZOLE AND DERIVATIVES

5-Amino-1,2,3,4-thiotriazole, called in Beil *1.2.3.4-Thiotriazolon-(5)-imid* or *5-Imino-1.2.3.4-thiothiotriazolin* and by Oliveri-Mandalà "Thiocarbamidsäureazid", $H_2N{\cdot}C(=N{-}N)-S-N$ or $HN{:}C(-HN{-}N)-S-N$, mw 102.12, N 54.87%. Crysts, mp–expl ca 128–30°; sol in warm alc, AcOH, ethyl acetate & aniline; more diff sol in chlf & CS_2; insol in benz; decompd on boiling in w. Can be prepd by treating a cooled aq soln of thiosemicarbazide hydrochloride with aq $NaNO_2$ soln

Its hydrochloride, $CH_2N_4S + HCl$ expl at 96°

Refs: 1)Beil **27**,781 2)M.Freund & A.Schander,Ber **29**,2502(1896) 3)E.Oliveri-Mandala, Gazz **44I**,672(1914)

Nitrosaminothiotriazole, CHN_5OS and *Nitraminothiotriazole*, CHN_5O_2S – not found in Beil or CA through 1956

AMINOTOLUENES AND DERIVATIVES

Aminotoluenes, Aminotoluols, Aminomethylbenzenes, Methylanilines, Tolylamines or

Toluidines, $CH_3 \cdot C_6H_4 \cdot NH_2$, are described in Beil **12**,772,853,880,(372,397,410 & [429, 463,482]

Azidoaminotoluene, $C_7H_8N_4$ and *Diazidoaminotoluene*, $C_7H_7N_7$ – not found in Beil or CA through 1956

Mononitroaminotoluenes, $CH_3 \cdot C_6H_3(NO_2) \cdot NH_2$, mw 152.15, N 18.41%, are described in Beil **12**,843–4,846, 848, 876–7,996,1000,(392, 394–5,408,438–9) & [458–60, 476,534–5]

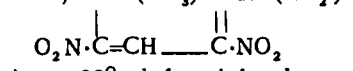
Nitraminotoluenes, $CH_3C_6H_4 \cdot NHNO_2$, are described in Beil **16**,670 & 672

Nitronitraminotoluenes, $CH_3 \cdot C_6H_3(NO_2),NHNO_2$, mw 197.15, N 21.32%, are described in Beil **12**,848,1000,1006 and **16**,671–2

Dinitroaminotoluenes, $CH_3 \cdot C_6H_2(NO_2)_2 \cdot NH_2$, mw 197.15, N 21.32%, are described in Beil **12**,851-2,878–9,1009,(396,409,442) & [462–3, 479–80,482,537–9]

Dinitronitraminotoluenes, $CH_3 \cdot C_6H_2(NO_2)_2 \cdot NHNO_2$, mw 242.15, N 23.14%, OB to CO_2 –72.7%, OB to CO –26.4%. The following isomers are described in the literature:

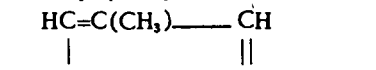
3,5-Dinitro,2-nitraminotoluene or **4,6-Dinitro, 2-methyl-phenylnitramine**,



Yel crysts(from benz), mp 92°, defgr violently when heated above its mp. Easily sol in alc, eth & benz. Can be prepd by treating 6-aminotoluene sulfonic acid with nitric acid(d 1.51) at –10° to 0°. It forms salts, such as $AgC_7H_8N_4O_6$ and $Ba(C_7H_8N_4O_6)_2$

Refs: 1)Beil **16**,671 2)T.Zincke & P.Malkomesius,Ann **339**,219(1905)

3,5-Dinitro-4-nitraminotoluene or **2,6-Dinitro-4-methyl-phenylnitramine**,

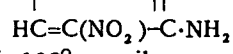


Col crysts, mp 104°, defgr when heated above its mp, easily sol in w; very sol in org solvents, except benz. Can be prepd by treating 4-aminotoluenesulfonic acid with nitric acid(d 1.48) at –5°. Forms salts, such as $AgC_7H_5N_4O_6$

Refs: 1)Beil **16**,672 2)T.Zincke & A. Kuchenbecker,Ann **339**,230(1905)

Trinitroaminotoluenes, $CH_3 \cdot C_6H(NO_2)_3 \cdot NH_2$, mw 242.15, N 23.14%, OB to CO_2 –72.7%, OB to CO –26.4%. The following isomer is described in the literature:

2,4,6-Trinitro-3-aminotoluene or **2,4,6-Trinitro-3-methyl-aniline**,



Yel prisms(from alc), mp 136–138°; easily sol in alc & eth; sol in aq solns of NaOH & $Ba(OH)_2$ and in NH_3. Can be prepd by treating 2,4,6-trinitro,3-ethoxy-toluene with alc NH_3 at ord temp(Ref 2) or by other methods listed in Refs 1 & 3. Its expl props were not examined

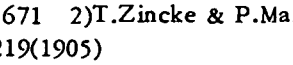
Refs: 1)Beil **12**,879 & 409 2)E.Noelting & E. van Salis,Ber **15**,1864(1882) 3)J.J.Blanksma, Rec **21**,332(1902)

Note: No other isomers of trinitroaminotoluenes and no higher nitrated derivs were found in Beil or CA through 1956

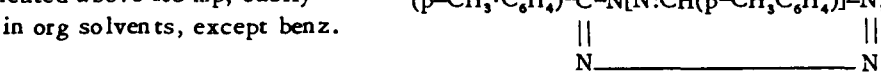
AMINOTOLYLTETRAZOLES AND DERIVATIVES

Aminotolyltetrazoles or *Tolyltetrazolonimides* (Cf with Tolylaminotetrazoles), $C_8H_9N_5$, mw 175.19, N 39.98%. The following isomers are described in the literature:

1-Amino-5-(p-tolyl)-a(or 1H)-tetrazole, [Amino-(1)-p-tolyl-(5)-tetrazol, in Ger]



Long ndls, mp 141°, decomp at higher temps; sl sol in cold w, fairly sol in hot w, moderately sol in eth, sol in alc & acet. Can be prepd by heating 1-p-tolyal-amino-5-(p-tolyl)-tetrazole,



with 30% hydrochloric acid (Ref 2)

Although not known as an expl, 1-ATTZ may serve as the starting material for the prepn of derivs, some of them expl(see below)

Refs: 1)Beil **26** – not found 2)R.Stollé et al, J PrChem **138**,2 & 6–7(1933)

2-Amino-5-tolyl-β(or 2H)-tetrazole,
$(CH_3 \cdot C_6H_4)$-C=N-N·NH_2 – not found in Beil
or CA through 1956

5-Amino-2-tolyl-β(or 2H)-tetrazole,
H_2N-C=N-N$(CH_3C_6H_4)$ – not found in Beil
or CA through 1956

5-Amino-1-(o-tolyl)-α(or 1H)-tetrazole,
H_2N-C-N(o-$CH_3 \cdot C_6H_4$)-N. Crysts, mp 191–2°.
Was prepd by cyclization of a substituted guanylazide as described in Refs 2 & 3. Its rate of isomerization is given in Ref 4. Relative basicities are given in Ref 4
Refs: 1)Beil – not found 2)W.H.Finnegan et al, JOC **18**,788(1953) 3)R.A.Henry et al, JACS **76**,92(1954) 4)R.A.Henry et al, JACS **77**,2265(1955) 5)P.Rochlin, D.B.Murphy & S.Helf, J ACS **76**, 1453(1954)

5-Amino-1-(m-tolyl)-α(or 1H)-tetrazole,
H_2N-C-N(m-$CH_3 \cdot C_6H_4$)-N. Crysts, mp 162–3°.
Was prepd by cyclization of a substituted guanylazide as described in Ref 2. Its thermal isomerization is discussed in Ref 2 and the rate of isomerization is given in Ref 3. Relative basicities are given in Ref 4
Refs: 1)Beil – not found 2)R.A.Henry et al, JACS **76**,92(1954) 3)R.A.Henry et al, JACS **77**,2265(1955) 4)P.Rochlin, D.B.Murphy & S.Helf, J ACS **76**, 1453(1954)

5-Amino-1-(p-tolyl)-α(or 1H)-tetrazole,
H_2N-C-N(p-$CH_3 \cdot C_6H_4$)-N. Col crysts, mp 175.5–177°; easily sol in hot alc, diff sol in hot w, very diff sol in eth. Was prepd by Stollé(Ref 2) on treating p-tolylthiourea with Pb monoxide and Na azide in hot alc. Henry et al(Ref 3) prepd it by cyclization of a substituted guanylazide. Its rate of isomerization is given in Ref 4

Refs: 1)Beil **26**,[249] 2)R.Stollé, Ber **55**, 1295(1922) 3)R.A.Henry, JACS **76**,92(1954) 4)R.A.Henry, JACS **77**,2265(1955)

Azidoaminotolyltetrazole, $C_8H_8N_8$ and *Diazidoaminotolyltetrazole,* $C_8H_7N_{11}$ – not found in Beil or CA through 1956

1,1-(N-Dichloramino)-5-(p-tolyl)-α(or 1H)-tetrazole(p-$CH_3 \cdot C_6H_4$)-C-N(NCl_2)-N, mw 244.09, N 28.69%. Crysts, mp–expl. Can be prepd by treating 1-amino, 5-phenyl-α-tetrazole with an aq soln of hypochlorous acid(which can be prepd by passing chlorine gas through an aq soln of Na_2CO_3)

It should be noted that the dichloramino compd is a very violent and extremely sensitive expl. It expl even when in the wet condition on being touched with a spatula or with the fingers

Refs: 1)Beil - not found 2)R.Stollé, et al, J PraktChem **138**,2,7 & 8(1933) & CA **27**,4798(1933)

1,1'-Azo-5,5'-di(p-tolyl)-tetrazole,
$(p\text{-}CH_3C_6H_4)\cdot C$(N–N:N–N)$C\cdot(p\text{-}CH_3C_6H_4)$,
mw 346.35, N 40.44%, OB to CO_2 -180.2%, OB to CO -106.3%. Lt yel ndls, explg violently on heating or on impact; insol in w, sl sol in MeOH & acet; sol in benz(decomp on standing). Was prepd by treating dichloraminotolyltetrazole (see above) with an aq soln of KI, followed by addn of thiosulfate to remove the iodine

This compd is very unusual as it contains ten nitrogen atoms linked together in a chain

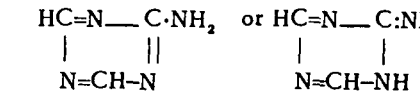
Refs: 1)Beil – not found 2)R.Stollé et al, JPrChem **138**,2 & 7–8,CA **27**,4798(1933)

Nitro- and/or Nitroso-Derivatives of Aminotolyltetrazoles were not found in Beil or CA through 1956

AMINOTRIAZINES AND DERIVATIVES

Aminotriazines or *Iminodihydrotriazines*, $C_3H_4N_4$, mw 96.09, N 58.31%. The following isomers of these high nitrogen compds are described in the literature:

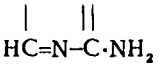
2-Amino-sym(or 1,3,5)-triazine,



Ndls decomg ca 228°(Ref2) or 224°(Ref 4). Was first obtained in a small amt by Diels and called "Monoaminocyanurwasserstoff' '(Ref 2). In the method patented in GtBritain (Ref 3), the 2-amino-s-triazine was produced by heating formylguanidine with $HCONH_2$ in the presence of an alkali metal hydroxide such as NaOH at 160° until the mixt became wholly liq. The liq was then distilled at 250°

Refs: 1)Beil **26**, 152 2)O.Diels,Ber **32**,696 (1899) 3)J.P.English & J.H.Paden,BritP 566,993(1945) & CA **41**,1716(1947) 4)R.Hirt et al,Helv **33**,1365(1950) & CA **46**,120(1952)

3-Amino-asym(or 1,2,4)-triazine,



Wh ndls, mp 171.5–172.5°; sol in w. Was prepd by adding an aq soln of glyoxal to an aq suspension of aminoguanidine bicarbonate, as described in Ref 2. Relative basicities are given in Ref 5
Refs: 1)Beil – not found 2)J.G.Erickson, JACS **74**,4706(1952) & CA **48**,8224(1954) 3)P.Rochlin, D.B.Murphy & S. Helf,JACS **76**,1452(1954) & CA **49**,3951(1955) (Some properties of 3-amino-as-triazine) 4)J.G. Erickson, The 1,2,3- and 1,2,4-Triazines, Tetrazines and Pentazines, Interscience, NY(1956),p 54 5)P.Rochlin, D.B.Murphy & S.Helf,JACS **76**,1453(1954)

Addnl Refs on Aminotriazines: a)C.Grundmann et al,ChemBer **87**,23(1954) & CA **49**, 6277(1955) (Prepn and props of 2-amino-1,3,5-triazine) b)R.C.Hirt & R.G.Schmitt, JChemPhys **23**,600(1955) & CA **49**,10058 (1955) (UV absorption spectra of amino-as-triazines) c)L.Paoloni,Gazz **84**,735(1954) & CA **50**,1039–40(1956) (Monoamino-, diamino- and triaminotriazines)

Nitrated and/or Nitrited Aminotriazines were not found in Beil or CA thru 1956

Azidoaminotriazine, $C_3H_3N_7$ – not found in Beil or CA through 1956

Nitro- and Nitroso- derivs – not found in Beil or CA through 1956

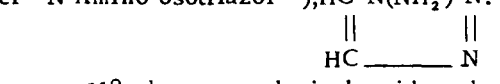
6-Amino-2,4-sym-triazinediol. Same as Ammelide

AMINOTRIAZOLES AND DERIVATIVES

Aminotriazoles, $C_2H_4N_4$, mw 84.08, N 66.64%, OB to CO_2 –114.2%, OB to CO –76.1%. The following aminotriazole isomers are theoretically possible: a)1-amino-*α*-vic(or 1H-1,2,3)-triazole b)2-amino-*β*-vic(or 2H-1,2,3)-triazole c)4-amino-*α*-vic(or 1H-1,2,3)-triazole d)5-amino-*α*-vic(or 1H-1,2,3)-triazole e)2-amino-*β*-as(or 2H-1,2,4)-triazole f)3-amino-*α*-as(or 1H-1,2,4)-triazole(also called 5-amino-2H-1,2,4-triazole or 5-amino-3H-1,3,4-triazole) and g)4-amino-*β*-as (2H-1,2,4)-triazole
No description was found in the literature for compounds b), c), d) or e)

Among the nitrated and/or nitrited derivatives only 3-amino-*α*-as(or 1H-1,2,4)-triazole nitrate and 3-nitramino-*α*-as(or 1H-1,2,4)-triazole were found in the literature

1-Amino-*α*-vic-triazole or **1-Amino-1H-1,2,3-triazole**[1.2.3-Triazolon-(1)-imid, in Ger](Called in Ber "N-Amino-osotriazol' '),



Crysts, mp 51°; decomp explosively with a sharp report at higher temps. Was prepd by von Pachmann by heating a mixt of 1-benzamino-vic-triazole

with dil HCl in a sealed tube at 90–100° (Ref 2). The constitution of his "aminoosotriazole" was established by Stollé (Refs 3 & 4). It forms salts, some of them expl, eg, the *picrate*, which melts with decompn ca 130° and then defgr(Ref 2)

Refs: 1)Beil **26**,12 2)H.von Pechmann & W.Bauer,Ber **42**,669(1909) 3)R.Stollé,Ber **59**,1742(1926) 4)J.G.Erickson et al, "The 1,2,3- and 1,2,4-Triazines, Tetrazines and Pentazines", Interscience,NY(1956), 154

3-Amino-α-sym-triazole; **3-Amino-1H-1,2,4-triazole** or **5-Amino-2H-1,2,4-triazole**[1.2.4-Triazolon-(3)-imid, in Ger] (Called by Thiele "Amidotriazol"), HC–NH–N
|| ||
N—— C·NH$_2$,

HC–NH–NH or H$_2$N·C=N–NH. This compd
|| | | |
N—— C:NH N=CH

is manufd by the Fairmont Co(Ref 5) under the name *5-Amino-1,3,4-triazole*. Yel crysts, mp 159°(Ref 2), 156–7°(Ref 4) or 156–9° (Ref 5); sublimes and partially decomp at higher temps. Sol in w, alc or chlf; very sl sol in acetic ester and nearly insol in acet or eth(Refs 1 & 2). Its Q_c^v is 343.10 kcal/mol and Q_f^v –18.3 kcal/mol(Ref 4). According to Ref 2b, its power is low and FI = 72% PA. Was first prepd by evaporating a soln of formylaminoguanidine nitrate with soda on a water bath(Ref 2)

HC–HN—HN + 2HNO$_3$ + Na$_2$CO$_3$ ⟶
|| |
O HN=C·NH$_2$

2NaNO$_3$ + CO$_2$ + 2H$_2$O + HC–NH–N
|| ||
N—— C·NH$_2$

This and some other methods of prepn are listed in Ref 1

In Ref 3 is given a detailed description of a method of prepn from formylaminoguanidine sulfate and soda soln. Rathsburg(Ref 2a) patented its use in primers, detonators, etc

The *silver* salt of 3-amino-α-sym-triazole is a weak expl(Ref 2)

Note: The amino triazole examined by Williams et al(Ref 4) was obtained from the Fairmont Chemical Co. This material was purified by dissolving in dioxane, decolorizing by charcoal and recrystallizing 3 times from dioxane

Refs: 1)Beil **26**,137,(38) & [76] 2)J.Thiele & W.Manchot,Ann **303**,46–7(1898) 2a)H. Rathsburg,BritP 185,555(1921) & CA **17**, 1147(1923) 2b)A.H.Blatt & F.C.Whitmore, OSRD 1085(1942) 3)OrgSynth **26**(1946), 11 or CollVol **3**(1955),95 4)M.M.Williams et al,JPhChem **61**,264 & 266(1957) 5)F. von Hessert, Fairmont Chemical Co, Newark, NJ; private communication Nov 18,1958

4-Amino-γ-sym-triazole or **4-Amino-4H-1,2,4-triazole** (Called in some papers, 4-Amino-4,1,2-triazole), HC=N–N . Hygr
| ||
H$_2$N·N—— CH

ndls, mp 82–3°; sol in w or HCl, sl sol in chlf & pet eth. Although this compd was claimed to have been prepd as early as 1899(Ref 1), its structure was not established until 1906(Ref 2). According to Erickson et al(Ref 6), S.Ruhemann & R.W.Merrimann,JCS **87**,1768–9(1905), believed that this aminotriazole was 1,4-dihydro-s-triazine, N–NH–CH, and named it *tetrazoline*, but
|| ||
HC–NH–N

this structure proved to be wrong

A detailed description of the prepn of 4-amino-γ-sym-triazole by heating formylhydrazine at 150–200° is given in Ref 3. Formylhydrazine was obtained by heating a mixt of ethylformate and hydrazine hydrate in alc for 18 hours. In Refs 4 & 5 is described a method of prepn of this aminotriazole starting with the treatment of hydrazine hydrate and carbon monoxide at high pressure and elevated temps

It forms numerous salts and additive compds, some of them expl, eg: a)C$_2$H$_4$N$_4$ + AuCl$_3$, yel amor pdr, mp 120°, expl at

higher temps b)$C_2H_4N_4 + AgNO_3$, wh ppt, defgr on heating

Ref: 1)Beil **26**,16 & [7] 2)C.Bülow,Ber **39**, 2620(1906) 3)Org Synth **24**(1944),12 or Coll Vol **3**(1955),96 4)G.D.Buckley & N.H.Ray, JCS **1949**,1157 & CA **44**,2942(1950) 5)Ibid, BritP 649,445(1951) & CA **45**,8561(1951) 6)J.G.Erickson et al, "The 1,2,3- and 1,2,4-Triazines, Tetrazines and Pentazines", Interscience,NY(1956),185–6

5-Amino-2H-1,2,4-triazole or **5-Amino-3H-1,3,4-triazole.** Same as 3-Amino-1H-1,2,4-triazole, listed here as 3-Amino-*a*-sym-triazole

Ref: Private communication from Dr Ronald A.Henry,NOTS,China Lake,Calif,5056/RAH: jj, 18 Nov 1958

Azidoaminotriazole, $C_2H_3N_7$ – not found in Beil or CA through 1956

3-Amino-*a*-sym-triazole Nitrate or **3-Amino-1H-1,2,4-triazole Nitrate**, HC–NH–N + HNO_3,
|| ||
N—— C·NH_2

mw 147.10, N 47.61%, OB to CO_2 −38.1%, OB to CO −16.3%. Crysts, mp 180.5 −181.5°(Ref 3), 174°(decomp)(Ref 2). Its Q_c^v is 318.0 kcal/mol and Q_f^v + 140.9(Ref 3). Was prepd by Thiele et al by treating the mother liquor left after removal of 3-amino-*a*-sym-triazole with nitric acid, as described in Ref 2. Rathsburg(Ref 2a) proposed its use in primers, detonators, etc

Refs: 1)Beil **26**,138 2)J.Thiele & W.Manchot,Ann **303**,47(1898) 2a)H.Rathsburg, BritP 185,555(1921) & CA **17**,1147(1923) 3)M.M.Williams et al,JPhChem **61**,264 & 266(1957)

3-Nitramino-*a*-sym-triazole, 3-Nitramino-1H-1,2,4-triazole or **5-Nitramino-2H-1,2,4-triazole** (Was called by Henry, *5-Nitramino-1,2,4-triazole*), HC–NH–N or $O_2NHN{\cdot}C{=}N{-}NH$,
|| || | |
N—— C·$NHNO_2$ N=CH

mw 129.08, N 54.26%, OB to CO_2 −43.4%, OB to CO −18.6%. Wh granular product decompg at 217°; Q_c^v is 317.5 kcal/mol and Q_f^v -26.9 kcal/mol(Ref 3). Was obtained by Henry(Ref 2) on heating 1-formamido-3-nitroguanidine with anhyd Na_2CO_3 in a small amt of water for 25 min according to the procedure developed by J.Thiele & K.Heidenreich,Ber **26**, 2599(1893) for the prepn of aminomethyltriazole. The reaction may be represented as follows:

HC–NH–NH + Na_2CO_3 ⟶
|| |
O HN=C·NH·NO_2

$2NaOH + CO_2$ + HC–NH–N
|| ||
N—— C·NH·NO_2

The formamido nitroguanidine was prepd by heating nitraminoguanidine with 3 times its weight of formic acid. The 3-nitramino-1,2,4-triazole may also be prepd by nitration of its parent compd, 3-aminotriazole

Refs: 1)Beil – not found 2)R.A.Henry, JACS **72**,5344(1950) 3)M.M.Williams et al, JPhChem **61**,264 & 266(1957) 4)Dr R.A. Henry,NOTS,China Lake,Calif; private communication 5056/RAH:jj, 18 Nov 1958

Note: Expl props of the above nitraminotriazole were determined by Henry et al and were supplied to SPIA for its propellant ingredients manual. OSRD 2014(1944) gives power & brisance less than PA and FI 72% PA

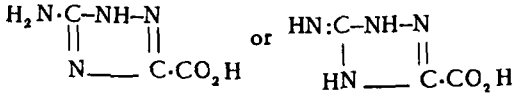
Addnl Refs on Aminotriazoles and Their Derivatives: a)J.Reilly & P.J.Drumm,JCS **1926**,1729–37[Prepn and props of aminopropyl -1,2,4-triazoles and their salts and derivs. One of the derivs described in this paper is expl. See also diazo-5-isopropyl-asym(1,2,4)-triazole, under D's] b)O.Dimroth & W. Michaelis,Ann **459**,39–46(1927) & CA **22**,423 (1928) (Discussion on intramolecular rearrangement of 5-amino-1,2,3-triazole derivatives:
$R^2HN{-}C{-}NR'{-}N$ ⇌ $R^1HN{\cdot}C{-}NR^2{-}N$)
|| || || ||
R·C—— N R·C—— N

c)J.Reilly & D. Madden,JCS **1929**,815–6 (Stability of diazonium salts of the triazole series) d)R.A.Henry,JACS **72**,5344(1950) & CA **46**,6088–9(1952) (Some derivs of 5-amino-triazole, including 5-nitro-amino-1,2,4-triazole) e)R.A.Henry & W.G.Finnegan, JACS **76**,291(1954) & CA **49**,10274(1955) (An improved proced for the deamination of 3-amino-1,2,4-triazole) f)J.E.deVries & E. St.Clair Gantz,JACS **76**,1008–10(1954) & CA **48**,7995(1954) (Spectrophotometric studies of dissociation constants of aminotriazoles, etc) g)M.R.Atkinson et al,JCS **1954**,4508–10 & CA **49**,15916–17(1955) (Derivs of 3-amino-1,2,4-triazole)

AMINOTRIAZOLECARBOXYLIC ACIDS AND DERIVATIVES

Aminotriazolecarboxylic Acids, $C_3H_4N_4O_2$, mw 128.09, N 43.74%. The following isomers and some derivatives are described in the literature:

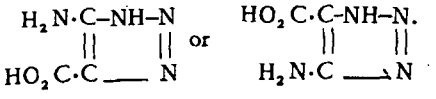
5-Amino-α-sym-triazole-3-carboxylic Acid or **5-Amino-1H-1,2,4-triazole-3-carboxylic Acid** [Called in Beil, 5-Imino-1.2.4-triazolin-carbonsäure-(3)],



Crysts with ½ H_2O(from w), decomp at 182–3° when heated rapidly. Can be prepd by prolonged heating of 4-amino-1,2,4-triazole-3,5-dicarboxylic acid with concd KOH on a water bath

Refs: 1)Beil **26**,311 2)T.Curtius et al,Ber **40**,818(1907) & CA **1**, 1418(1907)

5(4)-Amino-α-vic-triazole-4(5)-carboxylic Acid or **5(4)-Amino-1H-1,2,3-triazole-4(5)-carboxylic Acid**,



Crysts, mp 160–1°(decompn).

Can be prepd by treating 1H-vic-triazolo[d] pyrimidine-5,7-diol monohydrate with 25% NaOH soln in the manner described in Ref 2

Refs: See under next item

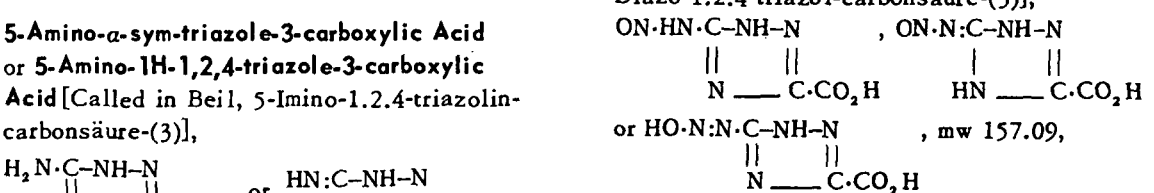
5(4)-Amino-α-vic-triazole-4(5)-carboxamide or **5(4)-Amino-1H-1,2,3-triazole-4(5)-carboxamide**,



mw 127.11, N 55.10%. Crysts, mp 226–7° (decompn). Can be prepd by treating 1H-vic-triazolo-[d]-pyrimidine-5,7-diol with NH_4OH in the manner described in Ref 2

Refs: 1)Beil **26** – not found 2)J.S.Webb, & A.S.Tomcufcik,USP 2,714,110(1955) & CA **50**, 12116(1956)

5-Nitrosamino-α-sym-triazole-3-carboxylic Acid or **5-Nitrosamino-1H-1,2,4-triazole-3-carboxylic Acid** [Called in Beil, 5-Nitrosimino-1.2.4-triazol-carbonsaure-(3) or 5-Diazo-1.2.4-triazol-carbonsäure-(3)],



N 44.59%. Solid, explg when dry at 120–130° as well as on friction. Can be prepd by treating the sodium salt of 5-amino-1,2,4-triazole-3-carboxylic acid with $NaNO_2$ in dil aq HCl at ca –3° (Refs 2 & 3)

Its *ethyl ester* is even more expl than the acid(Ref 3,p 8)

Refs: 1)Beil **26**,311 2)J.Thiele & W.Manchot,Ann **303**,54(1898) 3)W.Manchot & R. Noll,Ann **343**, 2–9(1905)

Nitraminotriazolecarboxylic Acid, $C_3H_3N_5O_4$ – not found in Beil or CA through 1956

AMINOTRIAZOLEDIONE AND DERIVATIVES

4-Amino-1,2,4-4H-triazole-3,5-dione, called

in Beil **4-Amino-3.5-dioxo-1.2.4-triazolin** or **Azodicarbonhydrazid**, OC–N=N , mw 114.07,
| |
$H_2N{\cdot}N$—CO
N 49.12%. Violet, very unstable pdr, mp–expl ca 72°. Was prepd by treating disilver salt of 4-aminourazol with ether soln of iodine in presence of BaO and MgO

Refs: 1)Beil **26**,(64) 2)R.Stollé,Ber **45**, 288(1912) & **46**,260(1913)

Nitrosaminotriazoledione, $C_2HN_5O_3$ and *Nitraminotriazoledione*, $C_2HN_5O_4$ – not found in Beil or CA through 1956

4-Amino-1,2,4-triazolon-(3)-imid. One of the Ger names for 4-Amino-3-imino-2,5-dihydro-α-sym-triazole

AMINOTRIAZOLOPYRIMIDINES AND DERIVATIVES

Aminotriazolopyrimidines, $C_4H_4N_6$, mw 136.12 N 61.75%. The following isomer is described in the literature:

7-Amino-α-vic-triazolo-[d]-pyrimidine or **7-Amino-1H-1,2,3-triazolo-[d]-4,6-diazine**, N=C(NH_2)–C–NH–N. Col crysts, decompg
| || ||
HC=N——C——N
without melting above 310°. Can be prepd by treating an aq soln of 4,5,6-triaminopyrimidine(acidified with some AcOH) with $NaNO_2$ in H_2O, followed by 20 min heating on a water bath(Ref 2). UV absorption spectra are given in Ref 3

Refs: 1)Beil – not found 2)R.O.Roblin et al,JACS **67**,292(1945) & CA **39**,1845(1945) 3)L.F.Cavalieri et al,JACS **70**,3875–8(1948) & CA **43**,1424(1948)

Nitrosaminotriazolopyrimidine, $C_4H_3N_7O$ and *Nitraminotriazolopyrimidine*, $C_4H_3N_7O_2$ – not found in Beil or CA through 1956

AMINOTRIAZOLOPYRIMIDINOLS AND DERIVATIVES

Aminotriazolopyrimidinols or *Aminohydroxypyrimidines*, $C_4H_4N_6O$, mw 152.12, N 55.25%. The following two isomers are described in the literature:

5-Amino-7-hydroxy-α-vic-triazolo-[d]-pyrimidine; 5-Amino-1H-1,2,3-triazolo-[d]-pyrimidin-7-ol or **5-Amino-7-hydroxy-1H-1,2,3-triazolo-[d]-4,6-diazine**, N=C(OH)–C–NH–N.
$H_2N{\cdot}C$=N——C——N
Col microscopic ndls, decomp above 300° without melting. Can be prepd by treating an alkaline aq soln of 2,4,5-triamino-6-hydroxypyrimidine with $NaNO_2$ + AcOH(Ref 2). Prepn of its hydrochloride is described in Ref 3, which also gives the UV absorption spectra
Refs: 1)Beil – not found 2)R.O.Roblin et al,JACS **67**,293(1945) & CA **39**,1846(1945) 3)L.F.Cavalieri et al,JACS **70**,3878–80(1948) & CA **43**,1424(1948)

7-Amino-α-vic-triazolo-[d]-pyrimidin-5-ol; 7-Amino-5-hydroxy-1H-1,2,3-triazolo-[d]-pyrimidine or **7-Amino-5-hydroxy-1H-1,2,3-triazolo-[d]-4,6-diazine**, N=C(NH_2)–C–NH–N. Its
HO·C=N——C——N
hydrochloride was prepd by treating an aq soln of 4,5,6-triamino-2-hydroxypyrimidine sulfate with $NaNO_2$ in HCl, followed by addition of HCl to the reaction mixture(Ref 2). Its UV absorption spectra are given in the same ref
Refs: 1)Beil – not found 2)L.F.Cavalieri et al, JACS **70**,3878–80(1948) & CA **43**,1424 (1948)

Nitrosaminotriazolopyrimidinol, $C_4H_3N_7O_2$ and *Nitraminotriazolopyrimidinols*, $C_4H_3N_7O_3$ – not found in Beil or CA through 1956

Aminotriethanol or Trihydroxyethylbenzene. See Triethanolamine

Aminotrimethylbenzenes. See Aminohemimellitene; Aminomesitylene and Aminopseudocumene

Aminotrinitrophenylmethylnitramine. See N-Methyl-N-nitro-2,4,6-trinitro-m-phenylenediamine, under Methylphenylenediamines

3-Amino-2,4,6-trinitrophenylnitramine. See N'-Nitro-2,4,6-m-phenylenediamine, under Phenylenediamines

AMINOURAZOLES AND DERIVATIVES

Aminourazoles, $C_2H_4N_4O_2$, mw 116.08, N 48.27%.

The following isomer is described in the literature:

4-Aminourazole; 4-Amino-3,5-dioxo-sym-triazolidine; 4-Amino-3,5-dioxo-1,2,4-triazolidine; (called *p-Urazine* in JCS **95**, 237) [Called in Beil, 4-Amino-3.5-dioxo-1.2.4-triazolin], OC-NH-NH. Crysts, mp 273 to 276°(decomp). Soly in w 0.032 g/100g at 0° and 4.02/100 g at 65°; diff sol in alc and insol in eth. Was first prepd in 1894 by Curtius and Heidenreich(Ref 2) but its correct structure was not established until 1907(Refs 3 & 4) (see note below). Several methods of prepn are listed in Beil(Ref 1) and the following two methods are described in detail in Ref 5: a)heating carbohydrazide, $OC(NH{\cdot}NH_2)_2$, with HCl and b)heating carbohydrazide N-carboxamide, , with HCl

Note: Curtius et al(Ref 2) assigned to p-urazine the structure HN-CO-NH and called it "Bishydrazicarbonyl" or "Diharnstoff" (Diurea)

Refs: 1)Beil **26**, 204,(60) & [109] 2)T. Curtius & K.Heidenreich,Ber **27**, 2684(1894) 3)M.Busch,Ber **40**, 2093(1907) 4)R.Stollé, JPrChem **75**, 422(1907) 5)InorgSynth **4** (1953),29–32

Nitrosaminourazole, $C_2H_3N_5O_3$ and *Nitraminourazole*, $C_2H_3N_5O_4$ – not found in Beil or CA through 1956

AMINOXYLENES AND DERIVATIVES

Aminoxylenes, $C_8H_{11}N$, mw 121.18, N 11.56%. All possible isomers are known and listed in Beil **12**,1101, 1103, 1106–7, 1111, 1131, 1134–5,1141,(478,480,482,483,487–8,490) & [601–4,606,613–14,618]

Azidoaminoxylenes, $C_8H_{10}N_4$ and *Diazidoaminoxylenes*, $C_8H_9N_7$ – not found in Beil or CA through 1956

Nitraminoxylenes, $(O_2N{\cdot}NH)C_6H_3(CH_3)_2$. One isomer, *eso-Nitramino-p-xylene* or *2,5-Dimethylphenylnitramine*, is mentioned in Beil **16**,675

Nitroaminoxylenes, $C_8H_{10}N_2O_2$. Various isomers are described in Beil **12**,1102–3, 1105–6,1110,1127–9,1132,1135,1140–2, (479,481,487–490) & [605–6,612–13,617–18, 620]

Nitronitraminoxylene, $C_8H_9N_3O_4$. One isomer, *5-nitro-4-nitramino-m-xylene*, is described in Beil **16**,[346]

Dinitroaminoxylenes, $C_8H_9N_3O_4$. Various isomers are described in Beil **12**, 1111, 1130, 1132, 1141, (479–82, 490) & [613]

Dinitronitraminoxylenes, $C_8H_8N_4O_6$, mw 256.18, N 21.87%. The following isomer is described in the literature:

3,5-Dinitro-2-nitramino-p-xylene or **4,6-Dinitro-2,5-dimethylphenyl-3-nitramine**, $HC{=}C(CH_3){-}C{\cdot}NH{\cdot}NO_2$ / $O_2N{\cdot}C{=}C(CH_3){-}C{\cdot}NO_2$. Ndls, mp 130° (decomp) and defgr when heated to higher temps. Sol in alc, ethyl acetate and ether. Was obtained(together with 3,5-dinitro-p-xylene-2-diazoniumnitrate) by treating 5-amino-p-xylene-2-sulfonic acid with HNO_3 (d 1.51) at −5°. Its expl props were not investigated

Refs: 1)Beil **16**,675 2)T.Zincke & E. Ellenberger,Ann **339**,207–9(1905)

Trinitroaminoxylenes, $C_8H_8N_4O_6$, mw 256.18, N 21.87%. The following isomer is described in the literature:

2,4,6-Trinitro-5-amino-m-xylene or **2,4,6-Trinitro-sym-m-xylidene** or **2,4,6-Trinitro-3,5-dimethylaniline**, $O_2N{\cdot}C{=}C(CH_3){-}C{\cdot}NO_2$ / $H_2N{\cdot}C{=}C(NO_2){-}C{\cdot}CH_3$

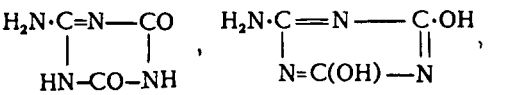
Yel crysts, mp 206°. Can be prepd by heating 2,4,6-trinitro-5-methoxy-1,3-dimethylbenzene for 2 hours with alc NH_3 in a sealed tube on a water bath(Ref 2) or by heating 5-bromo-2,4,6-trinitro-m-xylene with alc NH_3 at 130°(Ref 3)

Refs: 1)Beil **12**, 1133 2)J.J.Blanksma, Rec **21**, 329-30(1902) 3)Ibid, **25**, 374(1906)

Trinitronitraminoxylenes, $(O_2N{\cdot}HN)C_6(CH_3)_2(NO_2)_3$, mw 301.18, N 23.26% – Not found in Beil or in CA through 1956

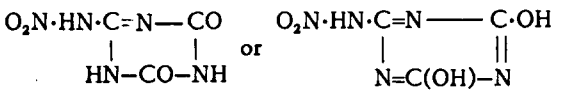
AMMELIDE AND DERIVATIVES

Ammelide; 6-Amino-s-triazine-2,4 diol or 5,6-Dihydro-6-imino-s-triazine-2,4(1H, 3H)-dione (called in Beil 4,6-Dioxo-2-iminohexahydro-1.3.5-triazin; Isocyanürsäuremonoimid; 2.4-Dioxy-6-amino-1.3.5-triazin or Melanurensäure)



mw 128.09, N 43.74% Prepn and props are in Beil **26**, 243, (73) & [132]
[See also CA **28**, 4706 (1934); **31**, 3490 (1937); **32**, 502 (1938); **40**, 7072 (1946); **43**, 698(1949); **44**, 1136(1950); **46**, 6163(1952); **50**, 1041, 9062 & 9151 (1956)]

Nitroammelide or 6-Nitramino-s-triazin-2,4-diol (Designated by Cason as TM-1),



mw 173.10, N 40.46%, OB to CO_2 – 32.4% OB to CO – 4.6% Col powd, decompg sharply without melting at 248°; appreciably sol in w(hydrolyzing slowly in cold and rapidly in hot w with formation of cyanuric acid). Was prepd by Cason on treating triacetylmelamine with a 1/1 mixt of fuming HNO_3 and Ac_2O at 25° for 5 hrs (Refs 2 and 3). The originally assigned empirical formula, $C_9H_9N_{15}O_{12}$ (Ref 2), proved to be erroneous and should be $C_3H_3N_5O_4$ (Ref 3). When analyzing a sample by combustion, it is necessary, in order to avoid explosion, to mix it thouroughly with copper oxide (Ref 2)

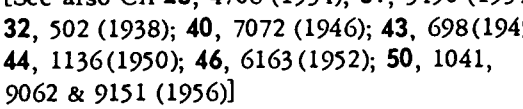
When nitroammelide was treated with ammonia an amorphous product contg 47.4% N was obtained

As nitroammelide and its ammonium salt are high nitrogen compds and as their OB to CO is close to zero, they may be of possible value in cool propellent compositions

Refs: 1)Beil-not found 2)J.Cason, JACS **69**, 497(1947) & CA **41**, 4158(1947) 3)E.R. Atkinson, JACS **73**, 4443(1951) & CA **47**, 138(1953)

AMMELINE AND DERIVATIVES

Ammeline; 4,6-Diamino-s-triazin-2-ol or 3,4,5,6-Tetrahydro-4,6-diimino-s-triazin-2(1H)-one, called in Beil 6-Oxo-2.4-diiminohexahydro-1.3.5-triazin; Isocyanürsäurediimid or 2-Oxy-4.6-diamino-1.3.5-triazin,



mw 127.11, N55.10% Prepn and props are in Beil **26**, 244, (74) & [132]

[See also CA **32**, 502(1938); **40**, 7072(1946); **41**, 1179 & 4003(1947); **43**, 698, 2786 & 5809(1949); **44**, 2158(1950); **46**, 6163(1952); **48**, 9413 & 9414(1954); **49** 14008, 14816 & 15734(1955); & **50**, 1041, 9062, 9151 & 10109(1956)]

Mononitroammelines, $C_3H_4N_6O_3$, mw 172.11, N48.83% – not found in Beil or CA through 1956

Dinitroammeline, called by Atkinson **N^4,N^6-**

Dinitroammeline, $O_2N{\cdot}HN{-}C(=N{-}C{\cdot}OH){=}N{-}C(NH{\cdot}NO_2){=}N$,

mw 217.11, N 45.16%, OB to CO_2 -18.4%, OB to CO + 3.7%. Wh crysts decompg sharply at 228°; can be detonated by a sharp blow (Ref 3). It is sol in w (slow hydrolysis) and in aq Na bicarbonate solns; insol in organic solvents. It was first prepd by Cason, but not properly identified (Ref 2). Atkinson (Ref 3) improved the method of prepn and identified the product as N^4, N^6-dinitro-ammeline. For its prepn, the finely ground melamine was added to a 1/1 mixt of HNO_3 and Ac_2O at 0-5° and stirred for 2 hrs. The resulting solid was filtered off and washed with AcOH and then with w. The purification was achieved by dissolving the product in aq Na bicarbonate soln followed by filtering and acidification with HCl as indicated in Ref 3. The yield was ca 50%

Dinitroammeline is an explosive, fairly stable in storage and comparable to tetryl in impact sensitivity

Following are the results of some tests conducted at PicArsn, Dover, NJ: *heat of combustn* at Cv 1673 cal/g; *explosion temp* (5 sec test) 275°; *impact sensitivity* 9" (PicArsn app, 2 kg wt, 0.014g sample) and 37cm (BurMines app, 2 kg wt); 100° *heat test* (%loss of wt) 0.14 in 1st 48 hrs, 0.0 in 2nd 48 hrs and no expln in 100 hrs; *100° vacuum stability* test 0.88cc gas evolved in 49 hrs per 5g sample

Refs: 1)Beil – not found 2)J.Cason, JACS **69**, 496(1947) & CA **41**, 4158(1947) 3)E.R. Atkinson, JACS **73**, 4443(1951) & CA **47**, 138(1953) 4)PicArsn, GenLab; private communication 5)Corinfskii, ZavodLab **12**, 418-21(1941)(Detn of ammeline by pptn as its picrate after separation of melamine)

Ammeline Picrate or Diamino-hydroxy-triazine Picrate, $C_3H_5N_5O{\cdot}C_6H_3N_3O_7{\cdot}H_2O$, golden-yel crysts, mp 266°. It loses water of crystn only when it is heated in vacuo over P_2O_5 at 140°. Can be prepd by treating ammeline with hot concd picric acid to which some oxalic acid is added to facilitate the soln of ammeline

Refs: 1)Beil – not found 2)A.Ostrogovich and G.H.Gheorghiu, Gazz **60**, 648(1930) & CA **25**, 957(1931)

Ammiachno-selitrennyi Porokh. One of the early Russian compns used as a propellant and as a blasting expl: AN 85 & powdered carbon 15%

Ref: N.N.Ushakov & I.V.Lebedev, "Manufacture of Explosives", Gosizdat, Leningrad(1934)

Ammiak. Russian for Ammonia

Ammiak or Ammoniakat. Ger names for Ammine

AMMINE OR AMMONIATE AND COORDINATION
(Ammoniakat in Ger and Ammoniacate in Fr)

The complex compds in which ammonia (NH_3) functions as a neutral group are very numerous and frequently very stable. They belong to the so called "coordination" compds, known since beginning of the 19th century but not properly systematized and explained until 1893 when Alfred Werner introduced his theory. In his theory, now nearly universally adopted(with slight modification, such as by Sidgwick and Lowry, etc), there are two types of valencies: a) *primary* (principal, main, ordinary or ionic) and b) *secondary* (auxiliary or non-ionic), now called "coordinate covalence". The difference between ionic and non-ionic valences is not as great as Werner at first supposed, and is one of degree rather than of kind. The same kind of anion radical or molecule may be held by either or both types of valence. There is, however, an upper limit for both types of linkages. In case of non-ionic linkages, the maximum number of atoms, radicals or molecular groups, which can be directly connected with central atom(metal), is called the "coordination number" of this atom. This number is for most metals six, but it can be also four, five, three and two for the same metal(as for instance Co). For Mo the "coordination number" was reported to be eight

In writing the formula of a "coordinated complex", the "coordinated group", called "nucleus" or the "first sphere" is enclosed in square brackets, while the acid radicals are placed outside in the so-called "second or ionization sphere". For instance in the formula $[M\,R_m]\,X_n$, M is a metal (such as Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni or Zn), R is a non-ionic(neutral) radical(such as NH_3, H_2O, ethylenediamine, diethylenetriamine, pyridine, etc), m is "coordination number" of M, X is a negative(acidic) radical(such as Cl^-, CN^-, NO_2^-, NO_3^-, ClO_3^-, ClO_4^-, IO_3^-, etc) and n is number of monovalent acidic radicals. An example of this type of complex is the compd $[Co(NH_3)_6](NO_3)_3$, in which the electrovalency of the nucleus is 3 (positive), the same as for the Co atom. If one or several NH_3 groups are replaced by neutral groups, such as H_2O, the electrovalence of the nucleus remains the same, as for instance in the compd $[Co(NH_3)_5\,H_2O](NO_3)_3$ If however, one or several negative(acidic) radicals replace neutral radicals in the nucleus, the electrovalency of the nucleus is reduced– one positive valence less for each negative valence introduced. For instance in the compd $[Co(NH_3)_5\,Cl](NO_3)_2$ the electrovalency of the nucleus is two and in the compd $[Co(NH_3)_3\,Cl_3]^\circ$ it is equal to zero. If the negative valency in the nucleus exceeds the positive valency of the central metal, as in the compd $[Co(NH_3)_2(NO_2)_4]\text{–}K^+$, the nucleus as a whole becomes negative and has to be associated with a corresponding number of positive ions(such as K) outside

In naming the complexes, the constituents of the nucleus are taken first(starting with the acid radical or extra neutral radical, if any present, and followed by the neutral group and metal) and then comes the name of the radical outside the bracket. For instance, the compd $[Co(NH_3)_5\,Cl](NO_3)_2$ is called "chloropentammine cobalt (III) nitrate" and the compd $[Co(NH_3)_5\,H_2O](NO_3)_3$ is "aquopentamminecobalt (III) nitrate", etc

Ammonia can form many "coordinated complexes" with the above mentioned metals and these complexes, called "ammines" or "ammoniates" are among the most important. Usually divalent metals form complexes with four NH_3 groups, while trivalent metals usually coordinate with six NH_3 groups. There are, however, many exceptions

Many metal ammines are explosive and those which were investigated in various

countries from that point of view are listed in tables A,B,C,D,E,F&G

It should be noted that until Werner's time, complexes were usually designated by names denoting color: *luteo* for yellow, *purpureo* for purple-red and *roseo* for pink. Some compds were called *praseo* to designate their green color

As a rule, the colors of Co and Cr ammines are largely independent of the nature of the metal, but depend on the number of neutral and acidic radicals. For instance hexammines are yellow(luteo), chloropentammines are purple-red(purpureo) and aquopentammines are pink(roseo). The compd $[Co(NH_3)_4Cl_2]Cl$ is green(praseo) and the compd $[Co(NH_3)_4(NO_2)_2]Cl$ is called the *flavo- or coceo salt*

Preparation of ammines. Most ammines can be prepd by passing NH_3 gas into a concd soln of a metal salt, such as chlorate, iodate, perchlorate, etc, as for instance:

$$Cd(ClO_3)_2 + 6NH_3 \rightarrow [Cd(NH_3)_6](ClO_3)_2$$

$$Cu(IO_3)_2 + 4NH_3 + 2H_2O \rightarrow [Cu(NH_3)_4](IO_3)_2 \cdot 2H_2O$$

$$Co(ClO_4)_3 + 6NH_3 + H_2O \rightarrow [Co(NH_3)_6](ClO_4)_3 \cdot H_2O$$

A detailed description of prepn of hexamminecolbalt (III) nitrate is given in Refs 33 and 41 and prepn of some other ammines may be found in Refs 3,7,9,10,11,11a,12,13, 14,15,16,17,19,20,21,22,24,25,26,30,31,34, 35,40,43,47,48,54,55,56,57,58,59,62,63, and 65

Explosive properties of metal ammines were studied by many investigators and references are given in the tables A,B,C etc

The NavOrd Rept 5639 (1957) (Ref 69) was not available to be utilized for this work

It seems that the most important ammines from the point of view of explosiveness and stability are chlorates and perchlorates. Their potential energy and velocity of detonation are usually between those of primary expls, such as MF and LA, and aromatic nitro compds, such as TNT. The chlorates are definitely more sensitive than the perchlorates. The expln temps of some ammoniates were reported to be higher for confined than for unconfined samples

According to Friederich and Vervoorst (Ref 11, p 49) the chlorate and perchlorate complexes decomp with explosive violence, when heated, and the reactions proceed as follows:

$$[M(NH_3)_4](ClO_3)_2 \rightarrow MCl_2 + 2N_2 + 6H_2O$$

$$[M(NH_3)_6](ClO_3)_2 \rightarrow MCl_2 + 3N_2 + 6H_2O + 3H_2$$

$$[M(NH_3)_4](ClO_4)_2 \rightarrow MCl_2 + 2N_2 + 6H_2O + O_2$$

$$[M(NH_3)_6](ClO_4)_2 \rightarrow MCl_2 + 3N_2 + 8H_2O + H_2,$$

where M represents a suitable metal

Uses: Some metal ammines were patented for use as primary explosives. This excludes those which are unstable at ambient temps (evolve NH_3) and are too hygroscopic

(See Tables A to G, pp A277 to A282)

References on Ammines: 1) A.Werner, ZAnorgChem **3**, 267-330(1893), "Beitrag zur Konstitution anorganischer Verbindungen," 2) R.Escales, SS **2**, 413-14(1907)[According to R.Escales, the "Kupfernitrat-Ammoniak", listed here in table E as "tetramminecopper (II) nitrate" and abbreviated as CuTAN, was proposed in 1887 by A.Nobel, BritP 16920, for use as follows: a) CuTAN 4, AN 91 & TNT 5% b) CuTAN 5, NG 30-35, Na nitrate 30.5 & meal 39.5% c)CuTAN 10, NG 48, CC 2, $NaNO_3$ 30, WM 9.6 & soda 0.4%.The first of these belonged to the class of "AN explosives", the second to "carbonites" and the third to "gelatin-dynamites". The firm G.Roth of Austria used the compns contg CuTAN 30-40, K nitrate 42-25, sulfur 10-7.0 & Al 18-28% for filling some blasting caps] 3)R.Salvatori, Chem Ztr **1910**, 1444 & CA **5**, 1568(1911)

TABLE A

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No.	Name of Compound	Formula	Color	Density g/cc	Melting Point, °C	Avg Expl Temp	Impact Sensitivity, cm: 2 kg wt	Impact Sensitivity, cm: 1 kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Cd1	Diamminecadmium (II) Azide	$[Cd(NH_3)_2](N_3)_2$	Note: Decomp on slow heating and expl violently, when heated on a metallic spatula										Dissolves in H_2O with decompn	22, pp 212 - 13
Cd2	Tetramminecadmium (II) Bromate	$[Cd(NH_3)_4](BrO_3)_2$	Col	2.53	–	192	2.5	–	–	–	–	–	–	7, pp 43 & 51
Cd3	Tetramminecadmium (II) Chlorate	$[Cd(NH_3)_4](ClO_3)_2$	Col	–	dec >120	195	–	40	67	–	–	–	–	7, p 49 & 11, pp 116 & 144
Cd4	Tetramminecadmium (II) Iodate	$[Cd(NH_3)_4](IO_3)_2$	Col	3.23	–	218	–	–	–	–	–	–	–	7, pp 43, 45, & 54 - 6
Cd5	Hexamminecadmium (II) Bromate	$[Cd(NH_3)_6](BrO_3)_2$	Col	–	–	192	15	–	–	–	–	–	–	7, p 43
Cd6	Hexamminecadmium (II) Chlorate	$[Cd(NH_3)_6](ClO_3)_2$	Col	1.78	–	205	15	46	57	Note: It loses NH_3 even at RT			–	7, pp 42 - 3 & 49 & 11, pp 66 & 144
Cd7	Hexamminecadmium (II) Perchlorate	$[Cd(NH_3)_6](ClO_4)_2$	Col	–	–	270	–	53	59	–	–	–	–	11, pp 66 & 144
Cr1	Triazidotriammine-chromium (III)	$[Cr(NH_3)_3(N_3)_3]^a$	Dirty olive	–	–	250 - 300 (deton)	Less sensitive than corresp Co complex		–	–	–	–	Insol in w, alc & eth.	65, pp 175 - 8 & 181
Cr2	Aquopentammine-chromium (III) Perchlorate	$[Cr(NH_3)_5H_2O](ClO_4)_3$	Orange	–	–	ca 300	Not sensitive		–	–	–	–	Soly in H_2O at RT: 10.9 g/ 100 cc of soln; insol in alc & eth	65, pp 178 - 9 & 180
Cr3	Azidopentammine-chromium (III) Nitrate	$[Cr(NH_3)_5N_3](NO_3)_2$	Dk red	–	–	250 - 300 (sizzles off with yel flame)	Not sensitive		–	–	–	–	Soly in H_2O at 20°: 0.762 g/100 cc of soln; insol in in alc & eth	65, pp 175 - 6 & 181
Cr4	Azidopentammine-chromium (III) Perchlorate	$[Cr(NH_3)_5N_3](ClO_4)_2$	Red-violet	–	–	250 - 300 (expl)	Less sensitive than corresp Co complex		–	–	–	–	Soly in w at 20°: 4.32 g/100cc of soln; insol in alc & eth	65, pp 175 - 6 & 180
Cr5	Chloropentammine-chromium (III) Azide	$[Cr(NH_3)_5Cl](N_3)_2$	Red	Note: Heated on a metal spatula it melts and then puffs off with report									Sol in H_2O	22, p 216
Cr6	Hexamminechromium Azide	$[Cr(NH_3)_6](N_3)_3$	Lt yellow	Note: Heated on a metal spatula it expl with loud report								–	Sol in H_2O	22, p 215 - 16
Cr7	Hexamminechromium (III) Nitrate	$[Cr(NH_3)_6](NO_3)_3$	–	–	–	265	32	–	–	97	0.20 g of MF	–	–	46, p 376
Cr8	Hexamminechromium (III) Perchlorate	$[Cr(NH_3)_6](ClO_4)_3$	Note: Its method of prepn is mentioned in Ref 65, but no props are given									–	–	65, p 180

TABLE 8

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No	Name of Compound	Formula	Color	Density, g/cc	Melting Point, °C	Av Expl Temp	Impact sensitivity, cm 2 kg wt	Impact sensitivity, cm 1 kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Co 1	Triazidotriammine-cobalt (III)	$[Co(NH_3)_3(N_3)_3]^\circ$	Green or blue -blk	–	dec >100	187° (deton)	Very sensitive		Note: May be detonated by rubbing the crysts even under water				Soly in H_2O at RT: 0.08 g/100 cc; sl sol in dioxane & acetone; insol in other solvents	57, pp 245–52
Co 2	Trinitrotriammine-cobalt (III)	$[Co(NH_3)_3(NO_2)_3]^\circ$	–	–	dec >158	305	44	–	–	–	–	–	–	21, p 1652; 9, p 2496; 46, p 376; 49, p 381
						Note: According to Ref 9, evolution of gas begins at 158 and at 162 the decompn becomes violent								
Co 3	Tetramminecobalt (III) Chlorate	$[Co(NH_3)_4](ClO_3)_3$	Dirty green	–	Unstable		–	–	–	–	–	–	–	7, pp 49–50
Co 4	Diazidotetrammine-cobalt (III) Azide (cis)	$[Co(NH_3)_4(N_3)_2]N_3$	Blk	–	150 (dec)	ca 182 (deton with sharp report)	Very sensitive		–	–	–	–	Soly in H_2O: 8.4 g/100 cc of soln at 20°; sol in 95% alc 0.028 g/ 100 cc at 20°	55, pp 235–6 239–40 & 243
Co 5	Diazidotetrammine-cobalt (III) Azide (trans)	Same as above	Brn -blk	–	170 (dec)	ca 182 (deton with sharp report)	Very sensitive		–	–	–	–	Soly in H_2O: 3.04 g/100 cc of soln at 20°; sol in 95% alc 0.029 g/ 100 cc at 20°	55, pp 235–6 239–40 & 243
Co 6	Diazidotetrammine-cobalt (III) Iodide (cis)	$[Co(NH_3)_4(N_3)_2]I$	Violet -brn	–	dec >80	180 (deton)	Not sensitive		Note: Detonation of both cis & trans iodide is attributed to formation of nitrogen iodide				Soly in H_2O at 20°: 2.57 g/100 cc of soln; v sl sol in 95% alc	55, pp 235–6 239–40 & 244
Co 7	Diazidotetrammine-cobalt (III) Iodide (trans)	Same as above	Brn -blk	–	dec >80	180 (deton)	Not sensitive						Soly in H_2O at 20°: 3.41 g/ 100 cc of soln; v sl sol in95% alc	55, pp 235–6, 239–40 & 243
Co 8	Diazidotetrammine-cobalt(III) Nitrate (cis)	$[Co(NH_3)_4(N_3)_2]NO_3$	Viol-brn -blk	–	dec >150	200 (sizzles off with yel flame)	Not sensitive		–	–	–	–	Soly in H_2O at 20°: 2.73 g/100 cc of soln; v sl sol in 95% alc	55, pp 235–6 239–40 & 244

TABLE C

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No	Name of Compound	Formula	Color	Density, g/cc	Melting Point, °C	Av Expl Temp	Impact sensitivity, cm 2 kg wt	Impact sensitivity, cm 1 kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Co 9	Diazidotetrammine-cobalt (III) Nitrate (trans)	$[Co(NH_3)_4(N_3)_2]NO_3$	Violbrn-blk	–	dec >150	180° (sizzles off with yel flame)	Not sensitive		–	–	–	–	Soly in H_2O at 20°: 1.71 g/100 cc of soln; v sl sol in 95% alc	55, pp 235–6, 239–40 & 243
Co 10	Diazidotetrammine-cobalt (III) Perchlorate (cis)	$[Co(NH_3)_4(N_3)_2]ClO_4$	Violet-blk	–	dec >180	200 (puffs off with yel flame)	Very sensitive		–	–	–	–	Soly in H_2O at 20°: 3.02 g/100 cc of soln; v sl sol in 95% alc	55, pp 235–6, 239–40 & 244
Co 11	Diazidotetrammine-cobalt (III) Perchlorate (trans)	Same as above	Green-blk	–	dec >180	220 (puffs off with yel flame)	Very sensitive		–	–	–	–	Soly in H_2O at 20°: 1.70 g/100 cc of soln; v sl sol in 95% alc	55, pp 235–6, 239–40 & 243
Co 12	Thiocyanatotetrammine-cobalt (III) Perchlorate	$[Co(NH_3)_4(SCN)](ClO_4)_2$	–	–	–	325	55	–	–	48	–	–	–	46, p 376
Co 13	Dithiocyanatotetrammine-cobalt (III) Perchlorate	$[Co(NH_3)_4(SCN)_2]ClO_4$	–	–	–	335	35	–	–	33	–	–	–	46, p 376
Co 14	Aquopentamminecobalt (III) Chlorate Monohydrate (Roseocobaltiammine chlorate)	$[Co(NH_3)_5,H_2O]-(ClO_3)_3,H_2O$	Pink	–	>100 (decomp)	130–150	Not sensitive		–	–	–	–	Soly in H_2O at 18°: 105.8%	3, p 1444
Co 15	Aquopentamminecobalt (III) Perchlorate Monohydrate (Roseocobaltiammine Perchlorate)	$[Co(NH_3)_5,H_2O]-[ClO_4)_3\ H_2O$	Pink	–	110 (decomp)	ca 250	Not sensitive		–	–	–	–	Soly in H_2O at 18°: 7.4%	3, p 1444
Co 16	Azidopentammine-cobalt (III) Azide	$[Co(NH_3)_5N_3](N_3)_2$	Red	–	dec >120	194–200 (deton)	Not sensitive		–	–	–	–	Soly in H_2O at 20°: 3.82 g/100 cc of soln; insol in alc & eth	54, pp 330–1, 338 & 341
Co 17	Azidopentammine-cobalt (III) Chromate	$[Co(NH_3)_5N_3]CrO_4$	Red	–	ca 300 (decrepitates)	ca 500 (sizzles off with yel flame)	Not sensitive		–	–	–	–	Soly in H_2O at 20°: 0.06 g/100 cc of soln; insol in alc & eth	54, pp 330–1, 339 & 343
Co 18	Azidopentammine-cobalt (III) Nitrate	$[Co(NH_3)_5N_3](NO_3)_2$	Red	–	200 (dec)	300 (defgr)	–	–	–	–	–	–	Soly in H_2O at 20°: 0.611 g/100 cc of soln; insol in alc & eth	54, pp 330–1, 339 & 342
Co 18a	Chloritopentammine-cobalt (III) Nitrate	$[Co(NH_3)_5(ClO_2)](NO_3)_2$	–	–	(See the original paper for description) Note: Its chromate, bichromate and picrate were also prepd									62

TABLE D

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No	Name of Compound	Formula	Color	Density g/cc	Melting Point °C	Avg Expl Temp	Impact Sensitivity, cm 2kg wt	Impact Sensitivity, cm 1kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Co19	Chloropentamminecobalt (III) Chlorate("Purpureocobaltiammine Chlorate")	$[Co(NH_3)_5Cl](ClO_3)_2$	Purple	Note: It was prepd by treating purpureocobaltimmine chloride with silver chloride. Its explosive props are not given in Ref 3								–	–	3, p 1444
Co20	Chloropentamminecobalt (III) Chlorite	$[Co(NH_3)_5Cl](ClO_2)_2$	–	–	–	(expl)	–	–	–	–	–	–	–	10, p 522
Co21	Chloropentamminecobalt (III) Perchlorate	$[Co(NH_3)_5Cl](ClO_4)_2$	Red	–	–	320	21	–	–	73	0.20 of MF	–	–	46, p 376
Co21a	Purpureocobaltiammine Perchlorate	Formula not given	Red-brn	–	–	200	Note: This compd seems to be identical with the Co21 compd						Soly in H_2O at 18°: 11%	3, p 1445
Co21b	Formatopentammine-cobalt (III) Nitrate	$[Co(NH_3)_5](HCO_2)](NO_3)_2$					(See the original paper for description)							47 and 56
Co22	Nitratopentamminecobalt (III) Nitrate	$[Co(NH_3)_5NO_3](NO_3)_2$	Red	–	–	310	48	–	–	84.5	0.24 of MF	–	–	46, p 376
Co23	Hexamminecobalt (III) Chlorate, Monohydrate or "Luteocobaltiammine Chlorate"	$[Co(NH_3)_6](ClO_3)_3 \cdot H_2O$	Yellow	–	–	120-150	–	–	–	–	–	–	Soly in H_2O at 18°: 7.87% and more at higher temp	3, p 1444
Co24	Hexamminecobalt (III) Chlorite	$[Co(NH_3)_6](ClO_2)_3 \cdot H_2O$	Orange-Yel	Note: Explodes on percussion					–	–	–	–	–	10, p 522
Co25	Hexamminecobalt (III) Hexanitrocobaltate	$[Co(NH_3)_6]Co(NO_2)_6$	–	–	Unstable		85	–	–	81	0.20 of MF	–	–	46, p 376 & 49, p 381
Co26	Hexamminecobalt (III) Iodate	$[Co(NH_3)_6](IO_3)_3$	Yellow	–	–	355	100	–	–	35	0.24 of MF	–	–	46, p 376
Co27	Hexamminecobalt (III) Nitrate	$[Co(NH_3)_6](NO_3)_3$	–	–	–	295	50	–	–	85	0.27 of MF	–	–	41, p 218 & 46, p 376
Co28	Hexamminecobalt (III) Perchlorate, Monohydrate or "Luteocobaltiammine Perchlorate"	$[Co(NH_3)_6](ClO_4)_3 \cdot H_2O$	Yellow	–	ca 110	–	–	–	–	–	–	–	Soly in H_2O at 18°: 0.967% and more at higher temps	3, p 14444
					Note: Looses H_2O and forms the anhydrous salt (See next item)									
Co29	Hexamminecobalt (III) Perchlorate	$[Co(NH_3)_6](ClO_4)_3$	Orange-red	–	dec >110	360	18	–	–	93	0.25 of MF	–	–	3, p 1444 & 46, p 376
Co30	Hexamminecobalt (II) Perchlorate	$[Co(NH_3)_6](ClO_4)_2$	–	–	–	275	–	55	87	–	–	–	–	11, pp 66 & 144
Cu1	Diamminecopper (II) Azide	$[Cu(NH_3)_2](N_3)_2$	Dk green	–	–	202 (hot block)	–	5-10	–	Less brisant than CuN_6	Init by coml detonrs	–	Insol in neutral solvents, water & methanol; decomp by hot water	22, pp 209-10; 35, p 343
Cu2	Diamminecopper (II) Nitrate	$[Cu(NH_3)_2](NO_3)_2$	–	Explodes on heating or on impact					–	–	–	–	–	11e, pp 479-80
Cu3	*Tetrammineocopper (II) Azide*	$[Cu(NH_3)_4](N_3)_2$	Blue	–	–	202 (hot block)	–	20	Note: It looses NH_3 in air			–	Sl sol in alc, sol in aq NH_3, decomp by H_2O; insol in neutral solvents	22, p 210; 34, p 336

TABLE E

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No	Name of Compound	Formula	Color	Density g/cc	Melting Point °C	Avg Expl Temp	Impact Sensitivity, cm 2kg wt	Impact Sensitivity, cm 1kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Cu4	Tetramminecopper (II) Bromate	$[Cu(NH_3)_4](BrO_3)_2$	Blue	2.31	–	140	2	–	–	–	–	–	–	7, pp 43 & 50
Cu5	Tetramminecopper (II) Chlorate	$[Cu(NH_3)_4](ClO_3)_2$	Blue	1.81	ca 125	ca160	15	39	80	–	–	–	–	7, pp 42 & 46; 11, pp 66 & 144
				Note: This compd is able to initiate TNT										
Cu5a	Tetramminecopper (II) Iodate	$[Cu(NH_3)_4](IO_3)_2$		(See description in the original paper)					–	–	–	–	–	15, p 329
Cu6	Tetramminecopper (II) Nitrate	$[Cu(NH_3)_4](NO_3)_2$	Purple	–	–	ca330 (ignited by a flame but not by a fuse)	19	–	–	41	0.24 MF or 0.19 tetryl	–	–	33 and 46, p 376
	Note: This sensitive explosive may form by the action of AN on Cu in presence of moisture (See Ref 33)													
Cu7	Tetramminecopper (II) Perchlorate	$[Cu(NH_3)_4](ClO_4)_2$	–	–	–	260	–	50	66	–	–	–	–	11, pp 66 & 144
Cu8	Pentamminecopper (II) Iodate	$[Cu(NH_3)_5](IO_3)_2$	Blue-green	2.72	–	210	–	–	–	–	–	–	–	7, pp 43 & 51
Cu9	Hexamminecopper (II) Chlorate	$[Cu(NH_3)_6](ClO_3)_2$	Blue	–	Decomp at room temp		–	–	–	–	–	–	–	7, pp 46-7
Mn1	Diamminemanganese (II) Fulminate	$[Mn(NH_3)_2](OCN)_2$	Yellow	–	dec	defgr weakly	–	Note: It is unstable and loses NH_3 in air				–	–	13, pp 2750 & 2751
Ni1	Pentamminenickel (II) Iodate	$[Ni(NH_3)_5](IO_3)_2$	Lt violet	2.97	dec	219		Note: This salt exists also as a trihydrate					–	7, pp 43, 45 & 52-3
Ni2	Hexamminenickel (II) Azide	$[Ni(NH_3)_6](N_3)_2$	Lt blue	–	turns green	expl viol	–	Note: It loses NH_3 at RT			–	–	Sol in H_2O	22, p 217
Ni3	Hexamminenickel (II) Bromate	$[Ni(NH_3)_6](BrO_3)_2$	Lt violet	1.99	dec	195	–	Note: It loses NH_3 at RT			–	–	–	7, pp 43 & 50
Ni4	Hexamminenickel (II) Chlorate	$[Ni(NH_3)_6](ClO_3)_2$	Blue	1.52	dec	210	25	47	68	Note: When ignited burns slowly. It would not initiate tetryl			–	7, pp 43, 45, 51 - 2; 11, pp 66 & 144
Ni4a	Hexamminenickel (II) Iodate	$[Ni(NH_3)_6](IO_3)_2$		(See description in the original paper)					–	–	–	–	–	15, p 329
Ni5	Hexamminenickel (II) Perchlorate	$[Ni(NH_3)_6](ClO_4)_2$	Blue	1.57	dec	275	–	55	91	–	–	5300 at d 1.36 (pressed)	–	11, pp 66, 85, & 144

TABLE F

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

No	Name of Compound	Formula	Color	Density g/cc	Melting Point, °C	Avg Expl Temp	Impact Sensitivity, cm 2 kg wt	Impact Sensitivity, cm 1 kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton, m/sec	Solubility in Various Solvents	References
Zn1	Diamminezinc (II) Fulminate	$[Zn(NH_3)_2](OCN)_2$	Col	–	dec	defgr	Note: It decrepitates when heated in a flame; the salt is stable even at 100						–	13, p 2750
Zn1a	Diamminezinc (II) Nitrite	$[Zn(NH_3)_2](NO_2)_2$		(See description in the original paper)					–	–	–	–	–	30
Zn2	Tetramminezinc (II) Bromate	$[Zn(NH_3)_4](BrO_3)_2$	Col	2.27	dec	170	2	–	–	–	–	–	–	7, pp 43, 45, & 50 - 1
Zn3	Tetramminezinc (II) Chlorate	$[Zn(NH_3)_4](ClO_3)_2$	Col	1.84	dec	220	15	69	79	–	–	–	–	7, pp 42 - 3, 45 & 48; 11, pp 66 & 144
Zn4	Tetramminezinc (II) Iodate	$[Zn(NH_3)_4](IO_3)_2$	Col	2.82	dec	214	–	–	–	–	–	–	–	7, pp 43, 45, & 53
Zn4a	Tetramminezinc (II) Nitrate	$[Zn(NH_3)_4](NO_3)_2$		(See the original paper)			–	–	–	–	–	–	–	23, p 526
Zn5	Tetramminezinc (II) Perchlorate	$[Zn(NH_3)_4](ClO_4)_2$	Col	–	dec	305	–	70	70	–	–	–	–	11, pp 66 & 144
Zn6	Hexamminezinc (II) Chlorate	$[Zn(NH_3)_6](ClO_3)_2$	Col	–	Note: Loses NH_3 at RT and forms the corresponding tetrammine (see above)									7, pp 44 & 48 - 9
Zn7	Hexamminezinc (II) Nitrate	$[Zn(NH_3)_6](NO_3)_2$		(See the original paper)			–	–	–	–	–	–	–	23, p 526

TABLE G

Some Properties (Approximate Values) of Explosive Metal Ammines Compared with Some Standard Explosives

Name of Compound	Formula	Color	Density g/cc	Melting Point, °C	Avg Expl Temp	Impact Sensitivity, cm 2 kg wt	Impact Sensitivity, cm 1 kg wt	Trauzl Test TNT = 100	Sand Bomb Test TNT = 100	Req for Initiation, g Primary Expl	Vel of Deton. m/sec	References
Cyclonite (RDX)	$C_3H_6(N \cdot NO_2)_3$	White	1.82	204	260	32	100+	157	125	0.19 of MF	8180 at d 1.65	71, p 76
Lead Azide (not dext)	$Pb(N_3)_2$	White	4.80	Dec	340	10	32	39	40	–	5180 at d 4.0	71, p 173
Mercuric Fulminate	$Hg(OCN)_2$	White	4.43	Dec	210	5	20	51	49	–	5000 at d 4.0	71, p 187
Tetryl	$(O_2N)_3C_6H_2N\begin{matrix}\diagup CH_3\\ \diagdown NO_2\end{matrix}$	Yellow	1.73	130	257	26	100+	125	113	0.20 of MF	7850 at d 1.71	71, p 300
Trinitrotoluene (TNT)	$CH_3 \cdot C_6H_2(NO_2)_3$	Buff	1.65	81	475	ca 100	Not affected	100	100	0.24 of MF	6225 at d 1.56 (pressed)	71, p 318

Notes: The tests for the ammines Nos: Cr7, Co2, Co12, Co13, Co21, Co22, Co25, Co26, Co27, and Cu6 and for the standard explosives listed at the end of the table were conducted according to the methods used at Picatinny Arsenal, Dover, N. J. and described in PATR 1401 (1950) by Wm. H. Rinkenbach and A. J. Clear. The tests for the other amines were conducted either by methods used in Germany and described in SS 21, 50 - 52 & 65 - 69 (1926) or by methods devised by individual investigators. Explosion temps were determined for unconfined samples, but there was no uniformity in methods of determination—in some cases the samples were heated very gradually, in other cases very slowly; in some cases the samples were heated on a metallic spatula, in other cases they were dropped on a preheated metal plate, etc. All this makes the values of explosion temps only approximate

[Prepn and props of amminocobalt (III) compds with chloric and perchloric acids. Three varieties are described: the luteo (yellow), purpureo(purple) and roseo(rose). The perchlorates are very stable at ord temps while the chlorates decomp slowly; the luteo-derivs being more stable than the roseo- and purpureo- compds] (See items Co14, Co15, Co19, Co21a, Co28 & Co29 in our tables C & D) 4) F.Ephraim, Ber **45**, 1322-31(1912) & CA **6**, 2709-10(1912) (The nature of partial valences in metal ammines) 5)F.Ephraim, ZPhysChem **81**, 513-42(1912-13) & **83**, 196-220(1913); CA **7**, 1658 & 3863(1913) (The nature of residual valence in ammines) 6) F.Ephraim, Ber **46**, 3103-31(1913) CA **8**, 597 (1914) (The nature of residual valence; influence of the anion on the stability of complex cations in metal ammonia compds) 7) F.Ephraim & A.Jahnsen, Ber **48**, 41-56(1915) & CA **9**, 2194-5(1915) {Various metal ammoniates of bromates, chlorates and iodates were prepd and examined at the Univ of Bern, Switzerland. The general method of prepn of hexammines was to pass ammonia gas into a concd aq soln of the corresponding oxyhalide salt: $6NH_3 + Cu(ClO_3)_2 \rightarrow [Cu(NH_3)_6](ClO_3)_2$. In some cases hexammines can be prepd by treating the corresponding tetrammine at room or lower temp. Many tetrammines can be prepd by passing ammonia gas into an aq soln of the corresponding oxyhalide salt, while others are prepd by heating the corresponding hexammine to above 100°. The compds described by E and J are explosive and they are listed in the above table. The explosion temps were detnd for confined and unconfined samples and were found in some cases, to be higher for confined samples then for unconfined ones} (See items Cd2, Cd3, Cd4, Cd5, Cd6, Co3, Cu4, Cu5, Cu8, Cu9, Ni1, Ni3, Ni4, Zn2, Zn3 and Zn6 in tables A,B,E & F) 8)F.Ephraim & E.Bollé, Ber **48**, 638-48(1915) & CA **9**, 2195(1915) (The nature of residual valence of zinc ammines. Table in Ber **48**, p 646 gives calcd values for absol temps of dissociation and heats of formation of various Zn ammines) 9)G.L.Clark et al, JACS **42**, 2496-8(1920) & CA **15**, 456(1921)[Cobaltammines such as trinitrotriamminecobalt (III)] (See item Co2 in table B) 10)G.R.Levi, Gazz **53**, 522-5 (1923); JCS **124**, II, 767 (1923) & CA **18**, 362 (1924) {Prepn and some props of several cobalt ammines, such as hexamminecobalt (III) chlorite $[Co(NH_3)_6](ClO_2)_3 \cdot H_2O$ and chloropentamminecobalt (III) chlorite $[Co(NH_3)_5Cl](ClO_2)_2$ are given. These complexes are less stable then the corresponding chlorates and perchlorates} (See items Co20 and Co24 in table D) 11)W. Friederich & P.Vervoorst, SS **21**, 49-52, 65-9, 84-7, 144-5(1926) & CA **21**, 1184(1927) [Metal ammine-Cd, Co, Cu, Ni and Zn chlorates and perchlorates were prepd by passing ammonia gas over the desired metallic chlorate or perchlorate solns, followed by cooling, agitation, filtering, and drying the pptd salt. Most of these compds proved to be explosives of the primary type. As a rule, they were deliquescent and hydrolyzed rapidly in moist air. Their explosive props are given in tables A,B,C,D,E & F. Methods of testing are described on pp 50-1 of this reference. Tetramminecopper (II) chlorate was the only one of the examined compds capable of detonating TNT] (See items Cd7,Co30,Cu5,Cu7,Ni5,Zn2, and Zn6 in tables A,D,E & F) 11a)Mellor, "A Comprehensive Treatise," v 8(1928), pp 228-52 479-80(See item Cu2 in table D) 12)J.N. Friend, edit, "Textbook of Inorganic Chemistry," Griffin, London, v 10(1928), "The Metal Ammines" by M.J. Sutherland 13) L.Wöhler & A.Berthmann, Ber **62**, 2750-1 (1929) & CA **24**, 1348(1930)(Prepn and props of diammine Mn and Zn fulminates)(See items Mn1 and Zn1 in tables E & F) 14)Gmelins Handbuch der anorganischen Chemie,Verlag Chemie, Berlin, System No 58, Kobalt, Tl B(Cobaltammines)(1932) 15)J.M.Cros & L.LeBoucher, Anales de la Sociedad Expañola de Fisica y Química(Madrid) **33**, 229-40(1935) & CA **29**, 4279(1935)[Prepn and props of some

ammines, such as tetrammine-copper(II) iodate and hexamminenickel(II) iodate](See items Cu5a and Ni4a in tables E & F) 16) G.Spacu & P.Voichescu, ZAnorgChem **226**, 273-88 (1935) & CA **30**, 3737(1936)(Ammoniates of some copper salts) 17)G.Spacu, ZAnorgChem **230**, 181-6(1936) & CA **31**, 2117(1937)[Ammoniates of uranium(VI) and uranium(IV) chlorides] 18)A.F.Wells, ZKrist **95**, 74-82(1936) & CA **31**, 1676(1937) {The cryst structure of silver tetranitrodiamminocobaltiate $[Co(NH_3)_2(NO_2)_4]Ag$ and of some other ammines} 19)N.R.Agafoshin, ZhObshKhim **7**, 2235-9 (1937) & CA **32**, 72 (1938){Complex compds of Cu ammines with picric acid and some other nitrophenols such as $[Cu(NH_3)_4]\cdot[C_6H_2(NO_2)_3O]_2$ and $[Cu(NH_3)_4]\cdot[C_6H_3(NO_2)_2O]_2$. Both compds seem to be explosive. They are not listed in our tables} 20)H.Brinzinger and H.Plessing, ZAnorgChem **235**, 110-14(1937) & CA **32**, 2450(1938) {Studies of nitrosopentamminecobaltic salts such as red complex $[Co(NH_3)_5(NO)](NO_3)_2\cdot 0.5H_2O$. They are not listed in our tables } 20a)J.L.Milward et al, JCS **1938**, 233-6 & CA **32**, 8296(1938) (Constitution of nitrosopentammine cobalt salts, among them the compd listed in the previous reference) 21)R. Duval, CR **206**, 1652-4(1938) & CA **32**, 5720 (1938) {Studies of trinitrotriamminocobalt(III) $[Co(NH_3)_3(NO_2)_3]$} (See item Co2 in table B) 22)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314(1939){Introduction of the azide group into complex compds to replace Cl was studied. Among the compds prepd were several ammines such as diamminecadmium(II)azide, diamminecopper(II) azide, tetramminecopper(II) azide, chloropentamminechrome(III) azide, hexamminechrome(III) azide and hexamminenickel(II) azide}(See items Cd1, Cr5, Cr6, Cu1, Cu3, and Ni2 in tables A, D & E) 23)D.B.Donskaya and M.A.Portnov, ZhObshKhim **9**, 526-31(1939) & CA **33**, 9091(1939) (The soly of Zn and Cd nitrates in liq ammonia and prepn of ammines such as tetrammine- and hexammine Zn nitrates) (See items Zn4a and Zn7 in table F) 24)A.Cirulis, Naturwissenschaften **27**, 583(1939) & CA **33**, 9175(1939) (Prepn and props of some Cu ammines) 25)C.O. Davis, USP 2,168, 562(1939) & CA **33**, 9648(1939) (An explosive obtained by dissolving a nitrate, such as AN, in anhyd liq ammonia, dispersing in the soln a comminuted sensitizer, such as Al, S or DNT, and evaporating free NH_3, thus leaving the nitrate in a continuous phase) 26)L.E.Agronomov, ZhObshKhim **10**, 1120-40 (1940) & CA **35**, 1333-5(1941)(Prepn and props of some boronhydride ammoniates) 27)M.A.Cook et al, USP 2,220,891-2(1940) & CA **35**, 1636(1941)(Blasting expls contg AN and ammine complexes are prepd by interaction of inorg nitrates, other than those of alkali metals, with ammonia) 28)T.W. Hauff & H.H.Holmes, USP 2,222,175(1940) & CA **35**, 1636(1941)(Nonsetting expls obtained by coating AN grains with tetramminezinc nitrate) 29)O.Schmitz-Dumont, ZElektrochem **47**, 221-2(1941) & CA **35**, 5052(1941)(Interaction of Co and Cr ammines with alkali metal amides results in formation of Co and Cr amides) 30)W.V.Smith, CanP 401,643(1941) & CA **36**, 1744(1942)(Prepn of diamminezinc nitrite)(See item Zn1a in table F) 31)O.Schmitz-Dumont et al, ZAnorgChem **248**, 175-207(1941) & CA **37**, 6205-7(1943) (Prepn of trivalent Co and Cr amides from corresponding ammines and alkali metal amides) 32)E.I.du Pont de Nemours and Co, BritP 544,582(1942) & CA **36**, 6804(1942) (Solid complexes obtained on treating metal nitrates with ammonia are recommended as ingredients of AN explosives) 33)A.J. Phillips, PATR 1302(1943)(Tetramminecupric nitrate; its prepn and explosive props; danger of formation when AN or its mixts come in contact, in presence of moisture, with copper or its basic salts)(See item Cu6 in table E) 34)M.Straumanis & A.Cirulis, ZAnorgChem **251**, 336-7(1943) and CA **37**, 6573(1943){Prepn and props of some Cu azide complexes among them, the tetramminecopper(II) azide} (See item Cu3 in table D) 35)A. Cirulis &

& M. Straumanis, ZAnorgChem **251**, 343-4 (1943) & CA**37**, 6574(1943){Prepn and props of some Cu azide complexes among them the diamminecopper(II) azide} (See item Cu1 table D) 36)H.J.Emeléus & J.S.Anderson, "Modern Aspects of Inorganic Chemistry," Van Nostrand, NY(1945), Chapter 4 "Coordination Compounds and Inorganic Stereochemistry" 37)L.Pauling, "Nature of the Chemical Bond," Cornell Univ Press, Ithaca, NY(1945) 38)Mellor's"Modern Inorganic Chemistry;" Longmans,Green,London(1946), pp 841-6(Complex compds including some ammines) 39)A.K.Dey,Nature **158**, 95(1946) & CA **40**, 6360(1946) (Composition of cupric ammino nitrates. Data are given leading to the inference of the existence of the tri-, tetra-, penta-, and hexammines of cupric nitrate) 40)C.A.Jacobson, "Encyclopedia of Chemical Reactions," Reinhold, NY, v 2 (1946), pp 14, etc 41)W.C.Fernelius in "Inorganic Syntheses," McGraw-Hill, NY, **2** (1946), p 218 {Prepn of hexamminecolbalt (III) nitrate} (See item Co27 in table D) 42) O.Schmitz-Dumont et al, ZAnorgChem **253**, 118-35(1947) & **254**, 329-42 (1947); CA **43**, 2887-8 & 6101 (1949)(Formation of hexamminecobalt(III) nitrate by the thermal decompn of cobalt(III) amide) 43)J.Brigando, CR **225**, 1319-20(1947) & CA **42**, 3725(1948) {Prepn of some hexamminecobalt(III) complexes} 44) W.Hückel, "Anorganische Strukturchemie," F.Enke Verlag, Stuttgart(1948), pp 56-138 45)W.C.Fernelius, "Structure of Coordinated Compounds" in Burk and Grummitt, "Chemical Architecture", Interscience, NY (1948), Chapter 3 46)W.R.Tomlinson, K.G.Ottoson & L.F.Audrieth, JACS **71** 375-6(1946) & CA **43**, 5187(1949)(Explosive properties of metal ammines)(See items Cr7, Co2,Co12,Co13, Co21,Co22,Co25,Co26,Co27, and Cu6 in tablesA,B,C,D&E) 47)K.B.Yatsimirskii, ZhObshKhim **20**, 1408-11(1949) & CA **44**, 10565-6(1950){Prepn of formatopentammine complexes of cobalt(III) among them the nitrate} (See item Co21b in table D) 48) M. Linhard & M.Weigel, ZAnorgChem **260**,65-83 (1949) & CA **44**, 5753(1950) {Prepn and props of some cis- and trans-diacidotetrammine complexes with fatty acid anions, such as $[Co(NH_3)_4 X_2]ClO_4$, where X represents acid residues from formic to caprylic acids] 49)Kirk & Othmer, "Encyclopedia,"v 4(1949), pp 379-9 (Coordination compounds, among them some ammines)(54 refs) 50)F.Ephraim, "Inorganic Chemistry," Interscience, NY (1949), p 292(Cu and Ni ammines); pp 320-32(Co and Cr ammines) 51)J.R.Partington, "A Textbook of Inorganic Chemistry," Macmillan, London(1950), pp 416-26(Coordination compounds) 52)W.Hückel, "Structural Chemistry of Inorganic Compounds" (translated by L.Long) ,Elsevier, Amsterdam v 1 (1950),pp 52 - 62(The coordination theory of complex compds) 53)A.F.Wells "Structural Inorganic Chemistry," Oxford Univ Press, London(1950),pp 7, 315, 638 & 649 54)M. Linhard & H.Flygare, ZAnorgChem **262**, 338-9, 341-3(1950) & CA **44**, 9853(1950){Azidopentamminecobalt(III) salts, such as azide, chromate, nitrate , etc}(See items Co16, Co17 and Co18 in table C) 55)M.Linhard et al ZAnorgChem **263**, 233-4(1950) & CA **45**, 3747-8(1950) {Diazidotetramminecobalt(III) salts such as azide, iodide, nitrate, perchlorate, etc}(see items Co4 to Co11 incl in tables B & C) 55a)N.V.Sidgwick, "The Chemical Elements and Their Compounds," Oxford Univ Press, London, v 1(1950), pp 156-7 & 279-8; v 2(1950), pp 1016-17, 1399-1400 & 1439-40 56)K.B. Yatsimirkii, ZhObshKhim **20**, 1465-8(1950)(in English) & CA **46**, 3444(1952){The pentammineformates of cobalt(III)}(See also Ref 47) 57)M.Linhard & M.Weigel, ZAnorgChem **263**, 245-52(1950) & CA **45**, 3748(1951){Prepn and props of triazidotriamminecobalt(III)}(See item Co1 in table B) 58)E.Weitz & H.Müller, AngewChem **62A**, 221-2(1950) & CA **44**, 8810(1950)(Nitrosopentammineferric salts) (They are not included in our tables) 59) O.Schmitz-Dumont, AngewChem **62**, 560-7 (1950) & CA **45**, 3749-50(1951)(A comprehensive survey of complex chemical reactions in liq ammonia resulting in the formation of ammines and other complexes)(36 refs) 60)Y.Tanito and Y.Saito, BullChemSoc, Japan **25**, 188-91(1952) & CA 47, 6213(1953)

(Crystal structure of trinitrotriamminecobalt (III)} 61)T.Moeller, "Inorganic Chemistry, An Advanced Textbook," Wiley,NY(1952), pp 227-73 62)G.R.Levi,R.Curti & G.Brignani, Gazz **84,** 753-8(1954) & CA **49,** 13813 (1955){Complexes contg chlorite groups coordinated with the metal, such as chloritopentammine-cobalt(III) nitrate,-chromate, -bichromate and -picrate)(See item Col8a in table C) 63)N.I.Lobanov, Izvest Sektora Platiny i Drugikh Blagorodnykh Metallov (Akad Nauk, Russia), No. 28, 277-81(1954) & CA **49,** 14555-6(1955)(Cobalt ammine iodates) 64)E.H.McLaren, Univ Microfilms (Ann Arbor, Mich), Publ No. 15174(1955)(73 pp) & CA **50,** 6130(1956){Structure of triamminochromium tetroxide $[(NH_3)_3CrO_4]$} 65)M.Linhard & W.Berthold, ZAnorgChem **279,** 173-81(1955) & CA **49,** 13812(1955) {Azidopentamminechromium(III) salts and triazidotriamminechromium(III)(See items Cr1,Cr2,Cr3,Cr4, and Cr8 in table A) 66) E.S.Gould,"Inorganic Reactions and Structure,"H.Holt and Co,NY(1955),pp 351-7 67)H.Remy,"Treatise on Inorganic Chemistry" (translated by J.S.Anderson),Elsevier, Amsterdam v 1 (1956),pp 392-410(Coordination theory) and v 2(1956),pp 304-6(Co ammines),pp 317-18(Ni ammines) & pp 389-90 (Cu ammines) 68)J.C.Bailar, "Coordination Compounds," ACS Monograph No. 131, Reinhold,NY(1956),pp 59-62(Ammines) 69)B.Taylor & B.Joyner,"Study of Explosive Sensitivity of Cobalt Ammine Complexes," NavOrd Rept 5639(1957)(U)(Not consulted) 70)F.Basolo & R.G.Pearson, "Mechanisms of Inorganic Reactions," Wiley, NY(1958), pp 1-90(Numerous refs) 71)W.R.Tomlinson, Jr,"Properties of Explosives of Military Interest," PATR 1740, revised by O.E.Sheffield (April 1958)(U)(Expls props of standard military explosives) 72)H.Taube & A.G.Maddock, edits,"Chemistry of the Coordinate Compounds,"Pergamon Press, NY,v1(1958)(638 pp)

Additional References on Ammines:
L. M. Orlova, ZavodLab **8,** 502(1939) & CA **36,** 6935(1942)Destruction of cobaltic ammines can be achieved by treating with thiosulfate in acid soln: $2[Co(NH_3)_6] + 2Na_2S_2O_3 + 12HCl = 2CoCl_2 + Na_2S_4O_6 + 12NH_4Cl + 2NaCl$

Ammissible(Ital). Permissible or "Permitted" (Explosive)

Ammoksil or Ammonxyl: Russian AN mining expls such as: a)AN 82 & TNX(called in Russ "ksilil" or "xylyl") 18% b)AN 82, TNX 12 & Al powd 6%. Properties of b) are: rate of deton 5300 m/sec, temp of expln 3380°, heat of expln 1180 cal/g and vol of gases evolved on expln 836 l/kg at STP

Ref. A. D. Blinov,Artillery Course, Voyennoye Izdatel'stvo, Moscow, 2(1949)

Ammon. Ger for Ammonia or Ammonium

Ammons were AN mining expls of varying compns developed in England prior to WW II during a shortage of glycerin to replace NG expls. Their strength was about 78% of blasting gelatin and vel of deton and sensitivity to shock and friction values were lower than those for NG expls of corresponding strength. They were appreciably hygroscopic and their oxygen balance was near zero

Refs: 1)Wm.Cullen & J.E.Lambert, Bull-InstMiningMet No **399,** (1937) & CA **32,**1933 (1938) 2)Ibid, MiningJ(London) **199,** 1125-6 (1937) & CA **32,** 3963(1938) 3)Ibid,Bull-MiningMet No **400,** 1-9(1938) & CA **32,** 2751(1938)

AMMONAL (USA)

[Ammonal, Alumatol, Burrowite, Minol and Nobel's 704(GtBritain); Ammonal (France); Ammonal and Fillers 19, 110 & 13-113 (Germany); Ammonal, Toluol-Ammonal and Nitramit (Italy) ; Amonal(Spain); Ammonal (Russia) and Ammonauru(Japan)]

Ammonals are explosive mixtures containing, as the principal ingredients, AN and powdered Al incorporated with high explosives, such as TNT, DNT, RDX and HNDPhA. Powdered carbon was also used in the first ammonals

Historical and Properties. The idea of incorporating powdered Al in expl mixts was originated by R.Escales in 1899 and by H.vonDahmen in 1900 but the explosives called "Ammonal" were patented in 1900 by G.Roth (Refs 1, 3 & 6). Originally all ammonals contained AN, Al and charcoal. The incorporation of charcoal was based on the assumption that the following reactions take place:

$3NH_4NO_3 + 2Al \rightarrow 3N_2 + 6H_2O + Al_2O_3 + 522\,kcal$

$Al_2O_3 + 3C \rightarrow 3CO + 2Al$ (Refs 3 & 17)

The reaction might also proceed as follows.

$4NH_4NO_3 + 2Al + C \rightarrow Al_2O_3 + CO + 8H_2$

(Refs 3 & 18)

If the above reactions occur, then the volume of gasous products is increased as a result of the presence of carbon and the high temp developed by oxidation of the Al

Later, a small quantity of TNT was added to make the expl compn more sensitive to deton and to increase its power and brisance. When ammonals were first tried for military purposes in Austria, it was found necessary to increase the amount of TNT in order to obtain higher loading densities

One of the earliest Ger ammonals was invented by Führer. It contained: AN 83, charcoal 3, Al 14%. In France, Lheure proposed the following mixture: AN 71, charcoal 4 & Al 25%, which was tested and approved in 1902 by the Commission des Substances Explosives. Finally(sometime after WWI), the Austrians, Germans, French and British omitted the charcoal altogether in military ammonals and these explosives became simply mixtures of amatols with aluminum. (See also Alumatol and Amatol)

In the ammonals not contg carbon, the mixture of TNT and AN detonate, developing a very high temp, which causes volatilization of the Al powder. Secondary reactions which follow, involve the oxidation of vaporized Al, either by air (if it is present) or by the products formed on deton of TNT and AN, such as CO_2 and H_2O:

$3CO_2 + 2Al \rightarrow Al_2O_3 + 3CO$

$3H_2O + 2Al \rightarrow Al_2O_3 + 3H_2$

These highly exothermic reactions develop much additional heat, causing greater expansion of the gases and consequently greater blast effect

Between WW I and WW II, ammonals were investigated in the Chemisch–technischen Reichsanstalt, Germany, by Haid & Schmidt (see Ref 20), who reported that Al reacts not only with O_2 but also with N_2 with the formation of Al nitride (80 kcal/Al_2N_2). For this reason the Germans developed aluminized explosives with a negative oxygen balance. The ammonals developed by the Austrians and used during WW I had positive oxygen balances. Expls contg Al were also investigated by the eminent Swiss chemist A. Stettbacher(Ref 14), who showed that the addition of Al increases the power while it slightly decreases the performance of the explosives. Stettbacher also found that Al-contg explosives(such as ammonals, etc) were most suitable in underwater performance and as such were used during WW II in depth bombs and torpedoes

As in the case of AN in amatols, the AN of ammonals might be hydrolyzed in the presence of moisture with the formation of ammonia but, due to the presence of Al, the amount of ammonia will be much greater. This amount might be as much as 3 times greater with ammonals than with 80/20 amatol.

The presence of ammonia is very undesirable because it reacts with TNT to form a complex addition compd which ignites at 67°. In addition, the reaction between Al and moisture produces hydrogen, which is highly inflammable in oxygen or oxygen-containing compds. If ammonia is detected in ammonals (identified by odor or brown coloration), the shells should not be washed out be steam but by means of a stream of cold water

The effect of incorporating Al in AN/TNT mixts was reported to be as follows: a) increase in sensitivity to impact, to friction and to rifle bullet impact b)increase of temp of deton from ca 1710° to ca 3914° and even to 4000° c) increase in strength (power) about 20% d)increase, in some cases, of total vol of gases evolved on deton e)decrease in vel of deton and, in some cases, in brisance(Ref 18, p 372 & Ref 19, pp 85-6)

In general, the current ammonals are fairly insensitive and stable mixtures but they are hygroscopic due to the presence of AN. Even if a small amount of moisture is absorbed, the Al(especially if impure) often reacts with it resulting in a slow evolution of hydrogen gas. This gas evolution usually occurs in storage, particularly at high temps. Ammonals are soluble(except the Al component) in water and acetone. They react (in the presence of moisture) with the same metals as the amatols, that is, copper, bronze, lead and copper-plated steel(Ref 19)

Preparation of Ammonals. In the USA ammonals were prepd by a method similar to that used for the prepn of amatols: the calcd amt of TNT(or other aromatic nitro-compd) was placed in a kettle (provided with a steam jacket and an agitator) and heated between 85° and 100°. To this was added gradually and with stirring, the calcd amt of powdered AN, previously preheated to the same temp (85 to 100°). Finally, Al powder was added and the mass cooled while continuing the agitn. The resulting mixt was in the form of a grayish pdr. If the ammonal mixt contained less than 40% TNT it had to be press-loaded but with 40% TNT or more such a mixt could be cast loaded directly into ammunition components. In preparing cast mixts a dry blend of the powdered Al and AN was added with stirring to molten TNT heated in a steam-jacketed kettle

According to PepinLehalleur(Ref 12), ammonals were prepd in France by a method similar to that used to prepare black powder. The calcd amt of the ingredients (total 35kg) were wetted with water to make about a 4% moisture content and then worked for about 1 hour on a wheel mill similar to that described in Ref 15,p 46

Composition and Uses of Ammonals as Commercial Explosives. Originally, ammonals were used as commercial blasting explosives and only with the outbreak of WW I did the use of ammonals for military purposes began. As commercial explosives they may be used for any blasting operation except in "gaseous" (fiery) coal mines (German regulation prohibit their use in such mines). Ammonals are especially useful in blasting soft rocks because the force of the expln does not break the rock into pieces which are too small for building or other purposes. The ammonals used in coal mining should contain reduced amts of Al, as well as some compds (such as a dichromate) which serve to reduce their temp of explosion. The density of ammonals used in commerce was ca 1.0 g/cc

The table, p A289, gives compositions of some commercial blasting ammonals

Composition and Uses of Ammonals as Military Explosives. Originally, ammonals were used as commercial blasting explosives, but with the outbreak of WW I they began to be used for military purposes such as filling grenades, aerial bombs, land and sea mines and trench mortar projectiles. The Austrians also used ammonals for loading cannon shells, but loading densities were too low (see Notes below) to achieve good explosive efficiencies

Commercial Blasting Ammonals

Components	Felixdorf Factory Ammonals (Austrian) a	b	c	d	Ammonals (German) A	B	Ripping Ammonal (British)	St Helen's Powder (British)	Nuevo Anagon (Spanish)	Ammonal (Spanish)	German Ammonal *
NH_4NO_3	80.75	90.0	88.0	80.0	72.0	93 - 95 5	84 - 87	92 - 95	70.0	84.5	91.8
KNO_3	–	–	–	–	–	–	–	–	–	1.5	–
$Ba(NO_3)_2$	–	–	–	–	–	–	–	–	–	0.5	–
Al(powder)	15.00	4.0	8.0	18.0	23.5	2.5 - 3 5	7 - 9	2 - 3	–	5.5	1.7
Alloy Al/Zn	–	–	–	–	–	–	–	–	20.0	–	–
Red Charcoal	–	–	–	–	4.5	–	–	–	–	–	–
Charcoal	4.25	6.0	4.0	2.0	–	2 - 3	2 - 3	–	10.0	8.0	–
Pitch	–	–	–	–	–	–	–	–	–	–	6.7
$K_2Cr_2O_7$	–	–	–	–	–	–	3 - 4	–	–	–	–
TNT	–	–	–	–	–	–	–	3 - 5	–	–	0.3
Moisture	–	–	–	–	–	0 - 1	0 - 1	0 - 1	–	–	–
References	Ref 4, p 394				Ref 4, p 393		Ref 4, p 393	Ref 4, p 393	Ref 17, p 247	Ref 18, p 372	Ref 19, p 84

*Was also used for military purposes, requiring a booster (Ref 23, p 4)

Marshall (Ref 4, p 393) cites the values for properties obtained by Bichel for ammonal A (see above table) compared with the same properties of dynammon contg AN 95.5 and red charcoal 4.5% (See table below)

Explosive	Density, d	Vel of Deton, m/sec	Heat of Expln, cal/g	Total Vol of Gases, l/kg	Vol of Gases at NTP (minus H_2O vapor) l/kg	Expln Press (Calcd), kg/cm^2	Power by Trauzl Test, cc
Dynammon	.865	3380	727.0	976	360	3668	250
Ammonal A	.900	3450	1600.5	594	418	9425	329

Notes: a) Although ammonals can be cast and compressed to densities as high as 1.65 (Ref 19, p 85), such highly compressed materials are very difficult to detonate and require very strong boosters. In order to assure their complete deton by conventional methods of initiation, the ammonals are usually pressed to a density of ca 1.2. However, this density is not sufficient for the safe firing of shells at high velocities because there is always the danger of premature explosions due to set-back b) According to Marshall(Ref 5, p 558), ammonal is said to be insensitive to blows and can be fired through the armour plating of a warship without exploding. This probably refers to the Austrian ammonal: AN 58.6, Al 21.0, TNT 18.0 & charcoal 2.4%, compressed to a high density – say 1.6

Originally, ammonals were not considered very favorably by military authorities due to their failure to realize the full effectiveness of Al in explosives. Also, Al was very scarce and very expensive during WW I. As soon as the price of Al dropped and the supply became plentiful (sometime after WW I), several new formulations for ammonals were reported. They were used during WW II and these mixtures were more effective than those used during WW I. The uses of ammonals during WW II included: demolition charges, block busters, aerial bombs (such as concrete-fragmentation bombs) and underwater ammunition, (such as mines, torpedoes and depth charges). Inasmuch as ammonals produce brilliant flashes on explosion, they are suitable for use in shells employed for testing purposes

The table, gives the compositions of some ammonals used for military purposes during WW I and WW II (See this page)

Note 1: American military ammonal, designated as 1 in attached table, was developed during WW I. It is a grayish powder which may be cast loaded. Its properties are: mw 102, OB to CO_2 -55%, OB to CO -22%, density 1.65, brisance (by sand test) 47.8g(TNT 48.0g), power (by ballistic mortar) 122% of TNT,

Military Ammonals of WW II

Components	American			Austrian		British		French		German	
	1	2**	3	I or T	2	Service Demolition	Roth 1904	S	C	1	2
NH_4NO_3	22.0	72.0	65.0	46.0	58.6	64.0	47.0	86.0	76.0	54.0	44.9
Al (powder)	11.0	16.0	32.0***	22.0	21.0	18.0	22.0	8.0	13.0	16.0	24.1
TNT	67.0	12.0	15.0 (added)	30.0	18.0	15.0	30.0 (or DNT)	–	–	30.0	31.0
Charcoal	–	–	3.0	–	2.4	3.0	1.0	–	–	–	–
Red Charcoal	–	–	–	2.0	–	–	–	–	–	–	–
Brown Coal											
Stearine	–	–	–	–	–	–	–	6.0	–	–	–
NH_4SCN*	–	–	–	–	–	–	–	–	11.0	–	–
References	Ref 22, p 12	Ref 19, p 84	Ref 25	Ref 9, p 152	Ref 5, p 558	Ref 9, p 152	Ref 3, p 71	Ref 12, p 352	Ref 12, p 352	Ref 15, p 368	Ref 8, p 195

*According to Davis (Ref 15), incorporation of NH_4SCN in ammonals produced mixts which were unstable to heat
**It was also used in Germany (Ref 23)
***Al 16% coarse and 16% fine powder

impact sensitivity 91cm (TNT 90 cm) with 2kg wt, Bur of Mines App and 11" (TNT 14"), with 2kg wt, Pic Arsn App; expln temp 265° (dec), sensitivity to initiation required 0.20g of MF, stability to heat- sl inferior to TNT. It was used as a shell filler (Ref 21, pp 12-13)

Note 2: Ammonal designated in the table, p A290, as 2 is similar in composition to the "British Service Ammonal" used for demolition purposes. It must be press-loaded because it does not contain sufficient TNT to permit cast loading (Ref 19, p 84)

Note 3: Properties of the Austrian ammonal listed as 1 and also known as "ammonal T," are given by Sukharevskii and Pershakoff (Ref 10), as follows: density ca 1.6 (Ref 13 gives 1.62), heat of formation +550 cal/g, heat of expln 1465 cal/g(Ref 13 gives 1485), vol of gases of expln(including H_2O vapor) 605 l/kg, temp of explosion 4050°, vel of deton 5400 m/sec(Ref 13 gives 5650), specific energy f (Abel) 9900(TNT 8080), power by Trauzl test 470cc(TNT 290); (Trauzl test values for ammonals and other Al-contg explosives are always unduly high due to the erosion of the lead testing block resulting from the enormous heat liberated on combustion of Al); brisance by copper crusher test 2.8 mm(TNT 3.6mm) or 77.7% of TNT; brisance by Kast formula 85.5×10^6 (TNT 86.1×10^6) or 99.2% of TNT; sensitivity to friction-insensitive; sensitivity to impact (2kg wt) 60cm (TNT 90cm). The Austrian "ammonal T" was invented in 1917 by R. Förg to be used for underwater ammunition such as torpedoes, sea mines and depth charges. This explosive has no advantage over TNT or PA when used in air, but underwater it is definitely more effective. It has been claimed that this ammonal was used in torpedoes which destroyed during WW I the French cruiser Léon Gambetta and the Italian cruiser Garibaldi (Ref 13)

Hofwimmer & Heckel(Ref 7) claimed that some German ammonals had vel of deton as high as 5650 m/sec(at d 1.62), total vol of gases of expln 740 l/kg, max press(calcd) 2693 kg/cm², temp of deton 3720° and specific energy (f) 10820. The Felixdorf Powder Works in Austria (Ref 2) reported that some Austrian ammonals gave better fragmentation test values than PA

Additional Information on Ammonals and Their Uses:

Germany In addition to the ammonals listed in the above tables and in Ref 23, p 4, there was *Füllpulver 19* (Fp 19): AN 35, TNT 55 & Al 10% used in some HE shells of mountain artillery; *Füllpulver 13-113* (Fp 13-113): AN 70, TNT 20 & Al 10% – used in some GP bombs and *Füllpulver 110* (Fp 110); AN 90, Al 2.5, naphthalene 5 & wood meal 2.5% – used press-loaded in some bombs (Ref 23, pp 47 - 8)

Great Britain Taylor & Gay (Ref 24) give the following compn and props of one of the current "non-permitted" expls: AN 83, TNT 12 & Al 5%. It is free-running powder of d 1.1 and power 88% of blasting gelatin. Used for blasting in quarries and for general work under dry to damp conditions

Italy Several pre-WW II ammonals were listed by Molina (Ref 8a), including a) AN 54, Al 24, MNB 20 a carbon 2% b) AN 65, Al 17 (of which 16% was coarse and 1% fine), TNT 15 & carbon 3%. It was not stated whether they were used by the Italians. All & EnExpls (Ref 19, p 84) lists the following ammonal used during WW II for military purposes: AN 84.5, K nitrate 1.5, Ba nitrate 0.5, Al powder 5.5 & BkPdr 8.0%. It required a booster for detonation Mangini (Ref 19a) lists the following compns suitable for military purposes: a) AN 72 to 95, Al powder 2 to 25 & carbon 0.8 to 3% b) AN 46 to 64, Al powder 17 to 22, carbon 3 & TNT 15 to 30%. The last expl was also called "toluol-ammonal". TNN was used in some compns in lieu of TNT. Belgrano (Ref 20b, p 163) lists a compn similar to ammonal, which is called "dinammon" (See also Nitramite)

Japan. An expl consisting of AN & charcoal and known as *ammon-yaku,* was listed as a "substitute powder". Its uses are unknown (Ref 17b, p 29 & Ref 21, p 212)

Russia. Several ammonals were used for civilian and military purposes, of which the following seem to be most common: a) AN 82, TNT 12 & Al powder 6% b) AN 80 & Al 20%, in which the Al was not too finely pulverized – otherwise it coated the AN grains thus impeding the reaction. This mixt had the following props: Trauzl test value 520 cc; brisance(by the Hess lead crusher test) 15.5 mm at d 1.0 & 22 mm at d 1.3, vs 13.0 for loose uncompressed TNT; tot vol of gases evolved on deton 700 l/kg at NTP; heat of expln 1680 cal/g (with Al_2O_3 as solid) and 1270 cal/g (with Al_2O_3 as gas) (Refs 19b & 20a)

Spain. In addition to the ammonals listed in the above tables and in Refs 17 & 18, it would be of interest to list here a fairly powerful and brisant expl developed in 1933 by Prof A. Blanko and called *amonal 1.* It contained AN 92.4 carbon 6.6 & Al 1% and was used by the Forces of Gen Franco during the Spanish Civil War. Its expl props compared favorably with those of 75% dynamite (Ref 17b)

Fire and explosion hazards of ammonal are briefly discussed in Sax (Ref 21a), but its toxicity is not mentioned. It would be safe to assume that toxicity of ammonals is similar to that of mixts of amatol (Ref 21a, p 266) with aluminum (Ref 21a, pp 259 & 261)

References on Ammonals: 1)G.Roth,GerP 172,327(1900) 2)Pulverwerk Felixdorf, SS **1**, 26-7(1906) 3)R.Escales,"Ammonsalpetersprengstoffe", Veit, Leipzig(1909),69-71, 97-8, 187 & 201-209 4)Marshall v1 (1917),393-4 5)Marshall v2 (1917),557-9 6)R.Forg, "Ammonal" Otto Klemm, Wien(1917) (200 pages) 7)F.Hofwimmer & F.Heckel, SS **13**, 169-72(1918), "Beiträge zur Kenntnis des Ammonals" 8)Barnett(1919),114 & 195 8a) Molina(1930), 339-40 9)Marshall v 3 (1932),116-18 & 152 10)Sukharevskii & Pershakov(1932),55 & 150 11)Stettbacher (1933), 308-9 12)Pepin Lehalleur(1935), 352 13)H.Muraour, Protar **9**, 62-3(1943) 14)A.Stettbacher,Protar **9**,212-18 & 233-42 (1943) 15)Davis(1943),368 16)Bebie(1943), 21 17)Pérez-Ara(1945),245-7 17a)PB Rept No 925(1945) 17b)Serrano de Pablo, Revista de Aeronautica **4**, 41-44(1943) & CA 39, 3159(1945) 17c)Anon, US Navy Dept, OPNAV 30-3M(1945), 29 18)Vivas,Feigenspan & Ladreda, v 2 (1946),372-3 18a)PBL Rept No 85160(1946) 19)All&EnExpls(1946), 83-7 19a)Mangini(1947),225 19b)S.B. Ratner, GornyiZhur **121**, No. 5, 21-5(1947) & CA **42**, 4347(1948) 20)Stettbacher(1948), 88-90 20a)Blinov v1(1948), 19 20b) Belgrano(1952),163 21)Anon, Dept of the Army TM 9-1910(1955), 184,214,269 & 315 21a) Sax(1957),273 22)PATR 1740(Rev1) (1958),12-13 23)PATR 2510(1958),4 24) Taylor & Gay (1958),26 25)Dr L.R.Littleton, Washington, DC; private communication (See also PicArsnTechRepts 1108,1286,1292,1308, 1372 & 1783)

Ammonals, Analytical Procedures

Identification of ammonals by color reactions described under Amatols (qv) and in Ref 2, pp 269-70, is also applicable to ammonals. This procedure does not, however, identify the Al always present in ammonals. For detn of Al it is necessary to apply one of the procedures listed below

Quantitive Analysis of Ammonals. Wogrinz (Ref 1) described the procedure which is essentially as follows: a) Thoroughly pulverize and dry the unknown material and weigh accurately a sample of ca 1g. Place it on a tared filter and rinse thoroughly with several portions of chloroform. Dry and weigh the residue on the filter. Loss in wt is equal to TNT or other nitrocompds in amatol b) Rinse the residue with several portions of cold water, dry and reweigh. The loss in wt is equal to AN c) Fold the filter with residue,

insert it in a small cylindrical glass vessel and introduce it in a vertical position into about 50 cc of 50% KOH soln in an Erlenmeyer flask which is a part of an apparatus consisting of a gas measuring burette and a levelling bulb d)Connect the flask by means of its ground glass neck to the ground glass fixture connected to a three way stopcock of the burette which is filled with water e) With the stopcock closed to the flask and open to atmosphere, move the levelling bulb until the level of water in the burette is at zero. f)Open the stopcock to the flask and tip the apparatus slightly to make the glass cylinder(inside the flask) fall. g)Contact of Al with KOH soln will cause the evoln of hydrogen and movement of the level of water in the burette. The reaction lasts about 1 hour. From the volume of hydrogen evolved the amt of Al is calcd. h)If there is any black residue in the flask, the presence of carbon is indicated and if the residue is yel or brown, the wood meal is present.

Quantitative Procedure Used at Picatinny Arsenal: a)Quantitatively transfer an accurately weighed sample (ca 1g)to a tared sintered glass extraction thimble and extract with anhyd methylene chloride (ca 1g) using a tared flask placed on a water bath b)Adjust the temp of the bath so that the solvent drips at the rate 2-3 drops per second c)When extraction is complete evaporate the liquid in the flask to dryness under a stream of dry air and then in a vacuum desiccator to const wt d) Subtract the wt of the flask from the tot wt, thus obtaining the wt of TNT e) Dry the thimble with the residue and extract AN with water into a second tared flask. f) Determine the amt of AN according to Spec JAN-A-175, Par F-4j, or identify it by one of the color reactions, such as with DPhA or thymol g) Dry the thimble with the residue, weigh it and extract the Al with concd HCl h)Determine the amt of Al as aluminum 8-hydroxyquinolinate, $Al(C_9H_6ON)_3$ according to Spec JAN-M-454, Par F-4c(2) or by other procedures described in the books on analytical chemistry i) Dry the thimble and reweigh. Total wt, minus the wt of thimble gives the wt of residue. If the residue is black, it is carbon and if the color is yellowish or brown, the possibility of presence of wood meal is indicated

Refs: 1)A.Wogrinz, SS **14**, 62(1919) 2) Anon, Dept of the Army TM 9-1910(1955), 269-71 3)N.Liszt, PicArsn; private communication

Ammonalmatrite. See under Almatrites

Ammoncahüsit. See Wetterammoncahüsit in PATR 2510(1958), p Ger 260, table 64

Ammoncarbonites. Belgian, Brit & Ger coal mining, permissible expls contg large amts of AN and small amts of NG, such as; a) AN 82.0, K nitrate 10.0, NG 4.0 & WM 4.0% b)AN 56.4 Na nitrate 7.0, NG 5.0, glycerin 5.0, WM 4.0 & Na chloride 22.6 c)AN 80.3, K nitrate 5.0, NG 4.0, NC 0.2, coal dust 6.0 & starch 4.5%

Note: Ger Ammoncarbonites are described in Ref 4

Refs: 1)Marshall **1**(1917), 396 2)Barnett (1919), 118 & 194 3)Naoúm, NG(1928),434 4)PATR 2510(1958), p Ger 5

Ammondynamit. A type of Ger mining dynamite contg large amts of AN and NG, such as AN 45 to 75, Na nitrate 5 to 0, NG 40 to 20 & WM 3 to 10%. This type of expl was also used in France, Gt Brit & USA (See also Ammonium Nitrate Dynamites)

Refs: 1)Naoúm, NG(1928) 2)Bebie(1943), 21 3)PATR 2510(1958), p Ger 5

Ammondyne. A type of mining expl: AN 45-51, Amm oxalate 17-19, NG 9-11, Na nitrate 8-10 & dry WM 11-13%

Ref: Cond ChemDict (1942), 287(not listed in the new editions)

Ammonex. Under this name are known thirteen castable AN expls, not contg TNT, prepd and examined during WWII at PicArsn, Dover, NJ. Their compns are given in Table p 294

Ammonexes

Compn and some props	A	B	C	D	E	F	G	H	I	J	K	L	M
Amm nitrate	60.5	78.0	78.0	36.3	38.0	38.0	43.0	43.0	42.0	32.5	42.0	36.0	36.0
Na nitrate	18.0	–	–	10.8	11.0	11.0	12.5	12.5	17.0	5.0	17.0	5.5	5.5
Urea	10.5	11.0	11.0	6.3	6.0	6.0	4.5	4.5	–	–	–	–	–
Al (flake)	1.0	1.0	–	–	–	–	–	–	–	–	–	–	–
Al (granular)	10.0	10.0	–	–	–	–	–	–	–	–	–	–	–
Ca silicide	–	–	11.0	6.6	–	–	–	–	–	–	–	–	–
Ca nitrate (anhy)	–	–	–	–	–	–	–	–	–	7.5	–	8.0	8.0
Gu nitrate	–	–	–	–	–	–	–	–	5.5	–	5.5	–	–
Dicyandiamide	–	–	–	–	–	–	–	–	5.5	5.0	5.5	5.5	5.5
Am pcirate	–	–	–	40.0	45.0	–	40.0	–	30.0	50.0	–	–	45.0
Tetryl	–	–	–	–	–	45.0	–	40.0	–	–	30.0	45.0	–
Avg d of chge	1.63	1.62	1.46	1.41	1.64	1.47	–	–	1.35	1.66	1.63	1.56	1.61
Appr pour point*, °C	86	98	98	95	92	92	–	–	97	98	100	94	92
Avg wt of chge (lbs) in 3" M42 Shell	0.91	0.88	0.80	0.77	0.90	0.80	–	–	0.74	0.91	0.89	0.85	0.89
% Tot fragms compd to TNT	24	28	41	73	87	86	–	–	69	95	84	85	100
Exudation in 3" Shell:													
7 days at 50°	–	–	–	None	Apprec	None	Apprec	Apprec	None	None	None	None	None
7 days at 65°	–	–	–	Apprec	Apprec	Apprec	Apprec	Apprec	None	None	None	None	None
Stability:													
100° Vac stab test	–	–	–	–	Satisf	Satisf	–	–	Satisf	–	–	–	–
100° Heat test	–	–	–	–	Satisf	Satisf	–	–	Satisf	–	Satisf	Satisf	Satisf
120°Vac stab test	Satisf	–	Satisf	Satisf	Satisf	Unsatisf	–	–	Satisf	–	Unsatisf	Unsatisf	Satisf
Rifle bullet test, (5 trials)	–	–	–	–	No detons	2 low order	–	–	1 low order	–	4 high & 1 low order	4 high order	1 high & 3 low order

*Note: Pour point is the temp below which mixt does not readily flow by gravity and pouring temp(min) is about 5°C above pour point

The following conclusions may be reached in regard to the table, p A294: a)Ammonex A developed by Phillips (Ref 1) while satisfactory with regard to stability and loading characteristics is of low brisance(when cast-loaded at high d in 3" M42 shell and subjected to fragmentation test) b)Ammonex M is comparable to TNT in brisance(fragmentation test), loading characteristics, stability and exudation and for these reasons may be considered as a suitable expl for shell-loading c)Ammonex J, while as satisfactory in brisance and exudation as M, cannot be poured below 100° d)Ammonex L, while not as stable as M or TNT, is comparable to pentolite and tetrytol in other props. It is very sensitive to rifle bullet impact e)Ammonex E, while favorable with regard to fragmentation and stability, exudes a dark-brown oily liq when stored at 50° or 65° for 7 days. This liq forms upon cooling needle-like crysts f)Ammonex F, while satisfactory in brisance and in 100° stability tests, is unsatisfactory with regard to exudation and 120° vac stab test g)Ammonex K, while satisfactory in regard to brisance and exudation, is unsatis in regard to 120° vac stab test and pour point. It is very sensitive to rifle bullet impact h)Ammonex D, while not too low in brisance, is unsatisf due to exudation and high pour point i)Ammonex I is unsatisf due to its high pour point j)Ammonexes B and C, as well as previously mentioned Ammonex A, are too low in brisance as detnd by the fragmentation test k)Props of Ammonexes G & H were not given in Refs 1 and 2

Refs: 1)A.J.Phillips, PATR 1106(1941) 2)R.D.Sheeline, PATR 1234(1943)

Ammongelatine. A type of Ger permissible gelatin dynamite. Several compns are listed in PATR 2510(1958), p Ger 5

Ammongelatine Dynamit. A type of gelatinous or plastic expl invented in 1879 by A. Nobel, in Sweden. One formulation contained NG 71, CC 4, charcoal 2 & AN 23%, another contained NG 25, CC 1, charcoal 12 & AN 62%. The 1st dynamite was gelatinous, whereas the 2nd was plastic and rather crumbly

Note: In these expls, AN particles were coated with NG-CC jelly. It was supposed that this jelly carries the expl impulse originating in the detonator to the AN causing it to decomp explosively with the formation of N_2, H_2O and O_2. The O_2 reacted with charcoal or other combustibles

Formulations of these dynamites were later changed to include HE's such as TNT, TNX, liq DNT, NS, NGc, etc. These compds act not only as sensitizers for AN but also as antifreezes

Compds similar to Ammongelatines are manufd by the DuPont Co under the name of **"Extra Dynamites"** (Ref 3)

Refs: 1)Naoúm, NG(1928), 11 2)Davis (1943), 335 3)Blaster's Handbook(1952), 59-62

Ammongelignite. A type of current Brit "non-permitted gelatinous AN expl. It is described under Ammonium Nitrate Gelatines

Ammongelit I. An expl mentioned in Explosivstoffe **1957**,168 (compn not given)

AMMONIA

(Anhydrous and Aqueous)

(Called Ammoniak in Ger, Ammoniaque in Fr, Ammiyak in Rus, Ammoniaca in Ital, Amoníaco in Span), NH_3, mw 17.03, N 82.25%, OB to H_2O + N_2 141.2%, mp −77 7°, bp −33.4°. Col gas with pungent odor at ord temp and press, col liq when compressed; usually stored in "Hortonspheres" at press 40 - 50 psi; critical temp 132.4°, crit press 111.5 atm, d 0.771 g/l at 0° and 0.817 g/cc at -79°; autoignition temp 651°, vap press 10 atm at 25.7°, expl range (in air) 16.0 - 25.0%. Fire and expln hazard moderate when exposed to flame(Ref82). Sol in water, alcohol and ether. Anhydrous ammonia is irritating to eyes, skin and mucous membranes of the respiratory tract. Ammonia gas at concns above 0.5% in the air is fatal to humans after prolonged breathing. When confined under pressure, anhyd ammonia may shatter the container with expl force (For a general discussion of the properties of ammonia see Refs 12,14,17, 30,32,34,59,72 & 86)

Ammonia is by far the most important of the hydride compds. It can be prepd by: a)synthesis from its elements b)hydrolysis of nitrides and amides c)reduction of oxides of nitrogen and d)the dry distillation of nitrogenous substs, such as coal, bones, etc. The history and occurrence of ammonia have been discussed in detail by Mellor (Ref 6), Gmelin (Ref 13), Gamburg (Ref 25), Kirk and Othmer(Ref 32) and Mittasch (Ref 47). The use of ammonia-water solns had been mentioned by the alchemists. In the 18th century Priestley isolated ammonia gas and demonstrated it could be decomposed by passing it over a hot wire or through an electric spark. Van Scheele first identified the component gases and Berthollet and also Henry discovered that they consisted of 3 parts hydrogen and one part nitrogen. The principal source of ammonia until about the twenties of this century was the by-product of distillation of coal for gas, coke and tar. Ammonia, in this process, was obtained as an aqueous soln(ammonia liquor) and could be recovered either as Amm sulfate or as NH_3 gas, which could be liquified. Other nitrogen contg materials, such as bones, horns, etc could be utilized for prepn of ammonia. Manuf of ammonia from coal is described by Kirk and Othmer(Ref 32), Riegel(Ref 38), Just et al(Ref 62), Little(Ref 69), Martin et al(Ref 71), Shreve(Ref 80), Osthaus(Ref 81) and Groggins(Ref 86). Fulton(Ref 45) and Groggins(Ref 86) describe the processes for the recovery of anhydrous ammonia from its aq solns

The first successful attempt to prep ammonia by direct synthesis of atmospheric nitrogen and hydrogen was made at the beginning of the century by *F. Haber*. He passed a mixt 1 vol N_2 and 3 vols H_2 at moderately high temps and under press over a contact catalyst. This procedure, which consists of reduction of nitrogen from N° to N^{---} was inspired by previously developed method of *Birkeland & Eyede* in which atmospheric nitrogen was oxidized to the state of its oxides. In this method, called "fixation of nitrogen," B & E passed air at a rapid rate through an electric arc spread out to form a flame and the resulting N oxides were removed as soon as they formed. It should be noted that as early as 1780 Cavendish prepd a small quantity of N oxides by passing a series of electric sparks through the air. The method of Haber is the "direct synthesis" methods, whereas Frank and Caro process, Serpek process and Bagnulo process are "indirect synthesis" methods

In the *Frank & Caro* process, invented in 1895 - 7 and known as the "calcium cyanamide method," a carbide, such as Ca carbide absorbs nitrogen to form cyanamide(fertilizer), which by a further treatment may be transformed either into cyanide or into ammonia. This process was used in Europe, USA and Canada (Refs 38 & 71). In the *Serpek* process introduced in France and not adopted in the USA, nitrogen was "fixed" as Al nitride and this gave ammonia when treated with steam(Refs 38 and 71). In the *Bagnulo* process, invented in Italy, ammonia and other N-contg substances

are obtained by a continuous method either from Ca cyanamide or from the reaction of hydrocarbons with nitrogen and water(Ref 43)

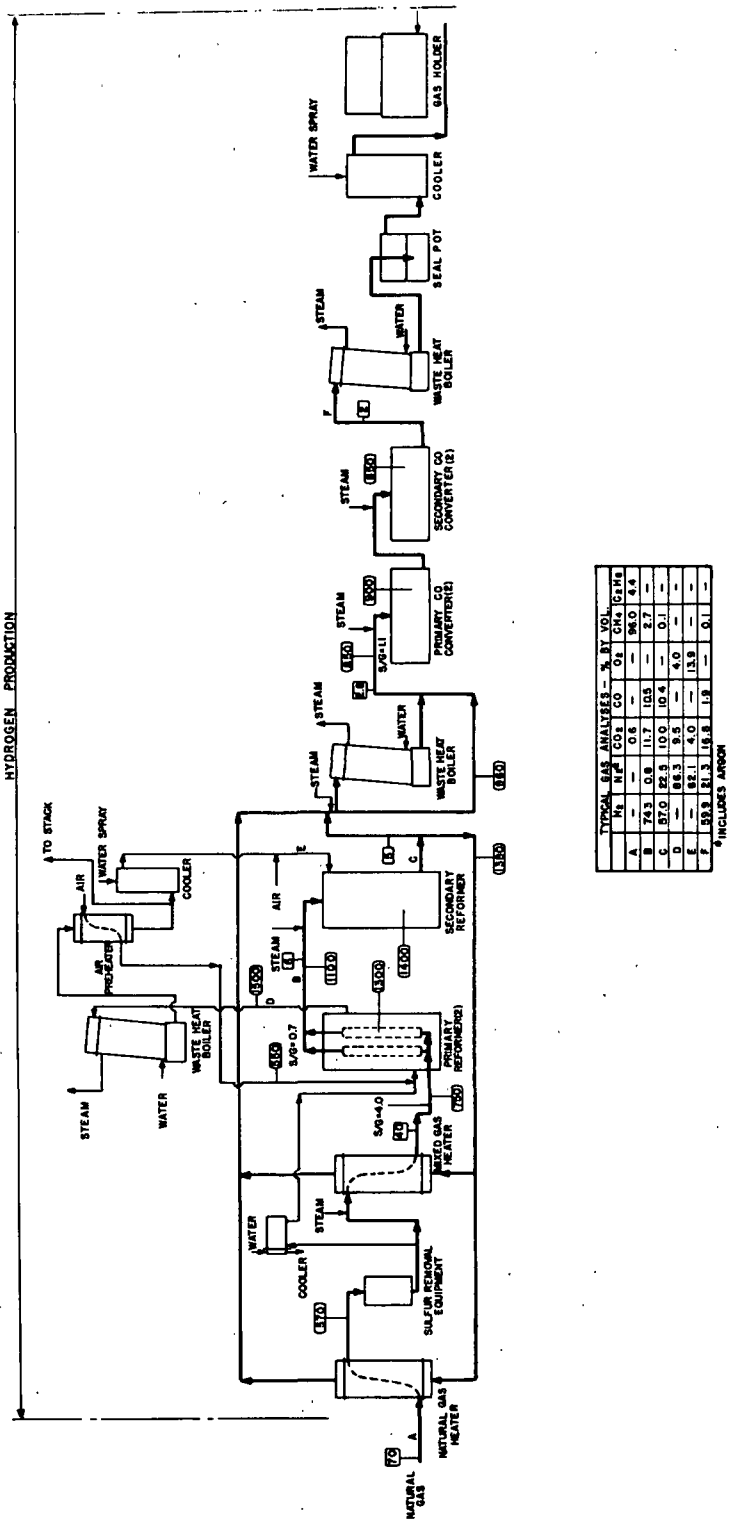
Of all the known methods of manuf of ammonia, the most important is the *"direct synthesis,"* which consists essentially of producing a gaseous mixt of 3 vols of hydrogen and 1 vol of nitrogen, purifying the mixt and synthesizing to ammonia by passing it at high press and temp through a converter contg a catalyst. The resulting anhydrous ammonia is stored in liq form and under moderate pressure in the so-called "Hortonspheres." It may be shipped to its destination in a special tank car or truck

Since the development of the Haber process, many patents were issued to various persons and companies, but essentially all the "direct synthesis" methods are based on the original process–the main differences are in construction of converters and in some minor details. A comprehensive description of "direct synthesis" methods and a general discussion on methods of manuf of ammonia is given in Kirk and Othmer(Ref 32) and Faith, Keyes & Clark(Ref 83) (See also Refs 4,7,19,20,21,23, 25,27,32,33,35,36,38,39,42,48,49,50,60,62, 70,71,77,79,80,81 & 82)

A flow diagram of the direct synthesis of ammonia as practiced at the TVA(Tennessee Valley Authority), is given on pp A298-9

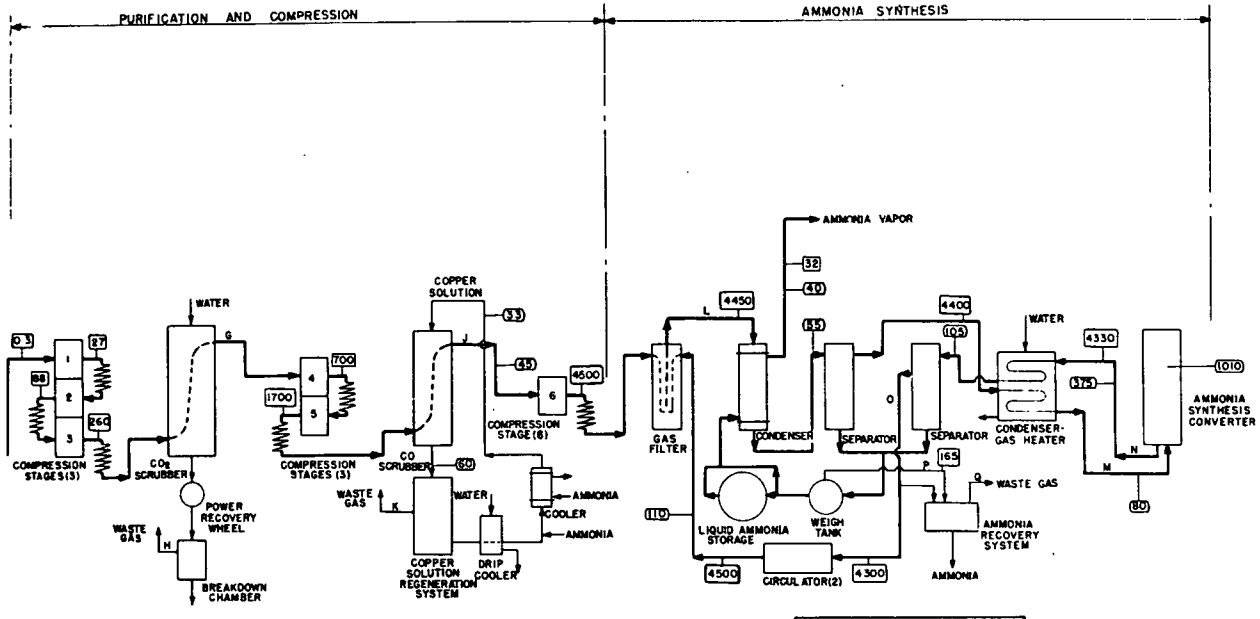
Following is the list of "direct synthesis" processes practiced currently in various countries: a)Haber-Bosch(Refs 19,32,38,39, 42,48 & 80) b)Casale(Refs 19,38,39 & 80) c)Claude(Refs 19,32,38,39,49,50,76 & 80) d)Fauser(Refs 19,32,39, & 80) e)Mont-Cenis(Refs 19,32,38 & 80) f)Air Liquide (Ref 80) g)DuPont(Refs 20& 80) h)GeneralChemical(Ref 19) i)Kellogg(Refs 64 & 80) j)Mathieson(Ref 38) k)NEC(Nitrogen Engineering Corp)(Refs 32,39,67 & 80) l) FNRL(Fixed Nitrogen Research Laboratory) (Ref 80) m)Uhde(Ref 39)

The following may be added to the information on the manuf of ammonia: Mitchell(Ref 26) discusses the manuf of ammonia from natural gas as practiced at the Lion Chemical Corp plant. Holroyd(Ref 27) discusses synthesis of ammonia by the Haber-Bosch process as practiced at the IG Farbenindustrie plants at Ludwigshaven and Oppau,Germany. Guillaumeron(Ref 33) reviews the production of synthetic ammonia during WWII in USA and Canada. Cope(Ref 36) discusses US production facilities for ammonia. Odelhög(Ref 44) patented an ammonia synthesis process using a granular catalyst consisting of either Fe + Ni, Fe + Co, Fe + Mg or Fe + V at temp 300° and press 50 kg/cm^2. Vergues & Patry(Ref 46) describe manuf of ammonia from natural gas in a plant located at Saint-Marcel, Pyrenées,France. Anon(Ref 48) gives a flow sheet and a brief description of the Lion Oil Co plant at El Dorado,Arkansas. The plant was designed to produce ammonia from natural gas by modification of the Haber-Bosch process. Shearon & Thompson(Ref 49) describe the modified high pressure Claude process plant, at Yazoo City, Mississippi. The authors also state that the first Claude units were installed in USA in 1927 and, with modifications from the Casale process, are now owned by the DuPont Co. The US Govt plant at Morgantown, West Virginia, was designed, built and operated during WWII by the DuPont Co using its own modification of the high pressure Casale process. Reidel(Refs 63, 64,65) describes some recently constructed ammonia synthesis plants, such as the Spencer Chemical Co plant at Vicksburg, Miss and the Kellogg process plant. The Spencer plant is also described in Refs 66 & 68. Anon(Ref 67) and Reidel (Ref 79) describe the Phillips Chemical Co plant near Houston, Texas. It is the NEC (Nitrogen Engineering Corp) medium pressure process utilizing natural gas as starting material. Resen(Ref 70) describes the manuf of ammonia at the Lion Oil Co plant in Louisiana. Frankenburg (Ref 51) studied the relationship between the nature and the effectiveness of ammonia synthesis catalysts. Other studies of synthesis catalysts were made by Odelhög(Ref 44), Enomoto & Horiuchi(Refs 53 & 54), Nielsen(Ref 60), Emmett(Ref 74) and Faith



TYPICAL GAS ANALYSES — % BY VOL.						
	H_2	N_2*	CO_2	CO	O_2	CH_4	C_2H_6
A	—	—	0.6	—	—	96.0	4.4
B	74.3	0.8	11.7	10.5	—	2.7	—
C	87.0	22.5	10.0	10.4	—	0.1	—
D	—	86.3	9.5	—	4.0	—	—
E	—	82.1	4.0	—	13.9	—	—
F	59.8	21.3	16.8	1.8	—	0.1	—

*INCLUDES ARGON



TYPICAL GAS ANALYSES - % BY VOL						
	H_2	N_2*	CO_2	CO	O_2	CH_4
G	72.2	24.4	0.7	2.4	0.1	0.1
H	17.8	5.1	76.4	0.6	0.0	0.1
J	74.7	25.2	<PPM	<PPM	0.0	0.1
K	5.3	2.2	20.9	71.3	0.0	0.1

* INCLUDES ARGON

TYPICAL GAS ANALYSES - % BY VOL					
	H_2	N_2	NH_3	A	CH_4
L	62.3	22.0	7.8	6.3	1.6
M	64.4	22.1	5.0	6.8	1.7
N	53.6	19.3	17.5	7.7	1.9
O	58.3	21.0	10.3	8.3	2.1
P	19.0	6.8	69.2	3.7	1.3
Q	62.4	23.9	0.0	10.6	3.4

PROCESS REQUIREMENTS PER TON OF AMMONIA

NATURAL GAS	
PROCESS GAS, MCF	21.3
FUEL GAS, MCF	8.9
STEAM, LB	6100
POWER, K WH	1050
WATER, GAL	176,500
OPERATING LABOR, MAN-HR	1.83
MAINT. LABOR, MAN HR	0.71

NOTES:
EQUIPMENT SHOWN FOR ONE TRAIN ONLY NUMBERS IN PARENTHESES ADJACENT TO EQUIPMENT NAMES INDICATE THE NUMBER OF ITEMS PER TRAIN

SYMBOLS

100	TEMPERATURE, °F
100	PRESSURE, PS I G
S/G	VOL RATIO, STEAM: GAS

FLOW DIAGRAM FOR THE PRODUCTION OF ANHYDROUS AMMONIA TENNESSEE VALLEY AUTHORITY, WILSON DAM, ALABAMA, U S A

et al(Ref 83). The thermodynamics of ammonia synthesis was reported by Oldham (Ref 22), Enomoto & Horiuchi(Ref 54), Jagannathan(Ref 56), Harrison and Kobe (Ref 61) and Schmidt(Ref 78)

Schlachman(Ref 21) lists the US War Dept sponsored synthetic ammonia plants operating during WWII and Faith Keyes & Clark (Ref 83) gives a list of current US ammonia plants

General properties of ammonia are discussed in Mellor(Ref 6), Gmelin(Ref 13), Thorpe(Ref 15),ChemRevs(Ref 17a), Kirk & Othmer(Ref 32) and in Ullmann(Ref 59). Franklin(Ref 1) described the reactions of liq ammonia. Von Braun et al(Ref 16) reported a violent and explosive reaction of liq ammonia with some organic halogen compds. For example, when the reaction between liq ammonia and $BrH_2C–CH_2Br$ was carried out at RT, there occurred a spontaneous evolution of heat resulting in a violent expln. Reeves & Giddens stabilized cellulose nitrate with ammonia(Ref 31). Sampey(Ref 28) observed that the residue resulting from the reaction of ammonia with Hg exploded in a steel U tube connected with a glass Hg manometer when an attempt was made to clean the tube with a steel rod. Some of the gray-brown solid residue was recovered and detonated by heating in a crucible. Analysis indicated that the residue consisted of dehydration products of Millon's base[See CR **140**, 853 (1905)] and was readily sol in $Na_2S_2O_3 \cdot 5H_2O$

The expln of mixt of ammonia with electrolytic gas and oxygen was reported by Partington and Prince(Ref 2) and with carbon monoxide and oxygen by Beeson and Partington (Ref 3). Jorissen et al (Refs 5,8 & 9) studied the expl regions of hydrogen-ammonia-air, hydrogen-ammonia-oxygen, ammonia-air and ammonia-oxygen-nitrogen mixts. Scliephake et al(Ref 10) also investigated the expl combustion of ammonia-air mixts while Franck and Döring(Ref 11) made similar studies under high pressure. Rozlovskii(Ref 24) detd the ignition limits of ammonia-oxygen mixts in a heated quartz vessel. When the pressure (P) of the gas mixt increased from 40 to 100 mm Hg, the min ignition temp (T) decreased from 1100° to 1025°. At a given P, T was the lowest for 33% or 50% ammonia; the minimum press at which the mixt could be ignited was at 33% concn of ammonia. Pieters(Ref 29) detd the expln limit of gases by a new type apparatus which gave values of 15.0 to 25% ammonia in air. Shaphorst(Ref 37) found that dry mixts of air contg 16.5 to 26.8% ammonia were combustible, whereas damp mixts were neither combustible nor explosive. Other studies of explosibility of ammonia-air mixts were made by Clarke & Wright(Ref 73) and by Banik(Ref 84)

The handling of ammonia safely has been discussed by Brandt(Ref 40) and the toxicity of ammonia fumes by inhalation, by Wheatherby (Ref 55). Ohno(Ref 57) noted that the toxicity of gaseous ammonia was much smaller than that of sulfur dioxide. Both gases produce irritation of mucous membrane and eyes, bronchitis, hyperemia, hemorrage, endema, exudation and pneumonia. Krop(Ref 58) lists the toxicity and health hazards of ammonia and other substances used as fuels in rocket propellants[Also see Sax(Ref 82) for additional information on the hazards of ammonia]

For use in the explosive industry, anhydrous ammonia is converted by oxidation, at high temp and pressure, in the presence of a suitable catalyst, to 60% nitric acid, called "dilute nitric" before being concentrated to 98 - 99% strength acid called "strong nitric." The "strong nitric" is used in the manuf of NG, NC, NS, DNT, TNT, PA, PETN, RDX and tetryl, whereas the "dilute nitric" serves the expl industry for the manuf of AN which is used either for prepn of various expl mixts(such as amatol, ammonal, etc) or for the prepn of fertilizers

There are many other uses of ammonia discussed by Kirk and Othmer(Ref 32). Serruys(Ref 18) investigated ammonia and some other substances as possible motor fuel substitutes. Canright(Ref 41), Clark

(Ref 52), Baker(Ref 75) and Gordon & Glueck Ref 85) investigated ammonia-oxygen systems as a liq propellant for rocket motors

Ammonia was also used during WWII in the USA for the neutralization of residual acidity in crude TNT prior to its purification by sellite(aqueous soln of Na sulfite). Use of ammonia in lieu of soda ash as a neutralizer produced a lighter colored TNT(See Trinitrotoluene, Manufacture of)

Requirements for ammonia used in the manuf of explosives are listed in Specification JAN-A-182

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AMMONIA, ANALYTICAL PROCEDURES

Detection of small quantities of ammonia in air, water, etc can be done by colorimetric methods using reagents such as Nessler's (Refs 1–6 and 8–10), phenol & hypochlorite(Ref 12). A detailed description of the colorimetric detn of NH_3 in air by use of Nessler's reagent is given by Jacobs(Ref 9, p 364). The concn of ammonia in air may be obtained also by passing a known vol of the air through two bubblers in series contg known vols of standard 0.02N sulfuric acid until the color of methyl-red indicator changes from yel to red. Detailed description of this method is given in Ref 9, pp 363–4. A midget impinger may be used instead of bubblers. Description of oxidimetric detn of ammonia is given by Hurka & Ruzdik(Ref 7)

Procedures described on the following pages are used at the US Ordnance plants for analyses of anhydrous and aq ammonias

1. Ammonia, Anhydrous, Synthetic. Commercial anhydrous ammonia contains as principal impurities: moisture, traces of oil and some rust, dirt, etc. The product intended for use at the US Ordnance plants for the manuf of nitric acid, Amm nitrate, Amm picrate and Na azide must comply with the following requirements of the US Specs(Ref 15): a)Moisture – max 0.5% by wt b)Oil – max 5 ppm

The procedures for sampling and testing of ammonia and calculations given in the above specs can be slightly simplified (especially in calcns), as was done during WW II at the Keystone Ordnance Works lab, to make them less time-consuming

Following is a description of the sampling and testing of anhydrous ammonia:

Sampling is usually done by two persons, each equipped with a full-face mask and heavy rubber gloves. Connect to the unloading valve in the dome of the tank a stainless-steel sampling line with a 1-inch union and a ½-inch valve, and attach to the sampling line by a rubber tubing connection the adapter equipped with a rubber stopper as shown on figs in the Spec(Ref 15). Attach to the side-outlet connection of the adapter a 4- or 5-foot length of rubber tubing to carry the NH_3 vapors, released by the sampling, to one side of the sample taker. Place yourself facing the sampling jet with your back against the wind and ask the operator to open the discharge valve slightly. Let the NH_3 run out for a few mins to sweep the pipes and then carefully fill two or three duPont special centrifuge tubes graduated from 0 to 100 ml. These tubes will serve for moist detn. If it is required to det oil content, fill with liq NH_3 two 2-l round-bottom flasks

Immediately after each sample is taken, tightly close the containers by means of rubber stoppers provided with Bunsen valves (in order to prevent the penetration of moist from the atm) and take the samples to the lab

Procedures:

a)Moisture Content. Wearing a full-face mask, loosen up slightly the stoppers and plunge each centrifuge tube slowly into a cooling bath contg salt-ice mixt. Remove after ca 1 hr one of the tubes and warm its tip by holding it between the fingers. If the liq starts to boil, continue the evapn of NH_3 for a little longer, etc. For the final test, remove the stopper, let the gas escape and warm the bottom of tube with the hand. If the smell of NH_3 is gone, take the reading. Each small division of the tube corresponds to 0.05% by vol. Divide the reading by the d of liq NH_3 = 0.682 in order to obtain % moisture by wt in the NH_3. Check if there is any oil or rust at the bottom of the tube

Note: A much more complicated formula for calcg % moisture is given in the Spec(Ref 15), but it is not necessary to employ it unless required by the Govt inspector

b)Oil Content. If an appreciable amt of oil is present in liq NH_3, some droplets will be

visible at the bottom of centrifuge tubes used in the previous operation. In this case proceed as follows:

Place in each of the 2000 ml round-bottom flasks(see under Sampling) a piece of 14–20 mesh washed coke(known commercially as ''anti-bump''), loosen slightly the stoppers and place the flasks in the cooling bath, as in proced a. When the bulk of NH_3 is gone, transfer the flasks into a bath contg tap water at RT and continue the evapn. Finally bring the temp of the bath to ca 30°(by adding some hot w) in order to complete the evapn of NH_3. Rinse the inside of each flask with four 10 ml portions of carbon tetrachloride delivered from a pipette, transferring each set of washings into a 75 ml separatory funnel contg no grease on its stopcock. After separation of layers and allowing evapn of w from the top layer, draw the liq in each funnel through a dry filter paper(previously washed with carbon tetrachloride) into a small (30–60 ml) tared dish. Evaporate the tetrachloride on a steam bath, cool each dish in a desiccator and weigh. Run a blank on 40 ml of tetrachloride and calculate oil in ppm from the equation $\frac{(W_1 - W_2) \times F \times 1000}{0.682 \times V}$, where W_1 = wt of residue in mg, W_2 = wt of blank, V = vol of sample in ml, 0.682 = d of sample and F = evaporation factor (0.871 for a press in the tank car of 60 psi; values for other pressures may be obtained from the chart in the spec)

II. Ammonia, Aqueous, Analytical Procedures

a)Ammonia Sales Car Analysis. Take two 8 oz samples from a tank car and fill two tared glass bulbs, each ca 4 ml capacity and provided with a capillary at least 3" long. Seal the bulbs and after reweighing, place each bulb in a known excess of N/3 HCl(usually 150.00 ml). Keeping the tip of the capillary completely immersed, drain the bulb as much as possible after breaking the tip, and then crush the bulb and the capillary with a stirring rod. Add a few drops of methyl-red indicator and titrate the excess of acid with N/3 NaOH. Calc % NH_3 from the formula $\frac{(V_1N_1 - V_2N_2) \times 1.703}{W}$, where V_1N_1 = vol and normality of HCl; V_2N_2 = vol and normality of NaOH and W = wt of sample (Ref 11,p 64)

b)Ammonia Recovery. Condensate from the evapn of crude Na azide at the KOW plant was sampled every 24 hrs and d was detd in the lab with a hydrometer, range 0.9–1.0. From a density-vs-concentration table (such as given in Lunge's Handbook), det the % NH_3 in the liq(Ref 11,p 63)

c)Ammonia in Steam Condensate of Ammonia Oxidation Plants can be detd by colorimetric method using Nessler's reagent. The following procedure was used during WW II at the Keystone OW:

To a 25 ml of condensate in a 50 ml Nessler tube was added from a pipette 5 ml of Nessler's reagent and distd w to the mark. The presence of ammonia was indicated by the appearance of brown turbidity due to the formation of $NHg_2I \cdot H_2O$ and the intensity of this turbidity was detd colorimetrically using the Nalco Phototester, which was previously calibrated by using freshly prepd standards

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Ammoniac, Ammoniacum or Sal Ammoniac. See Ammonium Chloride, under Chlorides

Ammoniacal Copper Nitrate. See under Ammines

Ammoniacal Liquor or Gas Liquor. Impure ammonia water obtained as a by-product in the distillation of coal, tar, bones, etc

Ref: Merriam-Webster's Unabridged Dictionary(1950),p 86

Ammoniacal Metal Complexes. See Ammines

Ammoniacal Nitrogen Iodide. See under Nitrogen Iodide

Ammoniacal Saltpeter Plastic Explosive. A plastic mixture consisting of AN 78–85% (with or without K nitrate) and a soln of nitrosemicellulose in nitrobenzene, nitrotoluene or nitroxylene

Ref: Vereinigte Köln-Rottweiler Pulverfabriken A–G,BritP 3,937(1909) & CA **4**, 1378(1910)

Ammoniacal Silver Compounds and Their Solutions. The following refs describe the explosive ammonical silver nitrate & silver oxide or their solutions:

1)C.L.Berthollet,CrellAnn **2**,390(1788) (Action of ammonia on silver oxide produced an extremely sensitive compd named "fulminating silver") (See next ref) 2)F.Raschig, (Liebig's)Ann **233**,93–101(1886) (Zur Kenntniss des Berthollet'schen Knallsilbers) (Investigation of Berthollet's fulminating silver showed that it is Ag_3N) 3)M.Berthelot & M.Delépine,CR **129**,326–30(1899) & JCS **76** II, 748(1899) (Ammoniacal silver nitrate $AgNO_3 \cdot 2NH_3$ was prepd by treating 1 mol $AgNO_3$ with 2 mols NH_3 in an aq soln and evaporating the water) 4)C.Matignon,BullFr, [4], **3–4**,618(1908) & CA **2**,245(1908) (An explosion at l'École de Sévres of a mixt of Ag nitrate alkali and ammonia, after it had stood quietly for 24 hrs, was evidently due to silver ammine formed by interaction of the foregoing ingredients) 5)A.Tingle,IEC **11**, 379(1919) & CA **13**,1152(1919) (Ammoniacal solns of silver oxide have to be handled with care because they might contain fulminating silver) 6)E.J.Witzemann,IEC **11**,893(1919) & CA **13**,2449(1919) (Several explosions took place while W was studying the oxidation of organic compds by silver oxide and was using ammonia to remove the non-reacted Ag_2O. It was claimed that silver fulminate, formed as a product of the reaction was the cause of these explosions) 7)J.Eggert,ZElektrochem **27**,547–58(1921) & CA **16**,1013(1922) (Investigation of sensitiveness of some expls, among them $Ag_2O \cdot 2NH_3$ and $NH_3 \cdot NI_3$) 8)H.Vasbinder,PharmWeekblad **87**,861–5 (1952) & CA **47**,4083(1953) (Discussion on hazards of ammoniacal silver solns. One of the solns obtained on mixing Ag nitrate, Amm hydroxide and gum arabic exploded on warming. Another soln, prepd by pptg Ag oxide from Ag nitrate soln with Na hydroxide, washing the oxide, dissolving it in Amm hydroxide, adding dropwise Ag nitrate until a permanent ppt was formed and then centrifuging, exploded on two occasions after standing for 10–14 days. Both these solns were used as permanent marking ink. It is suggested that the

explns were caused by an amorphous substance Ag_2HN, very sensitive to heat and shock even when wet, which formed as a result of the following reactions:

a) $Ag_2O + 4NH_4OH \rightarrow 2Ag(NH_3)_2OH + 3H_2O$
b) $2Ag(NH_3)_2OH \rightarrow Ag_2HN + 3NH_3 + 2H_2O$

Ammoniacate. French for Ammoniate(See Ammine)

Ammonia Derivatives of Polynitro-Alcohols. Several compds, some of them explosive, were patented recently by Swedish inventors. For their prepn, polynitro-alcohols(having all NO_2 groups attached to the same C atom and the OH group or groups attached to the adjacent C atom) were treated with ammonia(or an N-substitution deriv thereof) in aq or other solns

Following equations illustrate this process: a) $R{\cdot}C(NO_2)_2{\cdot}CH_2OH + NH_3 \rightarrow R{\cdot}C(NO_2)_2{\cdot}CH_2NH_2 + H_2O$ b) $R{\cdot}C(NO_2)_2{\cdot}CH_2OH + R{\cdot}C(NO_2)_2{\cdot}CH_2NH_2 \rightarrow [R{\cdot}C(NO_2)_2{\cdot}CH_2]_2NH + H_2O$ where R may be H, K, NO_2 or any other substituent. The conversion, carried out in soln, normally appears to cease when a secondary amine has been formed. For some compds the reaction may proceed to the formation of a tertiary amine, as for instance in the case of the K salt of dinitroethanol. Here, the reaction with ammonia may lead to formation of either a primary amine $H_2N{\cdot}C(NO_2)_2{\cdot}CH_2OK$(See 2-Amino-2,2-dinitroethanol, K salt) or a tertiary amine $N[C(NO_2)_2{\cdot}CH_2OK]_3$(See Tris(2,2-dinitroethanol)amine]

If instead of ammonia a deriv such as hydrazine is used, the reaction with dinitroethanol may proceed thus: $2(O_2N)_2CH{\cdot}CH_2OH + H_2N{\cdot}NH_2 \rightarrow 2H_2O) + (O_2N)_2CH{\cdot}CH_2{-}HN{\cdot}NH{-}CH_2{-}CH(NO_2)_2$

Another example is the reaction between urea and trinitroethanol: $2(O_2N)_3C{\cdot}CH_2OH + H_2N{\cdot}CO{\cdot}NH_2 \rightarrow 2H_2O + [(O_2N)_3C{\cdot}CH_2NH]_2CO$ (See also Azetidine or Cyclotrimethyleneimine, Dinitro)

Refs: 1)Beil – not found 2)F.R.Schenck & G.A.Wetterholm,SwedP 148,217(1954) & CA **50**,1893(1954) 3)Ibid,USP 2,731,460 (1956) & CA **50**,7125(1956)

Note: Some of the compds patented by Schenck & Wetterholm may be found suitable as ingredients of explosive and propellant compositions

Ammonia Dynamites are listed in this work as *Ammonium Nitrate Dynamites*, with the exception of the following:
a)Ammonia Dynamite or Ammonia Powder: AN 78, NG 18, paraffin & charcoal 4%. It was a powerful expl suitable for all types of blasting(Ref 1)
b)Ammonia Dynamite No 1. A French expl: AN45, NG 40, Na nitrate 5 & wood flour 10% (Ref 2)
c)Ammonia Dynamite No 2. A Fr expl: AN 75, NG 20 & wood flour 5%(Ref 2)
d)Ammonia Dynamite Pulverulent: AN 25, Na nitrate 36, NG 20 & rye flour 19%(Ref 2)
(See also Ammonique Dynamite)

Refs: 1)A.R.Ramsey & H.C.Weston, "A Manual on Explosives," Dutton, London–NY(1917),21 2)CondChemDict(1942),287 (not listed in newer editions)

Ammonia Gelatin A: AN 67, NG 30 & CC (collodion cotton) 3%

Ref: CondChemDict(1942),287(not listed in newer editions)

Ammonia Gelatin Dynamites or Ammonia Gelatins are listed in this work as Ammonium Nitrate Gelatin Dynamites and also as Ammongelatin Dynamites. See also Ammongelatine in PATR 2510,p Ger 5

Ammonia Gelignite: AN 70, NG 29.3 & NC 0.7%

Ref: ConChemDict(1942),287(not listed in newer editions)

Ammoniak(Ger). Ammonia(In old Ger chem terms it was equivalent to "ammonium")

Ammoniakat(Ger). Ammoniate described in this work as Ammine

Ammoniakkrut is the first known expl based on AN. It was patented in Sweden on

May 31, 1867 by J.H.Norrbin & C.J.Ohlsson. Slightly later(June 9, 1867) Bjoerkmann patented, also in Sweden, an expl contg AN 72.46, NG 18.12, sawdust(or charcoal) 8.70 & benzene or creosote 0.72%, and named *seranin*(Ref 1,p 713)

The original expl of Norrbin & Ohlsson consisting of AN 80 & charcoal 20% was difficult to ignite and was replaced by AN 80, charcoal 6–10 & NG 14–10%(Refs 2–5). The modified compn, in which NG served as a sensitizer for the insensitive AN, was much more powerful than the Guhrdynamite, previously invented(ca 1866) by A.Nobel, if it contd an equal amt of NG (Ref 1,p 21). Davis(Ref 6) listed Ammoniakkrut as an expl consisting of AN, either alone or in admixture with charcoal, sawdust, naphthalene, PA or NB

The modified ammoniakkruts were used to some extent in Sweden, but were found to be too hygroscopic and exudable. A.Nobel purchased in 1870 Norrbin & Ohlsson's patents and reduced the hygroscopicity and exudability of Ammoniakkrut by coating the particles of AN with paraffin, stearin or naphthalene (BritP 1570 of 1873). Still better results were obtained when AN particles were coated with NG gelatinized with collodion cotton. The resulting expls became known as *gelatin dynamites*(qv)

Refs: 1)Daniel,"Dictionnaire''(1902), 21 & 713 2)Anon,SS **2**,57–8(1907) 3)VanGelder & Schlatter(1927),340 4)Naoúm,NG (1928),11 5)PepinLehalleur(1936),342 6)Davis(1943),335

Ammonia Nitrate(Poudre): AN 80, nitroglucose 10, K chlorate 5 & coal tar 5%

Ref: Daniel, "Dictionnaire''(1902),24

Ammonia Oxidation consists of treating anhydrous ammonia with air (or oxygen) at high temp and pressure and in the presence of a catalyst(such as Pt gauze) to obtain nitrogen oxides and eventually nitric acid. Installations for this treatment are called "Ammonia Oxidation Plants'', and nearly every large plant using nitric acid for the manuf of explosives includes such facilities. Some information on ammonia oxidation plants is given under Nitric Acid, Manufacture

Ammonia Powder. See under Ammonia Dynamites

Ammoniaque(Fr). Ammonia

Ammoniaque(Dynamite à) (Fr). Ammonia Dynamite

Ammoniate. Same as Ammine

Ammonio-Nitrogen Iodide. See under Iodides

Ammonique Dynamite. A safety expl prepd by mixing guhrdynamite(75% strength) 50, Amm carbonate 40 & K nitrate 10%. The nitrate was incorporated to prevent the formation of CO

Ref: Daniel, "Dictionnaire''(1902),22

AMMONITE

(Ammonit in Germany and in Russia; Explosif Favier type N n° 1c in France; Explosif Favier n°1 in Belgium; Ammonite in Italy and Amonita in Spain)(Formerly called in England "Miner's Safety Explosive''). A type of AN expl known since 1884(Refs 1 & 2) and manufd since then in many varieties in several European countries. In England they were manufd by the Miner's Safety Explosives Co, Ltd at Stanford-le-Hope, Essex(Ref 5); in France by the Poudrerie d' Esquerdes; in Belgium by the Usine de Vilvorde(Ref 3) and in Germany by several plants(See Refs 9 & 12–14)

Most ammonites were used as commercial expls(particularly in coal mining) but some of them were used in military applications chiefly as substitutes for HE's based on aromatic nitro compds(such as TNT) or nitric esters(such as NG)(See also Ersatzsprengstoffe in PATR 2510)

According to Cundill(Ref 4) the first expl of this type manufd in England consisted of a cylindrical container, made by compressing a mixt of AN 91.5 and MNN 8.5%, and filled either with a pulverized mixt of AN and MNN or with dynamite or NC. The resulting cartridge was wrapped in paper and then waterproofed. In France similar cartridges were

prepd by using mixts of AN with either 8.5 – 12.6% DNN or with 4.5% of TNN(See also Ref 3). According to Marshall(Ref 7) the original expl invented by Favier consisted of an AN, MNN, paraffin and resin. Another compn listed by Marshall(Ref 7,p 389) and then by Cook(Ref 18) was the ammonite contg AN 88, DNN 12%, which passed the Brit Woolwich test and was on the "Permitted List". In order to pass the more stringent Brit Rotherham test(in which the expl was fired, without stemming, into the gas mixt), the previous compn had to be modified to AN 75, TNT 5 and NaCl 20%(Ref 7,p 390). This expl was placed on the "Permitted List" in 1914(Ref 6), under the name of *ammonite No 1*. Its compn was modified after WW I to AN 79.5, TNN 5.5 and NaCl 15%. A similar expl known as *ammonite No 2* contained AN 79.5, DNN 5.5 and NaCl 15%. Both of these expls passed the Brit Buxton Test(Ref 10). Barnett(Ref 8) gives for *ammonites No 1 and No 5* the same compn: AN 75, TNN 5, NaCl 20% plus 0.5% moisture and for *ammonite No 4*: AN 66, Na nitrate 10, DNN 4 & KCl 20%, plus 0.5% moisture. The only difference betn the No 1 and No 5 was that the latter was put up in waxed paper and the former in metal foil cases. Molina(Ref 9a) gives compn of Ital *ammonite No 1* as: AN 88, DNT 3, vegetable flour 6 & NG 3%, plus traces of DPhA

According to Naoúm(Ref 9) and Pepin-Lehalleur(Ref 14), seven types of ammonites were used in Germany. Their compn was, in general, 70 to 88% of AN(of which 10% could be replaced by K nitrate), 7 to 20% of aromatic nitrocarbons and 1 to 6% of a vegetable meal w/or wo a solid hydrocarbon. Besides these components the Ammonits 1, 3, and 6 contd up to 4% of NG, the Ammonits 3, 4 & 5 contd 3 to 10% of K perchlorate and the Ammonit 5 contd 2 to 12% of Al. These expls are described in Ref 9, pp 424–5 and in Ref 11,p 118. Ger Ammonit 1 contg AN 80, TNT 12, rye meal 4 and NG 4% was also known as *Astralit* or *Donarit*(Ref 12,p 309) and Ammonit 2 contg AN 81, TNT 17 and rye meal 2% was also known as *Aldorfit* (Ref 12,p 309). A variety of Ammonit 2, contg DNT, was also known as *Astralit ON*(See PATR 2510,p Ger 10). Slightly different compns for Ammonit 1, Ammonit 2 and Donarit 2 are given in Ref 13,p 94

Table 2,p Ger 6 in PATR 2510 gives compns of Ammonits in Germany developed during WW II

For the prepn of ammonites, the thoroughly pulverized and dried ingredients were stirred in a pan for 1 or 2 hrs, then heated(by indirect steam or hot water) above the mp of a nitrocarbon and then cooled while stirring. The result of such treatment was that the grains of hygroscopic ingredients(such as AN) were coated with nonhygroscopic nitrocarbons(See Ref 7,p 388 and Ref 14)

Ammonites are apparently no longer manufd in western European countries, but are still very much in use in Russia. Radevich (Ref 16) describes some Russian pre-WWII ammonites and Bebie(Ref 17) says that ammonites are the main types of expls used in industrial practice in Russia. The following are some Russian ammonites as described in additional Refs 20, 22 and 23 listed below: a)AN 88 & TNT 12%(called Ammonite No 2) b)AN 73, K nitrate 15 & TNT 12% c)AN 77.6, TNT 18.4 & WM 4% d)AN 54.5–57.5, TNT 8.5–9.5, finely ground pine bark(contg less than 12% of H_2O) 2.5–3.5 & NaCl 31–33% e)AN 59.5–62.5, TNX 9.5–10.5, pine bark 2.5–3.5 & NaCl 25–27%(See also Ammoksil or Ammonxyl, Ammonal, Ammonpek, Ammontol and Dynanammon in this section and Ammonit I in PATR 2510, under Commercial Explosives)

Refs on Ammonites: 1)Favier,GerP 31,411 (1884) 2)Ibid,BritP 2139(1885) 3)Charton, MP 4,159–60(1891) 4)Cundill,MP 5,334–5 (1892)(Favier explosives) 5)Daniel(1902),22 6)Anon,JSCI 33,986(1914) 7)Marshall 1 (1917),388–90 8)Barnett(1919),133 9)Naoúm, NG(1928),424–6 9a)Molina(1930),339 10) Marshall 3(1932),119 & 153 11)F.A.Pershakov,

Ugol' i Zhelezo **1933**, No 90–91,pp 94–104 & CA **28**,899(1933) 12)Stettbacher(1933), 246 & 309 13)Beyling & Drekopf(1936),94–5 14)Pepin Lehalleur(1936),351–2 15)Thorpe, **4**(1940),554 & 556 16)P.Radevich, Tekhnika i Vooruzheniye **1938**,No 7,86–94 & CA **34**, 1173(1940) 17)Bebie(1943),23–4 17a)J.M. Jiménez, ''Explosivos'', Ediciones Ejército, Madrid(1951),29 18)Cook(1958),10

Addnl Refs on Russian Ammonites: 19)A.G. Suvorov & V.L.Machkarin,Chim & Ind **36**,785 (1936) & CA **31**,2437(1937)(The higher the moist content of ammonites, the larger the amts of noxious N oxides formed. For ammonites having a positive O balance of not more than 5% and intended for use in underground work, the max permissible moist content is ca 0.5% because with larger amts of w, the amts of N oxides produced would be excessive. With ammonites having a negative O balance the amt of moist can be as high as 2%. This type of ammonite is therefore preferred for underground work under damp conditions) 20)K.K.Andreyev & M.M.Purkal'n,DoklAkadN **25**,394–9(1939) (in Engl); GornyiZhur **1939**, No 2,44–7 & CA **34**,4270(1940)[Investigation of some Russian ammonites by expln in a closed bomb to det the amt of toxic gases(such as CO and N oxides) showed that in case of ammonite No 2,(AN 88 & TNT 12%), the increase in fineness of the ingredients (from sieve No 16 to No 49) and their thorough mixing decreased the amt of N oxides but had practically no effect on the amt of CO formed. Increase of moist increased the yield of noxious gases especially if the ingredients were finely ground. Increase in propn of TNT, decreased the amt of N oxides formed but increased the amt of CO. Addn of 4% birch wood at the expense of the AN gave the same results as the increase of TNT] 21)I.A.Mukhin,Ugol' **20**, No 1/2,27(1945) & CA **40**,2629(1946) (Ammonites may be waterproofed by heating them to 65° and mixing with 2% paraffin. Such ammonites may be wrapped in ordinary paper) 22)K.K.Andreyev & M.M.Purkal'n, DoklAkadN **51**,445–8(1946) & CA **40**,6817 (1946) (Investign of deton of ammonite, contg AN 73, KNO_3 15 & TNT 12%, showed that the amt of sand surrounding the chge greatly influenced not only the decompn of the chge itself but also the formation of CO and N oxides. Since in actual practice it is difficult to duplicate the favorable conditions established by lab expt, the addn of K nitrate will usually not solve the problem of reducing noxious gases. More promising results were obtained by changing the compn of ammonites in order to obtain higher heat of expln and by more thorough pulverization and mixing of the ingredients. For instance the charge of ammonite contg AN 77.6, TNT 18.4 & WM 4%, when surrounded by quartz sand, gave smaller amt of noxious gases than a similar compd contg K nitrate) 23)B.D. Agranovich et al, RussP 67,692(1946) & CA **43**,3200(1949) [Non-brisant ammonites can be prepd by adding to a finely ground mixt of AN and a HE(such as TNT or TNX) in a mixing drum, first the ground pine bark (contg not more than 12% of moist) and then NaCl] 24)V.A.Assonov & E.P.Maksimova, GornyiZhur **122**,No 10,18–19(1948) & CA **43**, 3617(1949) (Expl props of ammonites were greatly improved by providing in the base of the chge a conical cavity. In storage the density of ammonites increased while the brisance and transmission of detonation decreased) 25)L.V.Dubnov & N.S.Bakharerevich,GornyiZh **1950**,No 12,20–2 & CA **45**, 5920(1951) (Expls safe for use in sulfur mines were prepd by combining ammonite No 8 with flame attenuators, such as Amm chloride, Na chloride, aq agar-agar jelly and Na sulfate dekahydrate. Na chloride proved to be ineffective)(Compn of Russ ammonite No 8 is not given in CA) 26)V.A.Assonov, GornyiZhur **126**,No 7,25–8(1952) & CA **47**, 319–20(1953)(It is claimed that compressed ammonite is superior to the powder in shattering effect and in resistance to moist, thus not requiring a water-impervious cover)

27)N.E.Yaremenko & A.V.Korenistov, Ibid 28–30(It is claimed that pressed ammonite is in no way superior to the powd and the added cost of pressing is not justified) 28)N.I.Kozlov,Ibid **126**,No 10,21–2(1952) & CA **47**,1391(1953)(It is claimed that there is no significant diff between pressed and powdery ammonites when used in very small bore-holes for blasting hard rocks)

Ammonite Goudronite. A Russian coal mining expl consisting of AN and tar(goudron). Its props are given by N.A.Shilling,"Explosives and Loading of Ammunition", Oboronguiz,Moscow(1946),107(See also Ammonpek and Dynammon)

Ammonium Azides. See under Azides(Inorganic)

Ammonium Bicarbonate. See under Bicarbonates

Ammonium Bichromate(Dichromate). See under Chromates, Bichromates, etc

Ammonium Borate. See under Borates

Ammonium Bromate. See under Bromates

Ammonium Carbamates. See under Carbamate

Ammonium Carbonate. See under Carbonates

Ammonium Chlorate. See under Chlorates

Ammonium Chloride. See under Chlorides

Ammonium Chromate. See under Chromates

Ammonium Cobaltic Hexanitrate. See Ammonium Hexanitrocobaltate

Ammonium Compounds. A general description of such comps is given in Kirk and Othmer, **1**(1947),810–26(29 refs) as well as in the books on inorganic chemistry and in Chemical Abstracts. The compds which are used either as components of expls or propellants or for prepn of such components are listed in this dictionary individually,as for instance Ammonium Nitrate

Ammonium Cyanide. See under Cyanides

Ammonium Dichromate(Bichromate). See under Chromates, etc

Ammonium Ethylenedinitramate. See Ethylenedinitramine, Ammonium Salt, under Ethylenediamine

Ammonium Haleite. See Ethylenedinitramine, Ammonium Salt, under Ethylenediamine

Ammonium Hexanitrocobaltate or Ammonium-cobaltic Hexanitrite(Cobalticammonium Nitrite),$(NH_4)_3Co(NO_2)_6 \cdot 1\frac{1}{2}\ H_2O$. It was prepd according to Mellor(Ref 1) in 1857 by O.W. Gibbs on mixing an acidified soln of cobalt chloride with ammonium nitrite. The following expl props were determined at PicArsn: expln temp(5 sec) 230°; impact sensitivity (BurMines app, 2 kg wt) 33 cm and sand test 45% of TNT. It requires 0.30 g of MF for its initiation(Ref 2)

Refs: 1)Mellor **8**(1928),504 2)W.R.Tomlinson, K.G.Ottoson & L.F.Audrieth, JACS **71**, 375–6(1949)

Ammonium Hydroxide. See Ammonia, Aqueous

Ammonium Hypophosphite. See under Hypophosphites

Ammonium, Metal(Metal Ammonium). Solutions of mercury and the alkali metals in liq ammonia were first prepd and studied by Weyl (Ref 1). According to his views, the metals are joined to the nitrogen of ammonia, forming substituted ammonium radicals. Many investigators later studied the solns of metals in liq ammonia and while a few of the investigators were in favor of Weyl's theory(Joannis in 1892, Moissan in 1898 and Benoit in 1923), others were against it(Seeley in 1871, Ruff and Geisel in 1906, Kraus in 1908, Biltz in 1920 etc)

Kraus & Johnson(Ref 2) reinvestigated the solns of lithium in liq ammonia and came to the conclusion that there is no evidence indicating that Li is joined to the nitrogen of ammonia, forming a substituted ammonium ion

Note: The compds formerly called *"metal-ammoniums"* are now called *ammines*(qv) or *"ammoniates"*

Refs: 1)W.Weyl,Ann Physik(Poggendorff's Ann) **121**,601(1864) & ChemZtr **1864**,601–4 2)C.A.Kraus & W.C.Johnson; JACS **47**, 725–31(1925)(16 refs) & CA **19**,1360(1925) 3)J.N.Friend,edit, "Textbook of Inorganic Chemistry", vol 10, Griffin,London(1928), "The Metal Ammines" by M. J. Sutherland

AMMONIUM NITRATE

(AN or Amm Nitrate)

(Ammonsalpeter in German, Nitrate d' ammoniaque in French, Ammoniynaya Selitra in Russian, Nitrato amónico in Italian, Nitrato amonico in Spanish, Hsiao Hsuan An in Chinese and Amonum Shosanen in Japanese). NH_4NO_3, mw 80.05, N35.00%, OB to H_2O and N_2 + 20.0% cryst d at 25° 1.725 (Ref 127a) (for densities of various modifications, see below), d of material used for manuf of amatol 1.06 or higher (Ref 127a), d of molten material 1.402 at 175° and 1.36 at 200°. Hardness on Moh's scale 1.1, mp 169.6 - 169.9° (with slight sublimation) (Ref 122a), mp of technical grade ca 165°, bp of pure material ca 210° at 11 mm Hg pressure and it distills practically without decomposition. It decomp at 230° and 760 mm Hg pressure, and above 325° it deflagrates. There may be some decompn even at as low at 100°, since constant weight cannot be obtained at this temp, and decompn is quite perceptible above the mp of AN. If confined, AN may explode between 260 and 300°. When liquid AN is cooled below its fr p, there are formed cubic crysts which, on further cooling, undergo (at 125.2°) transition into tetragonal crysts. There are five allotropic modifications of solid AN (Refs 122 & 122a)

The following table gives the crystallographic characteristics of the different forms of AN:

Form	Crystal System	Density, g/cc	Range °C
Liquid	–	–	Above 169.6
I Epsilon (ϵ)	Regular (cubic) (isometric)	1.594 at 130 ±5°	125.2 to 169.6
II Delta (δ)	Rhombohedral or tetragonal	1.666 at 93 ±5°	84.2 to 125.2
III Gamma (γ)	Orthorhombic	1.661 at 40 ±1°	32.1 to 84.2
IV Beta (β)	Orthorhombic	1.725 at 25°	–16 to 32.1
V Alpha (α)	Tetragonal	1.710 at –25 ±5°	–18 to –16

The heat of transition from form III to form II is 310 kcal/mol and that from form II to form I is 979 kcal/mol. Whetstone (Ref 113) studied the initiation of transition between forms III and IV. The effect of foreign substances on the transition IV $\rightleftarrows$ III was studied by Campbell and Campbell (Ref 81) who found that in the case of a solid solution of 8 to 10% of KNO_3 in AN the temperature of transition of form III into form IV is depressed by about 20°. Such solid solutions can be prepared either by fusion or by co-crystallization from aqueous solutions. Hendricks et al (Ref 40) found form III to be orthorhombic and form V to exist up to –18° and not to –16°. Bowen (Ref 30) showed that there is also a metastable inversion occurring at about 50° as follows: orthorhombic form (β)(32.1° to –16°) $\rightleftarrows$ tetragonal form (δ)(125.2° to 84.2°).

The heat of transition of various modifications of AN was studied also by Steiner and Johnston (Ref 36). According to Assonov and Rossi (Ref 68), beta orthorhombic crysts, which are stable up to 32.1°, do not cake provided that the moisture content is less than 0.5%. If the temp is raised above 32.1°, these crysts (d 1.725) undergo an increase of about 3% in volume and then break up into a fine, cryst powder having a d of 1.66. When stored in the open, this powder hardens (cakes) like cement. This is especially pronounced in the presence of moisture. Caking is also observed when crysts of the gamma form are cooled below 32.1°. This occurs with relatively dry samples having less than 0.15% moisture.

Historical (Refs 31, 62, 71 & 72)

AN is very seldom encountered in nature. The first description of its preparation and properties was in 1659 by Glauber, who treated $(NH_4)_2CO_3$ with HNO_3 and called the resulting salt "Nitrumflammans". Gmelin, at the start of the 19th century, called AN "Flammender Salpeter". Grindel and Robin were the first, in the beginning of the 19th century, to use it in explosives – as a replacement for KNO_3 in black powder. In 1840 Reise and Millon reported that a mixture of powdered AN and charcoal exploded on heating to 170°. In 1867 the Swedish chemists Ohlsson and Norrbin patented an explosive called "Ammoniakrut" which consisted of AN with a small amount of other ingredients. Nobel purchased the patent in 1870 and started to work on the possibility of rendering the AN less hygroscopic by various treatments. He partly succeeded in doing this in his "Extra Dynamites" or "Ammongelatin Dynamites", patented in 1879. In these explosives AN crystals were coated with a jelly consisting of NG and collodion cotton (Refs 5, 9, 10 & 84)

The study of AN was continued and in the eighties of the 19th century Berthelot published a theoretical equation for its decomposition (Refs 1 & 2). At the beginning of the present century Kast and Naoúm studied and described such explosive properties of AN as sensitivity to detonation, impact and heat, rate of detonation, Trauzl block value etc

Until the time of the Oppau disaster (1921), AN was not considered to be an explosive. This disaster called for more extensive research into its properties, and numerous works were published as a result of such investigations (Refs 15a & 15b). However, the findings of the various investigators were somewhat at variance. While some claimed that AN itself cannot be detonated unless it is strongly confined and a very strong initiating charge is used, others claimed that it can be detonated even on strong heating if confined, because the gases formed by decomposition are explosive, detonate first and cause the explosion of any remaining molten material. Although the Oppau disaster certainly showed that AN is an explosive, fires and explosions continued to occur throughout the world, although generally on a small scale

After the termination of World War II, the US Government began shipments to Europe of so-called FGAN (Fertilizer Grade Ammonium Nitrate), which consisted of grained AN coated with about 0.75% of wax and conditioned with about 3.5% of clay. Since this material was not considered to be an explosive, no special precautions were taken during its handling and shipment – workmen even smoked during the loading of the material. Numerous shipments were made without trouble prior to April 16 and 17, 1947, when one of the worst explosions in history occurred. The SS Grandcamp and the SS Highflyer, moored in the harbor at Texas City, Texas and loaded with FGAN, blew up. For description and probable causes, see under "Ammonium Nitrate Explosions and Fire Hazards"

As a consequence of this disaster, a series of investigations was started in the United States in an attempt to determine the possible causes of the explosions. At the same time a more thorough study of the explosive properties of AN and its mixtures with organic and inorganic materials was instituted

The explosion at Texas City had barely taken place when a similar one aboard the SS Ocean Liberty shook the harbor of Brest, France on July 28, 1947. As investigations following the Texas City and Brest explosions showed that AN is much more dangerous than previously thought, more rigid regulations governing its storage, loading

and transportation in the United States were promptly enacted

At present, AN is classified as "an explosive ingredient" and oxidizing material. Its manufacture, use, storage distribution and possession are regulated by the Federal Explosives Act, which is administered by the US Bureau of Mines. A yellow label, the same as for Amm perchlorate, is required on all railroad shipments. Since 1947, fortunately, there have been no major disasters with AN, only a few fires have occurred

Preparation of AN (General Discussion)

Until World War I, AN was manufd chiefly by neutralizing, with weak HNO_3, the NH_3 present in aqueous by-products of the artificial gas and coking industries. As the HNO_3 was then manufd from Chile saltpeter, it contained HCl, HNO_2 and boric acid as impurities, while the gas liquor NH_3 used contained pyridine and thiocyanates. Consequently, the AN also contained the same impurities. Such AN was used in blasting explosives and, to some extent, in mixed fertilizers. In 1913 the manuf of HNO_3 from NH_3 produced from atmospheric nitrogen was begun in Europe. This acid was of a higher degree of purity and, as it was neutralized with synthetic NH_3, the AN produced was much purer than that obtained by the earlier process

Some later achievements and problems in the manuf of AN and Amm sulfate have been reported by Perelman and Klevke (Ref 69). In a patent issued for nitrogen fertilizers from NH_3 and nitric, phosphoric or sulfuric acid, Lutz (Ref 100) described a procedure for the prepn of AN. Strel'zoff (Ref 98) described a process for the manuf of AN from NH_3 and aq HNO_3 in which the heat of reaction is utilized to evap and conc the soln so that the cryst AN can be withdrawn without any addition of external heat. By maintaining the reaction zone at a relatively high pressure and the concentrating zone at a low pressure, a sufficient temp gradient was set up to allow a rapid and efficient transfer of heat from the hot vapors of the reactor to the soln to be boiled and concd. A typical arrangement producing 32100 lb/hr is described in the patent. Seaman et al (Ref 102) reported the production of AN by continuous vac crystn. An apparatus to control the AN content of waste water from manufg plants has been described by Krichmar (Ref 117)

Laboratory Procedure. A. Pérez Ara (Ref 76) gives the following laboratory method of prepn of AN: Dilute in a beaker 100 ml of commercial nitric acid with an equal amount of water. Add coml Amm hydroxyde slowly, while stirring and cooling the soln, until it is alkaline to litmus paper. Evaporate on a steam bath to the formation of a crust. Cool the soln and separate crysts from the mother liquor. Dry the crysts and, if desired, purify by recrystn from distd water

Plant Processes. Following are some of the processes for manuf of AN:

1. Passing NH_3 gas into 40-60% HNO_3. This is the most common method of manuf and is described further with more detail. Symmes (Ref 19) was one of the first to describe this process in detail and later assigned the patent to the Hercules Powder Co (Ref 33)

2. By the double decomposition of calcium nitrate and ammonium carbonate (or sulfate) in solution: $Ca(NO_3)_2 + (NH_4)_2CO_3 \rightarrow CaCO_3 + 2NH_4NO_3$

3. By the double decomposition of ammonium sulfate and sodium nitrate in solution: $(NH_4)_2SO_4 + 2NaNO_3 \rightarrow 2NH_4NO_3 + Na_2SO_4$. This process, patented by Freeth and Cocksedge (Ref 13), was an economical one up to the time of the development of synthetic NH_3 and of HNO_3 by ammonia oxidation

4. By using sodium nitrate instead of common salt in the ammonia-soda process: $NaNO_3 + NH_4HCO_3 \rightleftarrows NH_4NO_3 + NaHCO_3$

5. By mixing the gases ($NO_2 + O_2 + H_2O$) obtained as by-products of ammonia oxidation plants, with ammonia gas and extra air in order to bring about the following reaction: $4NO_2 + O_2 + 2H_2O + 4NH_3 = 4NH_4NO_3$. In this process, AN is deposited as a powder

The first of these processes is now usually conducted on a plant scale as follows, the operation being continuous:

a. 40% nitric acid is gradually fed into a stainless steel open vessel (reactor or separator) and an equimolecular quantity of gaseous NH_3 is introduced simultaneously beneath the surface of the acid. The reaction $NH_3 + HNO_3 = NH_4NO_3$ is highly exothermic and causes the solution to boil, thus partially concentrating it

b. As soon as the reactor is full and/or the solution has reached a conc of about 50-55% AN, a valve at the bottom is opened and the liquor is continuously drawn to a cooler. From there it is run into a storage tank. While the solution is being removed from the reactor, it is continuously replaced with equivalent amounts of HNO_3 and NH_3

c. The AN solution in storage can either be shipped directly as such (to be used as fertilizer) or it can be converted into solid AN by evap. Several methods of evaporation and crystallization are used, as described below:

Methods of Evaporation of AN Solutions

A. *Batch Process* (Refs 93, 102). The saturated solution from the storage tank is gravity fed to open evaporating pans ("high pans") provided with stainless steel or aluminum heating coils and air agitation. The soln is evaporated to about 98% at a temp of 155-160°C (310-320°F). The evaporn is stopped when the material fudges. The fudge is transferred into flat grainers ("low pans") provided with slowly rotating stainless steel paddles. Stirring the syrup in the open, cools it, drives off the remainder of the water and produces small, rounded crystals of AN. When the granulation has reached a certain point, a coating material is added and the stirring continued for a while to insure uniform coating. One ton of AN so produced requires approx 440 lbs of NH_3 and 1630 lbs of 100% HNO_3 (Yield 98%)

B. *Continuous Process of Graining (Spray Granulation or Prilling* (Refs 104 & 118). The AN solution is transferred from the storage tank to evaporating pans ("high pans") where it is evapd to a concn of about 95%. The hot soln (about 140°) is pumped to the top of a spraying chamber (20 ft square by 70 ft high) and fed into the chamber through a sprayer. As the soln falls, the remainder of the water evaporates, leaving spherical grains about the size of buckshot, called "prills", which fall to the bottom of the tower. In order to prevent caking of these particles, they are dried further and coated with substances such as wax, paraffin and, in the case of FGAN, with diatomaceous earth, clay etc. The resulting product is called "Nitraprill" and can be stored without becoming caked (See Note, p A340)

C. *Continuous Vacuum Crystallization* (Refs 102, 118). The AN, after being concentrated in "high pans" to about 75-80% strength, is transferred to a special stainless steel vacuum crystallizer, similar to the "Oslo-Crystal" classifying type. Evapn is conducted at an absol press of 2 mm of Hg and a temp of 36°C (97°F). The resulting slurry, contg about 40% by weight of crystals, is continuously removed from the bottom and run through a centrifuge. The crysts, contg about 1% water, are dried to a 0.1% moisture content. The mother liquor is returned to the system (See also Ref 54) (See Note, p A340)

Other methods of manufg AN are based on complete utilization of the heat of reaction. They include the following:

6. *Fauser Process.* This method originated in Italy and has assumed considerable importance in Europe (Refs 52 & 85)

7. *Bamag Meguin A-G Process* (Refs 58 & 85)

8. *Caro and Frank Process* (Refs 41 & 85)

9. *Tomolo Process* (Refs 34 & 85). Some comparatively recent patents for the production of AN have been assigned to the du Pont Co (Refs 63, 64 & 66) (See also Refs 95 & 127)

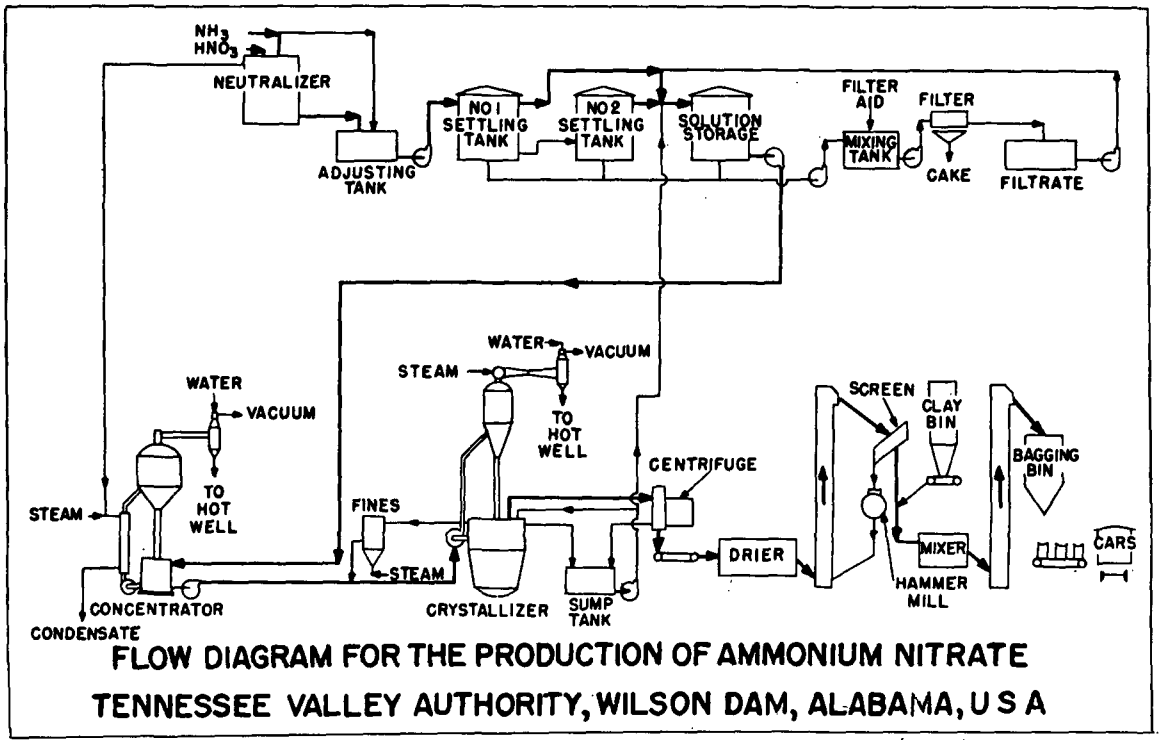
10. *TVA Process* (Refs 85, 93 & 102). In 1933 the Tennessee Valley Authority inherited a World War I plant designed to produce ammonia by a roundabout and obsolete method in the following steps: first the manuf of lime and subsequently Ca carbide, then Ca cyanamide, ammonia, nitric acid and finally AN. In 1940 a modern high-pressure ammonia plant was constructed, in which there were used an improved ammonia synthesis catalyst and a water-gas conversion catalyst. During WW II, the TVA produced 29000 tons of anhydrous ammonia, 10000 t of AN liquor, 64000 t of AN crysts and over 375,000 t of phosphate and nitrate fertilizer. When the Ordn Dept in 1943 reduced its demand for AN, TVA changed to the production of AN suitable for use as a fertilizer. In cooperation with the US Dept of Agric, the TVA developed an improved method of conditioning grained AN. This process was subsequently adopted by Ordn plants. In 1948 the batch graining process was replaced by a continuous crystg process operated at low temp; hence it is the safest known process for prepg cryst AN. In spite of the superiority of the coke-air-water process of ammonia production over the old cyanamide process, a more economical process is one based on the use of natural gas as the raw material. The TVA plant has been converted to the use of gas for this more efficient process of producing fertilizer and munition grades of AN. The TVA flow diagram for AN is included. (See next page)

11. *Stengel Process* (Refs 99, 105, 114 & 118). The neutralization of ammonia with preheated nitric acid takes place in a packed, tubular reactor at about 204°. The molten AN collects on the packing, flows to the bottom of the reactor and through a centrifugal separator, the bottom portion of which is also filled with packing. The heat of the highly exothermic reaction between NH_3 and HNO_3 serves to evaporate any water present. Air blown through the material controls the moisture content at almost any desired level. The molten product leaving the bottom of the reactor is essentially water free (0.2%) and goes to a weir box. A sheet of AN forms on water-cooled Sandvik belts, at the end of which is a breaker which reduces the sheet to flakes. The flakes are transferred to grinders, screens and a coating drum (See also the patented process of Davis, Ref 96). The Stengel process is in use at the Sterlington plant of the "Commercial Solvents" (See enclosed flowsheet)

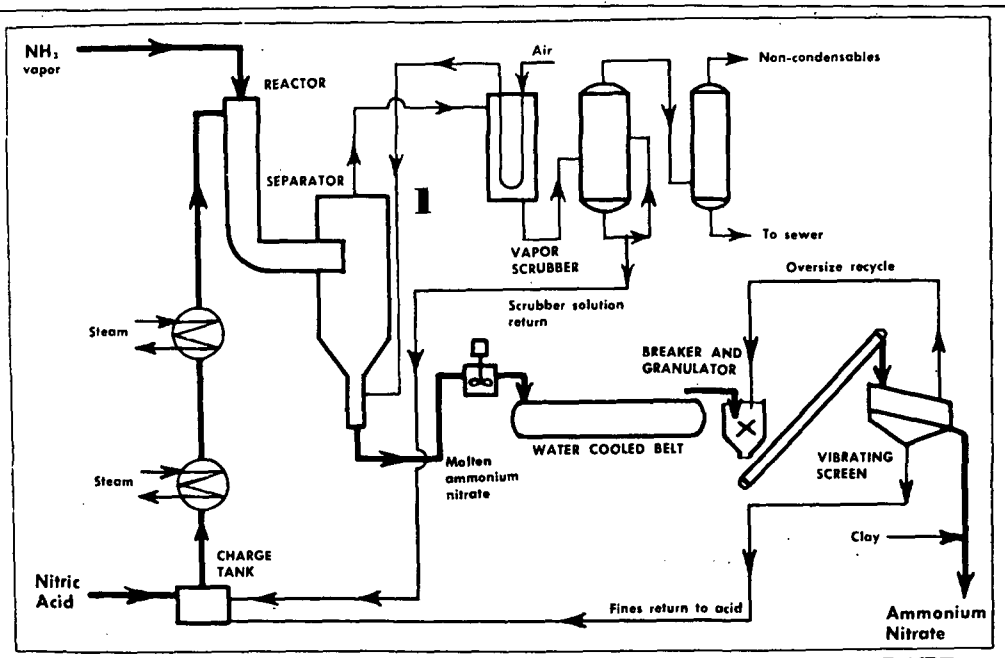
Besides the above Stengel Process, there are two other modern commercially important processes – the *Prilling Process* and the *Crystallization Process* (See inclosed flowsheets)

In the *Prilling Process,* such as practiced by the Lion Oil Co (Division of Monsanto Chemical Co, El Dorado, Arkansas), the production centers around a prilling tower, where a concd soln of AN forms into small droplets which flow downward against a stream of air. The resulting slightly moist prills are screened, dried and cooled by air and again screened

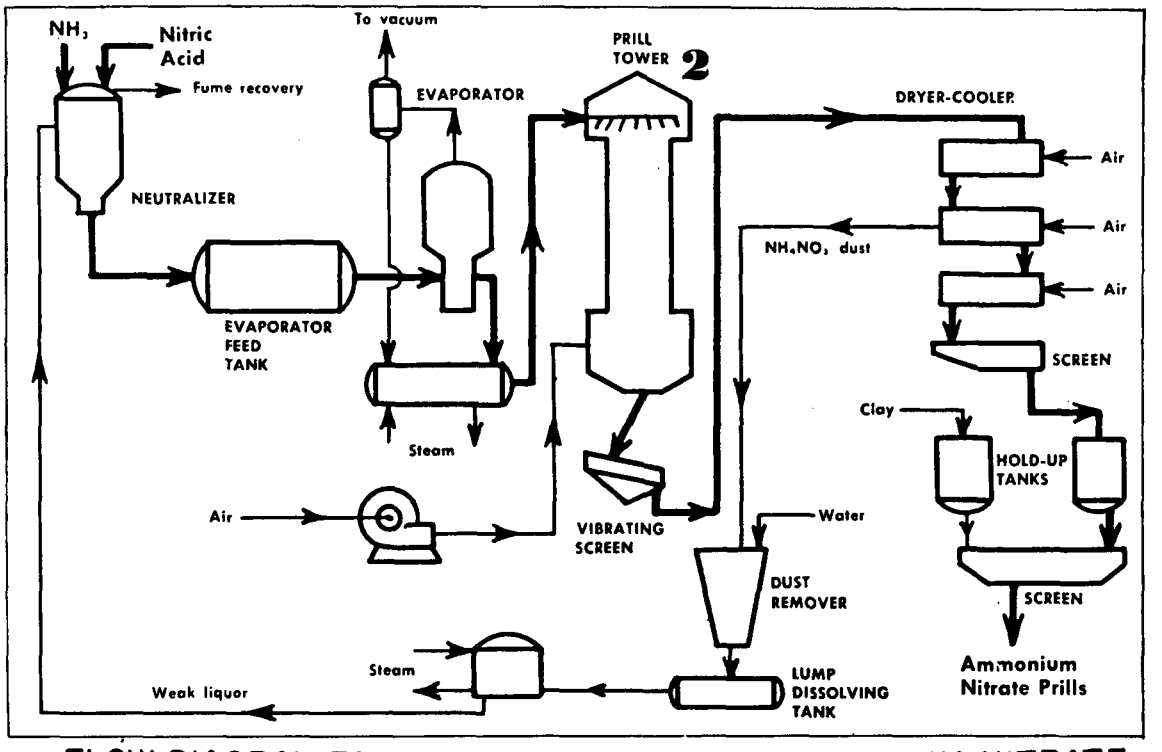
In the *Crystallization Process,* such as practiced by Aburdarverksmidjan HF plant,



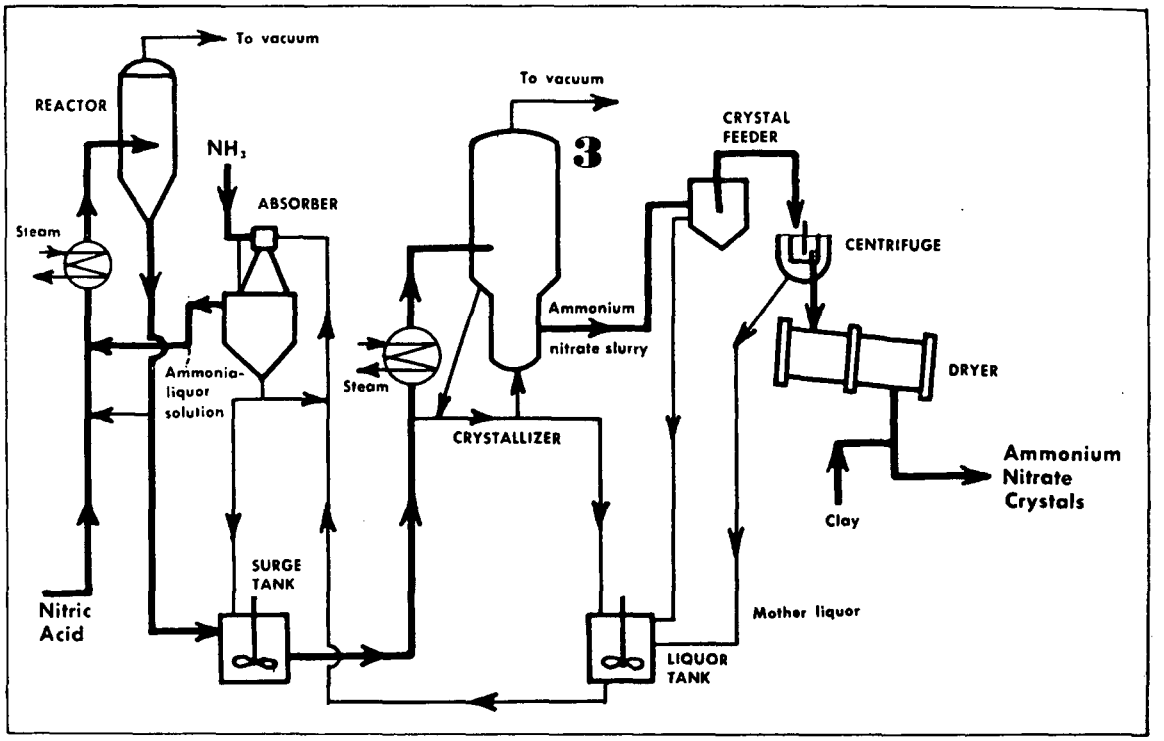
FLOW DIAGRAM FOR THE PRODUCTION OF AMMONIUM NITRATE
TENNESSEE VALLEY AUTHORITY, WILSON DAM, ALABAMA, U S A



FLOW DIAGRAM FOR THE PRODUCTION OF AMMONIUM NITRATE
BY THE STENGEL PROCESS (COMMERCIAL SOLVENTS)



FLOW DIAGRAM FOR THE PRODUCTION OF AMMONIUM NITRATE BY THE PRILLING PROCESS (LION OIL CO)



FLOW DIAGRAM FOR THE PRODUCTION OF AMMONIUM NITRATE BY THE CRYSTALLIZATION PROCESS (ABURDARVERKSMIDJAN HF)

the production centers around a vacuum crystallizer, where a satd soln of AN is cooled under vacuum to produce a slurry, which goes next to a centrifuge where crysts of AN are separated. After removal of moisture in a dryer, the crysts are coated with clay to prevent their caking

Note: High Pan Fires and Explosions. Numerous fires, sometimes resulting in explosions, have occurred during the concn of AN in "high pans". In view of this, it might be inferred that unconfined, pure AN may be detonated by heat alone. However, this is not the case, as investigations of such fires and explosions have shown that, there was confinement and there was definite proof of the presence of some organic inflammable materials such as TNT, carbonaceous matter, organic nitrates etc. The burning of these organic materials has been considered to yield so much heat that the bulk of the AN begins to decompose and the rate of such decomposition increases so rapidly that it becomes explosive. For more information on this subject, see Ref 86

Preparation of Fertilizer Grade Ammonium Nitrate (FGAN). As many explosives plants produce fertilizer grade AN, a short description of the American process is included. After transferring the AN fudge liquor from the "high pan" to the "low pan" (graining kettle) (see above under Methods of Evaporation), agitation and cooling are continued until the fudge breaks into grains. At this point, coating material [wax and clay; petrolatum, rosin and paraffin wax (PRP); or similar material] is added and stirring is continued until coating is complete. This coating (0.5 to 1.0%) with a waxy, water-repellant substance is necessary in order to render the nitrate less hygroscopic. In order to prevent caking of the coated grains, 3 to 5% of a so-called "conditioning" (anti-caking) agent such as powdered clay (kaolin), kieselguhr, "Celite," plaster of Paris, tricalcium phosphate etc is introduced and mixed with the AN. Very good results with a product called "Kittitas" (qv) have been reported. The AN is screened and the portion passing through an 8-mesh screen is bagged in moisture-proof bags at a temp not exceeding 93°C (200°F) (See Note, p A340)

The finished FGAN is required to meet the following specifications: moisture-0.25% (max), ether-soluble matter 0.75% (max), water-insol matter, such as clay 3.50% (max), nitrogen 32.50% (min); granulation: through US Std Sieve No 8 100% (min), on US Std Sieve No 35 55% (min) and through US Std Sieve No 100 8% (max)

For more information on preparation of FGAN, see Refs 74, 82 & 87

Properties of Ammonium Nitrate. Refs 5, 8, 11, 16, 56, 84, 109, 128, & 129 give one or several of the following properties of AN:

Ability to Propagate Detonation. AN has a very low ability to propagate a detonating wave. It happens very often that if a charge (cartridge) is long and of small diameter and unconfined, the detonating wave dies out (dampens) before it reaches the opposite end of the cartridge

Ballistic Mortar Test Value. See under Power

Behavior Towards Heat. See Sensitivity to Initiation by Heat

Behavior Towards Metals and Other Substances. According to Mellor, vol 7 (Ref 16), fused AN will not react at room temperature with As, Sn or Hg but will react with Al, Zn, Pb, Sb, Bi, Ni, Cu, Ag and Cd. Of these metals, Zn is attacked very rapidly and so is Cu. Fe reacts in the presence of moisture with the formation of ammonia (Ref 122a). According to Kast (Ref 31), the presence of $KMnO_4$ may cause the spontaneous ignition of AN. Investigations conducted at Pic Arsn (Ref 88) showed that different Cr compounds catalyze

the decompn of AN. According to Ref 122a, p 120, AN is decomposed by strong alkalis with the liberation of ammonia, and by sulfuric acid with the formation of $(NH_4)_2SO_4$ and HNO_3. In the presence of moisture, AN reacts with copper to form tetramminocupric nitrate, $[Cu(NH_3)_4](NO_3)_2$, which is of the same order of brisance and sensitivity to impact as lead azide. For this reason, tools of brass or bronze should not be used in operations with explosives contg AN. AN has little if any effect on coatings of acid-proof black paint, shellac, baked oil or NRC Compound (Ref 122a) (See Note, p A340)

Brisance, by the following methods: a) Lead Cylinder Compression Test, 54% of that of TNT b) 200 g Sand Test, only partial explosion takes place (Ref 122a) c) Kast Formula (Max Potential Work), 17,000 for complete and 5,800 for incomplete decomposition as compared with 86,000 for TNT (Ref 56) d) Fragmentation of 40 mm shell (average charge 44.5 g at d 1.0) - 16 fragments for AN coated with wax vs 66 fragments for TNT

Note: According to Ref 122a, p 123, because of its low rate of detonation, the brisance of AN is relatively low. Fragmentation tests in small shell loaded with AN and with TNT showed the nitrate to produce only 24% as many fragments as TNT

Coefficient of Expansion. See Thermal Expansion

Decomposition Reactions. Berthelot stated that AN can decompose according to any of the seven equations given below. The heats of decompn (indicating the heat evolved at const vol and 300°K) were calcd by C.G. Dunkle of Pic Arsn, based on the latest NDRC values. Unless otherwise stated, these values are for the solid salt. For molten AN, add about 4,000 cal/mol to these values. Values in *square brackets* are those of Scott and Grant (Ref 90), and were calcd from the data of Bichowsky and Rossini (Ref 57). The values for heat liberated are at const press and 18° for solid AN, with all products of decompn in gaseous form. It should be noted that the temps of decompn indicated here for some of the reactions are only approx and are not those originally given by Berthelot (Ref 1), but by later investigators. None of the Berthelot's reactions occurs as a single reaction, but is always accompanied by other reactions. The higher the temp of decompn, the more the reaction approaches reaction c (see below)

Berthelot's reactions are:

a. $NH_4NO_3 \rightarrow HNO_3 + NH_3 + 38.30$ kcal. This reaction takes place at a temp somewhat above the mp of AN(169.9°). The corresponding value for the solid salt is -41.70 kcal

b. $NH_4NO_3 \rightarrow N_2O + 2\,H_2O + 13.20$ kcal (H_2O gas) and 33.10 (H_2O liq). For the same reaction the value of -10.7 kcal is given in in Ref 122a, p 121. According to Berthelot, this reaction takes place at 180-200° when the AN is not confined. Other oxides (than N_2O) form at 230 to 285°. Berthelot also reported that AN decomp with puffs of smoke when heated to 260°

c. $NH_4NO_3 \rightarrow N_2 + 0.5\,O_2 + 2\,H_2O + 30.50$ kcal [28.47] (H_2O gas) or 50.40 (H_2O liq). For the same reaction, the value of +27.72 kcal is given in Ref 122a. This reaction is stated to take place when AN is heated under strong confinement or when initiated with a powerful detonator. It is the principal reaction of complete detonation of AN. According to calcn, this reaction developes temp 1500° and pressure 11200 kg/cm². The gas evolved is calcd to be 980 l/kg at STP

d. $NH_4NO_3 \rightarrow NO + 0.5\,N_2 + 2H_2O + 9.0$ kcal [6.87] (H_2O gas) or 28.90 (H_2O liq). This is supposed to be one of the side reactions taking place during incomplete detonation and one which developes a pressure of 4860 kg/cm² and a temp of 518°

e. $3\ NH_4NO_3 \rightarrow 2\ N_2 + N_2O_3 + 6\ H_2O + 20.80$ kcal [21.80] (H_2O gas) or 40.60 (H_2O liq). This reaction cannot take place alone because N_2O_3 exists only in the dissociated state as $NO + NO_2$

f. $4\ NH_4NO_3 \rightarrow 2\ NO_2 + 3\ N_2 + 8\ H_2O + 29.80$ kcal [24.46] (H_2O gas) or 49.80 (H_2O liq). This is another possible side reaction, occurring during incomplete detonation. For this reaction Ref 122a, p 121 gives the value of 96.0 kcal which does not agree with the value given by Berthelot. Ref 122a also gives the following endothermic reaction of decompn, which was not listed by Berthelot: $4\ NH_4NO_3 \rightarrow 3\ NO_2 + 5\ H_2O + N_2 + NH_3 + NO - 84.88$ kcal

g. $5\ NH_4NO_3 \rightarrow 2\ HNO_3 + 4\ N_2 + 9\ H_2O +$ 35.10 kcal [31.12] (H_2O gas) or ca 55 kcal (H_2O liq with HNO_3 dissolved therein). This reaction takes place under certain conditions, such as in the presence of spongy Pt and gaseous HNO_3 (Ref 1)

Later investigators have shown that the following other reactions of decompn of AN are possible:

h. $8\ NH_4NO_3 \rightarrow 16\ H_2O + 2\ NO_2 + 5\ N_2 +$ 16.58 kcal (207 cal/g). Saunders (Ref 21) found that this reaction takes place during incomplete detonation and is accompanied by a yellow flame. The calcd value for total gas developed by this reaction is 945 l/kg

i. $NH_4NO_3 \rightarrow 0.5\ NH_3 + 0.75\ NO_2 + 0.25\ NO +$ $0.25\ N_2 + 1.25\ H_2O - 21.20$ kcal. Kaiser (Ref 52) lists this reaction of decompn of AN when under confinement and at 200 to 260°. This endothermic reaction is followed, at 260 to 300°, by explosion of the gaseous products of reaction. This expln is an exothermic reaction liberating 48.94 kcal which is more than 1.5 times as great as the heat liberated by the reaction c (see above)

j. According to Ref 122a, p 121, the most important reactions of decompn of AN, when heated under various conditions, are:

$$NH_4NO_3 \rightarrow N_2O + 2\ H_2O + 10.7 \text{ kcal}$$

$$4\ NH_4NO_3 \rightarrow 2\ NO_2 + 8\ H_2O + 3\ N_2 + 96.0 \text{ kcal}$$

$$4\ NH_4NO_3 \rightarrow 3\ NO_2 + 5H_2O + N_2 + 2NH_3 + NO - 84.88 \text{ kcal}$$

In the last reaction the decompn is endothermic, and if the gaseous mixt of products of decompn is heated, these react exothermically with explosive effect
(See also Heat of Decomposition, Heat of Dissociation and Heat of Explosion of AN)

Detonation Rates (Velocities of Detonation). Rates as low as 1000 and as high as 3000 m/sec have been reported in the literature for AN depending on the conditions and methods of testing. The most important factors affecting the velocity are: density, degree of confinement, charge diameter, particle size (especially at low densities), strength of initiating impulse (nature and quantity of booster charge), temperature of sample and the presence of certain impurities such as organic materials or oxidizable metals (Refs 45 and 94)

According to Ref 122a, p 123, values from 1100 to 2700 m/sec were observed for AN, depending on whether the detonation was incomplete or complete. Because of the insensitivity of AN its rate of detonation is affected by its particle size, apparent density, degree of confinement, efficiency of booster charge and temperature of charge. Increase in rate is brought about by decrease

in particle size, decrease in apparent density of charge and increase in confinement. Increase in the temp of charge from 15 to 140° has been found to result in an increase of 400 m/sec in the rate

The following table gives values for rates of detonation of AN, and its mixtures under various conditions of testing. These values were taken from different sources as indicated

Rates of Detonation

Rate, m/sec	Density, g/cc	Charge dia, mm	Container	Initiation by	Charge temp, °C	Investigator	Ref
			Pure AN				
				No 8 cap plus:			
1140	0.68-0.73	26.2	Shelby tube	75 g tetryl	15	Gawthrop	29
1560	0.68-0.73	26.2	53.5 cm long, 4.85mm wall	75 g tetryl	71	Gawthrop	29
1230	0.69	50	Steel tube	100 g PA	Room	Kast	31
1310	0.84	25.	Steel tube	50 g tetryl	Room	Kast	31
1470	0.83	26	Steel tube	60 g tetryl	Room	Kast	31
1530	0.79	80	Steel tube	100 g PA	Room	Kast	31
1550	0.88	80	Steel tube	100 g PA	Room	Kast	31
1820	0.84	100	Steel tube	200 g PA	Room	Kast	31
1850	0.82	26.2	Shelby tube	100 g tetryl	Room	Perrott	37
1920	0.64	100	Steel tube	100 g PA	Room	Kast	31
2440	--	50	Lead tube	100 g PA	Room	Aufschläger	24
2700	0.98	80	Steel tube	250 g tetryl	Room	Kast	31
1200–1500	When incompletely decomposed					Stettbacher	44
2000–2500	When completely decomposed					Stettbacher	44
1500	Calculated value for incomplete detonation					Kast	31
3000	Calculated value for complete detonation					Kast	31
			AN + 1% Nitrostarch				
1940	0.82	26.2	Shelby tube	100 g PA	Room	Perrott	37
			AN + 5% Nitrostarch				
2060	0.82	26.2	Shelby tube	100 g PA	Room	Perrott	37
			AN + 10% Nitrostarch				
2470	0.82	26.2	Shelby tube	100 g PA	Room	Perrott	37
			Fertilizer Grade Ammonium Nitrate (FGAN)				
1106	0.90	114.3	Paper tube	225 g Comp C	Room	Macy	88
1350	0.91	31.7	Steel tube	50 g Comp A3	Room	Macy	88
2109	1.4	30	Glass tube	50 g Comp A3	>169°	Macy	88

Note: Booster charges of PA, tetryl, Comp A3, Comp C, etc were compressed

The above results show that rates of deton increase (within certain limits) with increased strength of initiating agent, increase in confinement, increased diam of charge, increase in d of charge, increase in temp and the presence of organic compds whether explosive or nonexplosive

According to Aufschläger (Ref 24) the rate of deton of AN increases with increasing diam of the charge, and if the diam is great enough, unconfined AN can detonate; the rate decreases with increased distance from the initiator

Aufschläger also found that: a) A 10 g charge of AN in a Trauzl lead block, under slight confinement, can be detonated completely by a No 8 blasting cap, but with a No 6 cap deton was not complete and with smaller caps no deton occurred. The Trauzl test value was about 165 cc b) Increasing the grain size of the AN seemed to improve propagation of deton, but if the crysts were too large the d was decreased to such an extent that the blasting effect was decreased. With AN of max d, such as is obtained by fusing and then solidifying the salt (d 1.65), only partial deton took place as indicated by the Trauzl test c) When large quantities of AN were tested under slight confinement or no confinement, they could be detonated only by a very strong initial impulse d) The sensitivity of AN is increased by the incorpn of even very small quantities (1-2%) of organic substances such as coal dust, wood pulp etc e) The vel of deton of AN decreased with decrease in confinement, decrease in diam of container and increase in distance from point of initiation. The influence of change in initiating agent is not very great. The lowest velocity (1270 m/sec) was obtained with No 5 to 8 caps in steel tubes of 40 mm diameter. The highest value (2450 m/sec) was obtained in 60 mm seamless steel tubes (Mannesmann's), using PA as the booster

Kast (Ref 23) reported velocities of detonation ranging from 1200 to 1900 m/sec when AN was loaded at d of 0.65 to 1.0 in wrought iron tubes having diams from 2.5 to 10 cm and the charges were detonated by means of boosters consisting of 50 to 300 g of tetryl or PA. Monroe (Ref 25) found that AN could not be detond at ord temps when unconfined, but when confined, it could be detond by several different detonators. The certainty with which deton could be effected increased with the degree of confinement. On the other hand, the certainty of deton by initiation decreased with an increase in d of the AN; that is, the salt tended to become "dead pressed" (See also studies by Parisot, Ref 67)

C.G. Dunkle of Pic Arsn conducted studies of the rate of deton of TNT and amatols at a d of 1.5 in Shelby steel tubing of 1¼" ID and initiated by pressed tetryl pellets. His data (Ref 70) indicated, by extrapolation, that the rate of deton of AN, under the experimental conditions given, should be 3700 m/sec. The amatols uniformly detond under the conditions mentioned, but the 50/50 amatol consistently failed to deton when extruded,lead tubing was substituted for steel tubing

"Limiting charge diameters" for AN under varying degrees of confinement were reported by Belyaev & Khariton (Ref 77) in studies summarized as follows: a) Deton of AN does not differ in principle from other explosives and is not unique b) In a series of experiments, dry AN (d 0.7-0.8) was packed in long, thin-walled glass tubes and cardboard casings of various diameters. These charges were initiated by means of a mixt of AN and 3% of TNT. This mixt, serving as a "booster", occupied the upper portion of the tube (or casing) and was set off by an electric detonator. Stable, nondamping detons of AN were observed where the charge diam ("limit charge diameter")

was greater than 80 - 100 mm. The smaller the diameter of the charge, the more abrupt was the damping c) When using heavy casings, such as concrete, or when exploding under water, the "limit charge diameter" decreased to 30 - 40 mm d) Explosives having lower heats of explosion than AN, such as mixts of AN with inert substances, have greater "limit charge diameter" values

Cook et al (Ref 119) measured the deton rates of pure AN (as well as of its mixts with TNT and with Comp B) as a function of charge diam. A hand-tamped charge of AN in paper tubing diam 12.72 cm, failed to deton. Average rates in larger diam paper tubes were: 1300 m/sec in 16.0 cm diam tube, 1500 in 19.99 cm, 1750 in 25.4 cm, 2150 in 35.7 cm, 2360 in 40.4 cm and 2760 m/sec in 46.0 cm diam tube

According to Rinkenbach (Ref 130), rates of deton of pure AN packed in steel tubes at approx d 0.96 g/cc were as follows: 2570 m/sec in 5" ID tube, 1681 in 4" and failure in 3" ID tube

Sakurai (Ref 120) determined the "*shock wave velocity*" of AN at an apparent d of 0.91 g/cc to be 3200 m/sec when measured in air and initiated by a TNT booster

Dissociation Pressures, as given in Ref 112a, are as follows:

Temp, °C	188.2	205.1	215.9
mm Hg	3.25	7.45	11.55
Temp, °C	223.1	236.7	249.1
mg Hg	15.8	27.0	41.0

Entropy (Absolute), as given in Ref 122A is 36.0 cal/mol/°C at 25°

Explosion by Heat. See Sensitivity to Initiation by Heat

Explosion by Initiation. See Sensitivity to Detonation by Detonators and Boosters

Explosion Temperature – cannot be determined (Ref 122a, p 121)

Fire and Explosion Hazard. According to Ref 122a, p 123, AN is a fire hazard, since it is a powerful oxidizing agent and will increase the intensity of combustion of any flammable material mixed with or adjacent to it. More information on the subject as well as on the handling of AN in storage and shipping may be found in Ref 127a and Ref 86a

Flash Point or Ignition Temperature may be detd by various methods such as those outlined in Refs 89 and 101. According to Ref 89, pure AN can be decompd by a flame at a temp of 395–6°, while a petrolated AN decomp at ca 380°. According to US Dept Agric Circ No 719, the fl p of AN is ca 500°

Formation, Heat of. See Heat of Formation

Fragmentation Test. See under Brisance

Fusion Heat. See Heat of Fusion

Friction Sensitivity. As shown by the US Bur Mines Pendulum Friction Test, neither pure AN or FGAN can be detonated when subjected to the test with a steel shoe

Gas Developed on Decomposition and Detonation. According to Kast (Ref 31) the total volume of gas produced by AN when complete deton takes place is 980/1 kg, while incomplete deton yields 945 l/kg. Macy (Ref 88) gives for incomplete deton 937 l/kg as an average derived from the values given by Brunswig (Ref 9), Berthelot (Ref 1), Colver (Ref 11) and Marshall (Ref 10). Ref 122a, p 120, gives 980 l/kg or 78.44 l/mol for complete deton. This is a calcd value for gas volume at STP

Heat of Combustion. According to Médard and Thomas (Ref 110) the values Q_c^v are 627.8 cal/g and 50.3 kcal/mol and Q_c^p are 616.9 cal/g and 49.4 kcal/mol

Heat of Decomposition. Kast (Ref 31) gives 207 cal/g for AN at d of 1.0 g/cc, as derived from the reaction: $8\ NH_4NO_3 \rightarrow 16\ H_2O + 2\ NO_2 + 4\ NO + 5\ N_2$. Dunkle of PicArsn, NJ, calcd the heat of decompn as 414 cal/g or 33.14 kcal/mol from the following Berthelots' reaction: $NH_4NO_3 \rightarrow 2\ H_2O$ (liq) + N_2O. One of the older literature values for the heat of decompn was 347 cal/g. The average of these three values is 323 cal/g or 25.86 kcal/mol

Heat of Detonation. See Heat of Explosion

Heat of Dissociation at Various Temperatures. Ref 112a gives the following values:

Heat of Dissociation (ΔH)

Temp, °C	169.6	200	250
ΔH, kcal/mol	39.33	38.92	38.25
Temp, °C	300	350	
ΔH, kcal/mol	37.65	37.12	

Heat of Explosion or Detonation. As calcd from constants given by Brunswig, Berthelot, Colver and Marshall, an ave value for Q_e^v is 630 cal/g (H_2O liq). According to Ref 122a, p 121, the detonation reaction is: $NH_4NO_3 \rightarrow N_2 + 2\ H_2O$ (gas) + $0.5\ O_2$ + 27.72 kcal. This is equivalent to 346.3 cal/g. One of the older literature values for Q_e^v was 375 cal/g with H_2O liq. Kast gave a value of 347 cal/g at a d of 1.0 g/cc (Ref 31)

Heat of Formation. Mellor gives for Q_f^p 88.1 kcal/mol or 1101 cal/g (Ref 16). As calcd from NDRC data (Rpt A–116), the value for Q_f^v is 84.5 kcal/mol or 1056 cal/g , which corresponds to 87.2 kcal/mol or 1090 cal/g for Q_f^p . Ref 122a gives 87.93 kcal/mol and 1098.46 cal/g respectively. Médard and Thomas (Ref 110) report a value of 1059 cal/g or 84.8 kcal/mol for Q_f^v and 1091 cal/g or 87.4 kcal/mol for Q_f^p. (See also values reported by Tavernier, Ref 123)

Heat of Fusion. 18.23 cal/g (Ref 122a, p 120)

Heat of Sublimation. See Latent Heat of Sublimation

Heat, Specific. See Specific Heat

Heat Tests. See under Thermal Stability

Humidity and Hygroscopicity. Pure AN is not deliquescent at RT and at RH up to about 75°, but at higher RH the salt starts to deliquesce. Moisture uptake of dried cryst AN exposed in a 5–7 mm layer for 7 days at 20.1° was as follows: 17% at 78% RH, 27.5% at 87.75% RH and 36.5% at 97.5% RH (Ref – Pic Arsn data)

The following values were taken from a curve in Ref 82 for the RH of air at various temps when in equilibrium with a satd soln of AN:

°C	5	10	15	20	25	30	35	40	45	50
% RH	82	75	70	67	63	59	56	53	51	48

The following values, detnd at Pic Arsn, give % gain in wt of AN stored in thin layers at 22.2° (72°F) at different RH's:

Hygroscopicity of AN

Exposure, days	1	2	3
% Gain at 52% RH	0.2	0.2	0.2
at 76% RH	14.1	26.1	29.1
at 90% RH	32	62	84
Exposure, days	6	7	8
% Gain at 52% RH	0.2	0.2	0.2
at 76% RH	69.1	74.1	78.4
at 90% RH	133	145	156

Hygroscopicity. See Humidity and Hygroscopicity

Ignition Temperature. See Flash Point

Impact Sensitivities at Room Temperature. PicArsn App, 2 kg wt: 31" for cp AN and 29–32" for AN coated with about 1% wax; BurMines App, 2 kg wt: no action for cp or wax-coated AN. Tests conducted at the same time with PicArsn App for standard explosives gave: 17" for Explosive D (Amm picrate), 12–15" for TNT, 8" for tetryl, 4" for LA and 2" for MF (See also Ref 122a)

Impact Sensitivity at Various Temperatures, PicArsn App, 2 kg wt (Ref 88):

Temp, °C	25	100	150	175
Impact Test, inches				
AN, cp	31	27	27	12
AN, wax-coated	30	22	23	12–13

The test at 175° shows that AN in molten condition is much more sensitive than the solid material. This had been previously shown by Kast (Ref 31) who gave 16–20 cm impact sensitivity with 10 kg wt and 12 cm with 20 kg wt. Ref 122a gives for molten AN 12" with 2 kg wt, PicArsn App

Initiation Sensitivity. See Sensitivity to Initiation

Latent Heat of Sublimation = 41.8 kcal/mol at 25° (Addnl Ref H) (See p A340)

Lead Block Test Value. See Trauzl Block Test value, under Power

Lead Cylinder Compression Test. See under Brisance and a Note under Power

Maximum Potential Work. See under Brisance

Oxidizing Properties of AN. Due to the fact that AN contains ca 20% of available oxygen, it serves as a powerful oxidizing agent. Various metals (especially Zn) react with AN even at RT, but the reaction is more vigorous when the mixture is heated. All organic compds, when heated with AN, burn with the evolution of CO_2, CO, N_2, N_2O etc (Ref 94). Hardesty and Davis (Ref 83) found that peanut-hull meal may be oxidized by AN with the formation of CO_2, N_2, N_2O, O_2 and small amounts of C_2N_2 and NO_2. Aqueous solns of AN corrode metals, such as Cu, but not as much as do NH_3 and HNO_3 (Ref 45)

Power. Values as detd by the *Trauzl Lead Block Test* (Lead Block Expansion Test) have been reported as high as 225 cc for complete detonation, as compared with 305 cc for PA and 300 cc for TNT. For incomplete detonation, a value of 165 cc has been reported (Ref,OSRD Rpt 2014). Comparing the Trauzl Block Test values of current Russian AN explosives used in open-pit mining, such as Ammonite No 2 and Dynamon K, which have test values of 280 cc and 300 cc respectively, Assonov and Rossi (Ref 68) concluded that it was cheaper to replace them with straight AN, for which they gave a test value of 225 cc. According to Ref 122a, p 123, both lead block expansion and lead cylinder compression tests gave 55% of TNT. In the standard *Ballistic Pendulum Test,* AN undergoes only partial detonation. If the test is modified so that a No 16 blasting cap is used, AN is found to be 79% as powerful as TNT (Ref 130)

Pressure Developed on Detonation. For the reaction: $NH_4NO_3 \rightarrow N_2 + 2\,H_2O + 0.5\,O_2$, the calcd pressure is 11200 kg/cm² at a temp of 1500°. Robinson (Ref 15) reported a pressure of 12.5 long tons/in² in 0.5×10^{-5} sec for straight AN and 15.2 lg t/in² for a mixt of AN with 0.5% TNT. The pressure rose steadily and rapidly with increasing amounts of TNT in mixts to a max value of 55 lg t/in² for straight TNT

Propagation of Detonation. See Ability to Propagate Detonation

Rates of Detonation. See Detonation Rates

Rifle Bullet Test. AN at a d of 1.2 g/cc was unaffected in 10 trials. The presence of small amts of wax-coating did not affect these results (Refs 122 a & 128)

Sand Test Value. See under Brisance

Self-Ignition. See Spontaneous Ignition

Sensitivity to Impact. See Impact Sensitivity

Sensitivity to Initiation by Detonators and Boosters. As a rule AN is much more difficult to detonate or explode than any of the standard explosives. Even with strong initiators, such as a No 8 cap, the process of explosn (or deton) does not always go to completion with the formation of N_2, O_2 and H_2O, but various quantities of nitrogen oxides are also formed. This is especially pronounced when confinement is not complete (Ref 26). If AN is unconfined, it cannot be initiated to complete deton by a No 8 cap (Ref 60). If AN is partly or completely confined, a No 8 cap may be considered in some cases as a sufficient initiator, but more complete deton is obtained either by combining a No 8 cap (or a weaker cap) with a booster (Ref 28), or by using a stronger cap, such as No 16, alone (Ref 130). It is much easier to deton the molten salt than the solid material (Refs 28 & 29). The pressed cryst material is much easier to deton than the cast material, and the sensitivity to deton of the cryst material increases with packing d. The dry salt is easier to deton than the moist. In finely divided, low-density form, pure AN with an apparent d of 0.75 deton more completely (and gives higher Trauzl block value), than does technical grade AN

The relative insensitivity to deton of pure AN was demonstrated at the Bur of Mines by Scott and Grant (Ref 90) by firing 5 g charges at d 0.84 with caps contg various amts of 80/20-MF/$KClO_3$ mixture. These tests were made in the sand test bomb with small paper cartridges in a manner similar to the miniature cartridge test described in Ref 78. The results of the tests were as follows:

Cap No	Wt, g, of MF–$KClO_3$	Ordinary Type Grams of Sand Crushed by 5 g of AN*	TNT*
6	1.0	21	253
8	2.0	43	254
10	3.0	58	261

*After correction for sand crushed by initiator

The above results show that only part of the 5 g sample of AN detonated. On the other hand, some tests conducted at the Bur of Mines with the ballistic pendulum, showed that, when properly confined, AN can be detond completely even with a No 6 cap (Ref 90)

Munroe (Ref 25) noted that an increase in temp results in increase in the sensitivity to initiation as well as the Trauzl lead block value of AN

Belyaev and Khariton (Ref 77) stated that sensitiveness to initiation is increased by increase in confinement and also by diam of the AN charge. For instance, charges at d 0.7–0.8 g/cc, under strong confinement such as steel tubing, could be initiated completely in smaller diameter containers than when the charges were relatively unconfined in glass or cardboard tubes

Sherrick reported (Ref 27) the following observations in studying the sensitivity of AN to initiation: a) Attempts to ignite 3 g of AN in a test tube by means of a black powder fuse failed b) Attempts to detonate AN loaded at d 0.7 in 1.5" id Shelby steel tubing having wall thickness of ¼" did not result in

more than feeble, partial deton when 100 g boosters of TNT, tetryl, PA or TNA were used c) At a red heat, 5 g of AN merely decompd without flame, leaving no residue d) The explosibility of AN decreased with increasing d and decreasing confinement

Sensitivity to initiation of AN is also increased when impurities, especially organic materials, are present (Refs 20, 21, 73 and 89)

According to Ref 122a, P 122, it has been practicable to deton large charges of properly confined AN by means of a booster charge of tetryl, but not by means of a LA or MF blasting cap. Sensitivity to initiation decreases with increase in loading d, and if this exceeds 0.9 charges of 1 to 3 lbs cannot be detond completely even by large booster charges. Charges larger than 3 lbs can be detond completely at d's not greater than 1.1. The admixture of up to 8% of non-explosive carbonaceous material somewhat sensitizes AN to initiation. Molten AN is much more sensitive than the solid material and can be detonated with practically no confinement

Sensitivity to Initiation by Heat. There is no agreement among various investigators as to what is the minimum temp at which AN under confinement explodes or detonates. It is safe to assume that this is above 260° and more probably nearer 300°. However, some investigators reported that pure AN can be initiated if heated above its mp (169.9°), if confined and under pressure of 2500 psi or more (Refs 21,106 & 122a). It is very probable that explosion starts, not inside the molten mass itself, but because the gases of decompn form an expl mixture which explodes (or deton) first and initiates the deton of the undecd, molten AN

It should be noted that heating pure unconfined AN never produces an explosion or deton, but only a more or less rapid decompn accompanied by a flash and a hissing sound (Refs 17, 75 & 86). For instance, if a cryst or a piece of cast AN is thrown upon a hot plate at a temp of about 500°, the material immediately catches fire and burns rapidly with a yellowish flame and a crackling or hissing sound, but leaves no residue. If a large piece of cast AN is thrown upon a red-hot plate, the decompn proceeds so rapidly and with so much noise that it resembles an explosion. This phenomenon apparently explains why some earlier investigators believed that AN could be exploded by heating, even if unconfined (Ref 18)

In experimental work conducted at the Bur of Mines after the Texas City disaster, it was shown that pure AN can be initiated when heated between 277 and 334°, FGAN at 114 to 350° and a mixture of FGAN and bag paper at 134 to 153°, when under confinement

The sensitivity of AN to initiation by heat may be increased or decreased by the presence of certain inorganic impurities. For instance, small amts of Cu increase sensitivity to heat because of the formation of a small amt of copper nitrite, which causes instability (Ref 90). The formation of Cu nitrite also was reported in Mellor, v 7 (Ref 16). Mellor also reports that the presence of Fe, Al or especially Zn in powdered form lowers the temperature required for the decompn of AN. Kast (Ref 31) reported that the presence of $KMnO_4$ in powdered form may cause the spontaneous heating of AN. Investigations conducted at Pic Arsn showed that different Cr compds such as the oxide and nitrate catalyze the decompn of AN, and in some cases explosions occurred at temps as low as 200°. Among the inorganic substances which lessen the sensitivity of AN to heat are clay, kieselguhr, powdered limestone etc. The same effect was expected of Amm sulfate until the disaster at Oppau in 1921 (Refs 15a & 15b) rendered the safety of such a mixt uncertain. Amm sulfate had been used for many years as a desensitizer for AN in fertilizers and it was considered that such mixts could not be

exploded or detond. This was disproved according to Scott and Grant (Ref 90) by Kast, who claimed that under certain conditions mixtures containing less than 40% of Amm sulfate may react exothermically according to the equation: $4\ NH_4NO_3 + 2(NH_4)_2SO_4 \rightarrow 16\ H_2O$ (gas) + $2\ SO_2$ (gas) + $6\ N_2$ (gas) + 145.2 kcal

The sensitivity of AN to heat is always increased by the presence of organic compounds, provided that the proportions of these do not exceed certain limits. Among the substances investigated are cellulose, paper, pulverized carbon, soot, sawdust, waxes, paraffin, TNT, NS, DNT and drip oil. For instance, the presence of 5% NS in AN increased the sensitivity to such an extent that the mixture detond when heated to about 150°. The presence of paper bags such as the containers for FGAN was a contributing factor to the Texas City disaster in 1947. In connection with this disaster, tests conducted at Pic Arsn and Aberdeen PG showed that transformation of the combustion of a mixture of FGAN and bagging paper into detonation required the building up of a gas pressure greater than a certain critical value. This was calcd to be about 100 psi (abs) or perhaps less

Sensitivity and Stability of Molten AN. (See also Decomposition Reactions).Klevke reported practically no decompn of AN during evapn of its solns at atmospheric or reduced pressures (Ref 53). Klevke also claimed to obtain the undecomposed AN in the gas phase (Ref 59). Tram and Velde (Ref 49) found that when AN (either neutral, slightly acidic or alkaline) was left standing in a molten condition at 175°, it underwent only slow decompn provided that no chlorine was present as an impurity. Samples of AN containing nitric acid and a small amount of chlorine decomposed spontaneously at temps as low as 140°

Shah and Oza (Ref 42) studied the decompn of AN when heated and reported that dissocn began before decompn started (180°) and NH_3 was evolved while HNO_3 accumulated in the residue. On heating to higher temps, other products of decompn, such as N_2, N_2O and H_2O were also formed. The decompn of AN was slow at 240°, but became rapid at 290°. Between these temps, N_2O and H_2O were the main decompn products and the amt of N_2O formed was directly proportional to the pressure. Gas phase explosions occurred at some point near 300°

Kretzschmar (Ref 48) also studied the thermal decompn of AN and reported that decompn began at 170° and became quite appreciable at 220°. During this time N_2O, H_2O and N_2 were evolved. At a higher temp (250°), a little oxygen was also evolved. In no case were other nitrogen oxides formed

The explosiveness of molten AN was studied also by Kaiser (Ref 52) who reported the following:

A small sample of AN in an evacuated tube was heated gradually to desired temps, and samples of the gas produced by decompn were pumped out, measured and tested. Decompn proceeded very quietly at temps below 200°, and only a small amt of gas was formed even on heating for several hours. The reaction proceeded more vigorously at higher temps and became rather violent at ca 260°. Between 260 and 269° a gray smoke was produced and, after a time, an explosion took place. This also occurred after heating above 250° for 10 hours, at which time more than 93.4% of the nitrate had decomposed. The main reaction of decompn of AN heated in an evacuated tube was: $4\ NH_4NO_3 \rightarrow 2\ NH_3 + 3\ NO_2 + NO + N_2 + 5\ H_2O$, but there were also found present small amts of N_2O and HNO_3 in the gaseous products of decompn. Some of the above products of decompn (ammonia and nitrogen oxides) interacted with the evoln of heat. This heat might raise the temp of the gases above the molten AN to such an extent that they could explode and cause the explosn of the molten AN in the tube. In order to avoid the danger of expln

it is necessary to remove the gases produced by decompn as rapidly as they are formed. There is a possibility that some substances present in impure AN act as catalysts and accelerate the reactions of decompn

Sensitivity to Initiation by Influence (Sensitivity to Sympathetic Detonation). According to Abinder and some other Russian investigators (Ref 60), cartridges or shells loaded with AN or explosives contg large amts of AN transmit deton by influence to other cartridges or shells provided they are laid side-by-side, even with a small air space between them; but detonation usually is not transmitted if the cartridges or shells are laid end-to-end. The presence of 1% or less or moisture does not affect the sensitivity of AN to initiation by influence, but the presence of more than 2.5% moisture renders AN insensitive to initiation. The presence of small amounts of organic substances or of inorganic reducing materials such as Fe increases the sensitivity of AN. Less than 0.1% of paraffin has practically no effect on the properties of AN

Shock Wave Velocity. See under Rate of Detonation (last paragraph)

Solubility of AN in Various Solvents
[Taken from Land-Börnst (Ref 22); ICT (Ref 35); Seidell (Ref 69a); Kirk & Othmer (Ref 84); PATR]

Solubility in Water

Temp, °C	g/100 g soln	g/100 g water	Temp, °C	g/100 g soln	g/100 g water	Temp, °C	g/100 g soln	g/100 g water
−20	40.0	66.7	30	69.9	232	80	85.2	576
−10	48.4	93.8	40	74.8	297	90	88.1	740
0	54.2	118	50	77.6	346	100	89.4	843
10	60.0	150	60	80.4	410	140	97.4	–
20	65.2	187	70	83.3	499	160	99.2	–

More data are given in Ref 69a, v 1, pp 1105–6 and Suppl, pp 385–7

Solubility in Aqueous Nitric Acid

See Ref 69a, vol 1, p 1107
A – Grams of HNO_3 per 100 g of soln saturated with AN
B – Grams of AN per 100 g of satd soln

At 0°

A	0.0	9.2	21.0	27.0	33.2	39.1	41.4	45.8	47.4
B	54.3	43.5	34.5	31.9	31.0	33.2	34.0	39.5	48.2

At 15°

A	0.0	9.0	21.7	27.1	36.7	39.0	44.0	45.0
B	62.4	52.0	42.2	40.9	39.9	40.8	46.8	52.5

Solubility in Aqueous Nitric Acid (continued)

At 30°

A	0.0	8.6	20.8	26.4	37.5	40.1	42.2
B	70.2	60.9	51.5	48.5	48.6	51.0	55.6

At 75°

A	0.0	12.8	16.0	26.3	31.6
B	84.1	73.7	71.8	67.5	66.8

Solubility in Pyridine (Ref 128)

20 to 25 g in 100 g of satd soln at 25°

Solubility in Acetone

Slightly soluble (Ref 84, v 1, p 818)

Solubility in Aqueous Solutions of Ammonium Sulfate, Ammonium Acid Sulfate, Sodium Chloride, Sodium Nitrate, Lead Nitrate, Uranyl Nitrate and Ammonium Thiocyanate

Ref 61a, Seidell, v 1, pp 1108–1112 (1940)

Solubility in Acetic Acid *

Temp, °C	Solubility	Temp, °C	Solubility	Temp, °C	Solubility
6	0.16	63.5	1.89	110.6	31.25
17.7	0.28	72.8	3.45	120.0	55.5
21.4	0.33	80.9	5.51	131.4	75.0
27.0	0.39	85.7	7.26	149.7	86.3
33.6	0.51	97.1	13.68	167.5	100.0
45.8	0.88	101.0	17.15		

*Gram moles of AN per 100 g mols of satd soln (Ref 46)

Note: According to Eichelberger (JACS 56, 801 (1934), one liter of a satd soln of AN in pure acetic acid contains 3.5 g of AN at 16.46°

Solubility in Ammonia (Ref 61a, v1, p 1108)

Temp, °C	−60	−30	−10.5	0	33.3	35.9	68.8	94.0	190.8
g AN	1.39	0.83	0.97	0.76	0.94	0.77	4.26	0.64	0.76
g NH_3	4.43	0.37	0.35	0.26	0.24	0.19	0.77	0.07	0.06

Solubility in Aqueous Ammonia at 25° (Ref 69a, v 1, p 1108)

g H_2O per 100 g H_2O + NH_3	g AN per 100 g H_2O + NH_3	g H_2O per 100 g H_2O + NH_3	g AN per 100 g H_2O + NH_3
0	390	21.8	316.8
2.80	381	47.5	247.0
4.82	372.8	68.0	220.0
10.1	354.7	100	214.0
15.9	337.4		

Solubility in Liquid Ammonia
(Ref 69a, v 1, p 1115)

Temp, °C	g AN per 100 cc NH_3	Temp, °C	g AN per 100 cc NH_3
−50.6	70.1	−40.8	75.1
−46.5	72.6	−36.6	77.0
−45.0	73.4	−34.0	77.9
−44.0	73.5	25	235.56

Note: According to Ref 84, v 1, p 818, the solns of AN in liquid NH_3 are called "Diver's Liquids". The amount of NH_3 taken up by 100 g AN is 42.5 g at −10° and 31.5 g at 18°, forming liquids containing 30% and 24% respectively of NH_3

Solubility in Absolute and Aqueous Ethanol
(Ref 69a, v 1, p 1112)

% Ethanol by Weight	°C	g AN per 100 g solvent					
		100	86.77	76.12	51.65	25.81	0
	20	2.5	11	23	70	140	195
	30	4	14	32	90	165	230
	40	5	18	43	115	196	277
	50	6	24	55	144	244	365
	60	7.5	30	70	183	320	–
	70	9	41	93	230	–	–
	80	10.5	56	–	–	–	–

Note: Kirk and Othmer (Ref 84, p 818) give the amt of AN dissolved in 100 g of abs ethanol as 3.8 g at 30° and 10.1 g at 80°. Additional data are given in Ref 69a, Suppl, p 390

Solubility in Absolute and Aqueous Methanol
(Ref 69a, p 1112)

	Grams per 100 g of satd soln						
H_2O	0	5	10	15	20	25	29.9
CH_3OH	83.3	74.8	63.8	50.7	35.2	19.8	0
AN	16.7	21.3	27.1	35	46.3	59	70.1

Note 1: Kirk & Othmer (Ref 84, p 818) give the amnt of AN dissolved in 100 g of abs methanol as 20 g at 30° and 39.6 at 60°; Note 2: Schiff & Monsacchi, ZPhysChem **21**, 277 (1896), give the soly of AN in 100 g of abs methanol as 14.6 g at 14°, 16.3 g at 18.5° and 17.1 g at 20.5°

Solubility in 95% Isopropanol
(Ref 69a, Suppl, pp 391–2)

Temp, °C	30	40	50	60	70	75
g/100 g soln	3.23	3.81	4.50	5.45	6.37	6.89

Solubility in Ethanol-Methanol-Water Mixtures at 30° (Ref 69a, v 1, p 1112)
(Various proportions of water were added to a mixt of 51.7% methanol and 48.3% ethanol by wt)

	Grams per 100 g of satd soln						
Water	3.4	5	10	15	20	25	29.9
Alcohol (mixt)	84.9	82.9	74.6	63.5	48.2	22.4	0
AN	11.7	12.3	16.4	24	35.1	54	70.1

Solubility in Ethyl Acetate at 19° – 0.050 g per 100 g of solvent (Ref 22)

Solubility in Furfural at 25° – 0.4 g per 100 g of soln (Ref 69a, Suppl, p 392)

Solubility in Carbon Tetrachloride and in Chloroform at 20° – Insoluble (Ref 22)

Solubility in Various Organic Compounds Determined in Russia. Khaishbashev et al (Ref 79) investigated about 200 organic compds and came to the following conclusions: a) AN is miscible in all proportions with compds contg OH or NH groups provided that the mol wt is not too high; b) Total or partial insolubility is observed with hydrocarbons and with the compds contg NO_2 or halogen groups. For instance, AN is miscible in all proportions with mannitol, resorcinol, urea and acetamide, while with p-phenylenediamine it forms a mol compd in the ratio of 3 parts AN to 1 p of the diamine. In the liquid state AN is partially miscible with m-phenylenediamine, PA, TNT and cholesterol. Eutectic mixts are formed with many of the organic compds investigated by K

Specific Gas Energy. Gmelins Handbuch (Ref 56) gives 5575 kg/l for complete deton and 3840 kg/l for partial deton

Specific Gas Volume. See Gas Volume Developed on Decomposition or Detonation

Specific Heat. Bellati in 1886 (Ref 3), Behn in 1908 (Ref 7) and Crenshaw and Ditter in 1932 (Ref 43) detnd sp heat values. The following values, in calories per gram per °C, represent average rounded figures taken from all three sources:

Specific Heats

Temp, °C	-200	-150	-100
Sp ht	0.07	0.19	0.30
Temp, °C	-80	-50	0
Sp ht	0.35	0.37	0.40
Temp, °C	50	100	
Sp ht	0.414	0.428	

Kirk and Othmer (Ref 84, 1, p 819) give the sp ht for temps from 0° to 31° as 0.407. Mellor (Ref 16) gives the following values for sp ht at atm pressures: Form II 0.426, Form III 0.355 and Form IV 0.407. The value for Form V at temps –190 to 20° is given as 0.423 ± 0.00143 cal/g

Ref 122a, p 120 gives the sp heat of AN as 0.397 cal/g at 0° and 0.428 cal/g at 100° (For additional information on sp ht values at low temps, see Refs 22 and 35)

Landolt & Börnstein, 3 Erg, 3 Teil, p 2276 gives sp heat 0.508 at 0°

Specific Volume. See Gas Developed on Decomposition and Detonation

Spontaneous Ignition, Self-Ignition, Heat of Spontaneous Combustion. As far as is known, there is no recorded instance of the spontaneous heating of pure AN, but there have been

several fires in which it was reported that impure AN underwent self-ignition. Laboratory tests have shown that self-ignition may take place when organic substances or easily oxidizable metals (such as Zn), plus some moisture, are present. The chance of self-ignition is greater if some free nitric acid is present or if the material is stored or transported at high temps. On the other hand, the presence of small quantities (0.5–1.0%) of organic materials, such as paraffin, wax or petrolatum (generally used for coating purposes) did not cause any spontaneous combustion at temps as high as 60°. However, if there is a fire somewhere in the neighborhood of AN or FGAN, which is in contact with some org material (such as sawdust), self-ignition may take place if the temp of material becomes sufficiently high (say 150°). Under these circumstances FGAN requires about 50 minutes for self-ignition

Stability. See Thermal Stability

Temperature Developed on Decomposition. Temps ranging from 800 to 1230° are reported, depending on how the reaction proceeds (Ref 56).

Temperature Developed on Detonation. For the reaction of complete deton to yield nitrogen, water and oxygen, the temp developed has been estimd to be 1500°. Macy et al (Ref 88) gave 2120° as the temp calcd from the data of Brunswig, Berthelot, Colver and Marshall

Thermal Conductivity. Golubev and Lavrent'eva (Ref 65) gave a value of 0.205 kcal/meter/hr/°C, which is equivalent to 0.1375 Btu/ft/hr/°F

Thermal Expansion. Cubical coefficient of expansion (γ) (Ref 56):

°C	−60	−20	0	18
$\gamma \times 10^6$	677	852	920	978
°C	20	60	100	
$\gamma \times 10^6$	982	1069	1113	

Thermal Stability. In general, pure AN may be considered stable up to its mp (169.9°). Reports of early investigators, such as Berthelot (Refs 1 & 8), that the salt begins to decompose at temps as low as 100°, were apparently correct because the substance was not pure. In the days when nitric acid (used for the manuf of AN) was prepd from Chile saltpeter, some nitrites and chlorides, as well as other impurities, remained in the AN, and it was apparently due to them that the stability of the product was not satisfactory. Tram & Velde (Ref 49) found that as little as 0.1% of Amm or Na chloride increases greatly the probability of decompn of AN, while 1–2% of such impurities are definitely the cause of increase of decompn of molten AN

In summarizing the present stability data on AN, prepd from synthetic ammonia and ammonia oxidation nitric acid, it may be said that up to temp of its mp, AN is fairly stable. At slightly above its mp (say ca 170°), slow decompn begins, but this is hardly perceptible until temps 200–210° are reached. From this point on the decompn, accompanied by evoln of gas, is fairly rapid and, if the substance is confined, explosion may take place above 260° (See Refs 91, 92 and 124 for more information on the thermal decompn of AN)

Following are the results of some tests conducted for the US Govt (after Texas City disaster) in order to determine the safety factors of AN: a) 100° Heat Test 0.74% loss in wt in the first 48 hrs, 0.13% loss in the 2nd 48 hrs and no explosion in 100 hrs b) 120° Vacuum Stability Test: 0.1–1.0 cc gas evolved from 5 g of pure AN in 40 hrs and 1.2 cc gas from AN coated with 1% wax c) 150° Vacuum Stability Test: 0.3 cc gas from 5 g pure AN in 40 hrs and 3.2 cc gas from FGAN d) Storage at 80° N content of pure AN decreased in 2 weeks from 33.3%

to 32.9%, but that of AN coated with 1% wax decreased in N content only from 32.7 to 32.6%
Note: According to Ref 122a, p 123, AN is a very stable material even at 150° as indicated by the Vacuum Stability Test at that temp. It can be heated at 100° for 100 days without appreciable decompn. This does not appear to begin until the compd melts. At 220° there are formed nitrous oxide, water and nitrogen, and this reaction is used for the manuf of nitrous oxide. If an organic material such as cellulose is present, decompn of the mixt begins at 100° and is pronounced at 120°. Admixture with TNT has little, if any, effect on the stability of AN at temps lower than 120°

Toxicity. Not toxic (Ref 122a, p 123)

Trauzl Block Test Value. See under Power

Uses. AN is one of the most widely used components of explosives. The most important explosives contg high percentages of AN include amatols, ammonals, ammonites, dynamites, dynammons, Favier type explosives, grisounites, grisoutines, schneiderites etc. In addition, AN and mixts containing it have been used extensively as fertilizers

For additional information on the uses of AN, see "Ammonium Nitrate Explosives and Propellants", and also "Ammonium Nitrate Fertilizer"
Note: According to Ref 128, AN has been used as an ingredient of mixtures for large bombs

Vacuum Stability Test Values. See under Thermal Stability

Vapor Pressure. See Dissociation Pressure

Velocity of Detonation. See Detonation Rate

Volume of Gases of Explosion. See Gas Volume Developed on Decomposition or Detonation

Water Resistance and Prevention of Caking. AN and its expl mixts are hygroscopic and absorb moisture unless this tendency is prevented by coating the AN particles with a water-repellent agent. Gorshtein (Ref 47) studied the hygroscopicity and caking tendency of AN and its admixt with Amm sulfate. He reported that a moisture content in AN varying between 0.5 and 30–40% showed no difference in the absorption of water. Thick layers of nitrate absorbed but little moisture and if the nitrate was covered with a thin layer of sand, the absorption of moisture was reduced still further. The size of the lumps of nitrate was almost without effect. At 32° AN changes from one modification to another. Dry AN does not cake at this point, but if more than 0.1% of moisture is present, severe caking accompanies the transition. Drying the moist salt results in caking. Although methods of mixing and small variations in the relative amts of AN and Amm sulfate in a mixt of these compds did not affect their absorption of moisture, increase in the relative amt of Amm sulfate decreased the caking tendency. If a double salt was formed, the mixture caked about as much as AN alone, but when these were mechanical mixtures, the caking was less

Snelling (Ref 14) suggested that the waterproofing of AN may be accomplished by allowing the crystals to fall through an atmosphere of nitronaphthalene vapor in such a manner that a water resistant coating is deposited. More recently Davidson and Rigby (Ref 97) proposed coating AN grains with a small amount of methylcellulose. Other coating materials such as clay, chalk, silica, alumina etc were used by Whetstone (Ref 103), but the best results were obtained by spraying AN crystals with "acid magenta". Goodale (Ref 107) found that coating of AN with 0.1–5.0% of 2,2-dinitropropane combined with about 5–30% of a nonvolatile liquid rendered the AN less hygroscopic and less water-soluble. Le Roux (Ref 108) noted improvement in resistance to water of explosives containing AN coated with transformer oil, paraffin, naphthalene acids, Al or Ca naphthenate, rosin, natural resins, Ca resinate, abietic acid,

beeswax, polyvinyl acetate dissolved in liq DNT, silicone grease dissolved in trichloroethylene, MNT, tetrachloronaphthalene, starches, Na or Al alginate, kaolin or Al stearate (See also Explosifs N)

According to Lytle (Ref 111), the water resistance of AN used in explosive compositions may be improved by the addition of a solid hydrophilic urea-HCHO-type reaction product such as Uformite, Cascamite, Melmac and other resinous products such as glues and adhesives. Taylor (Ref 112) obtained water-resisting explosives containing AN coated with 0.4–15% of a gel-producing mannogalactan such as guar flour, locust-bean gum or carob-bean gum. Improved resistance to water damage of coal-blasting explosives was achieved by Davidson and Rigby (Ref 116), who incorporated at the time of manuf a small amt of a water-swellable and water-soluble ether of a polysaccharide such as methylcellulose. Nylander (Ref 121), in order to prevent formation of hard lumps on storage, treated AN with 0.01–5% of Na polymetaphosphate. For example, an aqueous soln satd at 160° with AN was granulated at 70° until the water content was reduced to 0.1% and during the final stages of the drying there was added 0.25% of Na hexametaphosphate

Shneerson et al (Ref 126) recently determined the effects of cooling, moisture content, K nitrate content and compression up to 0.2 kg/cm² on the caking characteristics of AN. He concluded that caking occurred at the transition of forms III and IV into each other at 32.27°. Based on thermographic investigations of granulated AN, Alekseenko and Boldyrev (Ref 125) suggested rapid cooling and low moisture content as effective means for decreasing caking. Enoksson & Enoksson (Ref 122) decreased the tendency to lump formation in AN in incorporating 0.005 to 5% of an alkyl sulfate, alkyl sulfonate, alkylarylsulfonate of alkyl phosphate. In the examples given, the following compounds were added either before or after crystallization of AN: $(C_8H_{17})_2NaPO_4$, isopropylamine dodecylsulfate, $RCHMeOSO_3Na$ where R is an alkyl group having about 12 carbon atoms: $C_{18}H_{37}OSO_3Na$, $C_4H_9CHEtC_2H_4CH(SO_4Na)CH_2CH(CH_3)_2$ and $3,4(NaO_3S)(MeO)C_6H_3NHOCC_{17}H_{35}$

Note: The following information is included in Addnl Refs (See p340): The Hercules Powder Co patented a method of coating AN with nitrates of metals such as Cu,Pb,Zn, Cd,Ni or Fe to increase its sensitivity to detonation (Addnl Ref A). Lindsey proposed sensitizing AN by coating it with such materials as finely divided Al(Addnl Ref B). McGill examined, during WW II, the thermal stability of AN's melting betw 163 & 169° and found that at 100° and 135° the materials did not become acid and did not expl in 300 mins (Addnl Ref C). The NatlBurStds detd some thermodynamic props of AN(Addnl Ref D). Societe Technique de Recherches et d'Exploitation (Addnl Ref E) proposed to treat AN for storage by coating its crysts with diatomaceous earth and to incorporate some urea. It has been claimed that some org impurities in present commercial AN might form in storage formaldehyde, which would react with inorganic impurities such as NH_2OH and HNO_2. Urea is added to destroy formaldehyde. The NatlResCouncil gives compendium on the hazards of transportation, manuf etc (Addnl Ref F). Nitroglycerin AB (Sweden) proposed to crystallize AN from a soln contg a micelle-forming substance, such as alkylsulfonic acid. This is to prevent or reduce the tendency of AN to cake (Addl Ref G). Luft detd the latent heat of sublimation of AN as 41.8 kcal/mol (Addnl Ref H)

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Note: Comments on Plant Processes for manuf of AN (private communication, March 1960) from Dr Ralph D. Miller, Tech Director Agricultural Chem Div, Spencer Chemical Co, Kansas City, Missouri:

Under Continuous Process of Graining. There is no continuous graining carried on at this time and certainly none in the United States for the preparation of fertilizer materials. There may be some used for the preparation of dynamite grade AN where they can prepare exceedingly fine particle size material. Actually, all of the large producers and all of the materials made for fertilizer are made either by the Stengel process or by prilling through towers

For the prilling operation "high pans" have been completely eliminated and the AN solutions are evaporated to approximately 95% in vacuum evaporators. Exit the vacuum evaporators, the material is pumped to the top of the prilling tower dependent upon the size and design. The height may vary from 70 feet to 200 feet. The material is sprayed through nozzles or spray headers, and as it falls, some more of the water is evaporated. This leaves spherical particles called prills. These materials are dried further through various types of drying equipment, either counter or co-current flow with hot air and are then parted with diatomaceous earth or kaolin clay to prevent caking

There is no wax or paraffin used today in modern FGAN nor has there been since 1948

Under Continuous Vacuum Crystallization. This process is not used today, except for a small quantity which is manufactured by TVA. Here, the liquor is concentrated by vacuum crystallizers, and no "high pan" is used to concentrate the liquor prior to entering the crystallizer. All AN produced today in the neutralizers reaches about 80% to 85% concentration. Therefore, "high pans" are not necessary

Under Preparation of Fertilizer Grade Ammonium Nitrate. This process has not been used for making ammonium nitrate fertilizer since 1948, as mentioned above

Under Behavior Towards Metals and Other Substances. AN very definitely attacks shellac, baked oils and natural rubber compounds if applied as a coating. The only materials that we know of which can be used as coatings for AN and AN solutions are certain polyvinyl chloride coatings and a number of epoxy resins. The Ordnance Department used acid-proof black paint, shellac, baked oil and rubber paints. All were quite unsuccessful over long periods of time

AMMONIUM NITRATE BLASTING EXPLOSIVES, HIGH EXPLOSIVES AND PROPELLANTS

As was mentioned above under the heading "Ammonium Nitrate Historical", the use of AN in expls began about 1867 when Norrbin & Ohlsson patented their expl called "Ammoniakkrut" (Refs 2,3). This expl consisted of AN in mixts with combustibles such as charcoal, sawdust, naphthalene, picric acid, NG or nitrobenzene. Nobel acquired the patent and soon introduced a new series of AN expls called "extra dynamites", examples of which were 71/4/2/23–NG/collodian cotton/charcoal/AN and 25/1/12/62-NG/collodion cotton/charcoal/AN (Refs 52 & 94). AN has steadily increased in importance in the expl industry, especially in the "permissible expls" used in coal mining. One of the principle advantages of AN, besides its low cost, is its low temp of expln, which makes it suitable for use in gassy and dusty coal mines (Ref 16). The hygroscopicity of AN retarded to some extent its use in general expl mixts. During WW I AN found extensive application in military expls such as amatol, ammonal, sabulite etc. These expls are described under their individula names. Notwithstanding its hygroscopicity and tendency to pack, AN was recommended in Russia prior to WW II for use as a military expl (Ref 39)

AN Blasting Explosives allowed for use in American coal mines are called "permissibles", those in England "permitted", in France "explosifs antigrisouteux", in Belgium "explosifs S.G.P. (sécurité, grisou, poussière)", and in Germany "schlagwettersichere Sprengstoffe" (Ref 94). According to Taylor and Rinkenbach (Ref 14), one of the early "permissibles" was the duPont Monobel, consisting of 80/10/10–AN/NG/combustible and other ingredients. Early British "permitted explosives" included Ammonite (88/12–AN/DNN), Westfalite (95/5–AN/resin), Bellite (83.5/16.5 and 93.5/6.5–AN/DNB) and others. French safety expls included "grisounaphthaliteroche" (91.5/8.5–AN/DNN), "grisoudynamite" (70/29/1–AN/NG/colldion cotton) and "grisoudynamitecouche" (87.5/12/0.5–AN/NG/CC). Germany, Belgium and other countries developed many similar high-AN, non-NG safety expls (Ref 94)

Schmerber (Ref 5) examined mixts of organic ingredients with such proportions of AN as to give sufficient or excess oxygen for complete combustion. The calcd values for temp (t) developed on expln and also the specific gas pressure (f) were selected as characteristic of these mixts. It was observed that the (f) values for mixts contg excess 0 were proportional to the lead block expansion values in cc (Trauzl test). The approx calcd percentages of AN required to obtain maximum expln temps (t) of 1500° and 1900° (reqd in France for coal mining expls) are given in the table on the following page

Assonov and Rossi (Ref 40) compared the Trauzl block values of Russian expls used in open mining, such as "ammonit No 2" and "dynamon K", having Trauzl values of 280 and 300 cc respectively, and concluded that it was cheaper to replace them with straight AN, which gave a Trauzl value of 225 cc. Abinder and Andreev (Ref 41) disagreed with the conclusion of Assonov and Rossi, and reported a loss of about 35% in expl energy when AN is used without combustibles. Satisfactory agreement between calcd and exptl values for expl pressure was found for mixts of AN with TNT and with NG expls (Ref 35)

An AN expl, having no special name but which could be detonated, consisted of granulated AN (85 to 97%) coated with a small quantity of solid, nonexpl vulcanized oil (for example, the product obtained by treating cottonseed oil with sulfur chloride) and mixing with a sensitizing, detong subst such as

Organic ingredient	% AN for complete combustion	Calculated Values (t) °C	(f) kg/cm²	Approx % AN reqd to obtain t 1900°	1500°
Naphthalene	93.75	2122	8449	95.3	97.9
Carbon	93.02	2107	8179	94.7	97.3
Toluene	93.99	2100	8497	95.4	97.9
Nitronaphthalene(MNN)	90.86	2171	8553	93.0	97.0
Nitrotoluene(MNT)	90.05	2174	8632	92.9	96.6
Dinitronaphthalene(DNN)	87.45	2229	8650	91.2	95.8
Dinitrotoluene(DNT)	85.11	2244	8697	90.2	95.3
Trinitronaphthalene(TNN)	83.39	2298	8710	90.0	95.0
Trinitrotoluene(TNT)	78.10	2352	8833	87.0	93.5
Trinitroxylene(TNX)	81.75	2298	8797	89.0	94.0
Picric acid(PA)	69.42	2507	8932	83.5	92.0
Nitroglycerine(NG)	0	3469	10084	68.0	84.0
Guncotton(GC)	58.93	2561	9076	78.5	89.0

NS (Refs 11,13). Snelling also patented (Ref 17) an expl constg of AN intimately mixed with sensitizers such as TNT, NS and other expls. Wyler (Ref 18) patented a detonatable expl constg of AN (76 to 96%) and hexamethylenetetramine. To this there may be added one or several of the following: NS, $NaNO_3$, ZnO, hydrocarbons etc. Snelling and Wyler (Ref 22) proposed sensitizing AN by a coating of 1 to 6% of one of the following: aniline, toluidine, naphthylamine, anthramine, diphenylamine, ditolylamine, phenylenediamine, tolylenediamine, benzidine, tolidine, phenylhydrazine, diphenylhydrazine, tolylhydrazine, phenylhydroxylamine, azobenzene or azoxybenzene. Champney (Ref 25) used crystd AN having a packing d of 0.5 to 0.8 g/cc, as detd under a pressure of 10 psi, mixed with various other ingredients such as NG, $NaNO_3$ and bagasse pitch. McFarland (Ref 63) sensitized AN by the addition of 6 to 8% NG. The presence of 6% water was considered advantageous in blasting, as it appeared to decrease the brisance without affecting the strength of the expl

The popular duPont "Nitramon" blasting agents were invented in 1934 by Kirst et al (Ref 30). These expls contd AN as the principal ingredient with at least one sensitizer, such as DNT, paraffin or a mixt of fuels. The fineness and intimacy of contact of the constituents were so controlled that, at a d of 1.0 g/cc, the unconfined expl required a detong impulse greater than that from any common detonator. Kirst et al (Ref 32) also patented an expl of high blasting strength and relatively high density consisting of a mixt of coarse and fine particles of AN and a sensitizing agent such as NG, DNT, etc. Handforth and Johnson (Ref 33) developed a blasting expl of relatively low d, composed of AN having an apparent d less than 0.8 g/cc and a sensitizer such as NG. Hauff and Kirst (Ref 34) reported an expl suitable for blasting coal composed of AN with a puffed, porous texture and an apparent d less than 0.7 g/cc plus a sensitizer such as NG. The porous texture of the AN was obtained by subjecting the slightly moist salt to a special treatment under vacuum. During the past several years a number of additional expls of the "Nitramon" type have appeared. These included "Nitramon A", "Nitramex", "Vibronite B" and various trade name "Nitramon types" (Ref 94)

Many developments of AN coatings and dope additives have improved the resistance to water and moisture of high-AN, low-NG dynamites.

The more important coatings are the Ca stearate coating proposed by Baker (Ref 31) and the Cairns' "PRP" (petrolatum, rosin, paraffin) coating (Ref 47). Winning (Ref 51) described the use of pregelatinized starch products such as rye flour, cereals, meals and similar starch additives [See also the AN/paraffin expls by Stettbacher (Ref 43)]

One problem in the use of AN/fuel mixts for commercial blasting has been the difficulty in detong such expls and insuring the propagation of deton in long columns having small diams. Stoops (Ref 23) reported improved sensitiveness of AN dynamites obtained by making use of the water of crystn of some metal nitrates and heat to yield a molten mixture contg AN, which then was absorbed by a suitable combustible. Spaeth (Ref 24) described another method, in which AN/urea mixts were heated to the mp and absorbed by dynamite pulps. Coatings of trimethylolethylmethane trinitrate were also used by Spaeth (Ref 27). Stettbacher (Ref 20) suggested the use of PETN and other explosives as additives, as did Lewis and Johnson (Ref 26), for increasing the sensitivity of dynamites. Cairns (Ref 36) increased the sensitivity to deton by coating crystals of AN with a soln of inorg nitrates such as Ca, Mg or Zn, and drying the mixts. Davis (Ref 38) disclosed an expl of improved sensitivity to deton and of decreased sensitivity to unintentional initiation, which could be prepd by dissolving AN in liq ammonia and dispersing in it finely divided sensitizers such as Al, sulfur, DNT, NS etc. After evaporating the NH_3, a product remains in which the sensitizer is distributed in a continuous phase. Davis (Ref 45) also developed an expl of good sensitivity to initiation prepd by treating 92 p of AN with a solid aromatic compd such as DNT, and a solid fuel such as diphenylamine or p-toluidine which is soluble in the nitrocompound. The Davis' ammonia process (Ref 38) yields soft and pliable mixts that propagate deton reliably in regular dynamite cartridges and even in charges having small diams. However, these AN/fuel mixts were not easily waterproofed without excessive loss of sensitiveness (Ref 94)

Cook et al (Ref 48) developed another type of expl based on the reaction of AN with Ca cyanamide to yield Ca nitrate and ammonia. An organic substance is added to act as a fuel. During mixing at an elevated temp such as 80°, the freed ammonia renders the mixt plastic and facilitates intimate mixing of the AN with the fuel and/or expl sensitizer. Upon cooling, the mixt remains plastic, and while in this condition may easily be packed at any desired density. A few hours after packing, the free NH_3 reacts with the Ca nitrate to form Ca nitrate-amine and the product "sets" like concrete. Following are examples of mixts proposed by Cook

Composition, %	Density g/cc	Deton rate, m/s
83/12/5-AN/DNT/$Ca(NO_3)_2NH_3$	1.30	3830
83/12/5-AN/DNT/$Ca(NO_3)_2$	–	1750
81.5/12/6.5-AN/DNT/$Ba(NO_3)_2$-NH_3	1.30	3500
81.5/12/6.5-AN/DNT/$Ba(NO_3)_2$	–	2250
81.5/12/6.5-AN/DNT/$Sr(NO_3)_2$-NH_3	1.27	3325
81.5/12/6.5-AN/DNT/$Sr(NO_3)_2$	–	1900

One formulation of Cook's expls was used with satisfactory results when pressed wafers, sealed in cans, were subjected to trials in shooting of oil wells (Ref 54)

Two high-AN "permissibles" which had excellent safety properties for use in gassy and dusty coal mines and also nearly ideal blasting properties were developed by Wahl (Ref 44) and by Cook et al (Ref 46). These features were achieved by careful regulation of reaction rate, which was established by appropriate intimacy of mixing and by control of particle size of the product. Whitworth & Hornell (Ref 61) reported a low density blasting expl also suitable for use in gassy and dusty coal mines and particularly useful for obtaining lump coal. This expl compn consisted of a liq expl org nitrate (such as a nitrated 80/20 mixt of glycerol and glycol) 6–12%, an org expl sensitizer (such as NGu in needle like crysts) ca 10, a bulky, subdivided vegetable material (such as peat) ca 7, AN (or other inorg power producing salt) ca 57–59, resin ca 0.5, diammonium phosphate ca 0.5 and a salt (such as NaCl), which acts as a flame-coolant and retardant ca 11%. The expl described had a bulk d 0.73 –0.76 g/cc

Taylor (Ref 72), in prepn of low density AN expls used a special low d(bulk d < 0.99 at 1.77 atm) dendritic NaCl as the coolant. Such NaCl was prepd by rapidly adding KNO_3 to a boiling soln of equimol quantities of $NaNO_3$ & KCl satd with respect to the resulting NaCl. Using these NaCl crysts, the following expl compn was prepd by Taylor: AN (bulk d 0.71 at 1.7 atm) 55.5, $NaNO_3$ 10, mixt 80/20–NG/NGc 10, peat (bulk d 0.20 at 1.7 atm) 12, dendritic NaCl 12, diammonium phosphate 0.2 and rosin 0.3%

Farr (Ref 66) in 1951 developed a dynamite compn having high d and a low vel of deton when confined. This consisted of AN (coarse and coated with 1% microcryst wax) 82, NG 5, $NaNO_3$ 5, corn flour 2, wood pulp 1.5, apricot pip pulp 4 and chalk 0.5%. The same investigator developed an improved AN dynamite (which could be detonated by a No 8 blasting cap) of the following compn: AN 85.4, TNT 2.5, liq DNT 1.5, granulated soft coal 6.1 and $NaNO_3$ 4.5%. Farr also suggested that improvement in AN/NG expls was achieved by the use of cryst AN contg from 4 to 11% KNO_3 distributed in solid soln throughout the AN crysts (Ref 67)

Semigelatin dynamites represented a significant advance in the development of high AN dynamites. These dynamites made use of collodion cotton, nitrobodies and waterproofing agents in the form of a gelatin to which was added balanced-dope materials (Ref 28). These products had excellent physical and expl props for small diam blasting. Whetstone & Taylor (Ref 62) patented an expl incorporating AN in a liq mixt with certain low-melting eutectics. For instance, a liquid mixt consisting of AN 50, urea 25, Na acetate trihydrate 15 and $Na_2S_2O_3 \cdot 5H_2O$ 10 parts, was gelled by boiling with 3% starch soln and into this 20 p of NG contg 1% NC was dispersed. The resulting product was a soft plastic expl having greater sensitivity than similar compds using dry AN. Hauff & Holmes (Ref 49) prepd a nonsetting expl by coating AN crysts with Zn tetrammino nitrate. Sakurai & Sato (Ref 73) measured, by means of a plastometer, the stress-strain relationship resulting from the addn of 20 to 80% of AN to NG gels. A plot of these data gave an "S" shaped curve. The mechanical properties of these dynamites depended upon the amt of AN, its cryst form, density and surface props. Young's modulus became greater as the d of AN decreased

Some more recent safety AN blasting expls have been described by Taylor & Reid (Ref 86), Davis et al (Ref 84), Rinkenbach & Carroll (Ref 89), Scalera & Bender (Ref 90) and Taylor (Ref 91). Taylor & Reid described an expl prepd by grinding together AN 53.1 & TNT 11.9 parts so that all the mixt passed through a 150 mesh Brit Std Sieve. To this was added 35 parts of AN, all of which passed a No 60 sieve and only 50% of which passed a No 120 sieve. The resulting expl compn had a vel of deton of 2400 m/sec and was sensitive to a No 3 commercial fulminate detonator (Ref 86). Davis et al (Ref 84) described a NG-free blasting compn contg: AN/$NaNO_3$ mixt 60, DNT/TNT mixt 10–30 and finely divided metallic fuel 0.5–10%. The nitrate mixt contained 30–80% AN which passed a 100 mesh sieve. The DNT/TNT mixt contd 1–50% DNT, but the DNT content was not to exceed 10% of the entire compn. The fuel was preferably ferrosilicon contg at least 40% Si. The resulting mixt was blended at 160°F and packed into 1–2" diam cartridges at 120–150°F to obtain a cold d of 1.3–1.5 g/cc. It could be initiated by a cap sensitive primer to give a rate of deton 3500–3900 m/sec and had approx the same blasting strength as an equiv length of tamped dynamite. The compn was less toxic, much less shock sensitive and formed more rigid cartridges than dynamite. Rinkenbach & Carroll (Ref 89) proposed a blasting expl consisting essentially of mixed crysts of AN, urea and a dry pulverized absorbent for liq thoroughly mixed with the said crysts. They patented also a cast expl of high d consisting of AN, urea and an expl sensitizer. Scalera & Bender (Ref 90) rendered AN blasting compns

water-resistant by incorporating ethylenic monomers which polymerize on contact with water. Taylor & Reid's invention (Ref 91) was related to a safety AN blasting expl for use in dusty mines

The properties of AN and the recommended practices for the use of AN in field-compounded expls have been described by Cooley (Ref 96). Parrott (Ref 97) described the use of AN blasting agents in strip-mine operations and Cook (Ref 98) has dealt with large diam bore holes using AN expls not contg NG. One of the cheapest types of AN blasting expls is prepd by mixing prilled AN with 2–10% of No 2 or other fuel oil. The mixing can be done in the field or even in bore holes of large diameter. The O-balanced mixt consists of 94% AN and 6% fuel oil. Its properties are: d 0.8 g/cc, heat of expln 890 cal/g, max available energy 855 cal/g, av heat capacity 0.38 cal/g/°K, total number of moles of gas per kg 43.1, max pressure of gas 42 kilobars, deton vel 4200 m/sec and temp of expln 3000°K. A completely new expl, an aq slurry of AN and TNT, is being used for open pit blasting at the Iron Ore Co mine in Newfoundland. According to Canadian Industries, Ltd, which developed the expl in collaboration with the inventors Cook and Farnum, the slurry character of the material enables it to be loaded directly or in bags into the borehole. Because of its high d (1.4 g/cc) and good water tolerance, the expl can be loaded under water and performs effectively under wet conditions. Strength of the expl compares to that of 70% gelatin dynamite. However, the slurry is much less sensitive and requires a HE primer, the preferred primer being a six-ounce pellet of pentolite which can be initiated by an electric blasting cap or a "Primacord" detonating fuse. The explosive's high strength and efficient loading characteristics are expected to make it especially suited to open pit mining (Ref 93)

The so-called Ammoniakkrut originally patented by Norrbin and Ohlsson in 1867 consisted of AN to which could be added either a combustible (charcoal, sawdust, naphthalene) or an expl (PA, NG, NB). Practically the same AN/fuel compns have been patented over the years and recently for use in large diam blasting. These mixts may be prepd in advance like the Lee and Akrel mixt of AN with 1–12% carbon black packed in polyethylene tubes, or they may be prepd at the place of use (Ref 79). For instance, on the Mesabi Range, Minnesota, in blasting iron ore, good results were obtained by drilling 9" diam bore holes and pouring into each hole one 80 lb bag of AN, followed by one gal of fuel oil. It was found that in large diam holes (5" or more), AN is capable of propagating deton if it is sufficiently fine grained and adequately boostered. It was also found that, for large diam holes, it is not necessary to prepare an intimate AN/fuel mixt to make available at least most of the high explosive potential of such mixts (Ref 94). In very recent developments, Crater et al (Ref 92) reported that a compn consisting of AN mixed with about 5% fuel oil gave satisfactory results when poured into large diam holes and strongly primed with dynamite to insure deton. The holes must be dry, as propagation of deton under unfavorable conditions is less satisfactory than when a nitro-carbon-nitrate mixt, such as 91/4/5-AN/DNT/carbonaceous material is used. Present trends in the use of high-AN/fuel mixts suggest that important new developments can be expected in future AN blasting expls

AN High Explosives. As outlined above, AN is an important constituent of dynamites, but it is also a significant constituent of a number of other expls in which it is present in large proportions. These expls may be divided into two main types: a) expls in which a sensitizer, itself detonable, is used as a means of increasing the sensitivity to deton of AN and b) expls in which the material employed to sensitize AN is not detonable. In general, AN expls have low rates of deton (2000–

3500 m/sec) and high gas vols. Consequently, they have low shattering and good heaving or pushing effects. Their ballistic pendulum test values are 115–125% that of TNT. When used in quarries, it has been found that AN expls often show a shattering effect greater than expected on the basis of their low rates of deton, and this is ascribed to their large vols of gases having a disproportionate effect on shattering power. Although AN expls are rarely graded commercially in terms of strength percent, their strength may be as great as 65% dynamite, ie, equivalent to a 65% straight NG dynamite on an equal weight basis

AN Explosives Containing Detonable Sensitizers. There is no sharp line of division between AN dynamites and some other AN expls; the difference being mainly that AN expls are formulated with a lower percentage of NG or NS and therefore a higher percentage of AN. The presence of as little as 1% NG or NS in AN is sufficient to cause marked sensitization. Commercial AN expls are made by incorporating 3–10% NG or NS and 40–89% AN. The remaining component is usually a small amt of wood pulp or other carbonaceous material which would also be sensitized by NG. TNT, nitrotoluenes, naphthalene and other similar materials have been proposed for use as sensitizers, and some of them have actually been used, especially in foreign countries. However, in the US, NG and NS are the principle detonable sensitizers

AN Explosives Containing Nondetonable Sensitizers. Many substances which are not in themselves expls greatly increase the sensitivity of AN when added to it. To these belong: powdered metals (such as flaked Al) and combustible org materials (such as rosin, many metallic resinates, hexamethylenetetramine, many org bases, and paraffin oils and waxes). Usually these AN expls are prepd by coating granules of AN, either alone or mixed with NaCl, with a thin layer of sensitizer. If it is desired to make an expl of exceptional safety, there is employed an amt of sensitizer insufficient to make the expl capable of deton by the action of an ordinary blasting cap, but requiring either a US Corps of Engineers cap or a heavy booster charge such as TNT or amatol. A mixt of 92.5 AN, 4 DNT and 3.5% paraffin wax is representative of this type of expl. By controlling the d, bulk strength from less than 40 to 75% that of straight dynamite may be obtained. The vel of deton varies with the grade of AN and the diam of the charge, but it usually is between 3500 and 5000 m/sec. When properly initiated, the deton can be propagated across an air gap of 8–15 in. In order to exclude moisture, all AN expls must be packed in sealed metal containers. The simultaneous action of Zn, humidity and AN on nitrocompds such as TNT and TNX is apt to produce some dangerous products. As Al does not react under the same conditions, it is recommended for packing any mixt contg AN and nitrocompd expl (Ref 74)

In 1917 a patent was granted to Vereinigte Köln-Rottweiler Pulverfabriken (Ref 6) for a cast AN expl contg nitrocompds and "cell-pitch lye". One such compn contd at least 60 AN, less than 20 $NaNO_3$, less than 30 TNT and up to 10% "cell-pitch lye" (qv). The addn of "cell-pitch lye" lowers the mp considerably, aids in the mixing of the nitrates and nitrocompds, and prevents separation of the ingredients. Müller (Ref 12) proposed mixing molten AN, in an autoclave at a pressure of 0.5 to one atm, with hydrocarbons or hydrocarbon derivatives. Cook & Talbot (Ref 65) studied the sensitivity to explosion of AN/hydrocarbon mixts. Manuelli & Bernardini (Ref 8) melted together AN and GuN (guanidine nitrate) or biguanidine nitrate, or AN and dicyandiamide with/without the addition of NGu, in such proportions that the product melted below 130°. To the above could be added oxidizing agents, either with or without oxidizable substs. Heating AN and dicyandiamide together produces salts of biguanide and guanidine according to the temp and duration of the process. An expl compn consisting of AN 25 and urea nitrate 75%,

to which may be added some NC and Al, also was reported (Ref 15). In another procedure AN was melted with a nitrate or nitrates of aliphatic mono- or polyamines and then cast (Ref 21). Morris (Ref 29) obtained a nongelatinous HE by agitating hot, pulverized AN with a molten or partly molten mixt of a nitrocompd, such as TNT or DNB, and PETN at a temp not exceeding 100°. Davis (Ref 42) described a plastic expl consisting of granular AN and molten TNT for loading into shell. Some promising low-melting AN expls were developed at Pic Arsn. It was found that mixts of AN with Na acetate or with urea and $NaNO_3$ could be melted and cast at 103° (Ref 50). By the addn of Al to such mixts, expls were obtained having brisance and stability characteristics equal to TNT. However, when cast-loaded into 3" shell and detonated, the expl was of too low brisance for military use. Addn of Amm picrate to the basic ternary mixt resulted in an expl of satisfactory brisance; but upon storage at elevated temps, the expl exuded (See also Addnl Ref H on p A352)

In an effort to overcome the objection of exudation, two additional low-melting AN expls were developed at Pic Arsn (Ref 53). The most promising compns, designated as Ammonex I, J and M, had the following components and properties:

Composition, %	I	J	M
AN	42.0	32.5	36.0
Na nitrate	17.0	5.0	5.5
Ca nitrate, anhyd	–	7.5	8.0
Dicyandiamide	5.5	5.0	5.5
Guanidine nitrate (GuN)	5.5	–	–
Expl D (Amm picrate)	30.0	50.0	45.0
Avg chge d, g/cc	1.35	1.66	1.61
Approx pour point, °C	97	98	92
Av wt chge in 3" shell, lbs	0.74	0.91	0.89

Note: Compns, designated Ammonex K and L, were also developed with tetryl added in amounts equivalent to those of the Amm picrate used in the above mixts. These mixts were not superior to those contg Amm picrate when subjected to shell fragmentation tests

The three mixts shown below were approx equal to TNT in brisance, as indicated by fragmentation tests of cast-loaded 3" shell. Their stability was satisfactory, and they did not exude in storage tests at 65°

Campbell & Campbell (Ref 60) studied binary and ternary eutectics involving AN and found that compns contg AN, a mixt of nitrates of alkalies or heavy metals and TNT were unsatisfactory for filling shells. The eutectic consisting of 50/50-AN/ethylenediamine dinitrate, however, melted at 100° and satisfied nearly all requirements for a castable shell filler. A castable AN-based expl was reported also by Taylor & Whetstone (Ref 59). Early processes for the loading of AN into shell were described by Paris (Ref 9) and Olsen & Bain (Ref 19). Paris exhausted the air from a hermetically sealed mold having a riser, and siphoned the charge-forming material from a molten supply into the mold and riser. The material in the riser was maintained in a molten state while the charge solidified (Ref 9). Olsen & Bain suggested pouring into shell a predetermined amt of expl, such as TNT, followed by addn of AN pellets not larger than 10 mesh until the liquid just covered the pellets. Setting of the mixt was accelerated by absorption of heat from the molten expl by the AN pellets (Ref 19). The

process for the liquifaction of AN of mp 152° used in expl charges, by mixing AN with dicyandiamide (mp 205°) in the proportions 85/15 was described in 1917. The resulting mixt (fluid at 115°) was considered to be easier and less dangerous to use than AN alone (Ref 7). (See also Ref 70 for description of methods of prepn of AN expls by Médard and LeRoux)

Some miscellaneous AN expls having no special names include the following: a) A mixt of AN 93.6 and charcoal 6.4% similar to "thorite"(qv) with the addition of a little Al was used in bombs during the Spanish Civil War. This mixt had low brisance but comparatively high power (see also Ref 68) b)Mixts of AN with trinitrophenoxyethanol, $(O_2N)_3C_6H_2 \cdot O \cdot C_2H_4OH$(Ref 63) c)A stabilized AN/NG expl compn in which 4 to 12% of KNO_3 was distributed in solid soln throughout the AN crysts. The expl had stable rate of deton, small setting tendency and good thermal stability d)A 50/45.5/4.5 mixt of AN/RDX/beeswax that has the characteristics: OB to CO_2 –24%, power by Bal Mort 122% TNT, Q_c^v 209.3 kcal/mol, Q_e^v 94.5 kcal/mol, calc temp of expln 4890°K and gas vol 43.2 mol/kg e)A 60/40 AN/methylamine nitrate mixt that has the characteristics: OB to CO_2 –1.6%, power 122% TNT, Q_c^v 108.0 kcal/mol, Q_e^v 102.2 kcal/mol, calc temp of expln 3430°K and gas vol 43.7 mol/kg f)A mixt contg AN 60–40, RDX 15–20, TNT 0–10, hydrated potters clay 25 and water 2–6.25(Ref 75) g)A mixt of AN 35–60, TNT 0–10, NC 0–0.5, carbohydrate 0.5–5, NaCl with seaweed 5–25, WM(wood meal) with starch 0–10 and water 5–15%(Ref 76) h)An expl compn prepd by blending mixt A 8.8–12.5% with mixt B 91.2–87.5%. The mixt A consisted of Na cellulose glycolate 0.8, glutenous millet powd 2.5, glutenous rice powd 2.5, nonglutenous rice powd 3.0, flour 3.5 and potato starch 4.5 parts to which 8% of water was added. The mixt B consisted of NG 22, NC 1, AN 62.2 and WM 2 parts(Ref 77) i)An expl prepd by mixing 918 g of AN, 80 g of TNN or TNT dissolved in concd nitric acid, followed by neutralization of acid by blowing ammonia into the mixt(Ref 78) j)A compn consisting of AN 78.5–83.5, hydrazine nitrate 5–10, TNT 7.5, Al dust 0.5, coal 3 and chalk 0.5%. The introduction of up to 10% of hydrazine nitrate in an AN expl markedly increases sensitivity and deton rate. AN and hydrazine nitrate are preferbably mixed together prior to the addition of other ingredients, or they may be fused, solidified and subdivided prior to such addition (Ref 80) k)An expl obtained by neutralization of an acidic nitrated polyol such as NG by the addition of amines or amides such as urea, especially in the form of a melt with AN in a eutectic mixt. The expl is stable in storage, especially when the amine or amide is present in excess(Ref 87) l)An expl prepd by treatment which consists of neutralizing an acid-contg nitrated org prod, such as NG+nitroglycol or nitrosucrose, with a mixt or soln of AN(with or without other nitrates) in ammonia and/or org bases such as amines, amides and urea. The neutral mixt is then converted to a powerful expl by the addition of fuels, such as peat or wood flour and powd Al, and a gelatinizing agent such as NC(Ref 83) m)A cored AN expl cartridge having a granular base charge consisting of AN 88.5, TNT 7.5, coal dust 3, chalk dust 0.5 and Al powd (d 0.9–1.0) 0.5% is overlaid with a cast core of HE such as 40% RDX(100–200 mesh) in 60% TNT. The core diam may be varied so as to alter the strength from 60 to 90% of the same wt of NG and the deton rate is simultaneously varied between 4000 and 8000 m/sec(Ref 85) n)A Brit "non-permitted" expl contg 92 AN and 8% carbonaceous material(Ref 95)

It is known that expls contg AN possess the undesirable property of changing their vol if there are large variations in temp. If this expansion occurs in thin-walled ammunition components, considerable damage may result. This abnormal expansion is due to the fact that AN exists in five cryst modifications and great

changes in vol occur at the transition points. According to G. Romer(Ref 57), E. Janecke observed that if 5–10% KNO_3 is melted with 95–90% AN and the melt grained, mixed crysts are formed which do not possess any sharp transition points and undergo no abnormal expansion or contraction. It was also observed that, in mixts contg AN and PETN, if part of the PETN is replaced with "Tetra-Salt" (See Tetra-methylammonium Nitrate) the abnormal expansion of the expl is very much reduced(Ref 57)

AN Propellants. Propellants based on AN have had limited uses for military purposes, particularly in Germany and Austria, since 1885 when Gäns(Ref 1) patented a powd made from 35–38 AN, 40–45 KNO_3 and 14–22% charcoal. This compn came into use under the name "Amidpulver". An improved "Amidpulver" contained AN 37, KNO_3 14 and charcoal 49%. It gave a flashless discharge and only a moderate amt of smoke when fired in a gun. Another propellant, first manufd in Austria in 1890 and used during WW I by the Austrians and Germans, was called "Ammonpulver". This compn incorporated 85 AN and 15% charcoal in addition to a small amt of an aromatic nitro compd. "Ammonpulver" was cheap, powerful, flashless, smokeless, and insensitive to shock and friction but it had the disadvantage of being difficult to ignite, as well as giving irregular ballistic effects and tended to disintegrate on storage under widely varying temp conditions. According to one report(Ref 55), the "Ammonpulver" used during WW I contained AN 50, NC (12% N) 22, DEGN 22, hydrocellulose 5 and Centralite 1%(Ref 52).(See also "Ammonpulver" in PATR 2510, p Ger 5). Mayr(Ref 4) has discussed the advantages and disadvantages of using AN in gun propellants. DuPont(Ref 10) adopted a propellant for use in guns by using 92.5% AN imbedded in 7.5% of colloided NC

Roffey et al(Ref 56) formed a propellant by evapg a soln of AN, with small amts of KNO_3 or $Mg(NO_3)_2$ added, to dryness and grinding the resulting cake. Some AN propellants developed by the Aerojet Engrg Corp (Ref 58) include those shown on the following page. Taylor and Whetstone(Ref 59) prepd a rocket propellant by melting together at 90° AN 67 and magnesium nitrate 10 parts and adding, just before casting, 8 parts of ammonium dichromate to yield a gas-producing mixt. In a paper reported in the 3rd Symposium on Combustion(Ref 64) Taylor and Sillitto described the use of AN as a solid to provide gas for propulsive purposes. A cast of a compressed mixt of AN with 3% or more of potassium dichromate reacts completely, when ignited, to give gsseous water, nitrogen and a mixture of oxides of nitrogen. Hannum(Ref 81) described a liq propellant consisting of AN or ammonium perchlorate dispersed in a polynitro aliphatic hydrocarbon such as nitromethane, nitroethane, 1-nitropropane, 2-nitropropane, 1,1-dinitropropane, 2,2-dinitropropane, 1,2-dinitropropane, 1,3-dinitropropane or mixts thereof. The Std Oil Co of Indiana also has contributed to the development of solid propellants based on AN(Ref 88)

AN propellants assume importance in the type of compn known as "composite propellants". These contain no NC or NG, and are uncolloided, heterogeneous mixts consisting of org fuel, inorg oxidizing agent and org binder. Such compns can be manufd by a simple mixing operation and then molded in the desired form by pressing. The development of improved binding agents less affected by extreme temps will expand the future usefulness of this type of propellent compn(Ref 82)

Some AN propellent compns developed in the USA since WW II are classified and cannot be described in this work

The Table on next page lists some recently developed US AN propellants which are not classified

Aerojet Propellants

Composition, %	SP-42	SP-43	SP-45	SP-47	SP-49
AN	70.00	49.00	50.00	40.00	20.00
NH_4ClO_4	–	31.00	20.00	–	55.00
$KClO_4$	–	–	–	35.00	–
Paraplex P-10	29.83	19.90	29.80	24.85	24.85
t-Butyl hydrogen peroxide	0.17	0.10	0.20	0.15	0.15

Composition, %	SP-51	SP-52	SP-54	SP-55
AN	71.70	50.00	50.00	56.00
Nitromethane	–	23.00	23.00	20.00
Paraplex P-10	–	26.85	26.45	23.50
t-Butyl hydrogen peroxide	–	0.15	–	0.50
cat KNR-neoprene cement	28.30	–	–	–
Dibenzoyl peroxide	–	–	0.55	–

References on Ammonium Nitrate Blasting Explosives, etc: 1) D.R.Gäns, GerP 37, 631(1885) & Davis(1943),p 49 2)R.Escales, SS 1,456-7(1906) 3)O.Guttmann, SS 2,57 (1907) 4)J.Mayr, SS 2,401-3(1907) 5)H. Schmerber,SS 12,128-32 & 151-5(1917);CA 12,426(1918) 6)VKRP,GerP 303,980(1917) & JSCI 41, 199A(1922) 7)Dynamit AG, Ger P 305,567-8(1917) & JSCI 39,430A (1920); CA 14,2555(1920) 8)C.Manuelli & L.Bernardini,BritP 138,371(1917) & CA 14,2086 (1920) 9)P.G.Paris,USP 1,282,623(1918) & CA 13,188(1919) 10)F.I.duPont,USP 1,341,207(1920) & CA 14,2265(1920) 11) W.O.Snelling,USP 1,343,063(1920); J.B. Bronstein & C.E.Waller,USP 1,343,077(1920) & CA 14,2420(1920) 12)E.Müller,BritP 152,199(1920) & CA 15,756(1921) 13)W.O. Snelling,USP 1,395,775(1922) & CA 16,648 (1922) 14)C.A.Taylor & W.H.Rinkenbach, US BurMines Bull 219(1923) 15)Societé d' Explosifs et des Produits Chimiques, Fr P585,671(1924)(not found in CA) 16)E. Audibert, RevIndMinérale 1925,1-14 & CA 19,1198(1925) 17)W.O.Snelling,USP 1,617,182(1927) & CA 21,1013(1927) 18) J.Wyler,USP 1,720,459(1929) & CA 23,4344 (1929) 19)F.Olsen & C.J.Bain, USP 1,752,391(1930) & CA 24,2606(1930) 20) A.Stettbacher, SwissP 150,015(1930) & CA 26,4955(1932);USP 1,867,287(1932) & CA 26,4720(1932); NC 4,166(1933) 21)Dynamit A-G, BritP 384,966(1932) & CA 27,5981 (1933); FrP 742,312(1933) & CA 27,3612 (1933); P.Naoúm & R.von Sommerfeld u Falkenhayn,USP 1,968,158(1934) & CA 28, 5986(1934) 22)W.O.Snelling & J.Wyler, USP 1,827,675(1932) & CA 26.601(1932) 23)B.I.Stoops, USP 1,908,569(1933) & CA 27,3823(1933) 24)C.P.Spaeth, USP 1,920,438(1933) & CA 27,4930(1933) 25) H.Champney, USP 1,924,912(1933) & CA 27, 5542(1933) 26)H.A.Lewis & N.G.Johnson, USP 1,922,938(1934) & CA 28,7018(1934) 27)C.P.Spaeth, USP 1,932,050(1934) & CA 28,646(1934); CanP 340,401(1934) & CA 28, 3906(1934) 28)C.A.Woodbury, USP 1,944,910(1934) & CA 28,2538(1934); J.A.Farr, USP 2,602,026(1952) & CA 46,7771(1952) 29)W.Morris, BritP 437,035(1934)(not found in CA) 30)W.E.Kirst et al, USP 1,992,216 & -17 CA 29,2744(1935) 31)S.G.Baker,Jr, USP 2,048,050(1936) & CA 30,6200(1936) 32)W.E.Kirst et al, USP 2,069,612(1937) & CA 31,2010(1937) 33)S.L. Hanford & N.G. Johnson, USP 2,087,285(1937) & CA 31,6466 (1937) 34)T.W.Hauff & W.E.Kirst, USP 2,125,161(1938) & CA 32,7728(1938); CanP 380,363(1939) & CA 33,5662(1939) 35)H. Muraour & G.Aunis, MP 28,182-203(1938) & CA 33,8406(1939) 36)R.W.Cairns, USP 2,130,712(1938) & CA 32,9504(1938) 37)

E.F.Reese, PATR 946(1938) 38)C.O.Davis, USP 2,168,562-3(1939) & CA 33,9648(1939) 39)M.Ovchinnikov, KhimRefZh 2, No 4, 135 (1939) & CA 34,2173(1940) 40)V.A.Assonov & R.D.Rossi, GornyiZh 1939, No 7, 38–41 & CA 34,8283(1940) 41)G.A.Abinder & K. Andreev, GornyiZh 1939, No 7, 42–3 42)T.L.Davis, ArOrdn 20,91–4(1939) 43)A. Stettbacher, NC 10,109–10 & 128–30(1939) 44)M.H.Wahl, USP 2,171,379(1940) & CA 34, 265(1940) 45)C.O.Davis, USP 2,185,248 (1940) & CA 34,3092(1940) 46)M.A.Cook et al, USP 2,199,217&8(1940) & CA 34,6078 (1940) 47)R.W.Cairns, USP 2,211,738(1941) & CA 35,625(1941); USP 2,338,164(1944) & CA 38,3478(1944); USP 2,355,269(1944) & CA 38,6564(1944) 48)M.A.Cook et al, USP 2,220,891&–92;CA 35,1636(1941); Brit P 544,582(1942) & CA 36,6804(1942) 49)T.W. Hauff & H.H.Holmes, USP 2,222,175(1941) & CA 35,1636(1941) 50)A.J.Phillips, PATR 1106(1941) 51)C.Winning, USP 2,314,806 & 7,8,9,10(1943) and CA 37,5241(1943), 52)Davis(1943),p 49 53)R.D.Sheeline, PATR 1234(1943) 54)M.A.Cook, CanP 411,896(1943) & CA 37,3943(1943); USP 2,312,752(1943) & CA 37,4902(1943) 55) Anon, CIOS Rpt No 31–68,p 7(1945) 56)F. Roffey et al, BritP 573,147(1945) & CA 43, 5952(1949) 57)G.Römer, PBL Rept No 85160(1945) 58)W.E.Campbell,Jr. & W.H. Brown, AerojetEngrgCorp Rept No 192(1946) (15 pp) 59)J.Whetstone, USP 2,409,919 (1946) & CA 41, 865(1947); USP 2,460,375 (1949) & CA 43, 2776(1949); J.Taylor & J. Whetstone, USP 2,434,872(1948) & CA 43, 852(1949) 60)A.N.Campbell & A.J.R.Campbell, CanJRes 25B,90–100(1947) & CA 41, 3967(1947) 61)E.Whitworth & J.Hornell, BritP 595,443(1947) & CA 48,6699(1954) 62)J.Whetstone & J.Taylor, BritP 597,716&8 (1948) & CA 42,4349(1948); USP 2,548,693 (1951) & CA 45,10590(1951) 63)D.McFarland, USP 2,463,709(1949) & CA 43,6828(1949) 64)J.Taylor & G.B.Sillito, "3rd Symposium on Combustion", Williams &Wilkins, Baltimore,Md(1949),572–9 65)M.A.Cook & E.L. Talbot, IEC 43,1098–1102(1951) 66)J.A. Farr, BritP 649,473(1951) & 649,613(1951); CA 45,10589(1951) 67)J.A.Farr, BritP 662,346(1951) & CA 46,5320(1952) 68)A. Darche & E.Delemotte, FrP 986,807(1951) & CA 50,574(1955) 69)F.M.Lang, MP 34,189–94(1952) 70)L.Médard & A.LeRoux, MP 34, 195–204(1952) 71)J.A.Farr, BritP662,346 & USP 2,602,026(1952); CA 46,5320&7771 (1952) 72)J.Taylor, BritP 682,209(1952) & CA 48,6700(1954), 50,11672(1956); USP 2,736,262(1956) & CA 50,6796(1956) 73) T.Sakurai & Y.Sato, J IndExplSocJapan 14, 111–6,226–9(1953) & CA 49,11283(1955) 74)G.Bourjol, MP 36,41–5(1954) 75)A.Sato, JapP 988(1954) & CA 48,14210(1954) 76) T.Sakurai, JapP 989(1954) & CA 48,14210 (1954) 77)N.Sakurai, JapP 990(1954) & CA 48,14210(1954) 78)Y.Tsurusaki, JapP 148 (1954) & CA 48,13222(1954) 79)H.B.Lee & R.L.Akre, USP 2,703,528(1955) & CA 49, 15248(1955) 80)L.F.Audrieth, USP2,704,706 (1955) & CA 50,8208(1956) 81)J.Hannum, USP 2,721,792(1955) & CA 50,3740(1956) 82)Anon, TM 9–1910/TO 11A–1–34(1955), p 259 83)B.P.Enoksson, USP 2,736,742 (1956) & CA 50,6796(1956) 84)C.Davis et al, USP 2,752,848(1956) & CA 50,13444(1956) 85)J.Ruth & R.Wakefield, USP 2,754,755(1956) & CA 50,15087(1956) 86)J.Taylor & T.Reid, BritP 743,710(1956) & CA 50,14230(1956) 87)B.P.Enoksson, SwedP 154,069 & 70(1956) & CA 50,14230(1956) 88)Anon, Std Oil Co (Ind) Quarterly Rpt No 5, Dec 1956–Feb 1957 89)W.H.Rinkenbach & W.H.Carroll, USP 2,814,555(1957) & 2,817,581(1957); OffGaz 724, No 4, 774(1957) 90)M.Scalera & M. Bender, USP 2,826,485(1958) & OffGaz 728, No 2, 383(1958) 91)J.Taylor & T.J.Reid, USP 2,839,374(1958) & CA 52,14172(1958) 92)W.DeC.Crater et al, IEC 50,43A(1958) 93)Anon, C&EN 17 Nov 1958,p 43 94)Cook (1958),pp 10–4 95)J.Taylor & P.F.Gay, "British Coal Mining Explosives", G.Newnes Ltd, London(1958),p 26 96)G.B. Clark,ed, "Third Annual Symposium of Mining Research," Univ of Missouri School of Mines & Metallurgy (1958),pp 123–8, C.M.Cooley 97)Ibid,pp 129–34, F.W.Parrott 98)Ibid,pp 135–48, M.A.Cook

Addnl Refs: A)Société Roth (Germany), FrP 303,427(1900) & 353,864(1905); CA **1**, 1343 (1907)(An expl suitable for loading shells contained: AN 45, DNT or TNT 19.5, Al 22.0, BkPdr 4.5, PbO_2 1.0 sulfur 2.5 & KNO_3 with charcoal 5.5%) B)A.Haller, BullSocEncour-IndNat **119**, 761(1920) & CA **15**, 1401(1921) (Expls consisting of AN coated with paraffin were used by the French during WWI as bursting chges in airplane bombs, trench mortar projectiles and hand grenades) C) T.J.R.Alexander, BritP 297,375(1927) & CA **23**, 2827(1929) (A very intimate mixt suitable for use in expls can be obtained by heating to 140° equimol quantities of a concd soln of AN and Na perchlorate, followed by crystallization) D)Pepin Lehalleur(1935), 352-3 lists several AN expls manufdd betw WWI & WWII by the Poudreries de l'État(France) and supplied to industry for cartridges. These expls were known under the general name **Explosifs de Favier** and included the following: a)*Grisou-naphthalite couche* AN 95 & TNT 5% b)*Grisou-naphthalite salpêtrée* AN 90, K nitrate 5 & TNT 5% c)*Grisou-naphthalite roche* AN 91.5 & DNN 8.5% d)*Grisou-naphthalite roche salpêtrée* AN 86.5, K nitrate 5 & DNN 8.5% *e)Poudre Favier pour mines non-grisouteuses* AN 87.5 & DNN 12.5% f) *Grisou-tetrylite couche* AN 88, K nitrate 5 & tetryl 7% g)*Poudre de mine C 1b* AN 78 & Amm trinitrocresylate 22% E)A.G.White, USP 2,128,576(1938) & CA **32**, 8782(1938) (Blasting expls consisting of AN mixed with a faster burning substance such as black powder) F)Wm.E.Kirst, USP 2,145,399 (1939) & CA **33**, 3590(1939) (Expl, consisting of AN mixed with an org sensitizer such as DNT which is normally solid but fusible G) E.I. duPont de Nemours & Co, BritP 531,562 (1941) & CA **35**, 8299(1941) [Blasting expls consisting of AN sensitized with inorganic sensitizers (such as Al, Mg, Sb, Zr, ferro-silicon, Ca silicide, S etc) or organic sensitizers (such as DPhA, hexamethylenetetramine, acid amides, nitrocompds, alcs, nitrates, sugars, etc). These substances are dispersed in AN soln prior to crystn] H)A.J. Phillips, PATR 1106(1941) [A number of commercial AN expls tested at PicArsn, have been found to have satisfactory brisance values, but they were unsuitable for general military use because they could not be cast or extruded, but only compressed. When compressed to obtain suitable high density, they were found to be relatively insensitive to detonation. An effort had been made to develop AN expls which were highly brisant and castable. Two promising expls were developed: No 1 which contd AN 80.1 Na acetate 9.9 & Al 10.0% and No 2 which contd AN 60.5, Na nitrate 18.0, Al 11.0 & urea 10.5%. Both compns could be cast-loaded at ca 103° but the cast material was more difficult to initiate than the pressed material, especially at low d. Compsn No 2 was easier to initiate and it was of excellent stability. The sand test value of pressed material initiated with a No 8 elec detonator was almost equal to TNT, but the cast material gave lower results. The cast Compn No 2 fragmentation test value, listed in PATR 1234 was not as satisfactory as expected and new expls, known as Amatexes were developed (See Ref 53) I)CondChemDict (1942), p 287 lists the following expl compsn AN 80, NG 10, K chlorate 5 & coal tar 5% J)E.I. du Pont de Nemours & Co, BritP 535,137(1941) & CA **36**, 1496 (1942) (Coal blasting expl consisting of AN, a solid capable of functioning as both a detonation sensitizing agent and a binder and a liq medium capable of softening superficially the particles of binder) K)J.Barab, USP 2,280,360(1942) & CA **36**, 3650(1942) [A low d expl mixt detonable by a No 6 or No 8 cap is prepd by intimately mixing very finely divided AN and combustibles (such as coal, rosin, Al & TNT), compressing the mixt thus formed and crushing the resulting mass into a pdr, detonable by a No 6 or No 8 cap] L)A.J.Phillips, PATR 1302(1943) (Study of corrosive action AN expls, such as amatols on Cu in presence of moisture. In the first stage of reactions are formed basic Cu nitrates and ammonia and these readily combine giving tetrammine copper nitrate, which is expl)

N)Société Suisse des Explosifs, Swiss P 228,940(1943) & CA **43** 2437(1949) (An expl mixt consisting of AN 70, PETN 10, TNT 10 & $NaNH_4HPO_4 \cdot 4H_2O$ 10%. The phosphate is incorporated in order to lower the mp of the mixt and to give it higher stability) O)A.H. Blatt, OSRD Rept 2014(1944) lists the following mixts suitable for military purposes: a)AN 55 & ethylenediamine nitrate 45%; casting temp 105° and power by Trauzl test 126% PA b)AN 50 & ethylenediamine nitrate 50%; mp 102.5°, d 1.6333 at 25/4°; impact sensitivity-requires fall height 161% that of TNT; decomp with evoln of brn fumes at 278° c)AN 60, ethylenediaminedinitrate 20 & TNT 20%; the components were mixed dry and then molded at 108°; power by ballistic mortar is 135% TNT, impact sensitivity-requires fall height 170% that of TNT; decomp with evoln of brn fumes at 260° d)AN 60 & methylamine nitrate 40%; can be cast at 58°; power by Trauzl test 121% PA O)OpNav Rept 30-3M(1945), p 27, PBL Rept 53045(1945) and PB Rept 50394 (1946) list the following AN expls used by the Japanese during WW II; Ammonarū Anbenyaku (Shobenyaku), Angayaku, "E" Explosive, Shoanbakuyaku, Shoanayaku, Shonayaku, Shotoyaku, Torpex-Type Explosives, Type 4($Mk2K_2$), Type 4(Mk3), Type 4($Mk5K_5$) and Type 88 Explosives. These compns are listed in this work in alphabetical order P) PB Rept 1154(1945) & All & EnExpls(1946), p 134 list an AN expl used during WW II by the Italians for press-loading some 47 mm AP shell. Irs compn was AN 73.4-75.0, RDX 22.0 & Wax 5.0-4.6% R)T.Watanabe, JapP 176, 113(1948) & CA **45**, 4930(1951) (A blasting expl consisting of AN 71.7 NG 8.0, CC 0.3 & pulverized seaweeds 20.0%) S)Belgrano (1952), pp 33-6 & 45 lists many Italian AN expls, for example: a)AN 75, K perchlorate 10, DNN 10, Ca silicide 4 & wood flour 1% b)AN 69, K perchlorate 8, TNT 20 & Al 3% c)AN 82, DNN 9, TNT 3, cellulose 2 & Mn dioxide 4% d)AN 76, K perchlorate 8, TNT 12, sawdust 2 & Al 2% e)AN 68, K perchlorate 10, TNT 12, Ca silicide 8 & sawdust 2% f)AN 79.5, TNT 10, Ca silicide 8 & wood flour 2.5% T)K.K.Andréev & A.P. Glazkova, DoklAkadN **86**, 801-3(1952) & CA **47**, 10229(1953)(Additives such as NaCl and $BaCl_2$ exert an anti-firedamp action and increase the rate of combustion of AN) U) Kirk & Othmer **11** (1953), 777 stated that AN can be used as an oxidizer in rocket propellants in place of K perchlorate V)Warren (1958), p 1 lists a solid rocket propellant consisting of AN 80, elastomeric binder 18 & additives 2%. He also discusses on pp 34-5 the use of AN as an oxidizer in rocket propellants W)Dr L.Deffet, Belgium listed in private communication, 10 March 1954, the following AN expls as currently manufd in Belgium: Alkalite, Alsilite, Centralite TA, Cooppalite Dynamite III, Flammivore, Fractorite, Matagnite, Nitrocooppalite, Sabulite, Sécurite C and Sécurite G. Their compositions are given in this work in alphabetical order corresponding to their given names X)A.Stettbacher, Switzerland listed in private communication July 9, 1954, Aldorfit as a powdery AN expl manufd by the SweizSprengstoff-Fabrik A-G, Dottikon. The same plant manuf Gelatine-Aldorfit. Other Swiss AN expls are Gamsit and Telsit Y)J. Taylor, "Solid Propellent and Exothermic Compositions," Interscience, NY(1959), 118-20 lists the following ball-milled powder: AN 78.5, KNO_3 9.0, anhyd Amm oxalate 6.9 and Amm bichromate 5.6parts with 0.7p China clay added as a compn proposed for propulsion of the reciprocal energetic "William & James" motor, and with 2.5p China clay added as a compn proposed for driving a rotary blower motor Z)Spencer Chemical Co, Kansas City, Missouri reports:"Ammonium Nitrate Explosives for Underwater Applications", Jan 18, 1960 and "Safety Data", Feb 4, 1960 obtained through the courtesy of Mr. S.J.Porter give some expl properties of Spencer AN expls, such as 94/6-N-IV Prilled AN/Fuel oil No 2. These expls are the least sensitive of all military expls and of some commercial expls, such as 40% dynamite, Nitramite, Nitramon A, etc

The enclosed table gives some properties of typical Spencer expls, as compared with those of TNT, dynamite, etc. In all cases, Spencer expls were initiated by the Pentolite booster (675 g)

Explosive	Charge Density, g/cc	Cook-Off Temp (ave)	Deton Velocity, m/sec (max)	Wax-Gap, inches	Impact-Friction Pendulum: Drop Height, feet	Impact-Friction Pendulum: Number Exploded to Total Samples
N–IV Prill with	0.90	270	2,490	0	3½	0/5
6% Fuel oil	0.95	–	2,010	4	–	–
Ditto, coated	0.90	270	2,670	0	3½	0/5
with 3% diato-maceous earth	0.87	–	2,220	2	–	–
N–IV Prill without oil	0.86	292	No detonation		3½	0/10
Nitramite	–	255	–	–	3½	2/4
Nitramon	–	239	–	–	3½	3/6
40% gelatin extra dynamite	–	–	–		½	42/42
TNT	–	240 & 260	–	–	1	16/30
Explosive "D''	–	–	–	–	2½	7/20

Notes:

a)''Cook-Off'' Temperature is the temp at which the material decomp by heat and this provides a measure of its thermal sensitivity. In the test conducted at the Spencer Chem Co, a sample was placed in steel(or aluminum) cylindrical oven, 2½" long and ¾" ID and sealed with a threaded plug. The oven was wrapped with nichrome resistance wire and insulating asbestos. Elec energy supplied to the resistance wire raised the temp of the oven at the rate of 13 to 15° per min and was measured by a thermocouple and galvanometer. The point at which the material decompd was noted by a sharp rise in temp(caused by the energy release on decompn) and usually an audible reaction b)Detonation Velocity was detd with the apparatus using the Beckman-Whitley High-Speed Framing-Camera. This test was conducted in conjunction with the ''Wax-Gap'' test c)Wax-Gap Test. In this test a Shelby steel seamless tube(2" ID, 36" long and 0.120" wall thickness) was closed at the bottom with paper tape and charged to near the top with the test expl. Acrawax plugs of the same diam as the tube and of varying thicknesses may be placed on top of the charge. Two elec wires(connected to the deton velocity app) were inserted in the charge, one near the middle of the tube, marked ''Start'' and the other near the bottom of the tube marked ''Stop''. The charge was detonated by a No 6 elec blasting cap and pentolite booster(cylinder 2 × 8", weighing 675 g), placed on top of the Acrawax. If no deton took place with, say a 4" wax thickness, the test was repeated with a thinner wax and so on until a complete deton of charge was produced. When no deton was produced with any thicknesses of wax and only without wax, the gap was marked as 0. Those expls that were detond through longer wax-gaps were considered more sensitive d)Impact-Friction Pendulum used by the Spencer Chemical Co consists essentially of a hammer with a 9-ft handle and a 400-lb head. The hammer delivers energy to an expl sample by falling a specified distance before striking the sample. Variations in energy delivered are achieved by adjusting the height from which the hammer is released. The hammer is drawn to this height in an arc, by an electric winch and released from a distance by a lanyard connected to a trigger

AMMONIUM NITRATE DYNAMITE (AND) OR AMMONIA DYNAMITE (AMMONDYNAMIT, IN GER)

A type of "straight" (powdery) dynamite in which up to 50% of liq nitric ester (NG or NG + antifreeze additives) is replaced by AN and the remaining nitric ester acts as a sensitizer for the insensitive AN. These expls, which originated in the USA about 50 years ago, are notable for their heaving rather than shattering effect and their strength is lower than that of "straight" dynamites. AND's have been used for blasting soft rocks, clays and for earth excavations. They are not suitable, however, for use in coal mining except "strip-mining", called "open-cast mining" by the British

The included table gives compns of typical American AND's:

AND's were also used in some European countries and compn and props of some of these AND's are given in the tables shown on top of the next page

Some AND's were used for military purposes, such as demolition, excavation and cratering work

DuPont Co, Wilmington, Del has been manufg several brands of ammonia dynamites, such as "DuPont Extra", "Red Cross Extra", "Red Cross Blasting FR" etc. Some of their props are given in Ref 8, but their compn seems to be a trade secret

According to Stettbacher (Ref 7), the Ger AN expls contg more than 4% of NG were not considered as safe for use in coal mines. The name "Ammonsalpetersprengstoffe" was applied only to AN expls contg not more than 4% NG

AMERICAN AMMONIUM NITRATE DYNAMITES (AMER AND's)

Composition(%) and some properties	Designation and Strength: American –	American –	American "Ordinary" 20%	30%	40%	50%	60%	American Low-Freezing 30%	40%	50%	60%	Monobel (permissible USA)
NG	9.50	9.50	12.0	12.6	16.5	16.7	22.5	13	17	21	27	10
NGc, NSug etc	–	–	–	–	–	–	–	3	4	5	6	–
Amm nitrate	79.45	69.25	11.8	25.1	31.4	43.1	50.3	15	20	25	30	80
Na nitrate	–	10.20	57.3	46.2	37.5	25.1	15.2	53	45	36	27	–
Carbonaceous fuel	9.75(a)	9.65(a)	10.2	8.8	9.2	10.0	8.6	15(b)	13(b)	12(b)	9(b)	10
Sulfur	–	–	6.7	5.4	3.6	3.4	1.6	–	–	–	–	–
Anti-acid	0.40	0.50	1.2	1.1	1.1	0.8	1.1	1	1	1	1	–
Moisture	0.90	0.90	0.8	0.8	0.7	0.9	0.7	–	–	–	–	–
Density, g/cc	–	–	1.30	1.31	1.28	1.26	1.28	–	–	–	–	–
Rate of deton, m/sec	–	–	2700	–	3300	3900	4600	–	–	–	–	–
Ballistic pendulum, % TNT	–	–	89	–	91	99	109	–	–	–	–	–
Lead block expansion, cc per/g sample	–	–	19.9	–	27.5	–	34.7	–	–	–	–	–

(a) The carbonaceous combustible material (such as wood pulp and flour) contained 0.40% of grease or oil which was added to AN to counteract its hygroscopicity

(b) The carbonaceous combustible material of these Amer dynamites was mixed with some sulfur

EUROPEAN AND's

Designation of AND	Composition, % NG	Amm nitrate	Na nitrate	Wood- or cereal-meal	Charcoal
French 1	40	45	5	10	–
French 2	20	75	–	5	–
French 3	22	75	–	–	3
NG powder(Brit)	10	80	–	10	–

EUROPEAN AND's

Designation	Properties Density, g/cc	OB to CO_2, %	Pb block expansion, cc/10 g	Pb block crushing, mm
French 1	1.38	+0.75	400	22.0
French 2	1.20	+9.7	335	15.5
French 3	1.33	+7.8	330	16.0
NG powder(Brit)	1.0	–	Power: 78% of blasting gelatin	

These expls were also called "Sicherheits-sprengstoffe". [See also pp 6 and 177 in PATR 2510 (1958)]

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AMMONIUM NITRATE EXPLOSIONS FIRES AND HAZARDS

Glauber, who first prepd AN in 1659, identified the compd as "Nitrum Flammens", because it evolved luminous gas when heated but did not explode. In 1835 Turner (Ref 1) stated that when AN is exposed to fire it liquifies, emits aq vapor and detonates. However, Berzelius reported that when AN is heated to about 300° (572°F) or dropped into a glowing hot crucible, it burns with a weak hissing noise and a yellow flame (Ref 2). About the same time Gmelin (Ref 3) also observed that if AN is thrown on a red-hot porcelain plate, it burns with a pale yellow light, produces a very slight noise and gives off H_2O, nitrous acid and nitrogen. According to Munroe, AN explodes when thrown on red-hot charcoal (Ref 15); with pure AN, explosion does not occur (Ref 80)

The value of AN as an explosive was recognized in 1867 when Ohlsson and Norrbin patented a mixt of it with organic fuels such as charcoal, sawdust, naphthalene, nitrobenzene etc (Ref 49). These mixts were difficult to detonate until Nobel added NG to them. When unconfined, such mixts, called "extra dynamites" could be burned without detonation if spread in a thin layer or if the end of the cartridge were ignited

In 1883 Berthelot listed AN as an explosive and showed that one g-mol of AN liberated 10.3 kcal of heat when undergoing incomplete decompn and 30.7 kcal when completely confined (Refs 4, 6). According to Berthelot, the probable equation of detonation is: $2NH_4NO_3 = 2N_2 + 4H_2O + O_2$, because this reaction developed the highest temp. Brunswig (Ref 7) stated that the above reaction occurred as the result of sudden heating of AN to a very high temp under great pressure, and actually can be effected only with the aid of a strong detonator. The reaction: $2NH_4NO_3 = 2NO + N_2 + 4H_2O$ occurred as the result of insufficient initial impulse, as with a weak detonator

In 1921 Krase et al (Ref 8) reported that the decompn of AN proceeds at an appreciable rate at 210° and is accompanied by the evolution of heat in accordance with the equation: $NH_4NO_3 = N_2 + 2H_2O + 0.5O_2 +$ 29.5 kcal. The exothermicity of this equation accounts for the explosive properties of AN. Rosendahl in the same year (Ref 9) stated that when AN was heated above its melting point, decompn into H_2O and N_2O was greatest at about 200°. This decompn was local and did not result in deton, but if AN were subjected suddenly to a high temp such as is obtainable only with other expls, it can be exploded. The decompn of AN by heat was studied also by Saunders (Ref 18), who observed that at some temp near 300° the decompn proceeds explosively. Födransperg (Ref 13) stated in 1922 that even when large masses are ignited, AN burns quietly without expln. Although Kast (Ref 31) found that when AN was heated above 300° there resulted an evolution of heat at an accelerated rate followed by expl decompn, the work at Neubabelsberg (Rev 32) showed that AN could not be detonated when subjected to the heat of thermite or wood fires. The findings of Kaiser (Ref 40), who studied the explosiveness of molten AN, showed the following reaction to take place at 200 to 260°: $4NH_4NO_3 = 2NH_3 + 3NO_2 + NO + N_2 + 5H_2O$ and although this reaction is endothermic, Kaiser showed that the gaseous products could explode. In general, the findings of Kaiser were in agreement with independent studies made by Shah and Oza (Ref 36) and Kretzschmar (Ref 38), who also studied the thermal decompn of AN. In 1936 Torsuev (Ref 41) accepted the point of view that any deton of AN which is caused by thermal decompn results from the explosive decompn of the N_2O formed at the beginning of the reaction. Abinder (Ref 42) in the same year found that no expln resulted when samples of 53 and 1110 lbs of AN (loose and packed in bags, barrels or boxes) were burned in an open flame. The temp of combustion was found to be below 800° (See Note, p A363)

Although Parisot (Ref 46) in 1939 summarized the findings of other workers as establishing that AN cannot be detonated by either direct heating or heating in an open vessel, Nuckolls (Ref 48) stated that AN can be exploded at high temp under certain conditions, but that this was not accomplished readily. In 1945 Davis (Ref 51) observed that under favorable conditions of pressure, rapid heating and retention of heat, AN may be exploded when heated to approx 300°. Davis and Hardesty (Ref 52) also emphasized the fire hazard of AN, but referred to the expln hazard only obliquely. Summarizing the foregoing, it must be admitted that the literature, up to this time, was confusing and to some extent contradictory with respect to the fire and expln hazards of AN. Fortunately, more recent studies of the mechanism of expln of AN have been made by Delsemme (Ref 68), and kinetic studies relative to its rates of explosive decompn and hazardous behavior have been made by Henkin & McGill (Ref 71), Hainer (Ref 77) and Burns et al (Ref 74)

Between 1896 and 1948 there occurred about eighteen fires, serious explosions and disasters in which AN was involved (Ref 61). Of these, five were fires only and thirteen involved accidental explosions of various degrees of severity. A list of these incidents, together with a resumé of the conditions, and a complete bibliography of the fire and explosion hazards of AN have been reported by Scott and Grant (Ref 61). Other reports on the fire and explosion hazards of AN have been made by Sherrick (Ref 21), Munroe (Ref 25), Nuckolls (Ref 48), Davis (Ref 51) and Whetstone & Holmes (Ref 53)

A disastrous expln of 1,000,000 lbs of AN occurred during crystn in October, 1918 at the Morgan Plant of the T. A. Gillespie Co in New Jersey. Other explosions at the Oakdale and Repauno plants had occurred in 1916 during the evapn process. These explns were attributed to the presence of organic impurities. However, a fire occurred on board the SS "Halfried" at Brooklyn on Apr 14, 1920 in which about half of over 4,000,000 lbs of AN was consumed without expln (Ref 62). On July 26, 1921 two carloads of 99.3% pure AN exploded at the AG Lignose factory in Germany while being loaded and presumably being broken up by blasting (Ref 14). The most disastrous expln involving AN that has occurred to date was at Oppau, Germany on Sept 21, 1921, when 9,000,000 lbs of a mixt of AN & $(NH_4)_2SO_4$ detonated, killing more than 1,000 people and leaving a crater 250 ft in diam and 50 ft deep (Refs 10, 11 16, 18). The Oppau explosion aroused the world to a new awareness of the explosibility of AN, since $(NH_4)_2SO_4$ is not an explosive. Although the exact cause was never determined, it was suspected that blasting of the caked material into lumps was the cause although thousands of similar blasting operations had been carried out before. The mixt of salts had been assumed to be insensitive to detonation, but many subsequent investigations showed that a mixture of equal parts of AN & $(NH_4)_2SO_4$ can detonate with a velocity as high as 1400 m/s (Refs 26, 44, 73), and that sensitivity of the nitrate to detonation increases with an increase in temp (Refs 27, 29). It has been shown also that the effect of an increase in density of AN varied with the degree of confinement (Refs 21, 22)

Kast (Ref 30) studied the sensitiveness to impact and heat of AN, AN/salt mixts and AN/combustibles mixts. He reported in 1926 that the presence of up to 30% of $(NH_4)_2SO_4$ has practically no effect on the explosibility of AN, and that sensitiveness to impact as well as to heat increases with increasing amounts of $KMnO_4$ in AN mixts. Naoúm and Aufschläger (Ref 26) had found in 1924 that mixts of AN with $(NH_4)_2SO_4$ were explosive, but with increasing amts of sulfate deton became more difficult. An intimate mixt of equal parts of dry, powd nitrate and sulfate were detonated by an extremely strong impulse only. Torsuev (Ref 41) in

agreement with Kast, observed that addition of $(NH_4)_2SO_4$ rendered AN less sensitive and dangerous. Blinov (Rev 44) criticized the results of Torsuev and showed that it is possible to ignite nitrate-sulfate mixts with a Bickford fuse; and he detonated them, as had Naoúm and Aufschläger previously, when the ingredients were pulverized and thoroughly mixed. The effects of the following other inert and inorganic additives on AN have been reported: KCl (Refs 19, 30), acids (Refs 39, 76), H_2O (Ref 68), H_2O & Fe (Ref 42), Fe, Cu, Al, brass, Zn, & stainless steel (Ref 63), $KMnO_4$ or $K_2Cr_2O_7$ (Ref 45), inorg coating (Ref 70) and chalk (Ref 72). Stengel and Broadhacker (Ref 75) proposed preventing phys disintegration of AN, produced from NH_3 and HNO_3, by adding to the HNO_3 used sufficient H_2SO_4 to yield a product contg 0.25–5.0% $(NH_4SO_4$. The sulfate may also be mixed directly with the molten AN after its formation. Comparative tests made by alternately heating and cooling 10 g samples between 22 and 55° showed that 80 to 100% of pure AN disintegrated into particles having less than ¾ the size of those originally present; but samples containing 2% $(NH_4)_2SO_4$ showed no disintegration

Later incidents of fire or expln involving AN were the burning of two carloads of this material in transportation from Muscle Shoals, Alabama (Ref 28). Bashford (Ref 23) described an explosion at Nixon, NJ and attributed it to traces of TNT found in the AN. Shreve (Ref 35) described an explosion that took place during the graining of AN at a plant of the Ammonite Co, but no explanation of the cause was given. Fire and expln at Roseburg, Oregon is described in Ref 79

At Brest, France, a shipload of 6,600,000 lbs of AN exploded on July 28, 1947, killing 21 persons, injuring 100 others and doing major damage for a distance of three miles (Refs 61 & 78). Another cargo of AN fertilizer (FGAN) (2280 tons) on the SS Grandcamp at Texas City, Texas, detonated on Apr 16, 1947 and set off a series of fires and explosions that led to the deton (16 hours later) of 960 tons of FGAN on the SS High Flyer berthed 600 ft from the Grandcamp (Refs 59, 64, 66). These explosions took the lives of over 600 persons, injured 300 others and caused property damage of over $58,000,000. US Bureau of Mines personnel reached the scene some 12 hours after the initial expln, investigated the disaster and prepared a detailed description of the event. Suggestions were given for prevention of such accidents in the future (Ref 64). Investigations of different phases of this disaster were made at Picatinny Arsenal, the US Bureau of Standards, the Bureau of Explosives of the Amer Assoc Railroads and the National Board of Fire Underwriters. Detailed accounts of these investigations are given in a number of reports (Refs 54, 55, 56, 59, 62, 66). It has been accepted generally that the fire on board the SS Grandcamp had its origin in the careless handling of lighted cigarettes on the part of stevedores engaged in loading the cargo, and that other possible origins were highly improbable. The paper bags containing the FGAN were not marked to indicate the hazardous nature of the material, the stevedores handling it lacked knowledge of the danger in the presence of fire and the captain of the ship did not enforce the regulations against smoking in the hold of the ship. In May, 1947 the Secy of the Treasury appointed an interagency committee to develop additional information relative to the hazards involved in the transportation, handling and storage of AN and recommend a national safety policy in this connection. In 1947, the interagency committee issued two of three parts of its report (Refs 57, 58)

Irrespective of the origin of the fire on the Grandcamp, the following surmises were offered as possible explanations of the transformation of combustion of the nitrate and paper into detonation (Ref 62): a) abnormal sensitivity of FGAN b) impact of falling steel members on molten FGAN c) addition of steam to the gaseous combustion products

in the hold d) entry of fuel oil from adjacent tanks and its admixture with FGAN to form a sensitive explosive e) initiation by the fire of small-arms ammunition adjacent to the hold in which fire occurred f) formation of gaseous products of combustion or thermal decompn which were explosive and detonated upon ignition after reaching a lower limit of concentration and g) transformation of combustion into detonation as a result of increasing gas pressure due to conditions of partial confinement. Each of these possible causes was evaluated with respect to its probability as the cause of an explosion

PicArsn and BurMines data on sensitivity and some other expl properties of FGAN and pure AN (Refs 59 & 62) were as follows:

	FGAN	AN	TNT
PA Impact Test, 2 kg wt, inches			
At 25°	30	31	14
At 90° (molten TNT)	–	–	3
At 175°(molten)	12	12–13	–
Pendulum Friction Test, steel shoe			
Unaffected in 10 trials	10	10	10
Explosion Temp Test			
3 sec	333–85	325	–
5 sec	–	–	475d
Rate of Deton, m/sec			
At 25°	1350	1190	6800
Molten	2109	–	–

The above data show that FGAN is not abnormally sensitive to initiation by impact, friction or heat, but the molten FGAN is much more sensitive than the cryst material. However, a 52-lb wt allowed to fall 10 ft on a mixt of molten FGAN and bagging paper confined in a steel tube caused no expln. The impact on,and immersion in similar mixts of burning wood or charcoal also failed to cause deton of FGAN. The probability of expln by impact on molten FGAN appears to be unlikely

The injection of steam into the hold, as was done aboard the SS Grandcamp, was not a logical cause of the expln. This was not done aboard the High Flyer. Steam is one of the major products of decompn or expln of AN and is not reactive with the nitrate. Steam should retard rather than accelerate the decompn of molten FGAN, and its wetting effect on paper bagging should tend to retard the burning of this. The possible effect of fuel oil, which might have soaked into FGAN and formed a sensitive explosive mixture, was also considered. Tests at PicArsn (Ref 59) showed no reactivity between fuel oil and FGAN even at 120°. Numerous investigators have studied the effects of organic combustibles on AN, for example: TNT (Refs 12, 24, 43, 50), petrolatum or paraffin (Refs 24, 42, 44, 47, 67, 69), petrolatum-rosin-paraffin (PRP) (Refs 51, 52, 72), combustibles such as wood pulp, etc (Refs 17, 20, 30, 34, 42, 63, 70, 74) and other organics (Refs 37, 41, 60). In general, it has been found that such mixts are only slightly more sensitive than AN itself. Elliott (Ref 63) made an investigation of an expln that occurred during evapn by air agitation of an AN solution in a high-pan with the possibility that lubricating oil in the air compressor might have passed

through the air filter. His tests showed that no rapid exothermic reaction took place in mixts of compressor oil and AN at temps below 250°. Tests also showed that highly confined AN at 146° could be detonated by means of a No 8 detonator, but not when the AN was at 21°. The possibility that fuel oil may have been a factor in causing the Texas City explosion was dismissed as negligible

Since there were 16 cases of small-arms ammunition in a hold of the Grandcamp adjacent to that in which the fire started, their initiation and sympathetic deton of the burning cargo have been suggested as being responsible for the expln. As small-arms ammunition contains no HE charge and the propellant charges are small and separated by cartridges, fire involving the ammunition should result only in the combustion and low-order expln of the charges, with high-order deton of these only a remote possibility. Also, there was a heavy steel bulkhead between the two holds, and the transmission of such an explosive wave through this would be improbable. However, Abinder (Ref 42) found in 1936 that a shell loaded with AN and detonated,did transmit the deton by influence to other shells. The fact that a similar explosion following a fire took place on board the High Flyer, which had no small-arms ammunition cargo aboard, disposes of this possible explanation of the explosion on the Grandcamp

Sherrick (Ref 21) suggested in 1924 that the expl decompn of nitrous oxide, a product of the decompn of AN, or its mixt with carbon monoxide, might be capable of causing the deton of AN. Mixts of equal vols of nitrous oxide and carbon monoxide were detonated by means of an electric spark by Berthelot and Vieille (Refs 5&33), who found the rate of deton of the mixt to be 1106 m/s. As would be expected, some carbon monoxide, as well as carbon dioxide, was produced by the burning of a mixt of FGAN and bagging paper in tests made at Pic Arsn (Ref 59). In laboratory tests, the gaseous phase produced by heating the mixt at 175° underwent expln, but whether the residual solid material was detonated could not be determined. It can be postulated that as the burning of paper and wood dunnage progressed, the concn of nitrous oxide and carbon monoxide could increase until the limit of inflammability was reached, when deton of this would occur and cause deton of unburned FGAN

The final surmise as to the cause of transformation of fire into expln aboard the Grandcamp was an increase in gas pressure due to a condition of confinement. In an effort to simulate the conditions existing in the hold of the Grandcamp, small and intermediate scale tests in bomb cases were made at Pic Arsn and Aberdeen Proving Ground. These indicated that a certain minimum gas pressure must be developed before the deton of a burning FGAN-paper mixt can take place (Ref 65). Charges of 2,000 and 3,000 lbs of bagged FGAN in 12,000 lb T10 Bomb cases underwent only burning when some degree of venting was provided. However, when confinement was complete, deton of the entire charge occurred after 64 min of heating of an attached auxiliary tube contg approx 8 lb FGAN and bagging paper and also after only 45 sec of burning of a mixt of FGAN, excelsior and kindling in the center of the bomb charge. It was calculated that the gas pressure at the instant of expln was not greater than 235 psi in the case of the charge that detonated in 45 sec, approx 75 psi or less for the auxiliary bomb that detonated in 64 min and approx 22 psi for the vented bomb in which the FGAN-paper charge burned for 120 min without expln. Branconier and Delsemme (Ref 72) found that it is necessary to have sufficient confinement to build up a pressure of 1400 psi in order to cause the expln of AN solely by heating. If the AN is not stabilized or has a coating of the PRP type, the minimum pressure required for expln is only 42 psi

In the hold of the Grandcamp, the condition of confinement was one of venting, as the hatch was open at the instant of explosion. Whether

a gas pressure of 42 psi could be developed under this condition or whether the hull of the ship could have withstood this much pressure is not known. There is the possibility that collapse of part of the burning mass could have produced a localized pressure of 42 psi or more and caused transformation of burning in this region into detonation which was then transmitted to the rest of the FGAN in the hold

In conclusion, it might be said that most of the investigators of AN prior to the Texas City disaster recognized, especially after the Oppau disaster, that this compound may be exploded by impact or a powerful detonator, but very few recognized the possibility of exploding it by heat alone. With the knowlege of the explosibility of AN acquired after the disaster, it is now known that AN and mixts of it with organic materials and even ammonium sulfate can be caused to detonate by heating under conditions of confinement. With this knowledge, the problems of handling, storage and transportation of these materials can be solved by controlling conditions so as to insure safety and, in spite of potential hazard, prevent accidental explosions similar to those at Texas City and Brest

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Note: Dr R.D.Miller of Spencer Chemical Co, Kansas City, Missouri, pointed out (private communication, March 1960) that the Inter-Agency Rept which was issued after Texas City disaster stated that two reactions, one endothermic and the other exothermic, occur at the same time:

"AN melts at about 170° (338°F). If the melt is heated to about 210° (410°F) in an open container it will begin to decomp into N_2O (the "laughing gas" of commerce) and H_2O. This reaction produces heat, and it would, therefore, ordinarily be expected that a mass of decompg AN would become progressively hotter and decomp at an accelerating rate until finally the decompn would become expl. However, a process which absorbs heat is going on at the same time as the heat-producing decompn which gives N_2O and H_2O. This process includes the simultaneous vapzn and dissocn of AN into gaseous NH_3 and HNO_3."

AMMONIUM NITRATE, FERTILIZER GRADE (FGAN)

The history of restoring fertility to the soil shows that in 500 BC the Celts of Western Europe had learned the virtues of lime, wood ashes and compost. However, agricultural science as such is only a hundred years old. The foundations of soil chemistry were laid down in 1840 by Justus Liebig, a German chemist, in his classic text "Organic Chemistry in its Applications to Agricultural Chemistry". Complete, mixed fertilizers embodying various proportions of nitrogen, phosphate and potash were virtually unknown in England and Europe until 1925–30, but mixed fertilizers dominated the American market although most of the individual ingredients were imported prior to World War II. Both nitrogen and phosphorus have double roles: they are plant nutrients and materials useful in the production of war munitions (Ref 24)

Since 1930 increasingly larger quantities of nitrogen in the form of anhydrous ammonia and ammoniating liquors contg mixts of AN-ammonia-water or urea-ammonia-water have been used. Most of the AN used as fertilizer in the USA is applied directly to the soil. AN shows a marked tendency to cake and absorb moisture from the air (Ref 27) when the humidity exceeds 60% at 30°. A study by Gorshtein (Ref 4) of the hardening and caking of AN in storage showed that the granulated material with a max of 0.15% H_2O does not cake under any storage conditions; with a 0.4 to 0.5% H_2O content, caking is excessive at temps above 31.5°, while a higher H_2O content (0.5 to 1.5%) has no influence on the degree of caking. The rate of hardening is reduced sharply by an increase in particle size and is retarded by coating the nitrate with 0.1% paraffin. Gorshtein also describes a series of expts on producing granulated AN and mixed fertilizers (such as AN + $(NH_4)_2SO_4$ + CaO + phosphorite) employing Kestner evaporators and crystallizers (Ref 4)

During the past twenty years many developments of AN coatings and additives for improved moisture resistance have been disclosed. For instance, Baker (Ref 5) reported a Ca stearate coating, Cairns (Ref 10) the PRP (paraffin, rosin, petrolatum) coating, and Winning (Refs 11 & 12) a pregelatinized starch products coating. It was not until 1943 that the TVA (Tennessee Valley Authority) developed American FGAN, consisting of AN coated with 1% PRP and 4% kaolin dust (Ref 15). The method of prepn of Amer FGAN is described briefly under "Ammonium Nitrate". In 1945 the US Dept of Agric (Refs 13 & 14) published the results of investigations which showed the fire hazards of FGAN, but no definite statement was made with respect to its explosibility by heat. Previous to these publications, dynamite manufacturers for many years practiced the coating of AN with up to 2% of petrolatum or PRP. Cook (Ref 2) showed in 1924 that mixts of AN with 1% petrolatum were more sensitive to initiation than uncoated AN. US Bur Mines investigations indicated that 1% nitrostarch increased the sensitivity of AN to expln at elevated temp and under confinement (Ref 3). Torsuev (Ref 6) stated that the presence of org matter in AN facilitated its thermal decompn with the formation of an expl mixt of gases. His claim that even 0.1% of paraftin is dangerous was denied by Abinder (Ref 7) and also by Blinov (Ref 8), but both agreed that larger amounts of paraffin than 0.1% would considerably increase the sensitivity of AN

The explosibility of AN with approx 7% rosin was investigated by Hopper (Ref 9), who found that rosin increased the sensitivity to initiation and rendered the mixt less hygroscopic. The mixt was also satisfactory with respect to stability, brisance and impact sensitivity, being unaffected in the rifle bullet and pendulum friction tests. Marshall (Ref 23) and Banik (Ref 26) studied the explosibility of Amer FGAN contg 1% petrolatum and 5% kaolin in comparison with Brit FGAN contg

99.8% AN and about 0.15% H_2O. Marshall reported laboratory and field tests which showed that the Amer FGAN ignited more readily and decompd more quickly than Brit FGAN when these were subjected to heat; when confined, the Amer product exploded violently, whereas the Brit product either did not explode at all or did so only mildly. When heated in steel tubes 2 ft long which had an ID of 0.45" and a wall thickness of 0.08", the Brit FGAN burst the tube only at the end where it was heated, whereas the Amer FGAN burst the tube from end to end. This test showed that the explosion propagated throughout the unheated mass. Admixture of either type of FGAN with paper and wood (packing materials) in the form of small pieces did not cause the explosion of the unconfined mass. An intimate mixture of Brit FGAN and 1% hydrocarbon, when heated strongly under confinement, exploded in a manner similar to that of Amer FGAN (Ref 23). In general, the discussion of Bank (Ref 26) is in agreement with the findings of Marshall. Cook and Talbot (Ref 28) reported that AN grains coated with hydrocarbons were more sensitive to initiation than uncoated AN or AN coated with inorganic materials. Max sensitivity occurred in fine-grained AN coated with 0.75 to 1.5% wax. A No 6 blasting cap was sufficient to deton this material, and the expln propagated indefinitely in charges 1.875" in diam (See Note, p A340)

Early investigation of the sensitivity of mixed fertilizers was made by Rosendahl (Ref 1), who reported that mixts of Amm nitrate & sulfate decompd on heating according to the equation: $2NH_4NO_3 + (NH_4)_2SO_4 = 3N_2 + 8H_2O + SO_2$. Hardesty and Davis (Ref 16) observed the spontaneous development of heat in mixed fertilizers. Macy et al (Ref 17) at PicArsn, tested FGAN, consisting of AN nodules coated with 0.75% wax and 3.5% clay (identical with the material loaded on the SS Grandcamp and SS High Flyer), with the following results: a)FGAN at d 0.7 to 1.0 can be detonated by sufficiently strong boosters (50–100 g of tetryl) in columns 8–10 cm in diam when confinement is weak (paper) and in 3–4 cm diam columns when confinement is strong (steel). Rates of deton are between 1000 and 1350 m/s in the solid state and 2100 to 2500 m/s in the liquid state. Rate and sensitivity depend upon the particle size, density, confinement, charge diam and temp b)Due to the action of heat alone, gas phase explns have been noted at 260° and above c)Certain inorg and org contaminants in small amts can cause FGAN to decomp explosively at a temp as low as 200°, or it may be ignited at lower temps. For example, moist Zn dust and FGAN explode while in the presence of phosphoric acid at 30°, mixts of FGAN and combustible may ignite spontaneously or explode at elevated temps. FGAN is apparently reactive with substances like resins, starch, sugar, dry sawdust or bagging paper, with consequent charring or gassing. It is less reactive with iron, only slightly so with asphalt, and nonreactive with lubricating oil of SAE 30 at 120°C(248°F). The presence of 10% sawdust or bagging paper in FGAN reduces the ignition temp by 70 to 200° for exposure times from 0.5 to 6 min, and such mixts can ignite at 150°C (302°F) within 30 min. Mixts of FGAN with sawdust or bagging paper which react at 150° do so exothermically, but at temps of 135 to 150° the reactions are very slow and not exothermic d)Tests to det the susceptibility of FGAN to deton by impact or initiation show it to be, in the liquid state, as sensitive to impact as cryst TNT tested under the same conditions e)At a temp of about 50°(120–125°F), FGAN is far easier to explode than below this temp f)Liquid FGAN can be exploded by a booster charge in spite of its relatively high density (1.4 g/cc) g)The fragmentation effect of solid FGAN is about 24% that of TNT at the same density h)An electric spark passed through the gas over a 1.6 g sample of FGAN heated for 5 min at 175° in a partially closed glass tube, resulted in a flash

Eriksen (Ref 20) found that 2,000 and 3,000 lb charges of bagged FGAN can be detonated by the application of heat alone if confinement is such that the products of decompn can develop a gas pressure of about 100 psi. When some degree of ventilation was provided, FGAN could not be detonated by heat and underwent burning only. Eriksen also showed (Ref 21) that six-ply asphalt laminated paper bags stored with FGAN at 60°(140°F) were adversely affected after a period of eight weeks; similar bags without FGAN were not appreciably affected by the same storage conditions, and FGAN stored in such bags at a temp as high as 88° (190°F) remained unchanged. Rinkenbach (Ref 18) summarized these studies and others in a lecture delivered at PicArsn, Dover, NJ. The relative sensitivity of pure AN, wax-coated AN and FGAN to initiation by heat was studied by Varrato(Ref 25). These materials had the following characteristics:

Composition, %	Pure AN	Wax-coated AN	FGAN-A	FGAN-B
AN	99.83	94.45	96.03	96.22
Wax (PRP)	0.03	0.38	0.40	0.68
Clay	0.14	0.17	3.57	3.10
Moisture	0.04	0.02	0.04	0.12
Nitrogen content, %	34.62	34.81	32.98	33.40
Granulation				
Through No 8 US Std sieve	100	100	100	100
On No 325 US Std sieve	57.5	59.5	55.0	58.0
Apparent density, g/cc	0.97	1.06	1.01	1.01

Varrato's tests showed that AN coated with 0.38% wax was not more sensitive to initiation by heat than pure, uncoated AN. The sample of AN coated with 0.38% wax was more sensitive than FGAN-A. The most sensitive of the above mixts was that contg about 0.7% wax (FGAN-B)

Ottoson (Ref 19) investigated a mixt called "Cal-Nitro", contg 60% AN and 40% $CaCO_3$ supplied by the Semmett Solvay Co of Hopewell, Va. He found that Cal-Nitro was less reactive with paper and sawdust than ordinary FGAN. It did not yield significant amts of expl gases when decompd at realtively low temps, and bagged Cal-Nitro represented a slightly lesser fire hazard than bagged FGAN "Uraform" (95% AN and 5% urea-formaldehyde resin), also investigated by Ottoson (Ref 19), was found to be more explosive than FGAN, since its expl temp of 280° is considerably lower than that of FGAN (333–385°). The impact sensitivity values for "Uraform," obtained with PicArsn test apparatus, using a 2 kg wt, were 23" at 20° and 8" for molten material at 175°. These values are to be compared with 31" and 12" respectively for FGAN under the same test conditions. A detonation velocity of 1760–1910 m/s at d 0.80 was obtained for Uraform as against 1350 m/s at d 0.90 for FGAN

A more recent study by Burns et al (Ref 29) of the explosibility of AN may be summarized as follows:

	Explosion took place at: Temperature, °C	Pressure, psi
Pure AN, mp 169°	277 to 344	2600 to 3000
Pure AN with 1.5% bagging paper	134 to 153	250 to 300
FGAN	304 to 350	–
FGAN 1.5% bagging paper	140 to 153	–

According to Burns, the incorporation of about 25% of an inert material such as limestone materially decreased the expln hazard of AN

In conclusion it may be said that currently produced American FGAN, as well as some other ammonium nitrate fertilizers, can explode when confined and subjected to heat, impact or initiation by powerful detonators or boosters

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AMMONIUM NITRATE GELATIN (ANG) OR AMMONIA GELATIN DYNAMITE

ANG is a blasting type expl similar in compn to Ammonium Nitrate Dynamite, except that gelatinzed NG is used in lieu of straight NG. For the prepn of "gel", the NG is thoroughly mixed with 2 to 5.4% of collodion cotton (CC) and the mass is left for several hours at 40–45°. The resulting soft gel is mechanically incorporated with previously pulverized and thoroughly mixed AN, carbonaceous material, anti-acid, etc, to give a plastic mass. The above mixt of solid ingredients is called in the USA "dope" and it serves as an absorbent for NG. The presence of gel makes these explosives gelatinous or semi-gelatinous and more water-resistant than corresponding ammonia dynamites. As these expls produce comparatively small amounts of abnoxious gases, it is permissible to use them for underground quarrying, mining (except gaseous and dusty coal mines) and tunneling operations. Their velocity of deton is 4750 to 5750 m/sec, when confined

The table shown on next page gives compn and some props of ANG's

Refs: 1)Marshall 1(1917),372 2)Naoúm, NG(1928),328 & 349 3(Marshall 3(1932),108 4)Davis(1943),346&351 5)Bebie(1943),23 6)Stettbacher(1948),86 7)TM9–1910(1955), 206 8)Taylor & Gay(1958),26 9)PATR 2510(1958),p 5(Ammongelatin)

AMMONIUM NITRATE GELATINS(ANG's)

Composition(%) and some properties	American (Non-Permissible)					Belgian (Non-Permissible)		British (Not-Permitted)			French (Non-Permissible)		
	30%	40%	50%	60%	–	Forcites	Forcite Extra	Ammon-Gelatine Dynamite	Ammon-Gelignite	Semi-Gelatine	Dynamite O	Forcite Extra	Grisou-dynamite-roche
NG	22.9	26.2	29.9	35.3	45.0	36–64	63.0	33.0	25.7	15.0	50.0	64.0	29.0
Collodion cotton	0.3	0.4	0.4	0.7	2.5	2–3.5	2.0	1.3	0.9	0.3	2.5	3.5	1.0
Amm nitrate	4.2	8.0	13.0	20.1	45.0	0–25	30.0	59.2	57.8	78.7	44.5	25.0	70.0
Na nitrate	54.9	49.6	32.0	33.5	–	0–35	–	–	–	–	–	–	–
K nitrate	–	–	–	–	–	0–46	–	–	–	–	–	–	–
Carbonaceous fuel	8.3	8.0	8.0	7.9	7.5	6.5–15	5.0	6.5	5.8	6.0	3.0*	6.5**	–
Sulfur	7.2	5.6	3.4	–	–	–	–	–	–	–	–	–	–
Inert ingredients	–	–	–	–	–	–	–	–	9.8	–	–	–	–
Anti-acid	0.7	0.8	0.7	0.8	–	1.0	–	–	–	–	–	1.0	–
Moisture	1.5	1.4	1.6	1.7	–	–	–	–	–	–	–	–	–
Density, g/cc	–	–	–	–	–	–	1.44	1.5	1.5	1.2	–	–	–
Rate of deton, m/sec	4400	4900	5300	5700	–	–	7000	–	–	–	–	–	–
Ballistic pendulum, percent TNT	83	88	92	97.5	–	–	–	–	–	–	–	–	–
Pb block expansion, cc per 10g sample	–	–	–	–	–	–	485	–	–	–	–	–	–
Pb block crushing, mm	–	–	–	–	–	–	21.0	–	–	–	–	–	–
Power, % of blasting gelatin	–	–	–	–	–	–	–	90	78	82	–	–	–

*Carbonaceous fuel consisted of 2.5% cereal meal and 0.5% lamp-black

**Wood meal

AMMONIUM NITRATE, ANALYTICAL PROCEDURES

I. Qualitative Tests for AN. This can be done either by detecting the NH_4^+ ion or the NO_3^- ion

A)Detection of the NH_4^+ Ion. Add to 2-3 ml of liquid to be tested, a few drops of Nessler's reagent. If ammonia is present (free or bound) in small quantities, the liq turns dark yel to reddish-brn (Ref 3 & Ref 6, p 145). If a large amt of NH_4^+ ion is present, a brn ppt of $NHg_2I.H_2O$ is formed (Ref 3)

B)Detection of NO_3^- Ion. If it is desired to detect AN in the absence of nitric acid and inorg or org nitrates, use the diphenylamine method. For this pour about 5 ml of liquid to be tested into a narrow test tube of ca 25 ml capacity and while holding the tube in an inclined position (ca 45° angle), add slowly ca 2 ml of DPhA reagent (prepd by dissolving 1 g DPhA in 100 ml concd H_2SO_4) in such a manner that the reagent flows down the wall of the tube without mixing with the sample. If NO_3^- ion is present, a blue ring appears at the interface (Ref 3 & Ref 6, p 140)

Note: Instead of using DPhA, the ferrous sulfate reagent may be used. The reagent can be prepd by dissolving the max amt of powdered $FeSO_4.7H_2O$ in cold ca 60% sulfuric acid (previously prepd by slowly adding with stirring 1 vol of concd H_2SO_4 to 1 vol of w, cooled in ice). This test is conducted in the same manner as the DPhA test and the presence of a brown ring indicates the presence of a nitrate (Ref 6, p 140)

II. Quantitative Tests for AN. The following methods may be used:

A)Colorimetric Methods are applicable for detn of small quantities of AN such as 0.1-10 mg. This can be done by using one of the following reagents: Nessler's (Refs 3 & 6), m-xylenol (Refs 5 & 12) or sulfanilic acid & α-naphthylamine (Ref 6, p 140)

B)Other Quantitative Methods. For larger quantities of AN, the gravimetric, volumetric, gasometric and other methods can be used, such as:

a)Nessler's Reagent Method consisting of weighing $NHg_2I.H_2O$ precipitated on treating a soln of AN with the reagent (Ref 3)

b)Ammonium Chloroplatinate Method consisting of weighing $(NH_4)_2PtCl_6$ (Ref 3)

c)Direct Titration of Nitrates with Titanous Chloride Using Alizarin as an Adsorption Indicator, described by Wellings (Ref 1)

d)Volumetric Determination of Nitrates with Ferrous Sulfate as Reducing Agent, described by Kolthoff et al (Ref 2) (See also Ref 3, p 644)

e)Schulze-Tiemann Method for Determination of Nitrates, described in Ref 9, p 218

f)Oxidimetric Nitrate Analysis of Fertilizers and Other Commercial Products, described by Leithe (Ref 10)

g)Formaldehyde Method, proposed by Miller for rapid detn of nitrogen in AN fertilizers (Ref 11) (See also Ref 8, p 6)

h)Devarda Method, in Allen's modification, described in Ref 3, pp 640-3

i)Kjeldahl Method, described in Ref 6, pp 142-5

j)Nitrometer Method, described in Ref 8, pp 6-7

k)Refractometric Method, described by Miaud & Dubois (Ref 14)

l)Phase Analysis in Non-Aqueous Medium, described by Sartorius & Kreyenbuhl (Ref 18)

Gordon & Campbell (Ref 17) described the differential method of analysis in the investigation of thermal decompn of inorganic oxidants, such as AN

Jacobs (Ref 12) stated that AN is usually estimated in dusts by detg the NH_3 content of a sample either by nesslerization or by the m-xylenol reagent. When the dust also contains nitrocompds (such as nitrated derivs of toluene), it is necessary to make a distn in the presence of an alkali (as in the Kjeldahl method) and then test the distillate by one of the above reagents

Detn of moisture in AN is described by the following investigators: a)Guichard (Ref 4)–detn of small amts of w by continuous weighing on a specially designed analytical balance b)Roberts & Levin (Ref 13), –detn of small amts of w employing azeotropic distn and subsequent detn of w in the distillate by titration with Karl Fischer reagent c)Eberius (Ref 15)–detn of w by the Karl Fischer method in explosives chemistry d)Jensen et al (Ref 16)–detn of moist using a modified high frequency apparatus e)Engelbrecht et al (Ref 19)–a rapid method for detn of moist in AN using the Karl Fischer reagent f) Zil'berman (Ref 7)–cal'cn of moist content in AN melt by detg bp's at pressures of 500 to 560 mm Hg and interpolating the results from concn-temp curves prepd for pressures 520, 540 & 560 mm Hg

Kast-Metz(1946) described detn of AN in some expl mixts, such as blasting expls of black powder type, permissible expls, etc (See also under Amatol and Ammonal)

AN intended for military uses must comply with the following requirements of Spec JAN-A-175 (Ref 8, p 2)

	Grade I	Grade III
Moisture (max), %	0.15	0.15
Ether solubles (max)	0.10	0.10
Water insolubles (max)	0.18	0.18
Insolubles retained on US Std No 40 (420 micron) sieve (max)	0.01	0.01
Acidity, as HNO_3 (max)	0.02	0.02
Alkalinity, as NH_3 (max)	0.025	0.025
Nitrites	None	None
Sulfates as $(NH_4)_2SO_4$ (max)	0.05	0.02
Chlorides as NaCl (max)	0.50	0.02
Soly in nitric acid (min)	–	100.0
NH_4NO_3 content (min)	99.0	99.0

Note: Apparent density for Grade I, Class B AN shall be not less than 1.06

Granulation, %	Grade I Class A	Grade I Class B	Grade I Class C	Grade II
Through a 2.5" opening (min)	–	–	–	99.9
Through No 10 (2000 micron) sieve (min)	–	99	99	–
Through No 12 (1680 micron) sieve (min)	99.9	–	–	–
Retained on No 35 (500 micron) sieve	–	32-48	50-80	–
Through No 35 and retained on No 80 (177 micron) sieve	–	–	7-29	–
Through No 80 sieve	–	–	13-21	–
Through No 100 (149 micron) sieve	15-30			

Note: Grade II AN covered by US Army Spec 50-11-59E, July 1944 is a material of 97% purity

Grade I AN, Classes A & B are intended for use in the manuf of cast-loaded amatols, while Class C is for extrusion-loaded amatol (80/20). Grade III AN is intended for use as a nitrating agent in the manuf of expls

Following is a brief description of tests:
A)Moisture may be determined by one of the following methods:
a)Weigh accurately ca a 10 g sample in a tared wide-form weighing dish, heat at 100° for 2 hrs, cool in a desiccator and reweigh. Calc the loss of wt as %-age of moisture
b)Transfer a weighed 100 g portion of the sample to the 500 ml flask of the apparatus shown on Fig 1 and add 200 ml of perchlorethylene, which has been stored over anhydrous $CaCl_2$ in a moisture tube the interior of which has been cleaned with chromic acid. Connect a moisture tube (see Fig 2) and reflux condenser by means of tight cork stoppers, and support on a ring stand with the flask resting on a wire gauze. Heat to a brisk boiling and maintain so for 15 mins. Cool and disconnect the apparatus.

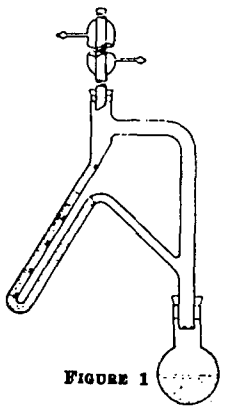


FIGURE 1

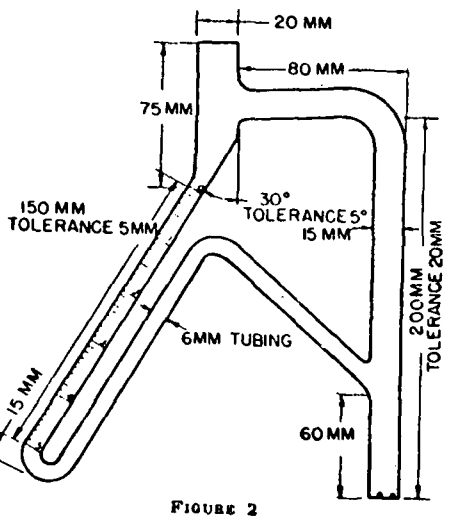


FIGURE 2

As the amt of w collected in the moisture tube usually will be too small to form two distinct menisci, add 1.00 ml of distd w from an automatic pipette, allowing it to run down the side of the tube and unite with the w collected from the sample. Read the vol of w in the moist tube, correct for the amt added, and calc the %-age of moist (Ref 8, pp 3-4)

B)Ether Soluble Material. Transfer a weighed portion of ca 25 g of the sample to an extractor (Soxhlet or other) and extract with anh ether. Evaporate the eth from the extract, dry at 100° to const wt, cool and weigh. Calc the wt of residue as %-age of ether-solubles. Save the extracted sample (Ref 8, p 4)

C)Water Insoluble Material. Transfer the extracted material of previous proced to a beaker and stir with hot w to dissolve the bulk of material. Filter through a tared sintered glass crucible and wash the residue in the crucible with hot distd w until it is free from nitrate. Dry to const wt at 100°, cool and weigh. Calc the residue as %-age of water-insolubles (Ref 8, p 4)

D)Insoluble Material Retained on US Std No 40 Sieve. Treat a 100 g sample in a beaker with hot dist'd w and pour the soln through the sieve. Transfer quantitatively any insol matter from the beaker to the sieve by means of a jet of hot distd w and when no more insol matter passes through the sieve, dry the sieve with residue at 100° for 1 hr. Transfer the dry residue to a piece of glazed paper and then to a tared weighing dish. Weigh the dish and calc the increase in wt as the %-age of insol matter retained on the sieve (Ref 8, p 4)

E)Acidity. Dissolve a 100 g sample in 400 ml of distd w, filter and titrate the filtrate with N/10 NaOH soln using methyl red indicator. Run a blank detn on the same amt of distd w and methyl red. Calc acidity as %-age of nitric acid (Ref 8, p 4)

F)Alklainity. If the previous detn showed acidity, report no alkalinity. If the soln prepd for acidity detn is alkaline toward methyl red, titrate with N/10 sulfuric acid and calc the alkalinity as %-age of ammonia (Ref 8, p 4)

G)Nitrites. Weigh to 0.1 mg ca a 1 g sample, dissolve it in 20 ml distd w, add 1 ml of 10% aq sulfuric acid and 1 ml of freshly prepd, colorless 0.5% m-phenylenediamine-hydrochloride soln. No yel or yel-brn color should develop.

Note: If the m-phenylenediaminehydrochloride soln is colored when prepd, decolorize it by treating with animal charcoal (Ref 8, p 4)

H)Sulfates. Weigh to 0.1 mg ca a 10 g sample and dissolve in ca 20 ml distd w in a porcelain dish. Add a little more Na carbonate

(free from sulfates) than necessary to transform AN to carbonate and evaporate to dryness. Cool the dish and acidify the residue with HCl, taking care to avoid loss during effervescence. Evap to dryness, moisten the residue with HCl and again evap to dryness to insure complete removal of nitrates. Dissolve the residue in distd w, filter if necessary, heat to boiling, and add 5 ml of a 10% Ba chloride soln. Allow the ppt to settle and transfer the liq and ppt quantitatively to a tared sintered glass crucible of medium porosity. Wash the ppt with hot dist w, dry the crucible at 100° for 2 hrs, cool in a desiccator and weigh. Calc the wt of Ba sulfate to %-age of Amm sulfate in the sample

Note: If the amt of sulfates in Grade III AN is not above 0.02%, the following turbidometric proced may be used

Transfer ca a 5.0 g sample, weighed to 0.1 mg to a tall-form beaker of ca 180 ml capacity, add 20 ml concd HCl and boil the mixt gently. From time to time replace the HCl lost by evapn, and continue boiling until a KI-starch paper moistened with distd w is no longer colored by the vapor. Evap the soln to dryness, dissolve the residue in a little distd w and transfer the soln to a 50 ml Nessler tube. Add 5.0 ml of 10% Na citrate soln, mix the contents and add 2.0 ml of 10% $BaCl_2$ soln. Dilute the mixt to the mark with distd w. Add 10.0 ml of 2% (1:20) HCl soln, mix and allow to stand 10 mins. Simultaneously and in the same manner prep in the 2nd Nessler tube a std contg 0.001 g of Amm sulfate, 5 g AN (sulfate-free) and the same reagents as in the 1st Nessler tube. Shake both tubes and compare the turbidity, while holding the bottoms of the tubes over a source of strong light. Consider the Amm sulfate content of the sample to be not more than 0.02% if the turbidity of the test soln is not greater than that of the standard. If the turbidity in sample tube is greater, use the gravimetric procedure described above (Ref 8, p 4)

I)Chlorides. Add to ca a 10 g sample weighed to 0.1 mg in a porcelain casserole, ca 80 ml of 10% NaOH soln and boil until all ammonia is driven off. Dissolve the residue in ca 150 ml cold distd w, neutralize with nitric acid and add a slight excess of Ca carbonate (free from chlorides). Titrate with N/10 Ag nitrate soln, using K chromate as an indicator. Calculate the chlorides found to %-age of NH_4Cl in the sample

Note: If the amt of chlorides in Grade III AN is not above 0.02%, the following turbidometric procedure may be used:

Transfer a 5.0 g sample, weighed to 0.1 mg, to a 100 ml low-form Nessler tube, add 50 ml of distd w and shake until the soln is complete. Add 10 ml of 10% HNO_3 soln, followed by 2.0 ml of 10% $AgNO_3$ soln and mix well. Protect the tube from the direct light. Simultaneously and in the same manner prepare in a 2nd 100 ml Nessler tube a std contg 0.001 g of NH_4Cl and 5 g of AN (chloride-free) and the same reagents as in the 1st Nessler tube. As soon as the $AgNO_3$ soln is added, shake both tubes and compare the turbidity while holding the bottoms of tubes over a source of strong light. Consider the Amm chloride content of the sample to be not more than 0.02% if the turbidity of the test soln is not greater than that of the std. If the turbidity in sample tube is greater, use the gravimetric test described above (Ref 8, p 5)

J)Solubility in Nitric Acid. Add an 80 g sample with stirring to 100 g of 97.5 to 99.0% nitric acid in a 200 ml beaker. After allowing the soln to cool, transfer a 100 ml portion to a 100 ml Goetz (or equal) oil sedimentation tube and centrifuge at 1800 rpm for 20 mins, using an 8-in centrifuge head. The sample is considered completely sol if the vol of sediment does not exceed 0.15 ml (Ref 8, p 8)

K)AN Content may be detd by one of the following methods:

a)Volumetric Method. Add to 100 ml distd w in an Erlenmeyer flask 25 ml of ca 40% formaldehyde soln and a few drops of phpht indicator. Neutralize the soln with ca 0.15 *N* NaOH soln and introduce ca a 1 g of sample weighed to 0.1 mg. Heat the mixt to 60° and titrate, after cooling it to RT, with ca 0.15*N* NaOH soln to a pink end point which persists for 30 secs

$$\% \text{ AN} = \frac{8.005AB}{Wt} - 1.494D - 1.212E, \text{ where}$$

A = ml of NaOH soln used in the titration B = normality of NaOH soln as detd by titration against AN of known purity D = %-age of Amm chloride in sample E = %-age of Amm sulfate in sample and Wt = weight of AN, on dry basis

b)Gasometric Method. In this proced the content of AN is calcd from nitrate nitrogen content as detd by the *nitrometer* method. The method depends on the following reaction taking place when nitrates or nitric acid are shaken with mercury in the presence of concd sulfuric acid:

$$2NH_4NO_3 + 4H_2SO_4 + 3Hg \rightarrow$$
$$(NH_4)_2SO_4 + 3HgSO_4 + 4H_2O + 2NO$$

The amt of nitric oxide liberated is measured and the amt of AN is calcd as will be shown under Calculation

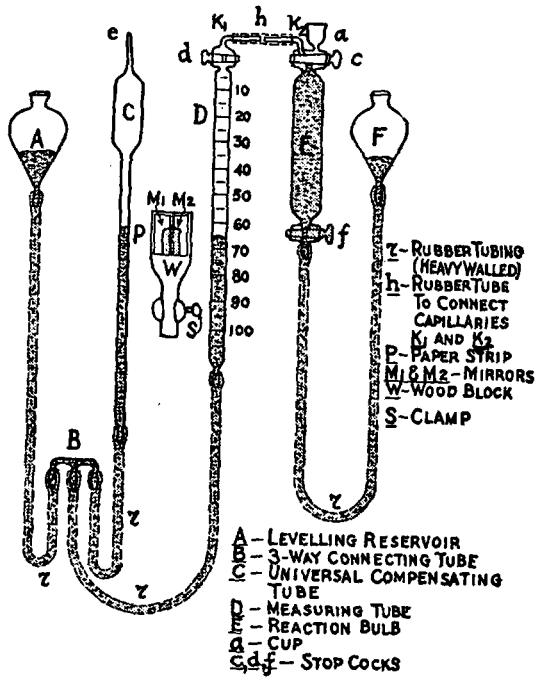
The apparatus used for this test, the du-Pont nitrometer is described in most books on Analytical Chemistry, eg Scott & Furman (Ref 3, p 651), but the description of proced is usually not sufficiently elaborate for anyone not familiar with the apparatus. The procedure described below was used during WWII for training technicians. It contains enough details to permit learning the handling of the apparatus by anyone without previous experience. The same proced can be used in detn nitric acid, K or Na nitrate, NC, NG, etc and it is not considered necessary to describe it again under these items

The assembled nitrometer is shown in the attached fig

If the nitrometer is in an unassembled condition, cut a 9½ ft piece of special thick-walled rubber tubing (such as AHTh No 8842), hang it vertically doubled up to form an upright U-shape, and fill it with 10% NaOH soln. After about ½ hour, drain the tube, rinse with tap water, and cut into the following lengths: 3 ft for tubing A to B, 3½ ft from F to E, 2 ft from B to D and 1 ft from B to C. Roll each piece under the foot and wash the inside and outside with tap water. Pass a long stiff round brush (such as made by trimming of AHTh No 2396) several times through the tubes to remove all dusty material, and then wash with w and with acet. Dry by passing, through the tubes, air thoroughly dehydrated by bubbling through two Drexel wash bottles contg concd H_2SO_4 and finally through glass wool (to catch acid droplets mechanically entrained)

Before assembling the glass parts of the nitrometer, fill them with a mixt of concd H_2SO_4 and K bichromate to which a little oleum is added. Let stand overnight, drain, and thoroughly wash first with tap w and then with distd w. After this, rinse three times with acet and dry by passing air dehydrated by bubbling through two Drexel tubes contg concd H_2SO_4. Wash the stopcocks in the same manner and grease them slightly when assembling the apparatus. Tie a piece of soft wire around each rubber-to-glass joint and attach the assembled apparatus by means of clamps to a vertical stand specially designed for the nitrometer

Preparation of Nitrometer for Standardization and Tests. Break the capillary *e* of the conpensating tube *C* and with the cock *d* open, raise the levelling bulb *A* and pour into it pure, dry, redistd mercury until *C* and *D* are completely filled and 2-3" of mercury are left in *A*. Close cock *d* and open *c* and *f* of the reaction bulb *E*. Raise the levelling bulb *F* and pour into it pure mercury until *E* is filled and 2-3"' of mercury remain in *F* and close *c*



Note: In order to obtain direct readings of NO without the necessity of correcting to std pressure (760 mm), the compensating tube *C* must be filled with dry air and sealed at the top. If the mercury in *E* is not thoroughly dry it must be dried as follows: fill the cup *a* with pure concd H_2SO_4, lower the compensating bulb *F* and, by communicating *a* with *E* by two-way cock *c* draw into *E* all the acid and a small amt of air through *a* and through the capillary K_2. Close the cocks *c* and *f* and shake *E* vigorously. Raise *F*, open *f* & *c* and remove the acid through K_2 into a small beaker. For prepn of dry air, fill the cup *a* with pure concd H_2SO_4 and draw it into *E* in the manner described above. By leaving the cock *c* open, draw into *E* enough air to almost completely fill it, leaving 1-2" of Hg reamining in the bottom of the bulb. Close cocks *c* & *f* and remove *E* from the stand. While holding the top of *E* with one hand and the bottom with the other, keep the stopcocks *c* & *f* in place by pressing against them with the fingers. Shake *E* vigorously for 1-2 mins, repeat the operation every hour during the day and allow to stand overnight. Attach to the capillary k_2, previously greased slightly on the outside, a piece of heavy rubber tubing sufficient to reach capillary *e* of *C*. Without attaching rubber tubing to *e*, expel the wet air in k_2 and in the rubber tubing by raising *F* slightly, opening *f* and then communicating *E* with K_2 for a few seconds by the 2-way stopcock *c*. Close *c* & *f* and immediately attach the rubber tube to *e* (previously greased on the outside with a stopcock grease. With the cock *d* remaining closed, lower the bulb *A*, reopen *c* & *f* and carefully transfer from *E* sufficient amt of air to fill *C* to the position ca *P*. Close the cock *c* and raise the bulb *A* slightly in order not to draw any more air into *C*. Detach the rubber tubing from K_2 and from *e*, wipe off *e* and heat its upper part until softening of the glass, draw out the end and seal the tube. In order to expel all the air from *E* and the capillary K_2 on top of it, raise *F* and by manipulating the two-way cock *c* (with *f* open), remove the acid through K_2 into a small beaker and as soon as the mercury fills the capillary, close the cock

Standardization of Nitrometer.

Weigh to 0.1 mg in a glass-stoppered bottle, 0.70-0.71g of finely pulverized CP K nitrate, previously dried at 100°. Dissolve it in ca 1 ml distd w and pour the soln into cup *a* of the nitrometer

Note: If KNO_3 is 100% pure, the quantity 0.7078 g will evolve in the nitrometer exactly 70 divisions of measuring tube *D*. This is because the tube *D* is designed to hold exactly 300.1 mg in its 100 divisions, which means that each division corresponds to 3.001 mg NO. As the mw of KNO_3 is 101.11 and that of NO 30.01 each 1.0111 g KNO_3 evolves 0.3001 g NO, which corresponds to 100 divisions of *D*, and each 0.7078 g KNO_3 will be equivalent to:

$$\frac{0.7078 \times 100}{1.0111} = 70.0 \text{ divisions}$$

By lowering *F* and opening *f* & *c* draw the contents of cup *a* slowly into *E* being careful not to introduce any air in *E*. Close the cock *c*, but leave the cock *f* open. *Caution.* From this point until further notice the stopcock *f* must remain open. If it is closed the pressure produced by the evolving NO will blow the reaction bulb *E* to pieces

Rinse the weighing bottle (after removal of KNO_3 soln) with 2 ml of cool 94.5% ±0.5 (H_2SO_4) and transfer it into *a*. By opening the cock *c*, accompanied by slight lowering of bulb *F*, draw a small portion of the acid from *a* to *E* and wait until the top of *E* cools. A gentle swirling of the mercury in the upper portion of *E* will hasten cooling. Introduce through *c* the balance of the acid slowly (to prevent overheating) and close *c*. Repeat the operation of rinsing two more times and then rinse the inside upper part of *a* 2 times with a stream of concd H_2SO_4 from a Schuster dropping bottle (AHTh No 2250), transferring the acid each time into the bulb. About 10 ml of 94.5% ±0.5 H_2SO_4 should be used altogether. This amt will dissolve ca 0.34 ml NO at RT *Note:* Soly of NO in sulfuric acid varies with concn and temp. In order to have concordant results the acid of the same concn must be used in all tests and its amt must be approx proportional to the vol of NO evolved in the reaction. Still better, is to use always approx the same amt of acid and the amt of sample which would evolve approx the same vol of NO (ca 70 divisions of the measuring tube *D*) as does 0.70-0.71 g of CP KNO_3 used in standardization

After introducing the last 2 ml portion of H_2SO_4, close the cock *c* and lower the bulb *F* as far as it will go. At this point, inspect the cock *c* to det if any air is leaking into *E*, which, of course would ruin the test. Make sure that *f* is open. Put on a full-view mask, wrap bulb *E* in several layers of toweling and remove it from the stand. Holding the bulb *E* vertically and much above *F* (one hand on the top of the bulb while the other at its bottom, place one of the fingers of each hand on the stopcocks *c* & *f* in such a manner as to prevent them from becoming loose. Shake the bulb *E*, gently at first and then more and more vigorously to start the reaction. After ca 1 min examine *E* to see if there is any gas formed. Continue to shake for another min and this is usually sufficient for evoln of the bulk of NO. Care must be taken not to shake the bulb so vigorously that the acid is carried down through *f* into the connecting rubber tubing

Remove the toweling and raise *E*. The mercury will flow into *F* as the vacuum is created. When 1-2 inches of Hg (not of emulsion) is left in *E*, close the cock *f* *Note:* If too much Hg is left in the bulb, the reaction will proceed slowly and a longer time will be required for the residue to settle. Also some of the gas is liable to be held in the emulsion bv the suspension

With the cocks *c* & *f* tightly closed and held in the manner described above, place *E* in a nearly horizontal position and shake vigorously for exactly 2 mins. By this time, the mass will be a nearly homogeneous emulsion. Replace *E* on the stand and allow the mixt to settle

While waiting, slightly grease the outside of capillaries K_1 & K_2 and see that they are filled with mercury. Raise or lower the measuring tube *D* so that K_1 will be on the same level as K_2. Slip a piece of heavy rubber tubing *h*, 7" long, over k_1 and push it all the way through. Place the end of K_2 just against K_1 and push the rubber tubing *h* over the junction until both capillaries are equally covered

Lower *A*, raise *F* above *E* and, by opening *f*, let the mercury enter *E* from *F* with formation of some pressure. Open the cock *d* and examine for any leakage. Normally the mercury in capillary will move slightly due

to the presence of rubber connections and traces of air

If there is no leak, leave *d* open and, by manipulating carefully with cock *c*, allow the gas to flow slowly from *E* to *D*. Towards the end of transfer, partly close *c* to slow down the rate of flow and just as soon as the acid starts to fill the capillary, close *c*. No acid shall enter *D* and no gas shall remain in the capillary. Close *d*, but leave *f* open

Allow the gas in *D* to stand until it acquires RT

Note: During this time, clean the reaction bulb *E* in prep for the test of the sample. For this, lower the bulb *F*, fill the cup *a* with concd H_2SO_4 and open the 2-way cock *c* to admit the acid into *E*. Suck in some air by manipulating *c* right and left and then create some vacuum in *E* by closing *c* and leaving *f* open, while *F* remains sl below *E*. Close *f*, remove *E* from the stand and shake it, while holding in two hands to wash the white deposit of mercuric sulfate from the walls. Replace *E* on the stand, raise *F* above *E*, open *f*, and, after the mixt has settled, let the acid and a few drops of mercury run through *c* and K_2 into a small beaker. Repeat the operation of cleaning once more and after the operations are completed, leave both capillaries in stopcock *c* and the capillary k_2 filled with mercury so as not to introduce any air in subsequent operations

Before proceeding with actual measuring of vol of NO evolved, calculate the divisions in *D* expected to be produced by the amt of KNO_3 used, from the following equation:

$R = \frac{Wt \times 100}{1.0111}$ divisions, where R = expected divisions in the measuring tube *D* and Wt = weight of KNO_3 sample

If the expected vol of NO is 70.0 divisions (which corresponds to exactly 0.7078 g KNO_3), manipulate *A*, *C* & *D* by lowering and raising them until the position is arrived at, in which the top of the meniscus in *D* is at 70.0 and at the same time the top of the meniscus in *C* is on the same level as in *D*

Note: A convenient arrangement for leveling the mercury in *D* & *C* is to paste on a block of wood *W* (approx 5 × 5 × 10 cm) two pocket-sized mirrors M_1 & M_2 so that the two long sides touch. Attach this block of wood by means of a clamp *S* in the center betw *C* & *D*. Move *W* forwards and backwards as well as up and down until the images of the two menisci form a single image as represented on the fig.

Paste a strip of paper *P* on the compensating tube *C* even with the top of the meniscus and this completes the standardization. Tighten the screws on the clamp holding *C* as this should never be moved, otherwise it will be necessary to restandardize the apparatus

Note: Another method of standardization called the "*absolute method*" is described in the Spec (Ref 8, p 7)

Determination of AN by Nitrometer Method

The best results are obtained with the nitrometer method when sufficient quantity of the sample is taken to produce 70 ±10 divisions of tube *D*. In case of AN, the calcn is as follows: 80.05 g of CP material produces 30.01 g NO and 0.8005 g produces 0.3001 g NO which corresponds to 100 divisions. In order to obtain 70 divisions, the amt AN must be

$$\frac{70.0 \times 0.8005}{100} = 0.5604 \text{ g}$$

If the sample is impure, larger amts are required to obtain 70 divisions

Procedure. Weigh to 0.1 mg ca 0.56 g or larger sample of finely pulverized material dried at 100° and proceed exactly as described under standardization of nitrometer.

Calculation

$$\% \text{ AN} = \frac{R \times 80.05}{Wt \times 100}$$

where R = divisions of D and Wt = weight of sample

Note: The Spec (Ref 8, pp 6-7) prescribes the taking of a sample of exactly 1 g and to proceed in practically the same manner as described above

L)Apparent Density. Transfer a 20 g sample to a stoppered glass cylinder ca 6" high, 0.08" ID and graduated in divisions of 0.5 ml. Drop the cylinder vertically 30 times from a height of 2.5", permitting the base to strike against a hard leather pad. Level off the surface of the column and note the vol occupied by the AN. Calc the d by dividing the wt by the vol occupied (Ref 8, p 9)

M)Granulation. Place a 100 g sample and a silver quarter (25¢) on a specified nest of sieves, properly superimposed and assembled with a bottom pan. Break up lumps or aggregates by gentle brushing with a camel's hair brush. Cover and shake for 3 mins by means of a mechanical shaker geared to produce 300 ±15 gyrations and 150±10 taps of the striker per min. Weigh the portions retained or passed by the various sieves and calc the results to a %-age basis (Ref 8, p 9)

N)Zinc Oxide Content. Weigh to 0.1 mg ca 10 g sample in a tared evapg dish and heat on a hot plate until the disappearance of fumes. Then heat it over a gas burner to a dull red heat, cool in a desiccator, and weigh. Calculate the wt of residue to %-age of ZnO. If the residue is red in color, det the %-age of iron oxide present by dissolving the residue in HCl, pptg any iron present by the addn of NH_4OH, catching the ppt on a filter, igniting and weighing. Correct the wt of residue by subtracting from it any iron oxide present (Ref 8, pp 8-9)

Note: ZnO is added sometimes to AN to prevent its caking. It is also added to AN in order to enhance its burning qualities in pyrotechnic compositions

Jet Propulsion Laboratory at California Institute of Technology, Pasadena, Calif, under the Dept of the Army, Ordnance Corps, ORDCIT Project, Contract No DA-04-495-Ord 18, has been investigating the methods of analysis

In their Progress Report No 20-310 (Ref 20) are given descriptions and evaluations of the following methods of moisture detn: a)Abderhalden Drying Procedure (by loss of wt) b)New Procedure for Determination of Moisture by Loss of Weight c)Oven-Drying at 130° d)Modified Karl Fischer Titration Apparatus for Use with AN Coated with ZnO e)Q-Metry Method (attempts to use this method were unsuccessful)

In the Progress Rept No 20-311 (Ref 21) are given the following procedures: a)Assay of AN by a Redox Titration with Alkali Hypobromite Solution [This method is based on the method described by G. M. Arcand & E. H. Swift in AnalChem *28*, 440(1956)] b)Determination of Total Ash in AN (using a proced of slow sublimation) and c)Determination of Zinc Oxide in AN (using a thioacetamide separation as ZnS and subsequent chelatometric titration with ethylenediaminetetraacetic acid) d)Determination of Zinc Oxide by a Short Procedure (for samples which do not require a sulfide separation)

Note: The Specification procedure described in Ref 8, pp 8-9) is not applicable if An contains other materials, such as an anticaking agent *Attasorb.* Zinc oxide acts not only as an anticaking agent but it also enhances the burning characteristics of AN which is important in case of pyrotechnic compns and may be of propellants

In the Progress Rept No 20-365 (Ref 22) are given the following procedures: a)Assay of AN by Quantitative Distillation of Ammonia (modification of the Kjeldahl method described in various books, such as in Ref 3) b)Assay of AN by Quantitative Reduction with Ferrous Ammonium Sulfate (modification of a method described in Ref 2)

c)Reduction of An with Titanous Salts (modification of Knecht & Hibbert Method described in various papers and books)
Note: It has been claimed in the Progr Rept 20-365 that none of the existing methods of reduction of nitrate by titanous ion are accurate when analyzing AN. The modification proposed in the rept is not as accurate as ferrous sulfate or other methods and for this reason cannot be recommended

References on Ammonium Nitrate, Analytical Procedures:

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12)Jacobs(1949), 364-6
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Ammonium Nitrate, Analytical Procedures Used by the Spencer Chemical Co, Kansas City, Missouri

I)Ammonia, Anhydrous. See under Ammonia

II)Ammonium Nitrate Solution. (Standard Procedure FP-4)

A)Sampling. When each car is completely loaded, it is sampled while the liq is still hot and before the sepn of any crysts. Four clean and dry 12 to 16 oz Pyrex bottles are placed on the sampling pole and lowered through the liq in the car and raised again to the top at a uniform rate, such that the bottles are filled approx ⅔rd full. The bottles are then tightly stoppered and sent to the lab for analysis

B)Preparation of Sample. Place three of the bottles on a steam heated water bath, retaining one bottle as a reserve sample. Heat until all crysts are completely dissolved and then pour all three bottles into a heated, dry 500 ml Florence flask. Mix well and analyze as follows:

C)Procedures:

1)Acidity or Alkalinity. Pour two 100 ml samples, accurately measured in a 100 ml graduate, into a 400 ml beaker contg 200 ml distd w. Add 5 drops of methyl-red indicator (prepd by dissolving 0.1 g of MR in 100 ml of 70% alcohol and neutralized to a salmon pink color). If the soln is yel, titrate with N/10 sulfuric acid and if red, titrate with N/10 NaOH soln. Calculate alkalinity as NH_3 and acidity as nitric acid

$$\text{Alkalinity} = \frac{\text{ml } H_2SO_4 \times N \times 0.017}{\text{SpGr of Sample}}$$

$$\text{Acidity} = \frac{\text{ml NaOH} \times \text{N} \times 0.063}{\text{SpGr of Sample}}$$

2)Ammonium Nitrate Content:
a)By Titration. Using a heated 1 ml graduated pipette, transfer ca 0.8 ml of hot sample into each of two tared and covered 30 ml beakers. Weigh quickly and wash into two 250 ml Erlen flasks. Dil each to ca 100 ml with distd w, add 25 ml neutral 20% formaldehyde and heat to 60°. Cool to 30° and titrate with N/10 NaOH soln using 5 drops of phenolphthalein indicator (prepd by dissolving 1 g phpht in 100 ml of 70% neutral alc)

$$\%\ \text{AN} = \frac{\text{ml NaOH} \times \text{N} \times 0.08005 \times 100}{\text{Wt of Sample}}$$

b)By Fog Point. Add ca 75 ml of hot sample into a well dried 100 ml beaker. Insert a thermometer, turn on an electric stirrer and run it until cloudiness appears in the AN soln. Check temp and det % AN from the table given on p 4 of Standard Procedure FP-4. For instance, if the Fog Point is at 55.5°C, the % AN is 78.89 and if it is at 58.0°, the % AN is 79.68, etc

c)By Specific Gravity. Fill to near the top of a preheated hydrometer jar with hot sample. Insert a 1.300-1.400 hydrometer and a thermometer, from 38° to 82° graduated in 1/10°. Stir the soln carefully with a thermometer and when the hydrometer has come to equilibrium, take a reading at the bottom of the meniscus and note simultaneously the temp to the nearest 1/10°. Det % AN from the table given on pp 5-6 of Standard Procedure FP-4. For instance, if sp gr at 60°C is 1.362, % AN is 78.76% and if sp gr at 70.1° is 1.360, % AN is 79.67 etc

III) Unparted Ammonium Nitrate Prills

In order to det if product meets specific purchaser's specifications, the following detns described in Spencer's Standard Procedure FP-5A are used:
1)Ammonium Nitrate Content
2)Moisture Content
3)Average Particle Size (ca 1.8 mm)
4)Water Insoluble Material (example given 0.0024%)
5)Chlorine as Cl (less than 5 ppm)
6)Ash
7)pH of 10% Solution at 20°
8)Acidity as Nitric Acid
9)Alkalinity as NaOH
10A)Ether Soluble
10B)CCl_4 Extractible Material
11)Foreign Material (visual)
12)Particle Size
13)Density
14)Nitrites
15)Sulfates

AMMONIUM NITRIDES

In order to explain certain anomalous behavior of cobaltous amide, $Co(NH_2)_2$, obtained as a result of the reaction:
$Co(SCN)_2 + 2KNH_2 = 2KSCN + Co(NH_2)_2$,
it was suggested by Bergstrom(Ref), that actually reaction proceeds further and cobaltous nitride and ammonia are formed:
$3Co(NH_2)_2 \longrightarrow Co_3N_2 + 4NH_3$.
As a large part of NH_3 is always retained by the nitride, the resulting compd, $Co_3N_2 \cdot xNH_3$, may be called "ammonium cobaltous nitride". A similar compd, $AlN \cdot xNH_3$, called "ammonium aluminum nitride" was prepd by pouring a soln of NH_4 Br in liq NH_3 into a soln of Na K ammonoaluminate. The amt of NH_3 retained by these complexes varies within certain limits depending on the temp and pressure. Attempts to prepare a similar complex from stannous nitride, Sn_3N_2, were unsuccessful
Ref: F.W.Bergstrom, JPhysChem 32,433–40 (1928)

AMMONIUM NITRITE

NH_4NO_2, mw 64.04, N 43.74%. Wh to yel ndl-like deliquescent crysts, d 1.69, mp – starts to sublime at 32–3° and decomp explosively >60°; fl p 158°F. Readily sol in w(with evoln of heat); sol in alc and nearly insol in eth, chlf or ethyl acetate. Was first prepd in 1812 by J.J.Berzelius by treating Pb nitrite with Amm sulfate or by treating Ag nitrite with Amm chloride. Later, 1874, M.Berthelot used the reaction betw Ba nitrite and Amm sulfate and also the interaction of ammonia with nitric oxide and oxygen. O.L.Erdmann & S.P.Sörensen prepd it by passing a mixt of nitrogen oxides(obtained by the action of arsenic trioxide on nitric acid) over coarsely ground Amm carbonate, kept cool by ice; the half liq mass was treated with alc, the unchanged carbonate filtered off and the Amm nitrite precipitated by the addn of ether. The nitrite so obtained was of 90–94% purity and was further purified by dissolving in alc and pptg by ether. All these methods are described in Mellor (Ref 1) and in Gmelins(Ref 4). Amm nitrite cannot be obtained by evaporating its aq soln(Ref 2). A procedure for prepg its aq soln was patented by Kahr(Ref 5)

Amm nitrite is an explosive sensitive to heat or shock. When a very small quantity of the dry salt is heated slowly on a spatula, the salt volatilizes and burns with a pale flame. When an appreciable quantity is heated above 60°, a violent expln takes place. Its concd aq soln decomps explosively when heated to 60–70° and if a similar soln is acidified with 1 drop of concd HCl, HNO_3, or H_2SO_4 a spontaneous decompn takes place even at room temp. Following props were detnd by Kast(Ref 3): heat of formation 65 kcal/mol or 1016 cal/g, heat of expln 803 cal/g, temp of expln 2210°, tot vol of gas evolved on expln 1050 l/kg, vel of deton(V)(calcd) ca4000 m/sec at d ca 1, spec energy(f) 9865 kg/l, brisance (by Kast formula fdV) 39500 vs 86000 for TNT. Heat of formation in Lange's Handbook is given as – 61.5, while in Hodgman's Handbook it is + 62,5, kcal/mol

Amm nitrite cannot be stored under ordinary conditions because it is very deliquescent and unstable, decompg slowly in winter and rapidly in summer into N and H_2O. The salt may be transported in dry ether, free from alc. The dry salt may be preserved for some time under an atm of hydrogen and in the presence of Amm carbonate and CaO. Its fire hazard and toxicity are discussed in Sax(Ref 6)

Refs: 1)Mellor 8(1928), 470–2 2)H.Kast, SS21,207(1926) 3)H.Kast, SS22,8(1927) 4)Gmelins, Syst No 23,Lfg 1(1936), 85–93 5)K.Kahr to Inventa A–G, USP 2,606,813 (1952) & CA 47,278(1953); BritP 685,726 (1953) & CA 47,4563(1953) 6)Sax(1957), 282 and under Nitrites, p 943

Ammonium Nitroform. See Ammonium Trinitromethane under Methane and Derivatives

Ammonium Oxalate. See under Oxalates

Ammonium Perbromate. See under Bromates, Perbromates, etc

Ammonium Perchlorate. See under Perchlorates

Ammonium Perchromate. See under Bichromates, Chromates, etc

Ammonium Periodate. See under Iodates, Periodates, etc

Ammonium Permanganate. See under Manganates, Permanganates, etc

Ammonium Peroxychromate. See under Bichromates, Chromates, etc

Ammonium Persulfate. See under Sulfates and Persulfates

Ammonium Picrate; Explosive "D" or Dunnite. See 2,4,6-Trinitrophenol, Ammonium Salt and Phenol and Derivatives

Ammonium Picrate Mixtures. See under Phenol and Derivatives

Ammonium Salts of Aromatic Nitro Compounds such as of Nitrophenol, m-Nitrocresol or of Picric Acid. For prepg phlegmatized and substantially shockproof Amm salts of the above aromatic nitro compds, ammonia gas (in excess) is dissolved in a viscous petroleum jelly or viscous lubricating oil having no substantial solvent effect upon the Amm salt and having a high bp. The above reaction medium is brought into contact at RT with the desired nitrocompd in the solid state and the resulting Amm salt of the nitrocompd is removed
Ref: E.Berl & W.Berl, USP 2,350, 322(1944) & CA **38**, 4961(1944)

Ammonium Salts, Quarternary. Several org quarternary Amm salts were prepd by Tadros et al. When, in the prepn of 4-formyl-2-nitrophenyltrimethylammonium salt, a mixt of 4-dimethylamino-3-nitrobenzaldehyde $(CH_3)_2N{\cdot}C_6H_3(NO_2){\cdot}CHO$ with methyl sulfate $(CH_3)_2SO_4$ was heated to ca 140°, a violent expln took place
Refs: 1)W.Tadros & A.Latif, JCS **1949**, 3337–40 & CA **44**, 4888(1950) 2)W.Tadros & A.Kamel, JCS **1951**, 1890–2 & CA **46**, 920(1952)

Ammonium Salts Which are Explosive, examined by H.Kast and described in SS **21**, 205–9(1926) and SS **22**, 6–9, 30–4, 56–61, 77–80, 99–102 & 131–5(1925) included: azide, bichromate, chlorate, nitrate, nitrite, perchlorate, permanganate and trichromate

Ammonium Sulfamate. See under Sulfamates

Ammonium Sulfate. See under Sulfates and Persulfates

Ammonium Sulfide. See under Sulfides

Ammonium Sulfite. See under Sulfites

Ammonium Tartrate. See under Tartrates

Ammonium Triazidocuprate. Same as Triazidocuprate, Ammonium

Ammonium Trinitrate, $NH_4NO_3{\cdot}2HNO_3$, prismatic ndls, mp 29–30° and **Ammonium Dinitrate,** $NH_4NO_3{\cdot}HNO_3$, lfts or plates, mp ca 12°. Both salts were obtained by Groschuff(Ref 1) from AN and anhyd nitric acid under cooling. Both salts were found to be hygroscopic and the dinitrate decompd by water. Duke & Llevellyn(Ref 2) gave a detailed description of prepn of trinitrate as well as its cryst structure as detd by means of X-rays. Its expl props were not examined
Refs: 1)E.Groschuff, Ber **37**, 1487–8(1904) 2)J.R.C.Duke & F.J.Llevellyn, ActaCrystallographica **3**, 305–11(1950) & CA **45**, 923(1951)

Ammonium Trinitrocresylate or Ecrasite. See Trinitrocresol, Ammonium Salt, under Cresol and Derivatives

Ammonium Ulmate or Ammonium Humate is a substance obtained in 1889 by Gaens on boiling peat(previously washed) with a soln of Na_2CO_3. An expl mixt contg this "ulmate", KNO_3 and collodion cotton(gelatinized by

ethyl acetate) was patented by Gaens(Ref 1) and another expl prepd by mixing the ''ulmate'' with molten naphthalene was patented by Reuland
Refs: 1)Daniel(1902),p 322(under Gaens) 2)Ibid,p 681(under Reuland)

Ammonium Urate. See under Urates

Ammonkarbonit. See Ammoncarbonit in PATR 2510(1958),p Ger 5

Ammon-Nobelit. See in PATR 2510(1958), p Ger 5

Ammono-Basic Lead Nitrate, $Pb_2N(NO_3)\cdot nNH_3$, N(detd) ca 7.6%. Wh to yel amorphous powder obtained by Franklin by the action of K amide on Pb nitrate, both dissolved in liq NH_3. The resulting ppt which settled with difficulty was dried and analyzed. Its expl props were not detd
Ref: 1)E.C.Franklin,JACS **27**, 846(1905)

Ammonolysis is the direct reaction of ammonia with an organic compd. This reaction is used for the prepn of various amines, nitramines, etc and some of these products are or may be converted into expls
Refs: 1)W.C.Fernelius & G.B.Bowman,Chem-Revs **26**, 3–48(1940)(286 refs)(Ammonolysis in liquid ammonia) 2)Kirk & Othmer **1** (1947), 826–44(89 refs) 3)A.C.Stevenson, IEC Sept 1948 to 1952, under ''Unit Process Review'' 4)G.H.Coleman,IEC Sept 1953, under ''Unit Process Review'' (No ammonolysis reviews appeared in the years 1954–1957) 5)Groggins(1958),388–485

Ammonpek. A Russian coal mining expl consisting of AN 95 and coal tar pitch (pek) 5%
Ref: A.D.Blinov, ''Kurs Artilleriee,'' Voyennoye Izdatel' stvo,Moscow,v 2(1949)

Ammonpentrinit or Ammonpenthrinite is a *Pentrinit* in which some AN is incorporated (up to 50%) in order to obtain expls suitable for blasting purposes. Pentrinits are plastic non-exudable expls invented ca 1928 by Dr A.Stettbacher, Zürich, Switzerland. One of the first mixts contained PETN 80 & NG 20% and was considered suitable for loading shells and as a base chge in detonators(using 0.04 g of LA as a primary chge). If CC(collodion cotton) is incorporated, the expl is called *Gelatinepentrinit*. Pentrinit was prepd and investigated at the Gamsen-Brigue plant of the Société Suisse des Explosifs and proved to be an outstanding expl, particularly effective for underwater explns. Incorporation of ca 15% of Al increases the efficiency but higher amts seem to decrease it. For low-freezing Pentrinit, NG is mixed with 20–25% of NGc(nitroglycol)

The enclosed table gives the composition and some props of Ammonpentrinits

Ammonpentrinits of Stettbacher

Compn & Props	1	2	3	4	5
PETN	40.9	40.6	37.0	31.0	33.8
NG	40.9	7.6	7.2	7.5	50.7
NGc	–	2.6	2.0	–	–
CC	1.6	–	0.8	0.5	0.5
AN	16.6	47.5	48.0	59.0	15.0
DNT(liq)	–	–	5.0	–	–
Vaselin	–	1.7	–	2.0	–
d(loading)	–	–	1.37	–	–
d(max)	–	–	1.45	–	–
Deton Vel, m/s	–	–	6600	–	–
Gas Vol at NTP,l/kg	–	–	430	–	–
Trauzl Test Value,cc	–	–	–	–	–

(See also Gelatinpentrinit and Pentrinit)

Refs: 1)A.Stettbacher, SS **23**, 345–8(1928) 2)Ibid,AngewChem **43**, 844–7(1930) 3)Ibid, Nitrocellulose **4**, 179,199,222-7(1933) & **5**, 6–12(1934) 4)Davis(1943), 281 5)Stettbacher(1948), 83–5 6)Stettbacher, Pólvoras (1952), 113 7)Dr A.Stettbacher,Zürich, Switzerland; private communication Dec 14,1953

Ammonpulver. See PATR 2510(1958), p Ger 5

Ammonsalpeter(Ger). Ammonium Nitrate

Ammonsalpetersprengstoffe(Ger). Ammonium Nitrate Explosives(see PATR 2510, p Ger 5)

Ammon Semi-gelatine or Semi-gelatine is one of the modern Brit ''non-permitted'' expls

based on AN: NG and NGc 15.0, NC 0.3, AN 78.7 & carbonaceous material 6.0%. It is a cohesive substance with d of 1.2 and its power is equal to 82% that of blasting gelatin
Ref: Taylor & Gay(1958), 26

Ammonsprenggelatine. A gelatinized dynamite consisting of NG(gelatinized with 2–3% CC) 38–47, AN 45–55, dry meal 3.5–5.0 and Na_2CO_3 0.5%
Ref: CondChemDict(1942), 287(not listed in newer editions)

Ammonsulfatsalpeter. Ger name for mixts of AN & Amm sulfate used in fertilizers
Ref: Stettbacher(1948), 81

Ammontol or Russian Mixture(Russkaya Smes'). A castable HE mixt used for filling projectiles: AN 50, TNT 38 and TNX(called "ksilil" in Russian) 12%.
Ref: A.D.Blinov, "Kurs Artilleriee," Voyennoye Izdatel'stvo, Moscow,v 2(1949), 64–5

Ammonxyl. See Ammoksil

Ammonyaku. A mixt of AN & charcoal used by the Japanese as a "substitute explosive"
Ref: Anon, "Handbook of Japanese Explosive Ordnance," OpNav 30–3M, GovtPrtg Off,Washington,DC(1945), 29

AMMUNITIONS AND WEAPONS OR ARMS

[Munitionen und Waffen in Ger; Munitions et Armes in Fr; Boyepripassy i Oruzhiye (Boyevyiya Sredstva) in Russ; Municiones y Armas in Span; Munizioni(Proietti) e Armi in Ital]. *Ammunition* is any material used in warfare and designed to inflict damage upon the enemy. The term includes the *complete round of ammunition* as well as other components or elements. Reduced to its fundamentals, ammunition usually consists of a container(metallic or other material) containing propellant and a missile with/or without explosive. *Weapons* may be subdivided into *small arms*(calibers up to about 0.60" in the US) such as pistols, revolvers, carbines, rifles, submachine guns & machine guns; *artillery ammunition*(calibers of 20 mm and larger in the US), such as guns(cannons), howitzers & mortars as well as recoiless rifles, rocket launchers & pyrotechnic pistols

The basic types of ammunition are:

I. Small Arms Ammunition(SAA), which includes various kinds of bullets to be fired by propellants enclosed in metallic cartridges from weapons called "small arms" and which are carried by one or two men. US small arms are weapons 0.60 inch or under in caliber and include: rifles(except recoiless), semi-automatic rifles, automatic rifles, pistols, revolvers, carbines, machine guns and submachine guns. This category also includes shells used in shot gun and rifle grenades
Note: The British also use inches to express calibers of their small arms and ammunition, whereas nearly all other countries of the world use the metric system

II. Artillery Ammunition consists of projectiles(shells) to be fired from weapons larger than 0.60 inch in caliber. Calibers of US artillery ammunition and weapons are given mostly in millimeters, some, however are given in inches(eg, most naval guns)

Artillery ammunition may be classified as follows:

A)According to Service Use: This includes: *a)Service Ammunition*–designed to inflict damage on the enemy *b)Practice Ammunition*–designed for training troops in marksmanship. The filler of the projectile may be inert or consist of a small explosive charge serving as a "spotting charge" *c)Drill or Dummy Ammunition*–designed to train gun crews in the motions of loading and firing a weapon without actually firing it; there is no explosive filling *d)Blank Ammunition*–designed primarily for saluting purposes and for simulated fire. It is also used for accustoming animals(such as horses, mules, dogs, etc) to the sound of fire; there is a propellant which is retained by a wad but no projectile
Note: This classification may also be applied to small arms ammunition

B)According to Tactical Use. This includes

the following projectiles(shells): **HE**(high explosive), **HE–T**(high explosive with tracer), **AP**(armor-piercing), **AP–T**(armor-piercing with tracer), **CP**(concrete-piercing), **SAP**(semi-armor-piercing), **HEAT**(high-explosive, antitank), **HEP–T**(high explosive-plastic, with tracer), **HEI**(high explosive-incendiary), **HVAP**(hyper-velocity armor-piercing), **HVTP–T**(hyper-velocity, target practice, with tracer), **TP–T**(target-practice, with tracer), **Incend**(incendiary), **Illum**(illuminating) and **Can**(canister)

C)According to the Type of Weapon from which the Projectile is Fired. This includes two types of artillery weapons: *a)Fixed Artillery or Artillery of Position*–designed for permanent emplacements. This may include: siege artillery, harbor and seacoast defence artillery, antiaircraft artillery and sometimes antitank artillery. Guns(cannons), mortars and howitzers of calibers 155 mm and higher are used for this type of artillery *b)Mobile Artillery*–designed to be movable from place to place to accompany or follow the troops. This may include: field artillery (self-propelled and towed), tank and anti-tank artillery, armored vehicles artillery and antiaircraft artillery. Weapons of smaller than 155 mm are used for this type of artillery but there are some mobile guns(usually towed) which are larger than 155 mm. Artillery used on gunboats, as well as naval and railroad artillery, may also be included in this type. Naval artillery is of all calibers(large and small) whereas railroad artillery comprises mostly large caliber weapons, such as the 155 mm gun and larger. Antiaircraft artillery may also be considered as one of the types of mobile artillery, but it is usually considered separately

Note: It is regrettable to say that there are no modern comprehensive treatises on US, Brit, French, Ital, Spanish, etc Artillery Weapons. Most of the existing books are either too brief or obsolete. The Russian book ''Kurs Artilleriee'' by Blinov(Ref 52) is fairly comprehensive but it is now out of print(A copy is available in the Library of Congress)

III. Aircraft Ammunition includes anything fired or dropped from a plane, such as shells, bullets, rockets, bombs, aerial torpedoes, aerial mines and pyrotechnic devices

IV. Rocket Ammunition is fired from a device called a ''launcher,'' such as the ''bazooka'' of WW II fame. Rocket launchers consist of either guide rails or guide tubes fitted with some electric ignition device. Each rocket carries its own propelling type motor and a warhead containing an HE or a chemical agent

V. Jatos consist of propelling-type motors used to furnish auxiliary thrust in the launching of aircraft, rockets, guided missiles, target drones and mine clearing detonating cables

VI. Guided Missiles consist of propelling-type motors fitted with warheads contg HE or other active agents and equipped with guidance devices

VII. Grenades are explosive- or chemical-filled projectiles of a size and shape convenient for throwing by hand or projecting from a rifle or a launcher

VIII. Bombs are containers filled with an explosive, chemical or other agent, designed for release from aircraft

IX. Land Mines are containers, metal, plastic or wood, filled with HE or chemical agents, designed for placing in or on the ground for initiation by and effect against enemy vehicles or personnel. This includes some booby traps (Other booby traps are not land mine types)

X. Demolition Materials consist of explosives and explosive devices designed for demolition purposes or for blasting in connection with military construction

XI. Pyrotechnic Ammunition includes devices used for signaling, illuminating or igniting purposes. It may be classified according to tactical use as ground devices, which are fired or used on the ground, and aircraft devices released from aircraft. Following are examples of pyrotechnic items: flares(trip, airport, ground, aircraft, parachute, reconnaissance & landing, observation, bombardment and tow range); photoflash cartridges and bombs; tracers in artillery projectiles; igniters (in incendiaries and for jet propulsion units); signal smokes and gunflash simulators

XII. Miscellaneous Ammunition includes items not listed in the above groups, such as torpedoes(sea, aerial and bangalore), depth charges, sea mines, destructors, cartridge-actuated devices(designed to facilitate an emergency escape from high-speed aircraft), etc

Ammunition: Complete Round of, includes all the components necessary to fire a weapon once. In the case of small arms ammo, a complete round consists of a primed cartridge with propellant and a bullet with/or without a tracer. In the case of artillery ammo, a complete round consists of propellant in a primed cartridge case or in bags, an igniter train and a projectile and/or high explosive shell. American artillery ammo may be divided into the following three classes, depending upon the type of enclosure used in loading the propellant charge:

a)Fixed Ammunition. The propellant charge is enclosed in a metallic cartridge which is provided at the base with a primer and an igniter and at the open end with a rigidly fixed projectile. The round is all in one unit *b)Semifixed Ammunition.* The propellant charge is contained in several cloth bags which are placed in the cartridge provided at the base with a primer and an igniter. The other end of the case is loosely attached to a projectile so that it can be removed before firing in order to adjust the number of bags to the desired muzzle velocity and range selected for the projectile. The unit is self-contained and ready to fire *c)Separate-loaded Ammunition.* The propellant charge is contained in several bags which are transported separately from the projectile. In loading the gun, the projectile is inserted through the breech of the gun and rammed into place; this is followed by bags of propellant, loaded one by one until the desired charge is reached; the breech is then closed and the primer-igniter and the firing mechanism inserted

The above classification applies only to the US artillery ammunition. Other countries may have different systems

For instance, the *German* artillery of WW II consisted of the following two types: *a)Einheitsmunition oder Patronenmunition* (One-piece Ammunition or Cartridge Ammunition), corresponding to US fixed ammunition *b)Kartuschmunition oder Getrenntemunition* (Canister Ammunition or Separated Cartridge Ammunition)–This was somewhat intermediate between US semi-fixed and separate-loaded ammunition. It consisted of a projectile which was placed in the weapon first and a canister(cartridge), provided with a primer and contg one or several bags with propellant charges, which was loaded into the breech afterward. The canister was not fixed to the projectile. The number of bags with propellant charges could be varied according to the range requirement at the place of firing

The *Russians*, according to Blinov, vol 5 (Ref 52), used the following types of artillery complete rounds:

a)Patronnoye Zariazheniye(Cartridge Loading). This corresponds to US "fixed" ammunition and is used for rounds up to and including 100 mm *b)Patrony s Sostavnymi Zariadami v Metalicheskikh Ghil' zakh*(Rounds with Composite Charges in Metallic Cartridges)–corresponds approximately to US "semi-fixed" ammunition, but the round is loaded in two operations; first the projectile (snariad) and then the metal cartridge case (ghil'za) with the propellant(porokh). This type of ammo was fired from 107, 122 and most 152 mm weapons *c)Razdel' noye Zariazheniye*(Separate-loaded Ammunition) – corresponds to the US "separate-loaded" ammo, but the round is loaded in three operations; first the projectile(snariad), then the cloth bag(kartoozy) with propellant(porokh) and finally igniter(vosplamenitel'). This type of ammo, also known as *Kartooznoye Zariazheniye,* was used in 152 mm M1935 cannon and in all cannons and howitzers of larger caliber

Note: More information on **German** ammo & weapons may be found in PATR 2510(1958) (Ref 100) and in Refs 25,45,73,75,76,89 & 102. A comprehensive treatise on **Russian** artillery may be found in the books of Blinov (12 vols)(Ref 52) and a brief description is given in conf PATR 2145(1955)(Ref 89a) and

in Ref 45. Russ ammo in general is described in Ref 44 and in conf Ref 74. Russ machine guns and other automatic weapons are described in conf vol 2 of Chinn's books(Ref 73). Some **French** ammo & weapons are described in Refs 12 & 14 and in a series of booklets by Pichené(Ref 63, 64, 65, 66, 67, 68, 81, 86, & 88). A few Fr items are described in TM 9–1985–6(Ref 79), which is identical with OP 1668(1946) (Ref 44a). Some info on **British** items(mostly obsolete) is given in Refs 1,2,3,4,5,6,7,28 & 96). Several Brit ammo items are described in conf TM 9–1985–6(Ref 79). Incomplete info on **Italian** ammo is given in Refs 44a & 79. Some info on **Japanese** ammo may be found in Refs 40, 77 & 78. No info at our disposal exists on ammo & weapons of Austria, Belgium, Egypt, Greece, India, Mexico, South American countries(except Argentina), Persia, Spain, Sweden, Switzerland, Turkey, and of the countries with communistic governments(Albania, Bulgaria, China, Hungary, Poland, Rumania and Yugoslavia)

Calibers and Uses of Small Arms Ammunition(USA)

Cal .22(0.223") (5.59 mm)–for cal .22 long and short rifles(TM 9–1990)
Cal 7.62 mm(0.300"), M61–for cal 7.62 mm NATO rifles M14 & M15 and for AR–10 "Armalite" rifles(Ref 100,pp 34–9)
Cal 7.62 mm–for cal 7.62 mm machine gun M60(Ref 100,p 44)
Cal .30(0.3075") (7.81 mm) M2–for rifle M1, carbine, Krag(subcaliber) machine gun and rifle-grenade cartridges(TM 9–1990)
Cal .32(0.314") (7.98 mm)–for automatic Colt revolver and S & W revolver (TM9–1990)
Cal 9 mm(0.354")–for Parabellum pistol (TM 9–1990)
Cal .380(0.356") (9.04 mm)–for automatic pistol, called 9 mm short(TM 9–1990)
Cal .38(0.359") (9.12 mm)–for super automatic Colt special and S & W (TM 9–1990)
Cal .38(0.375") (9.52 mm)–for short Colt (TM 9–1990)
Cal .45(0.4505") (11.44 mm)–for automatic pistols, revolvers and submachine guns (TM 9–1990)
Cal .50(0.5110") (12.98")–for machine guns, including multiple MG,M55 Shotgun shells: 12, 16, 20 and .410 gage (TM 9–1990)

Calibers and Uses of Artillery Ammunition and Rockets(USA)

Cal 20 mm(0.787") (fixed)–for 20 mm automatic guns AN–M2, M3, BRHS & M24 mostly used as aircraft cannons(TM 9–1901)
Cal 37 mm(1.457") (fixed)–for 37 mm automatic guns M1A2, M4, M6 & M9, used as AA and as aircraft cannon(TM 9–1901) & Ref 49, pp 140–1)
Cal 40 mm(1.575") (fixed)–for 40 mm automatic AA gun M1(Bofors) (Ref 49,p 146)
Cal 40 mm(fixed)–for 40 mm guns M1A2, Navy MK1 and for twin self-propelled gun M42(TM 9–1901 and Ref 100,pp 14 & 20)
Cal 57 mm(2.244") (fixed)–for 57 mm gun M1 (TM 9–1901)
Cal 57 mm(fixed, perforated cartridge)–for 57 mm recoilless rifle(TM 9–1901 & Ref 100, p 39)
Cal 2.36"(59.9 mm) rocket M6A3–for 2.36" rocket launcher "Bazooka" (Ref 49,p 178)
Cal 60 mm(2.362") mortar ammo–for 60 mm mortars M2 & M19(TM 9–1901 & Ref 49, p 161)
Cal 75 mm(2.953") (fixed)–for 75 mm guns M3, M6, M17 and the "Skysweeper" (Ref 100,p 15)
Cal 75 mm(fixed and semifixed)–for 75 mm howitzers M1A1(pack) and M3(TM 9–1901)
Cal 75 mm(fixed, perforated cartridge)–for 75 mm recoilless rifle M20(TM 9–1901)
Cal 76 mm(2.992") (fixed)–for 76 mm guns M1A1C & M29(TM 9–1901)
Cal 3"(76.2 mm)–for 3" AA gun M3 and A/T gun M5(Ref 49,p 148)
Cal 81 mm(3.19") mortar ammo–for 81 mm mortars M1, M21 & M29(TM 9–1901) & Ref 49,p 165)
Cal 3.25"(82.55 mm) rocket M2(referred to as "target rocket")–for 3.25" rocket projector M1(Ref 49,p 180)
Cal 3.5"(88.9 mm) rocket–for 3.5" rocket launcher(Ref 100,p 42)
Cal 90 mm(3.54") (fixed)–for 90 mm guns M1, M1A1, M1A2, M2, M2A1, M3, M3A1 & T8 (TM 9–1901 & Ref 100,p 15)
Cal 90 mm(fixed)–for 90 mm self-propelled

gun M56, also known as "assault gun" (Ref 100,p 40)
Cal 105 mm(4.134") (semifixed)–for 105 mm howitzers M2A1, M3 & M4(TM 9–1901)
Cal 105 mm mortar ammo–for 105 mm mortar T13(Ref 49,p 168)
Cal 105 mm(perforated cartridge)–for 105 mm recoilless rifle(Ref 100,p 40)
Cal 106 mm(4.17") (perforated cartridge ammo) –for 106 mm recoilless rifle M40A1(Ref 100, p 39)
Cal 4.2"(106.7 mm) mortar ammo–for 4.2" mortar M2, M3 and M30(TM 9–1901) & Ref 100,p 43)
Cal 4.5"(114.3 mm) (separate-loading)–for 4.5" gun and for howitzer M1(Ref 57)
Cal 4.5" rockets–for 4.5" rocket launchers M23, T36 & T66(Ref 49,p 182 & Ref 100, p 43)
Cal 120 mm(4.72") (separate-loading)–for 120 mm AA gun M1(TM 9–1901)
Cal 155 mm(6.10") (separate-loading)–for 155 mm guns M1 & M2 and howitzer M1 (TM 9–1901 & Ref 100,p 18)
Cal 155 mm mortar ammo–for 155 mm mortar T25(Ref 49,p 170)
Cal 155 mm(perforated cartridge ammo)–for 155 mm recoilless rifle(Ref 57)
Cal 175 mm(6.89")–for 175 mm self-propelled gun T235(Ref 100,p 11)
Cal 7.2"(182.9 mm) rocket–for 7.2" multiple rocket launcher M17(Ref 49,p 182)
Cal 8"(203 mm) (separate loading)–for 8" gun M1 and howitzer M2(TM 9–1901)
Cal 8" rocket T25–for 8" rocket launcher T53(Ref 49,p 194)
Cal 240 mm(9.449")(separate-loading)–for 240 mm howitzer M1(TM 9–1901)
Cal 280 mm(11.024") (separate-loading)– for 280 mm gun(Ref 100,p 9)
Cal 318 mm(12.91") rocket "Little John" – for 318 mm rocket launcher(Ref 100,p 24)
Cal 14"(355.6 mm) (separate-loading)–for 14" guns(TM 9–1904)
Cal 762 mm(30.29") rocket "Honest John" – for 762 mm rocket launcher(Ref 100,p 23)
Cal 914 mm(35.98") mortar ammo–for 914 mm mortar, nicknamed "Little David" (Ref 49,pp 172–3)
Calibers of guided missiles: Corporal, Dart, Hawk, Jupiter, Lacrosse, Nike(Ajax & Hercules) Redstone, etc are classified(Ref 10 pp 25–33 & 41)
Subcaliber and blank ammunition are described in TM 9–1901, etc
Notes: a)Mortars are either smooth-bore or rifled b)Some of the items listed above may be still in the development stage and some were used during WW II but not since(for example, the 914 mm mortar)
(See also Bangalore Torpedoes, Bullets, Bombs, Cartridges, Demolition Materials, Depth Charges, Grenades, Guided Missiles, Jatos, Land Mines, Projectiles, Pyrotechnic Devices, Rockets, Sea Mines, Shells and Torpedoes)

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Ammunition Component. Any part of a complete round of ammunition, such as a primer, cartridge, shell, etc. It is called "live" when loaded and "inactive" when inert
Ref: Anon, "Ammunition Inspection Guide," War Dept Tech Manual TM 9–1904(1944), p 899

Ammunition Inspection consists of tests to determine the current degree of serviceability or deterioration of ammunition as affected by the various conditions of manufacture, storage, handling, maintenance and renovation.
Ref: TM 9–1904(1944),pp 902–920

Ammunition Loading. See Loading of Ammunition

Ammunition Packing. See under Packaging, Packing, etc

Ammunition Priming Compositions. See Priming Compositions

Ammunition, Self-Destroying. In firing from guns(such as AA) against enemy aircraft, great damage could be caused if the shells which failed to explode in the air fell on friendly territory and exploded there. In order to prevent this, a feature is incorporated(either in the fuze or in the explosive train of a shell) which destroys such a shell in the air before it strikes the ground. Ammunition provided with this feature is called "self-destroying"
Ref: A.B.Schilling,PicArsn,Dover,NJ; private communication(1960)

Amonal 1. A Spanish expl proposed by Prof A.Blanco(See under Ammonal)

Amorçage(Fr). Priming; initiation

Amorçage(Explosif d') (Fr). Priming; initiating explosive

Amorce. A French word meaning primer, cap, detonator or fuse. In England the word has been used to mean a toy pistol cap. According to Ref 1, amorces consisting of a mixt of minute quantities of red phosphorus, K chlorate and gum are less dangerous than those contg MF. According to Ref 2, the word "amorce" is also applied to "snaps for bon-bon crackers." Amorces should not contain more than 60 grains(3.88g) of chlorate and 10 grains(0.65g) of red phosphorus per 1000. Some amorces contain Ag fulminate in an amount not exceeding 15 grains (0.97g) per 1000 amorces. Ref 3 gives the compn and prepn of some Ger amorces[See also PATR 2510(1958),p Ger 7]
Refs: 1)R.Hartenau,Sprengstoffe, Waffen und Munition, No 24,p 283(Sept 1909) & CA **4**,384(1910) 2)Marshall **2**(1917),611 3)BIOS Final Rept 1313(1947),pp 2–4

Amorce détonateur(Fr). Detonator; initiator

Amorce électrique(Fr). Electric primer; electric blasting cap

Amorce fulminate(Fr). Blasting cap

Amorce à percussion(Fr). Percussion cap

Amperometric Titrations(Polarometric Titrations). In a strict sense, the term "amperometric'' should be applied to titrations in which a polagraphic diffusion-controlled limiting current is measured, according to the procedures described in references 3 to 8. This method has to be differentiated from *galvanometric* titration of E.Salomon [ZPhysikChem **24**,55(1897) & **25**,366(1898)], or the *dead-stop end-point* method of C.W. Foulk and A.Bawden[JACS **48**,2045(1926)]

Heyrovsky and Berezický(Ref 1) were the first to perform titrations based on the measurement of polarographic diffusion currents and they used the term *polarographic titration.* Majer(Ref 2) simplified the technique by measuring diffusion currents at a constantly applied emf rather than recording a series of polarograms, and proposed the name *polarometric titration.* Kolthoff and Pan (Ref 4) proposed the term *amperometric titration* as more consistent with the terminology applied to other electrometric titrations, such as potentionometric and conductometric

Amperometric titrations can be conducted either with a dropping mercury electrode or with rotating platinum electrodes. Both procedures are described by Laitinen (Ref 8) and by others(see References given by Laitinen)

Refs: 1)J.Heyrovsky & S.Berezický, CollCzechoslovChemCommun **1**,19(1929) 2)V. Majer, ZElektrochem **42**,120, 122(1936) 3)I.M.Kolthoff & Y.D.Pan, JACS **61**,3402 (1939) 4)I.M.Kolthoff, TransElectrochem Soc **78**,191(1940) 5)I.M.Kolthoff & H.A. Laitinen, "pH and Electro-Titrations,'' J.Wiley,NY(1941) 6)I.M.Kolthoff & J.J. Lingane, "Polarography,'' Interscience,NY (1941) 7)J.T.Stock, Analyst **72**,291(1947) 8)H.A.Laitinen, AnalChem **21**,66–70(1949) (Amperometric Titrations; 59 references)

Amphibious Cargo Vehicle(BARC). See under Amphibious Trucks, Tractors and other Vehicles

Amphibious Devices for Tanks. During landing operations troops and their equipment are particularly vulnerable to enemy fire until they have arrived on the beach and have been able to set up their weapons for defence. Amphibious trucks(qv) were designed for bringing troops, weapons and supplies, and amphibious devices were developed to allow the tanks to move from ships to shore a few miles away. The first of these devices, called **DD Device**, was developed in England and then produced in the US for the invasion of France during WW II. The device consisted of a canvas framework(resembling a boat) attached to the top of a tank, thereby giving it the necessary flotation. A propeller arrangement was attached to the tank engine so that the ensemble could move under its own power. When not in use, the canvas folded down on top of the tank. The disadvantage of this device was that the tank and its occupants were submerged about 20 ft under water and the tank weapons could not be fired while moving through the water. In addition, the canvas framework was very vulnerable to wave action

An improved amphibious device was developed in the USA. It consisted of metal boxes filled with plastic foam and attached to the front, rear and sides of the tank in such a manner that they could be detached from the tank by the crew without getting out. The tank was propelled through the water at speeds up to 6 mph by simply driving the tanks in the normal manner. The advantages of this system were that the tank turret was above water and the weapons could be fired if necessary on approaching the enemy shore. This device was used for light and medium tanks

(See also Amphibious Vehicles and Amphtrack)

Ref: G.B.Barnes, "Weapons of World War II,'' Van Nostrand,NY(1947),232–3

Amphibious Vehicles are vehicles which can propel themselves through water and on land. Among these vehicles may be listed *amphibious trucks* which were made by installing amphibious bodies(resembling boats) on trucks. The smallest truck was ¼ ton and the largest 2½ tons. The latter truck was nicknamed

DUKW(Refs 1 & 2). These vehicles proved to be very valuable during WW II for bringing men, weapons and supplies ashore from ships anchored beyond the range of enemy land-based guns. The amphibious vehicle **BARC** is the largest wheeled vehicle(60 tons) utilized by the US Army. When in the water it resembles a boat. The BARC can haul large bulky cargoes(See also Amphtrack)
Refs: 1)G.B.Barnes, "Weapons of World War II," Van Nostrand,NY(1947),282–5 2)M.L.Worley,Jr, "A Digest of New Developments in Army Vehicles," The Military Service Publg Co,Harrisburg,Pa(1958),248–51

Amphipathic Substance. Same as Surface-active Substance

Amphoteric Substance. A substance having both acid and basic props, such as amino-acids, Al hydroxide, etc
Ref: Hackh(1944),50

Amphtrack. An amphibious track vehicle developed in the USA during WW II. This vehicle could land troops, carry supplies and weapons across the water and its armament is used for returning fire in the face of intense hostile resistance. Several models are described by V.J.Croizat in the Army Ordn **31**,265–7(Nov–Dec 1946)

Ampoule(Chemical) (also Ampule or Ampul). In addition to the usual meaning of a small sealed vessel for holding a liquid, this term was found listed without definition in a British paper entitled a "Comprehensive List of Government Explosives," 1955,Admiralty BR 819(1B/54),War Office Code No 11155

AMR–2504 is a code name for a rubber composite propellant described in classified "Propellant Manual SPIA/M2," The Johns Hopkins Univ,Silver Spring,Md(1959),Unit No 515

AMT–2035AX–3; AMT–2091–AX; AMT–2096–4AX; AMT–2106–AX and AMT–2109–BT are code names for fuel-oxidizer propellants described in classified "Propellant Manual SPIA/M2," (1959), Unit Nos 470, 473, 474, 475 and 476

AMTB. See Anti-Motor-Torpedo Boat

Amvis(Explosive). A Brit "permitted" expl patented in 1896 by W.J.Orsman and manufd beginning 1897 for some time by the Roburite Explosives Co. Its compn was: AN 88 to 91, DNB or chloronaphthalene 4 to 6 and wood flour 4 to 6%. It was packed in paper cartridges waterproofed by means of ceresine. A detonator contg 1 g of 95/5–MF/$KClO_3$ mixt was used for its initiation. Colver(Ref 2) gives the following compn for Amvis Powder: AN 90, chlorodinitrobenzene 5 and wood meal 5%
Refs: 1)Daniel(1902),27 2)Colver(1918),145

AMYL ACETATES

Amyl Acetate or Amylacetic Ester(commonly known as Banana Oil),$C_5H_{11}\cdot COOCH_3$, mw 130.18. Several isomers are known of which n-amyl acetate and isoamyl acetate are the most important. They can be prepd by heating amyl alcohols with acetic acid in the presence of some sulfuric acid. Industrial methods of prepn are given in Ref 9,pp 102–3. Commercial products are usually mixts of n- and iso-amyl acetates. When amyl acetate is prepd from "fusel oil" (a by-product obtained in the manuf of ethyl alc by fermentation), the chief component is isoamyl acetate

n-Amyl Acetate,$CH_3\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH_2\cdot COOCH_3$. Col liq with a pear- or banana-like odor, d 0.879 20°/20°, mp –70.8°, bp 148.4° at 737 mm, fl p(closed cup) 77°F(25°), ignition temp 750°F(399°), LEL(lower expln limit) in air 1.1% by vol, $n_D^{20°}$ 1.4012. Sl sol in w(0.2% at RT) and misc with alc or ether. Prepd from n-amyl alcohol and acetic acid in the presence of some sulfuric acid. Its Q_c^p is 1004.9 cal/g and Q_f^p 1011 cal/g(Ref 7,p 310)

iso-Amyl Acetate,$(CH_3)_2CH\cdot CH_2\cdot CH_2\cdot COOCH_3$. Col liq with banana-like odor, d 0.876 15°/4°, mp –78.5°, bp 142° at 757 mm, fl p (closed cup) 92°F(ca 33°), ignition temp 715°F(ca 380°). Prepd from iso-amyl alcohol and acetic acid in the presence of some sulfuric acid.

Amyl acetates are dangerous when exposed to flame or heat. When heated they emit acrid fumes. Their explosive hazard is moderate(Ref 8). Toxicity is discussed in Refs 6 & 8. Ritter et al(Ref 3a) detd the temp at which mixts of air with satd vapor of amyl acetate(in contact with some of the liq) in a stoppered flask are explosive. Pressure of expln was also detd. Amyl acetates are used for the manuf of fruit essences and as high-boiling solvent constituents of lacquers and other coating materials(Refs 5 & 9). They were also used as colloiding agents for NC(Refs 2, 3 & 4) employed in the manuf of smokeless propellants. For instance, one of the US shot-gun propellants was prepd by agitating at RT(in a jacketed vessel) a pulped, wet NC(contg 5% Ba nitrate & 2% K nitrate) with an emulsion of amyl acetate and water(contg some Ba- and K nitrate). After allowing to stand for a few minutes, the steam was turned into the jacket and agitation continued for 5–6 hours during which time most of the amyl acetate and some water were distilled off. The contents of the vessel were then run out and the grains of propellant dried and sieved(Ref 3)

Refs: 1)Beil **2**,131–2,(60–1) & [143–4] 2)Marshall **1**(1917),336 3)Barnett(1919),82 3a)R.Ritter et al, Jahresber CTR **8**,201–2 (1930) & CA **26**,4474(1932) 4)Davis(1943), 321 5)Kirk & Othmer **5**(1950),826 6)Ibid **7** (1951),854 7)P.Tavernier, MP **38**,310 (1956) 8)Sax(1957),287 9)Faith,Keyes & Clark(1957),102–3 10)US Specification TT–A–516(Amyl acetate for use in organic coatings)

iso-Amylacetyl Azide(Isoamyl-essigsäure-azid, in Ger),$(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}N_3$, mw 141.17, N 29.77%. Oil with a pungent odor, puffs off when heated on a spatula; easily sol in alc, ether and some other org solvents. Can be prepd by treating an aq soln of the HCl salt of isoamylacetylhydrazide with $NaNO_2$ + HCl

Refs: 1)Beil – not found 2)T.Curtius, JPraktChem **125**,159–60(1930) & CA **24**, 3217(1930)

AMYL ALCOHOLS

Amyl Alcohol, $C_5H_{11}OH$, mw 88.15. Eight isomers are known, all of them liquid except 2,2-dimethylpropanol, which is a solid. They are sl sol in water and miscible with oils or org solvents, such as alcs, esters, ethers, ketones and aromatic hydrocarbons. Its most important isomers are:

n-Amyl Alcohol; 1-Pentanol or n-Butylcarbinol,$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}OH$. Col liq with a mild odor, d 0.824 20°/20°, mp –79°, bp 138.1°, fl p(closed cup) 100°F (ca 38°), ignition temp 700°F(371°), LEL 1.2% by vol in air, $n_D^{25°}$ 1.4581 and sp heat 0.712 cal/g. Soly in w 2.7g per 100 ml at 22°. Its Q_c^p given in Hodgman's Handbook, is 793.7 kcal/mol, while its Q_f^p calc by Tavernier(Ref 6,p 309), is 980 cal/g

iso-Amyl Alcohol; iso-Butylcarbinol or 3-Methyl-1-butanol,$(CH_3)_2{\cdot}CH{\cdot}CH_2{\cdot}CH_2{\cdot}OH$. Col liq with a mild odor, d 0.813 15°/4°, mp –117.2°, bp 132.0°, fl p(closed cup) 114°F(ca 46°), ignition temp 450°F(232°), $n_D^{20°}$ 1.41

Amyl alcohols can be prepd either from fusel oil or by a synthetic method which involves hydrolysis of amyl chloride, which in turn is prepd by the chlorination of a mixt of pentane and isopentane obtained from petroleum. The alc prepd by synthetic method has, according to Ref 5,p 147, the following props: d 0.812 to 0.820 20°/20°, boiling range 120 to 130°, $n_D^{20°}$ 1.409 and fl p(open cup) 113°F(45°)

Industrial methods for the prepn of amyl alcohols are discussed in Ref 8

Their fire hazard and toxicity are given in Ref 7. The explosion hazard of amyl alcohols is moderate when exposed to flame(Ref 7). Ritter et al(Ref 3a) detd the temp at which mixts of air with satd vapor of amyl alcohol(in contact with some of the liq) in a stopped flask is explosive. Pressure of expln was also measured

Amyl alcohols are used as solvents for lacquers

and for the manuf of amyl acetates. At the end of WW II amyl alcohols and their derivatives were replaced to a great extent by other solvents such as methyl-isobutyl ketone and butyl alcohols and their derivatives(Ref 8,p 113)

Amyl alcohol was used in France, beginning in 1896–7, as a stabilizer for the military propellant called Poudre B and were known as *Poudres B(AM)*. The propellants were prepd from a mixt of sol and insol NC gelatinized with ether-alcohol to which some amyl alcohol was added. After incorporation, the mass was worked between rollers at 70° and the rolled sheets either cut into strips or extruded through a die into ribbons. It was then dried at 50° and washed with water. Amyl alc being less volatile than ether-alc remained in the finished product. *Poudre B (AM_2)*, used for small caliber guns, contained 2% of amyl alcohol, whereas *Poudre B(AM_8)* used for large naval guns contained 8% of amyl alcohol as a stabilizer. These propellants became porous in storage because part of the stabilizer evaporated. Experience showed that amyl alcohol acted only as a temporary stabilizer. Several disastrous explns of Poudres B(AM) occurred. The most notable were the total destruction of the French battleships, the Iéna in 1907 and the Liberté in 1911. At first the explns were attributed to appreciable loss of the stabilizer because of volatility. Later, it was realized that while amyl alcohol reacts with nitrogen oxides liberated from deteriorating propellant to form amyl nitrate or nitrite, these in turn were not very stable in the presence of acidic decomposition products and would decomp with the liberation of oxides of nitrogen which would further accelerate the decomposition of the propellant. Since any aliphatic alcohol could be expected to behave similarly, subsequent stabilizers were made from aromatic compds or derivatives which formed stable NO_2 compounds with oxides of nitrogen

Refs: 1)Beil **1**,383–5, 388, 392–3(193–6) & [416, 418, 420–2, 426] 2)Marshall **1**(1917), 295 3)Barnett(1919),81 3a)F.Ritter et al, JahresberCTR **8**,201–2(1930) & CA **26**,4474 (1932) 4)Davis(1943),307–10 5)Kirk & Othmer **1**(1947),844–9 6)P.Tavernier,MP **38**, 309(1956) 7)Sax(1957),288–9 8)Faith, Keyes & Clark(1957),106–14 9)US Specification TT–A–56(Secondary amyl alcohol for use in organic coatings)

Note: Nitration of amyl alcohol with mixed acid at low temp to obtain $C_5H_{11}NO_3$ is described by J.B.Hinkamp et al, USP 2,618,650 (1952) & CA **48**,1412(1954)

AMYLAMINE AND DERIVATIVES

Amylamine; Aminopentane; Aminomethylbutane, $C_5H_{11}\cdot NH_2$. Several isomers are known, including *Isoamylamine*, $(CH_3)_2\cdot CH\cdot CH_2\cdot CH_2\cdot NH_2$. They can be prepd by the reaction of amyl chlorides with ammonia in the presence of alcohol as a mutual solvent. Datta & Chatterjee(Ref 2) detd expln temps of *amylaminepicrate* and *amylaineperchlorate* and found them to be 270° and 262°, respectively

Refs: 1)Beil **4**, 175, 177–8,180,(377–80) & [641, 643–5] 2)R.L.Datta & N.R.Chatterjee,JCS **115**,1007–8(1919) 3)C.K.Hunt, IEC **35**,1050–2(1943) 4)Kirk & Othmer **1**, (1947),849–51

[α-Nitroisoamyl]-isonitramine, called by Traube *Nitropentylisonitramin*, $(CH_3)_2CH\cdot CH_2\cdot CH(NO_2)(N_2O_2H)$. Prepd as the Na salt from 4-nitro-2-methylbutane, 2 mols $NaO\cdot C_2H_5$ and NO. By treating aq solns of the Na salt with $Ba(OH)_2$(or $BaCl_2$), $CuSO_4$ and $Pb(CH_3COO)_2$, ppts of explosive Ba-, Cu- and Pb-nitroisoamylnitraminates were obtained

Refs: 1)Beil **1**,687 2)W.Traube,Ann **110**, (1898)

Amylazide; Azidopentane; Azidomethylbutane or Methyltriazobutane, $C_5H_{11}N_3$, mw 113.16, N 37.14%. The isomer *1-Azidopentane* or *d-1-Azido-2-methylbutane*, $N_3H_2C\cdot CH(CH_3)\cdot CH_2\cdot CH_3$, liq bp 72° at 138 mm, d 0.8770

at 25/4°(Ref 2) & 0.8695 at 25°(Ref 4); $n_D^{25°}$ 1.4240(Ref 2) & 1.4248(Ref 4); Q_c^p at 25°, 7794.6 cal/g or 882.1 ± 0.5 kcal/mol; Q_f^p −36.1 kcal/mol(Ref 5). Refs 2 & 3 give optical rotations and Ref 4 viscosities from 15 to 45°. The expl props of amylazide were not investigated

Refs: 1)Beil – not found 2)P.A.Levene et al, JBiolChem **115**,415(1936) & CA **30**, 8175(1936) 3)P.A.Levene & H.Rothen, JChemPhys **5**,985 & CA **32**,1151(1938) 4)O.L.I.Brown & H.E.Cary, Memorandum Rept No 112, US Naval Powder Factory, Indian Head, Md(1956) 5)J.W.Murrin & G.A.Carpenter, Memorandum Rept No 129 USNPF (1957)

Amyleneozonide,$C_5H_{10}O_3$; col viscous liq explg on moderate heating. Was prepd by ozonization of amylene dissolved in dry hexane cooled in salt-ice mixt. The resulting mixt was distilled in vacuo to remove the hexane. The product was called "Normales Ozonid." Another ozonide corresponding to the formula betw $C_5H_{10}O_3$ & $C_5H_{10}O_4$ was prepd from the crude product as described in Ref 2,p 3100. This ozonide expd more violently than the $C_5H_{10}O_3$

Refs: 1)Beil – not found 2)C.Harries & K.Haeffner, Ber **41**,3099–3100(1908) & CA **3**, 66(1909)

Amylether; Diamylether or Amyloxide, $(C_5H_{11})_2O$, mw 158.28. Several isomers are known. The commercial product is a mixt, principally of isoamyl ether and n-amylether, formed as a by-product in the manuf of amyl alcohols from amyl chloride(Ref 2). This ether is a clear and sl yell liq, d 0.78–0.81 20°/20°, mp <75°, boiling range 165–210°, fl p(open cup) ca 135°F(57°), $n_D^{20°}$ 1.42. Very sl sol in w and miscible with alc or ether. Its fire hazard is moderate when exposed to heat or flame. Toxicity details unknown(Ref 4). It is used principally as a solvent. When mixed with 15–20% of ethanol it dissolves ethylcellulose but not NC

Refs: 1)Beil **1**,401,(199) & [432] 2)Kirk & Othmer **1**(1947),846 3)Ibid **5**(1950), 862 & 874 4)Sax(1957),293

AMYLGUANIDINE AND DERIVATIVES

n-Amylguanidine,$CH_3(CH_2)_4{\cdot}NH{\cdot}C(:NH){\cdot}NH_2$, is listed in Beil **4**, [642]

1-Nitro-3-n-amylguanidine(NAmGu), $CH_3(CH_2)_4NH{\cdot}C(:NH){\cdot}NHNO_2$, mw 174.20, N 32.17%. Col leaflets, mp 98.8–99.3°; sl sol in alc, very diff sol in ether and cold w, decomp slowly in hot w. Was prepd by heating NGu at 60–70° with 10% aq soln of amylaniline. Its Q_c^v is 1004 kcal/mol(obs) and 884.8 kcal/mol(calcd)

Refs: 1)Beil **4**, [642] 2)T.L.Davis & S.B. Luce, JACS **49**,2304(1927) 3)A.D.Little, Report on the Study of Pure Explosive Compounds, Cambridge, Mass, v 4(1952), 541(C)

1-Nitro-3-tert-amylguanidine,$C_2H_5{\cdot}C(CH_3)_2{\cdot}NH{\cdot}C(:NH){\cdot}NHNO_2$. Col plates, mp 154.8–155.6°; sl sol in alc and very diff in ether and cold w, decomp slowly in hot w. Was prepd by heating NGu at 60–70° with a 10% aq soln of tert-amylamine

Refs: 1)Beil **4**, [644] 2)T.L.Davis & S.B. Luce, JACS **49**,2304(1927)

1-Nitro-3-iso-amylguanidine,$(CH_3)_2CH(CH_2)_2{\cdot}NH{\cdot}C(:NH){\cdot}NHNO_2$. Col ndls, mp 145.5–146.2°. Was prepd by heating NGu at 60–70° with a 10% aq soln of iso-amylamine

Refs: Beil–not found 2)T.L.Davis & S.B. Luce, JACS **49**,2304(1927)

Dinitroamylguanidine,$C_6H_{13}N_5O_4$, mw 219.20, N 31.95% – not found in Beil or CA through 1956(would probably be a weak explosive)

Amylmalonylazidic Acid(Isoamylmalonazidsäure, in Ger),$(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2{\cdot}CH(CO{\cdot}N_3){\cdot}COOH$, mw 199.21, N 21.10%. Lt yel oil expldg weakly when heated on a spatula. Diff sol in w, sol in alc and very sol in eth or chlf. Can be prepd by treating an aq soln of the K salt of isoamylmalonylhydrazidic acid with HCl+$NaNO_2$

Refs: 1)Beil – not found 2)T.Curtius & W.Wirbatz, JPraktChem **125**, 274(1930) & CA **24**, 3216(1930)

Amyl Nitrate(Mixed Isomers), $C_5H_{11}\cdot ONO_2$, mw 133.15, N 10.52%. Col to pale yel, liq with ethereal odor, bp 145 to 156°, d 0.99, fl p 118°F. Can be prepd by treating commercial amyl alcohol with mixed nitric-sulfuric acid

Its main constituent is *isoamyl nitrate*, $(CH_3)_2CH\cdot CH_2\cdot CH_2\cdot ONO_2$ called in Ger "Isoamylnitrat" or "Salpetersäure-isoamyl-ester." It is col to pale-yel liq, bp 147–8° and d 0.996 at 21.7°; very sl sol in w and insol in alc or eth. Was first obtained, accdg to Beil, in 1847–8 by Rieckner(Ref 1). A commercial method of prepn of isoamyl nitrate was patented during WW II by Olin (Ref 2). Vapor pressures in mm Hg at various temps are given for commercial product in Ref 4. They are 0.9 mm at 10°, 2.7 at 20°, 5.15 at 30°, 9.7 at 40°, 17.4 at 50°, 29.7 at 60°, 70.5 at 80°, 166 at 100°, 335 at 120°, 612 at 140° and 760 at 147.5°. Values given in Ref 5 are 1.6 mm at –10°, 2.4 at 0° and 3.3 at 10°

Commercial amyl nitrate has been used as an ignition accelerator for compression-ignition engine fuels (Ref 3). It also was investigated as a possible monofuel in rocket or ATO engines but found to be unsuitable. Amyl nitrate is capable of decompn on a Ni catalyst but the reaction is barely self-sustaining and large quantities of soot are deposited which quickly choke the pipes of motors(Ref 4).

Its toxicity and fire and explosion hazards are discussed in Ref 7

Refs: 1)Beil **1**, 403,(200) & [434] 2)J.F. Olin,USP 2,243,471(1941) & CA **35**,5511 (1941) 3)J.S.Bogen & C.C.Wilson, Petroleum Refiner **23**,118(1944) & CA **40**,6781 (1946) 4)A.C.Hutchinson, "The Use of Amyl Nitrates as Liquid Monofuels," ICI Ltd,Nobel Div,Stevenston,Ayrshire(1950) (a pamphlet) 5)T.E.Jordon, "Vapor Pressure of Organic Compounds," Interscience, NY(1954),pp 180, 193 & plate 5 6)R. Vandoni & M.Laudy,MSCE **40**,187(1955) 7)Sax(1957),295

Amyl Nitrite, $C_5H_{11}ONO$, mw 117.15, N 11.96%, exists in several isomeric forms of which *isoamyl nitrite*(Isoamylnitrit or Salpetrigsäure-isoamyl-ester, in Ger), $(CH_3)_2CH\cdot CH_2\cdot CH_2\cdot O\cdot NO$, is the most important. It is a pale yel liq with an ethereal odor, bp 99°, d 0.8528 at 20°/4°, $n_D^{20.7°}$ 1.38708. Sl sol in w and miscible with alc or eth. Can be prepd by treating isoamyl alcohol with a mixt of $NaNO_2 + H_2SO_4 + H_2O$ or by other methods(Ref 1). A detailed description of a lab procedure for the prepn of isobutyl nitrite is given in OrgSynth(Ref 3) and this method is suitable for the prepn of isoamyl nitrite Its Q_c^p is 812.64 kcal/mol and its Q_f^v is 44.66 kcal/mol(Ref 2). Its ignition temp in air is 408°F(209°) and in oxygen 396°F(202°)(Ref 4); temp range of flammability in air extends from 45.5° to 134.1°F(Refs 5 & 6). More info on limits of inflammability can be found in Ref 6

Amyl nitrite is used as a first aid treatment(by inhalation) until intravenous injections can be given. Its inhalation into the lungs presents, however, a danger because it might form an expl mixt with air(Refs 5 & 6)

Its fire and expln hazards are discussed in Ref 8 and its toxicity in Refs 8 & 9

Refs: 1)Beil **1**,402,(200) & [434] 2)J. Thomas, ZPhysChem **52**,348(1905) 3)Org-Synth **16**(1936),8 4)G.S.Scott et al,Anal Chem **20**,2381(1948) (Detn of ignition temp of combustible liquids) 5)M.G.Zabetakis et al,US BurMines RI 4824(1951) (Description of apparatus used for detn of limits of flammability of amyl nitrite in air and oxygen) 6)M.G.Zabetakis & G.W.Jones,US BurMines RI 4877(1952) (Flammability of amyl nitrite) 7)Kirk & Othmer **9**(1952),416 8)R.Chary, MP **37**,351ff(1955) (Cardiovascular props and the toxicity of nitrated derivs in their relation to the industry) (10 refs) 9)Sax(1957),295

Amylodextrin is an intermediate product of acid degradation(hydrolysis) of starch. It is

sol in water and gives a blue coloration with iodine. The final product of hydrolysis is D-glucose

Refs: 1)Merriam-Webster's Unabridged Dictionary(1951),p 92 2)Kirk & Othmer **12**(1954),767

Amyloid,$(C_6H_{10}O_5)_x$, mw $(162.14)_x$. Marshall (Ref 2,p 150) calls it "hydrocellulose" and gives its formula as $C_{12}H_{22}O_{11}$. It is a gelatinous cellulose hydrate produced when a freshly prepd soln of cellulose in concd sulfuric acid is diluted with w. Amyloid is also the name given to the following substances: a)Parchment paper formed by the action of sulfuric acid on sheets of cellulose b)substances produced in woody tissures as an intermediate stage in the process of lignification and c)a gummy substance found in seeds of the nasturtium and other plants

Marshall(Ref 2,pp 155–6) stated that nitration of "hydrocellulose" with mixed acid contg HNO_3 42.03, H_2SO_4 46.22, H_2O 11.50 and N_2O_4 0.25% gave a product contg about 13.3% N. Its soly in alc was 12.15% and its methylene-blue test(mg absorbed by 1g of nitrated product) was 2.4

Refs: 1)Beil – not found 2)Marshall **1** (1917),150 & 155–6 3)Hackh(1944),53 4)Merriam-Webster's Unabridged Dictionary (1951),92

Amylopectin is a branched polymer of the formula$(C_6H_{10}O_5)_x$. It is made up of 1500 or more glucopyranose units joined to each other through α-1,4-glucoside units. In addn to these normal or predominating linkages, an anomalous α-1,6-glucoside linkage is present at the point of branching in the ratio of about 1:25. Its structural formula is given in Ref 7, p 766. Amylopectin is a white mucilagenous substance present in starch granules together with amylose(qv). When starch is treated with water to obtain a paste, amylopectic forms a gelatinous soln which imparts to the paste its viscosity. Amylopectin may be separated from amylose by fractionating starch, as is briefly outlined under amylose, and then nitrated in a manner similar to starch (Ref 3). The nitrated product obtained by Ashford et al (Ref 4) contained 12.25% N and was sol in alc to the extent of 86%. Its alc-soluble portion had N=11.82%, whereas the insol portion had N=12.58%. The stability of amylopectin nitrate, judging by the Abel and Bergmann-Junk tests, was lower than for amylose nitrate or NC

In a later paper(Ref 5), Ashford et al described an amylopectin nitrate having N= 13.25% which was attacked by hot alkali to a smaller degree than amylose nitrate

Refs: 1)Beil – not found 2)Hackh(1944), 53 3)W.R.Ashford, et al, CanJRes **24B**, 242–3(1946) 4)Ibid, 250–3(1946) 5)Ibid **25B**,151–4(1947) 6)Merriam-Webster's Unabridged Dictionary(1952),92 7)Kirk & Othmer **12**(1954),764 & 766

Amylose(also called Polyamylose) is a linear polymer with the formula $(C_6H_{10}O_5)$. It consists of 200–1000 glucopyranose units joined together through α-1,4-glucoside linkages. Its structural formula is given in Ref 8, p 766. It is a white substance contained in the inner part of starch granules together with amylopectin(qv). Amylose may be separated from amylopectin by fractionating starch. Most existing methods of fractionating employ swelling agents which may bring about hydrolysis of glucosidic linkages so that the products isolated from starch may not be the true constituents of native starch

The "selective absorption method" employed by Ashford et al(Refs 5 & 6) in the study of starch avoids the use of harsh swelling agents but employs adsorbing agents (such as cotton activated charcoal or Fuller's earth) to remove amylose from amylopectin when in aq soln. The cotton-amylose adsorbate is formed instantly when a cold corn starch paste(2%) is brought into contact with cotton and can be washed free of amylopectin with w. The cotton-adsorbate is then readily decomp by boiling w to give a clear soln of amylose and the solid is obtained by concg the soln to

ca 1/20th of the original vol at a temp not exceeding 55°, adding an equal vol of alc, separating the flocculent amylose by centrifuging and grinding the product under absol alc. The soln of amylopectin was concd to ca 1/40 at 50–55° and 20–25 mm Hg and treated with an equal vol of alc. The resulting flocculent ppt was centrifuged, dehydrated with alc, washed with ether and dried in a desiccator(Ref 5, pp 248 & 251–2)

Nitration of amylose was carried out according to the Will & Lenze method, using mixed nitric-sulfuric acid as described under nitration of starch(Ref 4). The resulting product had N=12.96%, soly in alc 20% and stability(by Abel and Bergmann-Junk tests) higher than for aminopectin nitrate(Ref 5). In the later paper(Ref 6), Ashford et al described amylose nitrate with N=13.25%. Hot alkali attacked it slightly more than amylopectin nitrate

The purpose of Ashford et al(Ref 5) in fractionating corn starch was to obtain homogeneous products giving on nitration more stable products than crude starch. This was accomplished to a certain degree. In order to make possible a more thorough study of the props of amylose and amylopectin, a fractionation of nitrated starch was undertaken and a study was made of the characteristics of the resulting amylose nitrate and amylopectin nitrate. Separation of these two products was achieved by fractional dissolution, employing alcohol as a solvent. A detailed description of the procedure is given in Ref 5,p 252. The separated nitrates of amylose and amylopectin had props similar to the nitrated starch fractions

Amylose and its nitrated starch products were also studied by Pringsheim et al(Ref 2). They claimed that nitration of amylose, even with a large excess of nitrating acid, always gave a mixt of compds nitrated to varying degrees. The yield of crude nitrates was always good. On superheating the solns, they often spontaneously underwent decompn with evoln of nitrous fumes. All the nitrates described in Ref 2 were unstable. They became yel in a desiccator and finally lost up to 75% of the N

Refs: 1)Beil – not found 2)H.Pringsheim, Ber **58**,1889–93(1925) & CA **20**,380(1925) 3)Hackh(1944),53 4)W.R.Ashford et al, CanJRes **24B**,242–5(1946) 5)Ibid 248–53 (1946) 6)W.R.Ashford & H.Hibbert,CanJRes **25B**, 153–4(1947) 7)Merriam-Webster's Unabridged Dictionary(1951),92 8)Kirk & Othmer **12**(1954),764 & 766(under Starch)

Amyloxide. See Amylether

Amylphthalate. See Diamylphthalate

iso-Amylpicrate or iso-Amyl-(2,4,6-trinitrophenyl)ether(Pikrinsäureisoamyläther, in Ger),$(O_2N)_3C_6H_2 \cdot O \cdot C_5H_{11}$, mw 299.24, N 14.04%. Nearly col hexagonal plates, mp 68–9°. It is decomp by air but may be stored in a vacuum; decomp by water or acids. Can be prepd by treating dipicrylsulfide with boiling sodiumisoamylate

Refs: 1)Beil **6**,290 & [281] 2)C.L.Jackson & W.F.Boos,AmChemJ **20**,452(1898) & JCS **74i**,517(1898) 3)C.L.Jackson & R.B.Earle, AmChemJ **29**,105(1903)

iso-Amylureidoacetyl Azide(Isoamyl-ureidoessigsäure-azid, in Ger), $(CH_3)_2CH \cdot CH_2 \cdot CH_2 \cdot NHCO \cdot NH \cdot CH_2 \cdot CON_3$, mw 213.24, N 32.85%. Wh solid; expl on heating but stable at RT; easily hydrolyzed. Can be prepd by treating isoamylureidoacetyl hydrazide with HCl + $NaNO_2$

Refs: 1)Beil – not found 2)T.Curtius et al, JPraktChem **125**, 199(1930) & CA **24**,3217(1930)

Amyrin or Amyrol, $C_{30}H_{50}O$. A cryst resinous substance occurring in some gums. It exists in α- and β-forms(Ref 1). On treating their solns in CCl_4 with ozone, Ruzicka et al(Ref 2) obtained powdery substances corresponding to the formula $C_{30}H_{50}O_{16}$. They were called α- and β-amyrin ozonides. Both substances were stable at RT but decompd ca 100°. These ozonides were probably mild expls

Refs: 1)Beil **6**,593(304) & [568–70] 2)R. Ruzicka et al, Ann **471**,32(1929)

AN. Abbreviation for Ammonium Nitrate

AN–507. A resin-based solid rocket propellant contg Amm perchlorate 75 and fuel 25%. The fuel consists of styrene 50 & "A–10 Polyester Resin'' 50%. The d of AN–507 is 0.058 lb/in.^{2}, Isp 195 secs, burning rate 0.365 in/sec, temp sensitivity 0.16%/°F. Its smoke is light
Ref: Armament Engrg(1954),42

AnAc. Ger designation of ethylideneaniline (Äthylidenanilin, in Ger), $CH_3{\cdot}CH{=}N{\cdot}C_6H_5$, which was used during WW II as one of the numerous fuels in liquid propellants
Ref: Dr H.Walter, PicArsn; private communication

AN–525; AN–525J; AN–557; AN–565J; AN–579Y; AN–581W; AN–583AF; AN–584J. Code names for cast fuel-oxidizer powders described in classified "Propellant Manual SPIA/M2,'',The JohnsHopkinsUniv,Silver Spring,Md(1959), Unit Nos 2, 255, 299, 256, 356, 357, 358 & 359

AN–586Y; AN–628BF; AN–2011; AN–2017; AN–2030; AN–2035AX; AN–2502EB. Code names for fuel-oxidizer propellants described in classified "Propellant Manual SPIA/M2'' (1959), Unit Nos 477, 478, 257, 298, 360, 479 & 489

Anagon Powder(Anagon-Sprengpulver, in Ger). According to Escales(Ref 1) it is a mixt of neutral inorg nitrates with pulverized Al, charcoal & alizarin or resinified linseed oil. Médard(Ref 2) gives compn of Anagon as: AN 84.5, K nitrate 1.5, Al 5.5, charcoal 8.0 & Ba nitrate 0.5%
Refs: 1)Escales, Ammonsprengstoffe(1909), 104 2)L. Médard,MAF **22**,596(1948)

"Analmatic'' is an automatic lab system which does away with routine lab work. This system was invented by the Ger firm Achema and is now available in US through Chicago Apparatus Co
Ref: Anon, C&EN **36**, 48(9 June 1948)

ANALYTICAL CHEMISTRY

Following is a selected list of books on this subject:
1)Collective, "Allen's Commercial Organic Analysis,'' Blackiston, Philadelphia, vols 1–10(1923–33) 2)W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis,'' Van Nostrand, NY(1939) 3)F.P.Treadwell & W.T.Hall, "Analytical Chemistry,'' Wiley, NY(1937–1942) 4)I.M.Kolthoff et al, "Volumetric Analysis,'' Interscience,NY, 1(1942), 2(1947), 3(1957) 5)A.L.Olsen & J.W.Greene, "Laboratory Manual of Explosive Chemistry,'' Wiley,NY(1943) 6)F.D.Snell & F.M. Biffen, "Commercial Methods of Analysis,'' McGraw-Hill,NY(1944) 7)R.E.Burk, edit, "Recent Advances in Analytical Chemistry,'' Interscience,NY(1949) 8)W.G.Berl, "Physical Methods in Chemical Analysis,'' Academic Press,NY(1950) 9)H.H.Willard et al, "Instrumental Methods of Analysis,'' Van Nostrand,NY(1951) 10)A.I.Vogel, "A Textbook of Quantitative Inorganic Analysis,'' Longmans,Green,London(1951) 11)D.F. Boltz, edit, "Selected Topics in Modern Instrumental Analysis,'' Prentice-Hall,NY (1952) 12)I.M.Kolthoff & E.B.Sandell, "Textbook of Quantitative Organic Analysis,'' Macmillan,NY(1952) 13)M.Pesez & P.Poirier, "Méthodes et Réactions de l'Analyse Organique,'' Masson,Paris(1952) 14)I.Specht, "Quantitative Anorganische Analyse in der Technik,'' VerlagChemie, Weinheim(1953) 15)J.J.Lingane, "Electroanalytical Chemistry,'' Interscience,NY (1953) 16)W.F.Hillebrand & G.E.F.Lundell, "Applied Inorganic Analysis,'' Wiley,NY (1953) 17)S.R.Young, "Industrial Inorganic Analysis,'' Wiley,NY(1953) 18)Collective, "Organic Analysis,'' Interscience,NY, v 1 (1953), v 2(1954) & v 3(1956) 19)F.Feigl, "Spot Tests,'' Elsevier,Amsterdam(1954) (translated by R.E.Oesper) 20)C.W.Griffin, "Inorganic Quantitative Analysis,'' Blakiston,Philadelphia(1954) 21)L.F.Hamilton & S.G.Simpson, "Calculations of Analytical Chemistry,'' McGraw-Hill,NY(1954)

22)G.W.Ewing, "Instrumental Methods of Chemical Analysis," McGraw-Hill,NY(1954) 23)J.H.Harley & S.E.Wiberley, "Instrumental Analysis," Wiley,NY(1954) 24)C.R.N. Strouts, J.H.Gilfilan, "Analytical Chemistry," ClarendonPress,Oxford, vols 1 & 2(1955) 25)G.Charlot & D.Bézier, "Méthodes Modernes d'Analyse Quantitative Minérale," Masson, Paris(1955) 26)R.Belcher & C.L.Wilson, "New Methods in Analytical Chemistry," Reinhold,NY(1955) 27)A.F.Dagett & W.B. Meldrum, "Quantitative Analysis," Heath, Boston(1955) 28)M.G.Mellon, "Quantitative Analysis," Crowell,NY(1955) 29)W. Wagner, C.J.Hall & G.E.Markle, "Advanced Analytical Chemistry," Reinhold,NY(1956) 30)S.Siggia & H.J.Stolten, "An Introduction to Modern Organic Analysis," Interscience, NY(1956) 31)R.L.Shriner, R.C.Fuson & D.Y.Curtin, "The Systematic Identification of Organic Compounds," Wiley,NY(1956) 32)G.Charlot & D.Bézier, "Quantitative Inorganic Analysis," translated from the French by R.C.Murray, Methuen Co,London (1957) 33)L.Meites, H.C.Thomas & R.P. Bauman, "Advanced Analytical Chemistry," McGraw-Hill,NY(1958) 34)L.F.Hamilton & S.G.Simpson, "Quantitative Chemical Analysis," Macmillan,NY(1958) 35)I.M.Kolthoff, P.J.Elving & E.B.Sandell, "Treatise on Analytical Chemistry," Interscience,NY, vol 1(in three parts) (1959) 36)C.L.Wilson & D.W.Wilson, "Comprehensive Organic Analysis," Elsevier,Amsterdam(1959) 37)Collective, "Anal Reviews of Analytical Chemistry," AnalChem **21**,196–84 (1949); **22**,2–153 & 206–71(1950); **23**, 1–257(1951); **24**,1–300(1952); **25**, 1–380 (1953); **26**,1–440(1954); **27**,1–476(1955); **27**,577 et seq(1956) (See also Activation Analysis, Chromatographic Analysis, Colorimetric Analysis, Microanalysis, Polarographic Analysis, Semi-Micro Analysis,etc)

Analytical Procedures for Acids. See under individual acids, such as Acetic Acid, Nitric Acid, Sulfuric Acid, etc and also under explosives and propellants for manuf of which these acids are used

Analytical Procedures for Explosives and for Primary Materials Used for their Manufacture are described under corresponding primary materials, such as Aniline, Anisole, Benzene, Toluene, etc

Analytical Procedures for Propellants are described under Propellants

Anarchists' and Revolutionists' Explosives and Weapons. See Explosives and Weapons Used by Anarchists and Revolutionists

Anasite. An expl compn consisting of AN, K nitrate, myrobalans and a small amt of agar-agar

Ref: CondChemDict(1942), 287(not listed in newer editions)

ANB. Code name for a cast double-base propellant described in classified "Propellant Manual SPIA/M2," JohnsHopkinsUniv, SilverSpring,Md(1959), Unit No 407

Anbenyaku. A Japanese expl: An 55 & DNB 45% used during WW II as a bursting chge in some projectiles. It was manufd at Nanman Arsenal,Manchuria, under the name of **Shobenyaku**(Refs 1 & 2). A blasting expl also called Anbenyaku was patented after WW II by Watanake. It consisted of AN 71.7, NG 8.0, CC 0.3 & powdered seaweed(Ref 3)

Refs: 1)G.C.Tibbitts et al, PB Rept 50394 (1945) 2)R.A.Cooley et al, PBL Rept 53045 (1945) 3)T.Watanabe, JapP **176**,113(1948) & CA **45**,4930(1951)

Anchorite. One of the Brit "permitted" expls: AN 34, Na Nitrate 33, TNT 12, Amm chloride 20 & moisture 1%; max chge 14 oz; ballistic pendulum swing by 4 oz of expl 2.73" vs 3.27" for std Gelignite contg 60% NG

Ref: Barnett(1919),132

Anderson's Explosives, patented in 1908 in Denmark had the following compositions: a)Pb peroxide 60 & PA 40% b)Pb peroxide 45, PA 20, TNT 10, GC 15 and NG 10%

Ref: Colver(1918),324–5

André's Explosive, patented in 1895, contained AN 85, K nitrate 3 and wood flour 12% cf Electronite No 2
Ref: Daniel(1902),28

ANE. Code name for a cast double-base propellant described in classified "Propellant Manual SPIA/M2" (1959), Unit No 408

Anello di forzamento(Ital). Driving band(of a projectile)

Anello graduato(Anello mobile) (Ital). Time ring(of a fuze)

Anello superiore della spoletta(Ital). Upper time train ring

Anesthetic Agents, Ignition and Explosion of. Some anesthetic agents used by inhalation (such as ether) may ignite or explode when brought in contact with air or oxygen. The subject of ignition and explosion of anesthetic agents is discussed in the following papers:
Refs: 1)M.Thalheimer, Anasthésie et Analgésie **4**,382–9(1938) & CA **32**,8781(1938) (Explns of anesthetics in operation rooms) 2)W.P.Morrill, BullAmAssocNurseAnesthetics **7**,302–9(1939) & **8**,285–95(1940); CA **34**, 1486(1940) & **35**,624(1941) (Hazards and prevention of anesthetic explns) 3)D.F.March, UnivCalifPubPharmacal **1**,369–74(1940) & CA **35**,2276(1941) (Explosibility of inhalation anesthetics and related compds) 4)H.B.Hass et al, Anesthesia and Anelgesia **20**,1–14(1941) & CA **35**,2217(1941) (Studies relating to anesthetic expln hazards) 5)J.W.Horton,Anesthesiology **2**,121–37(1941) & CA **35**,4208(1941) (Present status of the problem of preventing anesthetic explns) 6)B.A.Greene, Anesthesiology **2**,144–60(1941) & CA **35**,4954(1941) (The hazard of fire and expln in anesthesia) 7)W.P.Morrill, Hospitals **15**,No 4, 42–9(1942) & CA **36**,2413(1942) (Anesthetic explns–their incidence and prevention) 8)G.W. Thomas, et al, US BurMinesTechPaper **653**, 47pp(1943) & CA **38**, 487(1944) (Expln hazards of combustible anesthetics) 9)S.D. Miller, NebraskaStateMedJ **29**,9–11(1944) & CA **38**,3477(1944) (The expln hazard in anesthesia) (A lecture) 10)F.Cole,Surgery **18**,7–26(1945) & CA **39**,4487(1945)(Expln in anesthesia) (A review with 77 refs) 11)W.A.Low, ProcRoySocMed **44**,219–24 (1951) & CA **45**,6844(1951) (Fires and explns connected with anesthesia) 12)B.A.Ribeiro, Arqivos de Facultade de Hygiene e Saúde Pública da Universidade de São Paulo (Brazil)**6**,61–84(1952) & CA **47**,9013(1953) (A review with 53 refs) 13)A.S.Jackson, JInternlCollegeSurgeons **23**,398–401(1955) & CA **49**,7853(1955)(A discussion of the causes and prevention of explosions of inhalation anesthetics during their use)

Anethole, p-Allylphenylmethylether or p-Propenylanisole("Anisoin" or "Anise Camphor"),$CH_3{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}OCH_3$. Wh crysts tending to melt at warm RT. It was the first org compd to be treated with fuming nitric acid to produce a nitrocompound. This nitrocompd$[C_{10}H_{10}(NO_2)_2]_x$, mw 222.20, N 12.60%, was prepd in 1842 by Cahours(Ref 2) and is called in Beil "Dinitroanisoin," because the name of polymeric anethole $(C_{10}H_{12}O)_x$ is "anisoin". Anethole combines with PA to yield a mild expl, $C_{10}H_{12}O{+}C_6H_3N_3O_7$, red ndls(from alc), mp 70°(decomp)
Refs: 1)Beil **6**,566–70,(280–1) & [522–3] 2)A.Cahours,Ann **57**,73(1842) 3)Colver (1918),82–3

ANF–58. Code name of one of the liquid fuels of rocket propellants listed by Bloom, Jr, et al, without giving its compn
Ref: R.Bloom et al, JAMRocketSoc No 80, 14(1950) & CA **44**,8110(1950)

Angayaku. Japanese expl compns: a)AN/RDX–75/25, 78/22 or 84/16 b)GuN/RDX/AN–34/15/51 or 32/20/48 used during WW II. All of them were white, nontoxic mixts comparable in performance to amatols. The expls(a) were used for press-filling some bombs, while expls(b) were used for cast-loading some shells and bombs. It was claimed that expls(b) had low coefficient of shrinkage and therefore could be poured in quite large casts in a single pour, whereas

with similar expls based on TNT, it was necessary to break down between increments in the larger casts

Refs: 1)Anon, OpNav 30-3M(1945),27 2)Anon, Allied and Enemy Explosives,Aberdeen Proving Ground,Md(1946),135 3)G.C. Tibbitts et al, PB Rept 50394(1946),48 & 62 and Appendix A

Angeli, Angelo(1864–1931) An Italian professor of chemistry who made some contribution to the explosives industry(See "Angeli Test" below)

Ref: L.Cambi,Gazz **63**,527–60(1933) & CA **28**,1231(1934) (An obituary and a bibliography of Angeli's 238 publications from 1889 to 1931)

Angeli Test(Saggio Angeli in Ital) (Detection of Acidity in NC Propellants). The presence of small amts of acids, which are not easily detectable by indicator papers or by means of tests involving heating may readily be detd by the following procedure:

Cut ca 0.5g of sample in very thin slices (shavings), place them in a small beaker and add a few mls of distd w contg 3–4 drops of 0.2% alc soln of p-dimethylaminoazobenzene("butter yellow"), $(CH_3)_2N\cdot C_6H_4\cdot N{:}N\cdot C_6H_5$, serving as an indicator. Stir the mixt in cold and observe the color of shavings: if it is yel, the sample is alkaline, if it is red, the sample is acid and if the color is buff, the sample is neutral. In the last case it is advisable to heat the mixt slightly in order to be sure that the sample is neutral. If the sample is acidic, the degree of reddening indicates the extent of decompn of the propellent. If the red color appears and then disappears almost immediately, it might indicate that the material is in an advanced stage of decompn and that oxides of nitrogen, being evolved during the test, are attacking the indicator with probable formation of a compd such as $(CH_3)_2N{:}C_6H_4{:}N\cdot NH\cdot C_6H_5$(?) where R is the acid group(Ref 2)

This test can also be used for detg acidity or alkalinity of expls

If it is required to examine the decomposing effects of light on expls or propellants, the same test can be used and if the resulting coloration is too intensely red, a weaker soln of indicator is advisable

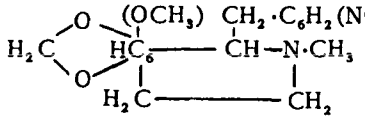
Refs: 1)A.Angeli,AttiRAccadLincei **27**, 164(1918); JSCI **37**,608A(1918) & CA **13**, 262(1919) 2)A.Angeli & G.Erani,Gazz **50** I, 139(1920) & CA **14**,2858(1920) 3)A.Angeli, SS **17**,115(1922) & CA **16**,4064(1922) 4) Molina(1930),428–30 5)Reilly(1938),90

Ångström, A.J.(1814–1874) A Swedish physicist, noted for optical research. He proposed a unit of wave length equal to 10^{-7} mμ or 10^{-1} mμ. This unit is called the ångström and is abbreviated to å, Å or AU

Ref: Hackh(1944),57

ANH. Code name of a cast double-base propellant described in classified "Propellant Manual SPIA/M2," JohnsHopkinsUniv, SilverSpring,Md(1959), Unit No 409

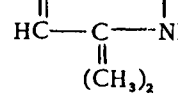
Anhydro-[cotarnine-(2,4,6-trinitrotoluene)] or 1'-(2,4,6-Trinitrobenzyl)-hydrocotarnine



mw 446.37, N 12.55%. Yel prisms, by pptg with MeOH from chloroformic soln; melts when slowly heated to ca 130° with expl decompn. Sparingly sol in most neutral solvents but readily dissolves in cold chlf. Can be prepd by grinding a mixt of TNT, alc and cotarnine

Refs: 1)Beil **27**,(459) 2)E.Hope & R. Robinson,JCS **99**,2133(1911)

Anhydrodiacetoneurea or 2-Oxo-4,4,6-trimethyltetrahydropyrimidine



when nitrated with mixed nitric sulfuric acid yielded **Trinitroanhydrodiacetoneurea**, $C_7H_9N_2O(NO_2)_3$, mw 275.18, N 25.45%. Wh ndls(from alc), which flash when heated in a flame. It is diff sol in cold w and decomp by boiling w. Its Ag and Ba salts are powerful expls

Refs: 1)Beil **24**,71 2)W.Traube & H. Lorenz, Ber **32**,3161–3(1899)

ANHYDROENNEAHEPTITOL AND DERIVATIVES

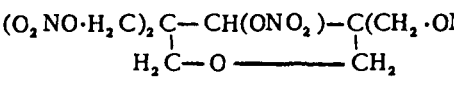
Anhydroenneaheptitol or Tetrahydro-3,3,5,5-tetrakis(hydroxymethyl)-4-oxypyrane, $C_9H_{18}O_6$, mw 222.23. Wh crysts (from alc), mp 156°. Can be prepd by the interaction of acetone with a large excess of formaldehyde in water in the presence of slaked lime as a condensing agent(Refs 1–4). Its Q_c^v is 1158.1 kcal/mol and Q_f^v 300.0 kcal/mol(Ref 5)

Refs: 1)Beil **17**,208 2)M.Appel & B. Tollens, Ann **289**,46–9(1896) 3)A.H.Blatt, OSRD Rept 2014(1944) 4)J.F.Walker, "Formaldehyde," Reinhold,NY(1953),226–7 5)L.Médard & M.Thomas, MP **38**,51 & 62(1956)

Anhydroenneaheptitol, Azido-$C_9H_{17}N_3O_6$ and Diazido- $C_9H_{16}N_6O_6$ Derivatives were not found in Beil or CA through 1956

Anhydroenneaheptitol, Mononitro- $C_9H_{17}NO_8$ Dinitro- $C_9H_{16}N_2O_{10}$ Trinitro- $C_9H_{15}N_3O_{12}$ and Tetranitro- $C_9H_{14}N_4O_{14}$ Derivatives were not found in Beil or CA through 1956

Anhydroenneaheptitol Pentanitrate(AEHP) or *Tetranitroxy-3,3,5,5-tetrakis(nitroxymethyl)-4-oxypyrane*, called also *2H-Pyran-3,3,5,5-(4H,6H)-tetramethanol; 2,2,4,4-Tetrakis (hydroxymethylnitrate)-1-pyrano-1-nitrate* (Pentanitrate de l'anhydro-ennéaheptite, in French),

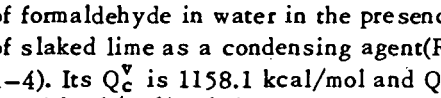


mw 447.23, N 15.64%, OB to CO_2 -30.4%, OB to CO +1.8%. Crysts, mp 132.5–136°; explodes at higher temps. Can be prepd by nitration of AEH as described in Refs 2 & 4. It was patented by von Herz for use in blasting caps(Ref 2). He claimed that AEHP possessed greater brisance and lower sensitivity than PETN. According to Blatt(Ref 3), the power of AEHP, detd by ballistic mortar, is 137% of TNT and the impact sensitivity, when detd with BurMines app and 2 kg wt, is 30 cm vs 16 cm for PETN. Médard & Thomas (Ref 5) give for Q_c^v at 18° 1105.2 kcal/mol and Q_f^v 184.1 kcal/mol

Refs: 1)Beil – not found 2)E.von Herz, GerP 856,527(1913) 3)A.H.Blatt, OSRD Rept 2014(1944) 4)J.F.Walker, "Formaldehyde," Reinhold,NY(1953),473 5)L.Médard & M.Thomas, MP **36**, 123–5(1954) & CA **50**, 3763(1956)

ANHYDROFORMALDEHYDEANILINE AND DERIVATIVES

Anhydroformaldehydeaniline; Trimeric Methyleneaniline or 1,3,5-Triphenyl-trimethylenetriamine,



Crysts, mp 140–3°; nearly insol in w; very sl sol in alcs more sol in eth and easily sol in chlf & benz. Its prepn is described in Refs 1–4. It has been used in plastics and as an accelerator in the vulcanization of rubber

Refs: 1)Beil **26**,3 & [3] 2)Ullmann **3**(1953), 494 3)Kirk & Othmer **11**(1953),874 4)Walker (1953),10 & 288

Anhydroformaldehydeaniline, Azido- $C_{21}H_{20}N_6$, Diazido- $C_{21}H_{19}N_9$ and Triazido- $C_{21}H_{18}N_{12}$ Derivatives were not found in Beil or CA through 1956

Nitroanhydroformaldehydeaniline, $C_{21}H_{20}N_4O_2$, mw 360.40, N 15.55%. Red solid, mp 136–41°; burns slowly in open flame leaving large amt of ash; sl sol in 95% alc. Was prepd by Shriner et al(Ref 2) in 90% yield by nitration with mixed 98% nitric acid and acetic anhydride, followed by pouring the mixt into crushed ice. The yield was only 50% when mixed nitric-sulfuric acid was used. The substance does not seem to possess expl props worthy of future testing

Refs: 1)Beil – not found 2)R.L.Shriner et al, OSRD Rept 2054(1943), 11 & 15 3)CA through 1956 – not found

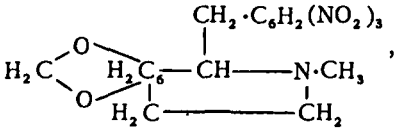
Dinitroanhydroformaldehydeaniline, $C_{21}H_{19}N_5O_4$ and higher nitrated derivs were not found in Beil or CA through 1956

Anhydroglucose, $C_6H_{10}O_5$; wh ndls, mp 117–18°; v sol in w, sol alc, v sl sol in ethylacetate. Was prepd by Fischer & Zach by hydrolysis of glucosides(Refs 1 & 2)

Note: It is logical to assume that this compd can be nitrated to form explosives. None of

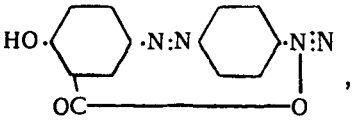
the nitrated derivs was, however, found in Beil or CA through 1956
Refs: 1)Beil **18**,386 2)E.Fischer & K. Zach, Ber **45**,459–61(1912)

Anhydrohydrastinine-[2,4,6-trinitrotoluene] or 1'-[2,4,6-trinitrobenzyl]-hydrohydrastinine,



mw 416.34, N 13.64%. Or-yel prisms(from ethyl acetate); mp decomp explosively ca 143°. Can be prepd by grinding a mixt of TNT, methanol and hydrastinine
Refs: 1)Beil **27**,[535] 2)R.Robinson & H.West, JCS **1926**,1987

Anhydro-(4-hydroxy-3-carboxy-azobenzene-4'-diazoniumhydroxide, called in Beil Anhydro-[4-oxy-azobenzol-carbonsäure-(3)-diazoniumhydroxyd-(4')]

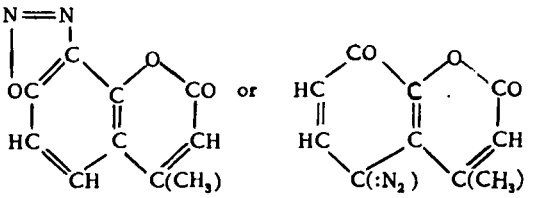


mw 268.23, N 20.89%. Brn-blk powder, mp – deflagrates at 130–2°; fairly stable; insol in most of org solvents; dissolves in aq pyridine with formation of salts. Can be prepd by the diazotization of anilinosalicylic acid as described in Ref 2
Refs: 1)Beil **16**,375 2)C.Bülow, Ber **44**, 608–10(1911)

1,4-Anhydro-D-mannitol. Same as Mannitan

1,5-Anhydro-D-mannitol. Same as Styracite

Anhydro-(7-oxy-4-methylcumarin-8-diazoniumhydroxide), called in Beil *8-Diazo-4-methylumbelliferon* and in Ref 2 *β-Methylumbelliferone-3-diazoanhydrid,*



mw 202.16, N 13.86%. Red-yel crysts of two isomers, one defl at 173–5°, the other(which is not always present) at 135–6°. The 1st isomer gives red prisms with PA, deflg at 176–80°, while the 2nd gives ndls deflg at 160–5°. Both isomers were prepd by treating 8-amino-4-methyl-umbelliferone(Beil **18**, 624) with $NaNO_2$ +HCl, as described in Ref 2
Refs: 1)Beil **18**,652 2)H.von Pechmann & J.Obermiller, Ber **34**,668–70(1901)

1,5-Anhydro-D-sorbitol. Same as Polygalite or Acerite, formerly called Styracite

3,6(?)-Anhydro-D-sorbitol, $C_6H_{12}O_5$, crysts, mp 113°, obtained by treating **anhydro-D-glucose** with Na amalgam in weak alk soln. It seems logical to assume that it can be nitrated to form expls but none of such products was found in Beil or CA through 1956
Refs: 1)Beil **17**,(129) 2)E.Fischer & K. Zach, Ber **45**,2070–1(1912)

Anhydrous Ammonia. See under Ammonia

Anhydrous Powder. Same as Baked Powder

Anilides are compds contg the monovalent C_6H_5NH-radical, such as acetanilide, $C_6H_5NH{\cdot}COCH_3$, nitranilide, $C_6H_5NH{\cdot}NO_2$, etc. Some of the nitrated products are expl
Refs: 1)Hackh(1944),58 2)Kirk & Othmer **1**(1947),913–14

ANILINE

(Aminobenzene or Phenylamine)

$C_6H_5NH_2$, mw 93.12, N 15.04%, OB to CO_2 -266%. Col oily liq, fr p -6.2°, bp 184.4°, d 1.022 at 20°/4°, n_D 1.5863 at 20°, sp ht 0.518 cal/g at 20-25°, at ht of vapn 113.9 cal/g, H_c^P 815 cal/g, H_c^V 810.4, fl p 168°F (75.5°) & vap press at various temps(Refs 4&6). It is sl sol in w and very sol in alc, eth or benz. Was first prepd in 1826 by Unverdorben. The usual method of prepn of aniline is the reduction of nitrobenzene with iron in dil HCl or ammonolysis of chlorobenzene. Tech methods for its prepn are given in Refs 4 & 10. Aniline was formerly used as a stabilizer for smokeless propellants but its strongly basic character and volatility are serious objections to such use (Ref 2). Snelling & Wyler(Ref 3) proposed the use of aniline as a sensitizer for AN. The principal commercial use of aniline is in the manuf of dyes and synthetic rubber additives(Refs 4 & 10), while its military uses are: a) as a fuel in liq rocket propellants(Refs 5a, 7, 9 & 12) b)as an intermediate in the manuf of diphenylamine and Centralites, employed as stabilizers in NC propellants (Refs 4 & 10) and c) as the starting material for the production of tetryl

Its higher nitrated compds are powerful expls (see below). Military requirements of aniline are discussed in US Spec MIL-A-10450A and its toxicity in Refs 4,7 & 11. Aniline is extremely poisonous and if adsorbed through the skin may lead to cyanosis and death (Ref 5)

Aniline forms salts, some of which are explosive (see below)

Refs: 1) Beil **12**, 59, (131) & [44] 2) Marshall **1**, (1917), 272 3)W. O. Snelling & J. A. Wyler, USP 1, 827,675(1931) & CA **26**,601(1932) 4)Kirk & Othmer **1**(1947), 914-20 4a)J.B.Willis,TrFaradSoc **43**,100 (1947)(Heat of combustion) 4b)W.C. Lothrop et al, JACS **73**,3583(1951)(Infrared spectra) 5)J.D.Clark,Ordn **36**,661-3(1952) 5a)Kirk & Othmer **11**(1953), 770 6)Jordan (1954), 181 & 196 7)S.Krop,JetPropn **24**, 224(1954) 8)M.Kilpatrick & L.L.Baker, "5th Symposium on Combustion," Pittsburgh 1954, published in 1954, pp 196-205 & CA **50**, 574(1956) 9)Sutton(1956), 168 & 177 10) Faith,Keyes & Clark(1957), 115-28 11)Sax (1957),300 12)Warren(1958), 21 & 25

Salts of Aniline with Inorganic Acids:

Aniline, Azidoderivatives. See under Azidoaniline

Anilinochlorate, $C_6H_7N + HClO_3$; col prisms exploding ca 75-6° or on impact; sol in w and v sol in alc or eth. Can be prepd by treating aniline with aq chloric acid. It is unstable

Refs: 1)Beil **12**,116-7 2)M.Beamer & F.W.Clarke,Ber **12**,1066(1879)

Anilinoiodate, $C_6H_7N + HIO_3$; scales, d 1.48 at 13°, expl at 125-30° on rapid heating but remains unchanged on slow heating; sol in hot w & v sol in hot alc. Can be prepd by treating aniline with aq iodic acid

Refs: 1)Beil **12**,117 2)M.Beamer & F.W. Clarke,Ber **12**,1066(1879)

Anilinonitrate, $C_6H_7N + HNO_3$; col crysts, mp 182-4°, yields nitraniline when heated to 190°, d 1.356 at 4°; very sol in w, alc or eth. Can be prepd by treating aniline with 70% nitric acid as described in Ref 2,p 1798. Its Q_c^V is 787.9 kcal/mol(Ref 2,p 100), Q_c^P 795.1 (Ref 3) and Q_f +42.5 kcal/mol(Ref 3)

Note: According to Daniel(1902),pp 462-3, aniline nitrate served in France for the prepn of a primary compd, *diazobenzene nitrate,* known in France as *aniline fulminante.* The reaction proceeded as follows:

$C_6H_5NH_2 . HNO_3 + HNO_2 \rightarrow C_6H_4N_2 \; HNO_3 + 2H_2O$

Nitrous acid was obtained by the interaction of nitric acid with arsenic acid

Refs: 1)Beil **12**,117,(141),[66] 2)J.B. Willis,TrFaradSoc **43**,98 & 100(1947) 3)T. L.Cottrell & J.E.Gill,JCS **1951**,1798-9

Anilinoperchlorate, $C_6H_7N + HClO_4$, plates,

decomp slowly at 180° and expl at 250° (Ref 3) or 275°(Ref 4); deton on impact, sol in w, alc, acet & hot AcOH, insol in eth. Can be prepd by treating aniline with aq perchloric acid

Refs: 1)Beil **12**,117,(141),[66] 2)M. Beamer & F.W.Clarke,Ber **12**,1066(1879) 3)R.L.Datta & N.R.Chatterjee,JCS **115**, 1008(1919) 4)F.Arndt & P.Nachtwey, Ber **59**,446(1926)

Anilinoperchromate, $C_6H_7N + HCrO_5$; crysts similar in appearance to $KMnO_4$; very explosive and unstable; sol in eth, insol in w and nearly insol in benz & ligroin. Can be prepd by treating aniline with aq perchromic acid, as described in Ref 2

Refs: 1)Beil **12**,117 2)O.F.Wiede,Ber **30**,2187(1897)

Dianilinocupric Nitrite, $2C_6H_7N + Cu(NO_2)_2$; green plates, deflagrates on heating to 85° or on treatment with liq ammonia or with cold concd sulfuric acid; insol in w, alc or eth. Can be prepd by treating aniline with the green soln obtained when a mixt of equiv solns of K nitrite and Cu sulfate is treated with alc and filtered(Ref 2)

Refs: 1)Beil **12**,[67] 2)H.J.S.King, JCS **1929**, 2593

p-N,N-Trilithioaniline, $Li.C_6H_4.N(Li)_2$; bright yel ppt; when dry expl violently on contact with air. Was prepd by adding an ethereal soln of n-butyllithium to p-bromaniline, as described in Ref 2

Refs: 1)Beil- not found 2)H.Gilman & C.G.Stuckwisch,JACS **71**,2933(1949) & CA **45**,5127(1951)

Salts of Aniline with Organic Compounds:

Aniline fulminante (Fr). Diazobenzene Nitrate (See Note under Anilinonitrate on previous page)

Anilinopicrate (AP),$C_6H_7N + C_6H_3N_3O_7$; yel crysts, darkened ca 168°, melted at 181° and exploded at 398°(Ref 3); d 1.558. Can be prepd by mixing equim quantities of aniline and picric acid in w

Note: According to Daniel (Ref 1a), aniline picrate was used in the following expls proposed by Street: a)AP 13.5, K chlorate 67.3, castor oil 9.6 & starch 9.6% b)AP 1.8, K chlorate 80.0, MNB 9.1 & castor oil 9.1%

Refs: 1)Beil **12**,120,(143), [72] 1a) Daniel(1902),742 2)O.Silberrad & G.Rotter JCS **89**, 169(1906) 3)R.L.Datta & N.R. Chatterjee, JCS **115**, 1008(1919) 4)T.Curtius & A.Bertho, Ber **59**, 583(1926)

Anilinotetranitrobenzene, $C_6H_7N + C_6H_2N_4O_8$, red ndls, very unstable(Beil **12**,115)

Anilinotrinitrobenzene, $C_6H_7N + C_6H_3N_3O_6$; red ndls, mp 123-4°; can be prepd by mixing equim quantities of aniline and 1,3,5-TNB in alc

Refs: 1)Beil **12**,115 2)P.Hepp,Ann **215**, 356-8(1882)

Anilinotrinitrotoluene, $C_6H_7N + C_7H_5N_3O_6$; red crysts, mp 83-4°, can be prepd by mixing equim quantities of aniline and 2,4,6-TNT in alc

Refs: 1)Beil **12**,115,[71] 2)P.Hepp,Ann **215**,365(1882) 3)C.A.Taylor & W.H.Rinkenback,JACS **45**,54(1923)

Nitrated Derivatives of Aniline

Mononitroanilines, $C_6H_6N_2O_2$, mw 138.12, N 20.28%, OB to CO_2 −150.6%. If the substitution takes place in the ring, $O_2N.C_6H_4.NH_2$, the compd may be called *nitroaniline, mononitroaniline or nitroaminobenzene.* If the substitution takes place in the $-NH_2$ group, the compd may be called *nitraniline, N-nitroaniline* or *phenylnitramine.* In order to avoid confusion the last group of nitrated anilines is described under *phenylamine*

Mononitroanilines or Nitroanilines, $O_2N.C_6H_4.NH_2$, exist in three isomeric forms: ortho-, meta-, and para-. They are commercially available and extensively used in industry. None of these isomers can be obtained in good yield by direct nitration of aniline and it is necessary to employ indirect methods such as the Holleman method of acetylation of aniline to acetanilide followed by nitration(Ref 2) or by ammonolysis

of chloronitrobenzenes, similar to the ammonolysis of chlorobenzene described in Refs 4 and 10, listed above under Aniline

o-(or 2-Mononitroaniline(o-MNA); yel-orange rhmb crysts, mp 71.5°, bp 284.1°, d 1.442 at 15°, Q_c^p 765.9 kcal/mol(Ref 3); for vap press at various temps see Ref 8. Other props and prepn are given in Refs 1,7,7a & 9

m-(or 3-)Mononitroaniline(m-MNA); yel, rhmb crysts, mp 114°, bp 306.4°, d 1 43, Q_c^p 766.3 kcal/mol(Ref 3) & 754.1(Ref 6), Q_f 16 kcal/mol; for vap press at various temps see Ref 8. Other props and prepn are given in Refs 1,4,7 & 9

p-(or 4-)Mononitroaniline(p-MNA); yel, monocl ndls, mp 146.7°, bp 331.7°, d 1.437 at 14°, Q_c^p 760.2 kcal/mol(Ref 3); for vap press at various temps see Ref 8. Other props and prepn are given in Refs 1, 4a,5,7&9

Mononitroanilines serve for the prepn of higher nitrated derivs, some of which are used in the expls industry

Note: According to Daniel (Ref 1a), commercial mononitroanoline was used in the following expls proposed by Street: a)MNA 13.5, K chlorate 69.4, azobenzene 14.3 & castor oil 2.8% b)MNA 7.7, K chlorate 79.1, MNB 11.0,CC 1.1 & castor oil 1.1%

Refs: (Mononitroanilines) 1)Beil **12**,687, (339) & [367] (o-MNA); Ibid 698,(345) & [374] (m-MNA); Ibid 711,(349) & [383](p-MNA) 1a)Daniel(1902),742 2)A.F.Holleman et al, Ber **44**,704 ff(1911) 3)F.Swarts, Rec **33**, 281-98(1914) 4)Marshall 1(1917), 273 4a)R.Robertson,JCS **119**,18(1921) 5)Org-Synth **9**,64-5(1924)& CollVol 1(1941),pp 388-9 6)W.H.Rinkenbach,JACS **52**, 116(1930) 7) Pepin Lehalleur(1935), 260 . 7a)Kirk & Othmer 1(1947),921-2 8)Jordan(1954),181 & 196 9)Sax(1957),943-4

N-Alkylnitroanilines, which contain groups such as Me, Et or iso-Pr and a nitrogroup in either the 3 or 4 position on the ring, were proposed(in quantities of 0.5 to 5%) as stabilizers of NC in smokeless propellants. A eutectic mixt of N-ethyl-and N-methyl-4-nitroaniline was found to improve the plasticizing qualities of propellants

Ref: J.A.Gallaghen & I.Pincus,USP 2,696,430 (1954) & CA **49**,5845-6(1955)

4-Nitro-N-nitrosaniline, $(O_2N)C_6H_4.NHNO$ also called 4-Nitro-phenylnitrosamine, is described under Phenylamine

Nitro-N-nitranilines, $(O_2N)C_6H_4.NHNO_2$, also known as *Nitrophenylnitramines,* are described under Phenylamine

Dinitroanilines(DNA)or Dinitroaminobenzenes, $(O_2N)_2C_6H_3.NH_2$, mw 183.12, N 22.95%, OB to CO_2 -91.2%. Six isomers exist of which the 2,4-DNA and the 3,5-DNA are of interest either as components of expl compns or as intermediates in the prepn of higher nitro derivs

2,3-Dinitroaniline, orange-yel ndls, mp 127°. Other props and prepn are given in Ref 1a

2,4-Dinitroaniline(2,4-DNA); yel, monocl prisms, ndls of plates; mp 186-8°, explodes when heated in a flame at ca 548°(Ref 26); d 1.615 at 14°; insol in cold w, v sl sol in hot w, sl sol in alc; Q_c^p 718.9 kcal/mol(Ref 4), Q_c^v(average) 711.5(Refs 13 & 14), Q_e(calcd) 103.2 and Q_f (calcd average) 18.1 kcal/mol(Refs 13 & 14); temp of expln (calcd) 2110°K(Ref 14). Was first prepd in 1870 by heating 4-chloro-1,3-dinitrobenzene with ammonia(Ref 1b). More recent methods of prepn are described in Refs 6,8&12. The X-ray diffraction pattern is given in Ref 11 and a spectrographic method for its detn in Ref 9

The 2,4-DNA is a weak expl. Its power is ca 88% TNT as detd by BalMort. Its expl props were investigated by Robertson(Ref 3), Rinkenbach(Ref 4) and by Wöhler & Wenzelberg(Ref 5). Its FI(figure of insensitiveness) is > 120% PA(Ref 3) and the 50% impact sensitivity figure with a 2-kg wt was reported as 12.8 m kg/cm² or 112% TNT(Ref 5). Its thermal stability at 140° is satisfactory

DNA was used by the Germans during WW II as an additive to TNT, presumably to render the TNT less brisant so that excessively small fragments would be eliminated (Ref 6a). Another reason for use of DNA was unquestionably to stretch the available supply of TNT

2,5-Dinitroaniline, orange-yel crysts, mp 137°. Other props and prepn are in Ref 1c

2,6-Dinitroaniline, yel ndls, mp 138-142°, or leaflets, mp 137°. Other props and prepn are in Ref 1d

3,4-Dinitroaniline, lemon-yel ndls (from w), mp 154°, Other props and prepn are in Ref 1e

3,5-Dinitroaniline(3,5-DNA), yel ndls, mp 158-61°, sl sol in cold w, sol in hot w, alc & eth; sl sol in benz(Ref 1f). Was prepd in 1891 by Bader(Ref 2) from 1,3,5-TNB and by Flürscheim(Ref 2a) who improved the method. A synthesis from 3,5-dinitrobenzoic acid utilizing the Schmidt reaction(Ref 7) is described in Ref 10. 3,5-DNA is a weak explosive, similar in props to 2,4-DNA. Nitration of 3,5-DNA yields a very powerful explosive, 2,3,4,5,6-pentanitroaniline

Blanksma & Verberg (Ref 5a) prepd 3,5-DNA and a number of its derivs, some of which proved to be explosive

Refs(Dinitroanilines): 1a)Beil **12**, 747 & [405](2,3-DNA) 1b)Beil **12**, 747, (361) & [405] (2,4-DNA) 1c)Beil **12**, 757, (365) & [413](2, 5-DNA) 1d)Beil **12**, 758, (365) & [413] 1e) Beil **12**, 758 (3,4-DNA) 1f)Beil **12**, 759, (366) & [414](3,5-DNA) 2)R.Bader, Ber **24**, 1654(1891) 2a)B.Flürscheim, JPraktChem **71**, 537(1905) 2b)R.L.Datta & N.R.Chatterjee, JCS **115**, 1007 (1919) 3)R.Robertson, JCS **119**, 18(1921) 4) W.H.Rinkenbach, JACS **52**, 116(1930) 5)L. Wöhler & O.Wenzelberg, AngChem **46**, 173(1933) 5a)J.J.Blanksma & G.Verberg, Rec **53**, 988-1000 & 1037-46(1934) 6)OrgSynth **15** (1935), 22 or CollVol **2**(1943), 221 6a)Anon, "Allied and Enemy Explosives," Aberdeen PG, Md(1946), 90 7)H. Wolff, "Organic Reactions," Wiley, NY, v 3(1946), 307 7a)Kirk & Othmer **1** (1947), 922 8)M.F. Bühler, AnalesAsocQuímArgentina **38**, 252-4 (1950) & CA **45**, 5116(1951) 9)L.S.Harrow, JAssocOffAgrChemists **33**, 390-6(1950) & CA **44**, 9155(1950) 10)W.C.Lothrop et al, JACS **73**, 3583(1951) 11)H.M.Rice & F.J.Sowden, CanJChem **30**, 575-80(1952) & CA **47**, 2567 (1953) 12)A.L.Beckwith & J.Miller, Australian JSciResearch **5A**, 786-9(1952) & CA **47**, 12333 (1953) 13)L.Médard & M.Thomas, MP **36**, 97-127(1954 & CA **50**, 3763(1956) 14)ADL Punch Cards (1954)(C) 15)Sax(1957), 628 16)Blatt, OSRD 2014(1944)

2,4-Dinitro-N-nitraniline or N,2,4-Trinitroaniline, $(O_2N)_2C_6H_3.NHNO_2$, also called *2,4-dinitrophenylnitramine*, is described under Phenylamine

Trinitroanilines(TNA) or Trinitroaminobenzenes, $(O_2N)_3C_6H_2.NH_2$, mw 228.12, N 24.56%, OB to CO_2 -56.1%. The following isomers are known of which only 2,4,6-TNA is of importance in the expl industry

2,3,4 Trinitroaniline(2,3,4-TNA); lt yel crysts, mp 190°. Can be prepd by oxidation of 2,3,4-trinitrodimethylaniline with chromic acid in AcOH

Refs: 1)Beil **12**, [419] 2)P.van Romburgh & D.W.Wensink, PrAkadAmsterdam **17**, 1036 (1915)

2,4,5-Trinitroaniline(2,4,5-TNA); lt yel ndls, claimed to be prepd from 3-nitroacetanilide as described in Ref 2 According to Ref 3 this procedure yielded the 2,3,4,6-tetranitroaniline instead of 2,4,5-TNA

Refs: 1)Beil **12**, 763 2)O.N.Witt & E.Witte, Ber **41**, 3095(1908) 3)C.F.van Duin & B.C.R. Lennep, Rec **39**, 148(1920)

2,4,6-Trinitroaniline(2,4,6-TNA) or Picramide. Orange-red (with bluish tinge) monocl-prism-crysts(from AcOH), mp 189-92°, bp- explodes; d 1.762 at 20°; Q_c^p (av) 678 4 kcal/mol (Refs 8 & 17), Q_c^v (av) 680 (Refs 7,8 & 17), Q_e 191.2(Ref 19) and Q_f(av) 21.6 kcal/mol(Refs 12 & 17) It is nearly insol in w, sl sol in alc or eth and sol in benz, acet, liq NH_3 or hot ethyl acetate (some numerical data is given below under solubility). Apparently was first prepd in 1854 by Pisani by treating picryl chloride with Amm carbonate(Refs 2 & 3). Witt & Witte prepd TNA by nitrating with concd nitric acid a soln of aniline in glacial AcOH (or in concd H_2SO_4) at about 5°, but the yields were poor. Better yields were obtained by nitrating mono- or dinitro- anilines (Ref 14). Other methods of prepn are described in Refs 13 & 15. Sancho(Ref 12a) described prepn of TNA, called in Span "picramina," at the Fábrica de Granada. Oglobin and Markina (Ref 20) described the separation of 2,4,6-TNA

from its mixts with mono- and dinitroanilines. Lothrop et al(Ref 18) detnd infrared spectra of 2,4-TNA. Toxicity is given in Ref 21

Following are some expl props of 2,4,6-TNA:

Brisance–no data found in open literature

Detonation Rate–by the Dautriche method, for compressed TNA in cardboard cartridges 30 mm diam and 3 mm thick: at d 1.5 7000m/sec for TNA vs 7300 for PA and at d 1.7 7600 vs 8200 for PA (Ref 16)

FI–see under Impact Sensitivity

Gap Test Value(Coefficient de self-excitation, in Fr). A paper cartridge 30 mm diam & 78.5 mm long contg 50g TNA(d ca 0.85) and initiated by 2g of MF caused the deton of another cartridge placed at a distance of 7.5cm (PA gave 13cm and TNAns 6.5cm in the same test)

Hygroscopicity- considerably higher than for TNT(Ref 16)

Impact Sensitivity. FI(figure of insensitiveness) 111% PA(Ref 6); impact work for 50% expln with 2kg wt is 10.4 kg m/cm^2(Ref 11); impact sensitivity by the French method with 10kg wt for 50% point 36cm vs 17cm for PA (Ref 16). Blatt(Ref 22) gives FI = 122% PA

Initiation Sensitivity(Sensibilité à l 'amorce, in Fr). A 50 g sample loaded at d 0.85 in a Kraft paper cartridge, 30mm in diam, required ca 1g MF for complete deton(Ref 16)

Power. The pressure bomb method gave ca 110%TNT(Ref 14, p 132), while the Trauzl test,Fr modif,gave a value(called CUP) equal to 92%PA(Ref 16). Blatt(Ref 22) gives Trauzl test value 98% TNT

Sensitivity to Impact. See Impact Sensitivity

Sensitivity to Initiators. See Initiation Sensitivity

Solubility. Desvergnes(Ref 10) gives solys in g per 100 g in the following solvents: water 0.106 at 19.5°, 0.120 at 50° & 0.123 at 100°; ethyl acetate 1.898 at 19.5°; acetone 4.798 at 19.5°; 96% alcohol 0.120 at 19.5°;abs alc 0.127 at 19.5°; methanol 0.245 at 19.5°; benzene 0.907 at 19.5°; chloroform 0.322 at 17°; ether 0.121 at 17°; pyridine 13.64 at 17°; CS_2 0.013 at 17.5°; CCl_4 0.003 at 17.5° and toluene 0.108 at 21.5°

Stability. According to Daniel(Ref 2a) stability of TNA is satisfactory and according to Robertson(Ref 6,p 13), the vacuum stability test at 140° gave values comparable to TNT

Temperature of Explosion is, according to Burlot & Tavernier(Ref 16,p 122), ca 3700°C

Uses. According to Daniel(Ref 2a) one of the first patents on the use of TNA in expls was issued to the Chemische Fabrik Griesheim as well as to Pierre & Pottgiesser. TNA has advantages over PA because it is nearly insol in w and it is not acidic. Marshall(Ref 4a) and Colver(Ref 5a) stated that although TNA is a powerful expl it has not been widely used. This probably because other HE's such as TNT, are more available and cheaper to produce. Sancho (Ref 12a)stated that TNA was used in mixts with TNT for cast loading some projectiles. Derivs of aniline are in great demand in industry other than as expls

Refs (Trinitroaniline): 1)Beil **12**,763,(368) 8 [421] 2)F.Pisani,CR **39**,853(1854) 2a) Daniel(1902),475 3)J.Meisenheimer & E. Patzig,Ber **39**,2534(1906) 4)O.Witt & E.Witte, Ber **41**,3091(1908) 4a)Marshall **1**(1917), 273 5)C.F.van Duin, Rec **37**,111(1917) 5a)Colver(1918), 364 6)R.Robertson,JCS **119**,13 & 18(1921) 7)Land-Börnst, Eg 3,T1 3(1923), 291 8)W.H.Rinkenbach,JACS **52**, 116(1930) 9)A.Holleman,Rec **49**,112-20(1930) & CA **24**,2440(1930) 10)L.Desvergnes, Rev-ChimInd **44**,34-7(1931) & CA **25**,2980(1931) 10a)Vennin, Burlot & Lécorché (1932),461 11)L.Wöhler & O.Wenzelberg,AngChem **46**, 173(1933) 12)A.Schmidt, SS **29**,262(1934) 12a)E.Sancho,"Química de los Explosivos," Aguado,Madrid(1941), 164-5 13)E.Macciotta, Gazz **71**,81-94(1941) & CA **36**,1593-4(1942) 14)Davis(1943) 132,137 & 173 15)E.Y. Spenser & G.F.Wright,CanJRes **24B**,204-7 (1946) & CA **41**,723(1947) 15a)Kirk & Othmer **1**(1947),p 922 16)E.Burlot & P. Tavernier,MP **31**,121-9(1949) & CA **46**,11685

(1952) 17)L.Medard & M.Thomas,MP **31**, 173-96(1949) & CA **46**,11684(1952) 18)W. C.Lothrop et al, JACS **73**,3583-4(1951) 19) ADL Punch Cards(1954)(C), Compd No 270 20)K.A.Ogloblin & G.V.Markina,ZhObshKhim **26**,93-101(1956); CA **50**,9235 & 15347(1956) (in Russ & in English) 21) Sax(1957), 1222 22)Blatt, OSRD 2014(1944) 23)Vivas, Feigenspan & Ladreda **2** (1946) (Trinilina – as replacement for PA)

2,4,6-Trinitroaniline Nitrate, $(O_2N)_3C_6H_2 \cdot NH_2 . HNO_3$, mw 389.12, N 24.23%, OB to CO_2 -30.5%. Crysts (from alc), which defl on heating. Was prepd by adding a chilled mixt of p-nitroaniline with fuming nitric acid to chilled oleum and allowing to stand until crysts are formed

Refs: 1)Beil–not found 2)E.Macciotta & Zaira Orani,Gazz **60**,408-14(1930) & CA **24**, 4280(1930)

2,4,6-Trinitro-N-nitraniline or N,2,4,6-Tetranitroaniline, $(_2N)_3C_6H_2 . NHNO_2$, is described as *2,4,6-Trinitro-phenylnitramine* under Phenylamine

Tetranitroanilines(TeNA), $(O_2N)_4C_6H . NH_2$, mw 273.12, N 25.64%, OB to CO_2 -32.2%. The following two isomers are theoretically possible, of which only the 2,3,4,6-TeNA is of interest to the expl industry

2,3,4, 5-Tetranitroaniline(2,3,4,5-TeNA)– not found in Beil or in CA through 1956

2,3,4,6-Tetranitroaniline(TeNA)(Chishokianin in Jap). Golden-yel crysts (from acet), mp 216-17°(dec), puffs off at 222°; d(cryst) 1.867; Q_c^v 654.3 kcal/mol(av values of Refs 22,27 & 33), Q_c^p 653 kcal/mol (Ref 27), Q_e 265.1 and Q_f 14.0 kcal/mol(Ref 45). It is a neutral compd. Solys of TeNA in various solvents were reported in Refs 4,16 & 21. Ref 21 gives solys of TeNA at RT in g per 100 g of the following liquids: water 0.07, acetone 7.50, benzene 0.13, CS_2 0.0056 CCl_4 0.0036, ethanol 0.34, ether 0.81,methanol 0.45 and toluene 0.188. TeNA is also sol in ethyleneglycol(Ref 37)

Hygroscopicity for pure lab prepd TeNA is very low (gain in wt 0.1% for a sample exposed for 48 hrs in highly humid atm at 32°) and noticeably higher for commercial products(Refs 4,16 & 19)

Behavior toward metals: TeNA, whether wet or dry, did not attack steel, iron, tin, copper, brass, lead, aluminum or zinc in 14 days at RT (Refs 8 & 16). TeNA did not attack inorganic nitrates in storage(Ref 16,p 99T)

No change in color or props was observed after exposure to light at RT for periods as long as 17 years(Ref 16,p 102T). Crystallographic data are given in Ref 44 and infrared absorption spectra values in Ref 43. According to Ref 16,p 103T, TeNA is non toxic, while according to Ref 46, it is highly toxic

Additional info on chem and phys props of TeNA can be found in Refs 4,7,9,11,16,17, 20,40,41, & 42

According to Flürscheim (Ref 16 p 97T), TeNA was discovered in 1904, but he does not indicate where this discovery was described. The first patent on prepn and uses of TeNA was granted in 1910 to Flürscheim (Ref 2). He prepd TeNA by one-stage nitration of m-nitroaniline as described in Refs 2,3, 4 & 5. Essentially, the same method was used recently by Lothrop(Ref 43) and by van Duin (Ref 10). Later, Stettbacher(Ref 31) described a method of prepn starting with aniline. Van Duin & Lennep(Ref 12) prepd TeNA starting with 3-acetanilide. Purification of TeNA can be achieved by washing crysts with a 40-70% aq soln of acetone at a temp not over 30°(Ref 34), or by crystn from ethyleneglycol (Ref 37)

Good descriptions of prepn and props of TeNA are given by Colver(Ref 11a) and by Flürscheim(Ref 16)

Explosive Properties of TeNA:

Brisance. According to Stettbacher(Ref 16) and some other investigators (Refs 6 & 7), TeNA is more brisant than tetryl, while Clark(Ref 32),gives its brisance, as detnd by the sand test, as only 102% that of TNT. According to Flürscheim(Ref 16,p 105T), the brisance, as detnd by the steel plate test

and by the fragmentation test, is much greater than that of TNT

Detonation Rate. According to Flürscheim (Ref 16, p 104T), the velocity of deton is "great" and exceeds that of tetryl and NG, but he did not give any numerical data. The value given in Blatt (Ref 41a) is 7630 m/sec at d 1.6

Detonation Temperature. See Explosion Temperature

Explosion Temperature. According to Ref 4, p 186, a 0.1g sample of TeNA in a test tube deflagrated at 220° when heated in an oil bath. In Ref 12, the expln temp is given as 237° for a sample heated at the rate of 5°/min and 247° when heated at the rate of 20°/min. According to Ref 21, there is a violent combustion at 225°

Gas Volume. 1g of TeNA evolved on expln 10.55 l at 3238°C vs 6.76 at 2217° for TNT (Ref 16,p 103T)

Impact Sensitivity. According to Flürscheim (Ref 16,p 102T), TeNA is sl less sensitive than tetryl and sl more sensitive than PA. This agrees with tests conducted by Robertson(Ref 18,p 18), who gives for FI(Figure of Insensitiveness) 86%PA for TeNA, 70% PA for tetryl and 115%PA for TNT. The values given in Ref 12 are 54-55cm fall for TeNA vs 51-53cm for tetryl, when using the Kast app and a 2kg hammer(See also Ref 41a)

Initiation Sensitivity. According to Ref 32, p 667, for 0.5g TeNA compressed at 3400 psi in a No 8 cap with reinforcing cup, the min wt of initiator reqd was 0.175g MF or 0.05g LA. According to Ref 16, p 105T a 100g cartridge of TeNA requires ca 0.5g MF for complete deton (See also Ref 41a)

Power: a)by Trauzl Test 140%TNT(av value of Refs 5,7,16 & 31) b)by BalMort 121% TNT (av value of Refs 13 & 15) c)by BalPend 146%TNT(Ref 16,p 104T) and 155%TNT, as calcd by Waller using the formula of Mallard & LeChatellier(Ref 16, p 104T) (See also Refs 41a and 41b)

Note: Although some of these data show higher values for TeNA than for tetryl, the Manometric Bomb(Druckmesser) Test of Bichel gives values lower than for tetryl (Ref 16,p 104T)

Stability of Dry Material. According to Flürscheim(Ref 16,pp 97T-99T), a sample of dry TeNA, produced on an industrial scale and without any purification other than washing with water, proved to posses satisfactory stability for use in military and commercial explosives. The following tests were used: a)Abel Test at 65.5°–satisfactory b)Vacuum Stability Test at 100° for the commercial product predried 18 hrs at 30-40°–loss of wt 0.034 cc/g per hr in the first 16 hr period, 0.020 in 17 to 32 hr period and 0.015 in 33 to 48 hr period c)135° German Test–no fumes in 60+ hrs d)British Storage Test at 90°F(32°C)–satisfactory e)International Test at 75°–satisfactory f)French Litmus Test–neutral litmus paper was not reddened in 5 days at RT g)French Railway Test–neutral litmus paper was not reddened by 100cc of w with which 1g of TeNA had been shaken for ½ hr at 15° h)Refs 12 & 36 state that TeNA starts to evolve nitrous acid fumes after 18 hrs at 70° i)Ref 12 stated that the vacuum stability test at 120° was 1.39 cc/g gas evolved in 30 hrs for a sample after about 10 months storage j)Hinschelwood & Bowen (Ref 14) reported that TeNA in small crysts required about 350 hrs at 120° for complete decompn, while at 140° the time was reduced to 70 hrs. The vel of decompn increased steadily throughout, falling sharply near the end of the reaction (See also Ref 41a)

Stability in Presence of Moisture is unsatisfactory(Refs 6,7,19,28 & 38). According to Carneiro(Ref 38) this poor stability is due to the presence of an NO_2 group in the 3rd position of the aniline ring. This labile group allows moisture to slowly hydrolize it with the formation of HNO_2 and 3-hydroxy-2,4,6 TNA. This phenolic compd attacks metals similarly to PA. Bradley (Ref 7), reported that this instability can be remedied by coating the crysts of TeNA with a small

of TeNA with a small amt (about 5%) of paraffin (see also Refs 41c & 43a)

Strength. See Power

Temperature of Explosion Two calcd values are given in Ref 16,p 103T: 3238° & 3500° vs 3127° & 3370° for tetryl and 2217° for TNT

Uses. According to Flürscheim(Refs 4,5,6, 11a & 16), TeNA is suitable as a substitute for tetryl in composite detonators and detonating cord as well as a filler for projectiles, bombs, mines and torpedoes. It is also suitable as a component of HE compositions, where it serves to sensitize cheaper insenitive compds, such as nitrobenzene, nitronaphthalenes and AN. Formulations of some mixts contg TeNA are given below. As a military explosive it was used to some extent by the Germans during WW II as a "substitute" explosive (Ersatzsprengstoff) in order to conserve TNT and other scarce nitroaromatic HE's. It could be used to a much greater extent, even in peace time, if it were more stable in the presence of moisture(Ref 47) (see above under Stability in Presence of Moisture). TeNA was called *Chishoki-anin* in Japan but there is no info whether or not it was used there for military purposes. O'Neil & Schuricht (Ref 29) proposed the use of TeNA for coating grains of some NC propellants. According to Ref 26a, TeNA was used in Russia as a booster. It was manufd in US by Aetna Expl Co and by Verona Chem Co

Refs(Tetranitroaniline): 1)Beil **12**, 763 (footnote),(372) & [427] 2)B.Flürscheim, BritP 3224 & 3907 (1910), USP 1,045,011 & 1,045,012; CA **7**, 258(1913) 3)B.Flürscheim & T. Simon,ProcChemSoc **26**, 69(1910) & CA **5**, 865 (1911) 4)B.Flürscheim,SS **8**, 185-8(1913) 5) B.Flürscheim,GerP 243,079 & SS **9**, 195(1914) 6)B.Flürscheim, CA **9**, 1391(1915) 7)W. Bradley, ProcUSNavalInst **42**, 533-41(1916) & CA **10**, 1596(1916) 8)C.Storm & W.Cope, BurMinesTechPaper **125**, 59(1916) 9)A.Stettbacher,SS **11**, 114(1916) 10)C.van Duin,Rec **37**, 111(1917) 11)Marshall I (1917), 169 11a)Colver(1918), 371-80 12)C.van Duin & B.van Lennep, Rec **39**,146 & 165-177(1920) 13)A.Marshall, IEC **12**,336(1920) 14)C.N.Hinshelwood & E.J.Bowen,PhilMag **40**,573 & 578(1920) 15)W.C.Cope,IEC **12**,870(1920) 16)B.Flürscheim,JCSI **40**,97T-107T(1921) & CA **15**, 2548 (1921) 17)A.Stettbacher,SS **16**,137-8 (1921) 18)R.Robertson,JCS **119**,13 & 18 (1921) 19)L.G.Marsh,ChemMetEn grg **27**, 1261-2(1922) & CA **17**,469(1923) 20)C.W. Davis & T.C.James,Aberystwyth Studies **4**, 213-6(1922) & CA **17**,3487(1923) 21)C.A. Taylor & W.H.Rinkenbach,JACS **45**,1218-20 (1923) 22)Land-Börnst,Eg III,Tl 3(1923), p 2914 23)B.Flürscheim,BritP 226,913(1923) & CA **19**,2133(1925) 24)C.A.Taylor & Wm. H.Rinkenbach,BurMinRI No 2513(1923) 25)C.A. Taylor & Wm.H.Rinkenbach,ArOrdn **5**,464(1924) 26) L.Desvergnes,MonSci **14**,121(1924) & CA **18**,2810 (1924) 26a)VanGelder & Schlatter(1927),953 27)Wm. H.Rinkenbach,JACS **52**,116(1930) 28)W.T. Ingraham,ArOrdn **11**,59-60(1930) & CA **24**, 4933(1920) 29)A.S.O(Neil & A.G.Schuricht, USP 1,849,355(1932) & CA **26**,2867(1932) 30)Vennin,Burlot & Lécorché(1932),p 461 31)Stettbacher(1933),291 & 363 32)L.R.V. Clark,IEC **25**,666-7 & 1385-90(1933) 33)L. Wöhler & O.Wenzelberg,AngChem **46**,173 (1933) 34)C.W.Davis,CanP 339,579(1934) & CA **28**,2727(1934) 35)A.Schmidt,SS **29**, 262(1934) 36)Pepin Lehalleur (1935), p 261 37)W.A.Smith,USP 2,024,396(1936) & CA **30**,1233(1936) 38)A.Carneiro,Rev BrasilQuim(São Paulo)**13**,287-8(1942) & CA **37**,261(1943) 39)Davis (1943),p 173 40) T.Katsurai & S.Yamaguchi,BullInstPhysChem Res(Tokyo) **22**,750-1(1943) & CA **42**,370(1948) 41)Thorpe **4**(1949), p 487 41a)A.H.Blatt,OSRD Rept 2014(1944) 41b)Vivas,Feigenspan & Ladreda **2**(1946)(Tetralina) 41c)Giua,Dizionario v2(1949), 151 42)E.Burlot & P.Tavernier, MP **31**, 121-9(1949) & CA **46**, 11685(1952) 43)W.C.Lothrop et al, JACS **73**, 3583-4(1951) 43a)Belgrano(1952),141 44)W.C.McCrone, AnalChem **25**, 1774-5(1953) 45)ADL Punch Cards, Compd No 233 46)Sax(1957), p 1170 47)Dr Hans Walter, PicArsn; private communication

Tetranitroaniline Explosives. Flürscheim (Ref 1) proposed the following expl compns contg TeNA: a)Molten mixt of TeNA/DNB (1:2) 6 & AN 94%; Trauzl test 265cc vs 254 for TNT or 430 for TeNA b)Molten mixt of TeNA/MNN/DNN(5:3:7)4.5 & AN 94.5%; Trauzl test 220cc c)Molten mixt of TeNA/DNN (1:2) 4 & AN 96%; Trauzl test 205cc d) Molten mixt of TeNA/DNB(1:2) 20 & AN 80%; Trauzl test 360cc e)Molten mixt TeNA/DNB (1:2.5) 12 & AN 88%; Trauzl test 500cc f) TeNA 35 & AN 65% and g)TeNA 50, K nitrate 10 & Ba nitrate 40%. Bradley (Ref 2) proposed the mixt TeNA 95 & paraffin 5% for use as burster chges in shells, bombs & mines and as a booster charge. Colver(Ref 3) lists the following TeNA blasting expls: a)TeNA 13, AN 81 & charcoal 6% and b) Mixt TeNA/DNB(23:77) 7, AN 87 & charcoal 6%. Flürscheim(Ref 4) patented expl compns prepd by heating pulverized mixts of TeNA 60 to 95% with TNT (or other low melting nitro-compd) 40 to 5% in such a manner that the TNT melts and permeates the TeNA crysts, thus rendering them waterproof. Oxidizers, such as inorganic nitrates, may also be incorporated

Refs: 1)B.Flürscheim,SS **8**, 187-8(1913) 2)W.Bradley,ProcUSNavalInst **42**, 533-41 (1916) & CA **10**, 1596(1916) 3)Colver (1918), pp 328-9 4)B.J.Flürscheim,BritP 226,913 (1923) & CA **19**, 2133(1925)

2,3,4,5,6-Pentanitroaniline(PNA), $(O_2N)_5C_6NH_2$, mw 318.12, N 26.42%, OB to CO_2 -15.1%. Golden-yel prisms, mp 196-8° (dec)(Ref 7), Q_c^v (H_2O liq) 686.8 kcal/mol (2159 cal/g)(Ref 4). When dropped into a red-hot crucible, a few centigrams at a time, PNA flashes without deton. It is insol in w, sol in acet (~25g in 100cc at 20°), AcOH (~10g in 100cc at 20°), benz (~5g in 100cc at 20°) and sl sol in conc H_2SO_4 (~2g in 100cc at 20°)(Ref 3)

In most respects the chemical and physical props of PNA are similar to those of TeNA. The 3-and 5-NO_2 groups of PNA are reactive and may be easily hydrolyzed. In the 100° Heat Test (Ref 5) PNA lost 1.58% in the 1st 48 hrs, 5.90% in the 2nd 48 hrs and did not explode in 100 hrs

PNA was prepd for the first time in an impure state in 1910 by Flürscheim and Simon (Ref 2). In 1928 Flürscheim and Holmes (Ref 4) prepd PNA in a pure state(mp 192°) by nitration of 3,5-dinitroaniline using 100% H_2SO_4 and concd HNO_3 at 75° for 3 hours. The structure assigned from chemical evidence was confirmed by infrared spectra data (Ref 7)

PNA is a powerful and brisant expl, being comparable to RDX or PETN. Its sensitivity to impact is between values for PETN and LA, which would make this expl unsuitable for use as a filler for projectiles. It could be used, however, as an initiating expl. Some numerical data for its power and sensitivity are given in Ref 9, such as Trauzl Test value 164% PA and FI(figure of insensitiveness) 36% PA

Note: The toxicity of PNA is not discussed in Sax(Ref 8) but it is logical to assume that toxic effects of PNA are similar to those of TeNA(Ref 8,p 1170)

Refs: 1)Beil **12**,[428] 2)B.Flürscheim & T.Simon, PrChSoc **26**, 81(1911) & CA **5**, 865 (1911) 3)B.Flürscheim & E.Holmes, JCS **1928**, 3041 & CA **23**, 823(1929) 4)Detd at PicArsn by Mrs. L.E.Neuman, PACLR 124,-138(1948) 5)Detd at PicArsn by B.Kanouse, PACLR 124,345(1948) 6)P.Karrer(1950), p 465 7)W.C.Lothrop et al, JACS **73**, 3583-4 (1951) & CA **46**, 6093(1952) 8)Sax(1957), p 987(PNA is not listed), p 1170(TeNA) 9) A.H.Blatt, OSRD Rept 2014(1944) 10)L. Pauling, OSRD Rept 5953(1945) (Absorption spectra of nitrated anilines)

ANILINE AND DERIVATIVES, ANALYTICAL PROCEDURES

Aniline intended for US military use must comply with the requirements of Specification MIL–A–10450A(Ref 12) which prescribes the following tests:

A)Aniline Content(minim 99.5%) is detd volumetrically by the potassium bromate-bromide method based on the following reaction:

$$C_6H_5NH_2 + KBrO_3 + 5KBr + 6HCl \rightarrow$$
$$C_6H_2Br_3NH_2 + 6KCl + 3H_2O + 3HBr$$

The original bromination method was described in 1876 by Koppeschaar(Ref 1)

a)Preparation and Standardization of 0.2N $KBrO_3$–KBr Solution. Dissolve in distd w contained in 1 l volumetric flask, 5.56 g of $KBrO_3$ and 30 g of KBr and make up to the mark. Standardize this soln against purified aniline(prepd by distn of commercial aniline as described on p 2 of Spec MIL–A–10450A) as follows:

Weigh to 0.1 mg 2.5 to 3 g of the purified aniline, transfer to a 1 l volumetric flask contg 900 ml distd w and 50 ml concd HCl. Fill the flask to the mark and shake it until the soln is uniform. Transfer a 100 ml aliquot to a 400 ml beaker, add 10 ml concd HCl and cool the soln to 15°. Titrate slowly with the 0.2*N* $KBrO_3$–KBr soln. Near the end of titration, test, after the addition of each drop of the soln, by spotting KI-starch test paper. The end point is indicated by the lt blue coloration which can be duplicated after a 2-min period. When the end point is reached, an addnl drop of the $KBrO_3$–KBr soln will color the mixt yel, thereby corroborating the end point indicated by the test paper. Calculate the normality of $KBrO_3$–KBr by the following formula:

$$N = \frac{\text{Grams aniline in aliquot}}{(\text{ml } KBrO_3\text{–KBr soln}) \times 0.01552}$$

b)Procedure. Conduct the test in exactly the same manner as for standardization and calculate percentage of aniline by the following formula:

$$\% \text{ Aniline} = \frac{(\text{ml } KBrO_3\text{–KBr soln}) \times N \times 1.552}{Wt},$$

N = normality of $KBrO_3$–KBr soln and *Wt* = weight of sample in aliquot

B)Nitrobenzene(NB) Content(max 0.2%) is detd by titrating the steam distd NB with std titanous chloride, based on the following reaction:

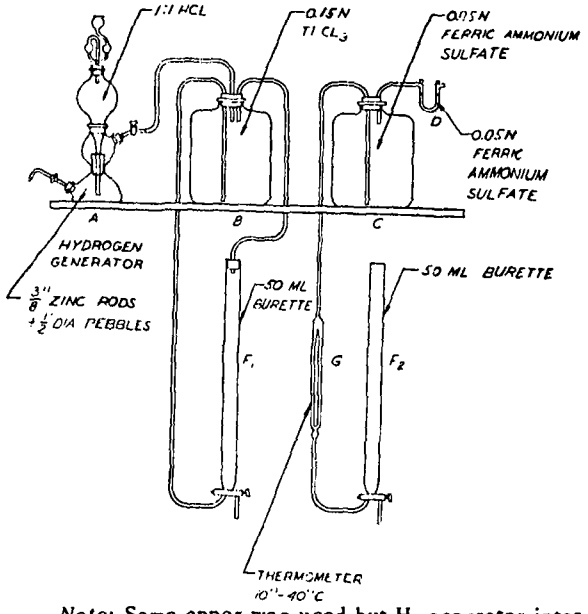
$$C_6H_5NO_2 + 6TiCl_3 + 6HCl \rightarrow$$
$$C_6H_5NH_2 + 6TiCl_4 + 2H_2O$$

a)Apparatus used in the standardization and test is shown in the fig on the following page

b)Preparation of Methylene Blue Indicator (MBI). Weigh into a 500 ml Erlen flask 0.5 g of MB dye, especially prepd for oxidation and reduction reactions. Dissolve it in 50 ml dist w and 50 ml of 25% sodium tartrate soln ($Na_2C_4H_4O_6 \cdot 2H_2O$). Heat to boiling in an atm of CO_2 and add dropwise the $TiCl_3$ soln to disappearance of blue color. Filter rapidly, dil to 500 ml and store in a tightly stoppered bottle. If the blue color reappears due to oxidation, discolor the soln before using by adding a few drops of $TiCl_3$ soln

c)Preparation of 0.05N Ferric Ammonium Sulfate Solution. Weigh into a large beaker 24.1 g of $Fe(NH_4)(SO_4)_2 \cdot 12H_2O$ for each liter of std soln desired. Dissolve in a mixt of 50 ml 40% sulfuric acid(by wt) and 50 ml distd w and filter into 1 l vol flask. Dil to the mark with distd w and thoroughly mix. Store it in the stock bottle C(see fig)

d)Preparation of 0.15N Titanous Chloride Solution. Filter the commercial 15–20% soln of $TiCl_3$ in strong HCl through asbestos, place the filtrate(*stock solution*) in a gas washing bottle and pass CO_2 through the soln to remove air. Close both exits of the bottle with rubber tubing and pinch cock assemblies. Det the strength of this "stock soln" by pipetting 5 ml into a 300 ml Erlen flask contg 25 ml distd w and 25 ml of 40% sulfuric acid, boiling the soln and titrating hot with std ferric ammonium sulfate soln using MBI(methylene blue indicator). The vol of stock soln required for the prepn of



Note: Same appar was used but H_2 generator interchanged with CO_2 generator

the desired amt of 0.15 *N* soln is obtained from the following formula:

$$V = \frac{0.15 \times V_1 \times 5}{N_2 \times V_2}, \text{ where}$$

V_1 = ml of std $TiCl_3$ desired; N_2 = normality of std $Fe(NH_4)(SO_4)_2$ soln; V_2 = ml of this soln consumed in the titration of 5 ml stock $TiCl_3$

Assemble the app as shown in fig and transfer to a thoroughly clean stock bottle B, 150 ml of concd HCl(for each liter of std soln desired), diluted with an equal vol of freshly boiled and cooled distd w. Add the accurately measured vol of stock soln(value V calcd from the above formula), dilute with w to the required vol and pass a rapid stream of CO_2 through the soln for 1 hr. Connect bottle B to hydrogen generator(see fig) and fill the burette F_1 with $TiCl_3$ soln and then discharge it. Leaving the stopcock of burette partly open, pass a moderately rapid stream of hydrogen through B and F_1 for 30 mins in order to remove any air. Close the stopcock of F_1 and allow the soln to age for several weeks in an atm of hydrogen before using it

e)Relation(R) of $TiCl_3$ and $Fe(NH_4)(SO_4)_2$ Solutions. Add 25 ml of 40%(by wt) sulfuric acid to a mixt of 150 ml distd w and 50 ml 95% ethanol in 500 ml Erlen flask equipped with a 3-hole rubber stopper and connected through one of the holes to the CO_2 generator(see fig). Sweep the air from the flask for 10 mins with a strong current of CO_2 and add through another hole of the stopper from a burette 15–20 ml of 0.15*N* $TiCl_3$ soln. Place the flask in a 2 l beaker contg 200–300 ml of cold tap w and heat the beaker at such a rate that the w boils in not less than 15 mins. Maintain at boiling temp for 15 mins and while continuing the current of CO_2 but at a slightly slower rate than before, add 2 ml MBI(colorless) and titrate hot with std $Fe(NH_4)(SO_4)_2$ to the appearance of a blue color persisting for 30 secs. Apply temp and calibration correction to both $TiCl_3$ and $Fe(NH_4)(SO_4)_2$ burette readings(see table at top of following page) and calculate R(relation) as:

$$\frac{\text{ml } TiCl_3 \text{ soln}}{\text{ml } Fe(NH_4)(SO_4)_2}$$

f)Standardization of 0.15N $TiCl_3$ Solution. The soln is standardized against purified p-nitroaniline, based on the reaction:

$$H_2N{\cdot}C_6H_4{\cdot}NO_2 + 6TiCl_3 + 6HCl \rightarrow$$

$$H_2N{\cdot}C_6H_4{\cdot}NH_2 + 6TiCl_4 + 2H_2O$$

Note: Purification of a good grade commercial p-MNA can be achieved by crystallizing twice from 95% ethanol and three times from distd w. This should give a product having a mp betw 147.3 & 148.2°. This will be called standard p-MNA

Accurately weigh 0.3 g of std p-MNA, transfer to 250 ml vol flask, dissolve in ca 100 ml distd w & an equal amt of 40% sulfuric acid and dilute to the mark. After thorough mixing, pipette two 25 ml aliquots into two 500 ml Erlen flasks, add to each flask 25 ml 40% sulfuric acid, 50 ml 95% ethanol & 100 ml distd w. Sweep the flask for 10 mins with a strong current of CO_2, add 50 ml of 0.15N $TiCl_3$ soln and proceed as described above under (e). Calculate normality of $TiCl_3$ by the following formula:

$$N = \frac{Wt}{V_1 - RV_2 \times 0.02302}, \text{ where}$$

Temperature Corrections for $TiCl_3$ and $Fe(NH_4)(SO_4)_2$ Solutions

Burette reading, ml	Corrections added, ml		Corrections to be subtracted, ml						
	16°	18°	22°	24°	26°	28°	30°	32°	34°
5	0.01	0.00	0.00	0.01	0.01	0.01	0.01	0.02	0.02
10	.01	.01	.01	.01	.02	.02	.03	.03	.04
15	.02	.01	.01	.02	.03	.03	.04	.05	.05
20	.02	.01	.01	.02	.03	.04	.05	.06	.07
25	.03	.01	.01	.03	.04	.05	.06	.08	.09
30	.04	.02	.02	.03	.05	.06	.08	.09	.11
35	.04	.02	.02	.04	.05	.07	.09	.11	.12
40	.04	.02	.02	.04	.06	.08	.10	.12	.14

Note: These temp corrections are applied to burettes calibrated at 20°. The corrections for $TiCl_3$ and $Fe(NH_4)(SO_4)_2$ solns are different from those applicable to aq solns generally due chiefly to the large amt of HCl present. The cubic coeff of expansion of the solns is on the average 0.000254 ml per degree per ml betw 18° and 25°C. If all calibrations are done at the same temp, no temp corrections are necessary

N = normality of $TiCl_3$ soln; Wt = weight of p-MNA used; V_1 = ml of $TiCl_3$ used; R = relation(see procedure e); V_2 = ml of $Fe(NH_4)(SO_4)_2$ used

g) Procedure for Determination of NB. Pipette a 50 ml sample(ca 51g) into a 1 l round bottom flask to which is attached a 24-inch reflux condenser cooled with tap w. Pour slowly through the inner tube of condenser 200 ml concd HCl and cool the mixt in the flask to RT. Disconnect the condenser and connect the flask to steam distn app, provided with a Kjeldahl spray trap and condenser and distil with steam into a 500 ml Erlen flask contg 50 ml 95% ethanol until the total vol in the receiver is ca 200 ml. Stop the distn, transfer the distillate to a 500 ml vol flask and dil to the mark with 95% ethanol

Note: If it is suspected that the NB content is not above 0.1%, the entire distillate may be titrated with $Fe(NH_4)(SO_4)_2$, directly in the receiver without transferring it to vol flask

Pipette an aliquot(100 ml or more) into a 500 ml Erlen flask equipped with a 3-hole rubber stopper and connected through one of the holes to CO_2 generator(see fig). Sweep the air from the flask for 10 mins with a strong current of CO_2 and add from a burette, through another hole in the stopper, 40–50 ml std 0.15N $TiCl_3$ soln. While continuing to pass CO_2 over the soln, quickly remove the rubber stopper and insert the one carrying a Hopkins condenser cooled with tap w, in order to prevent loss of NB in subsequent operations. Place the flask in a 2 l beaker with a porcelain grid in the bottom and contg 200–300 ml cold tap w. Heat the beaker at such a rate that w boils in not less than 15 mins, and continue boiling for 15 mins while still passing CO_2 but at a sl slower rate than at the beginning of the operation. Add 2 ml MBI (colorless) and titrate, while hot and still passing CO_2, with 0.05N $Fe(NH_4)(SO_4)_2$ (see proced c) to the appearance of a blue color which persists for 30 secs. Calculate NB content from the following formula:

$$\% \text{ NB} = \frac{(V_1 - RV_2) \times N \times 2.0518}{\text{Wt}}, \text{ where}$$

V_1 = ml of $TiCl_3$ soln used; N = its normality; R = relation(see proced e); V_2 = ml of

$Fe(NH_4)(SO_4)_2$ soln; Wt = weight of sample titrated and 2.0518 = grams of NB corresponding to 100 ml of normal $TiCl_3$ soln

C)Moisture Content(max 0.25%). Det moist by the Method 3001.5 of Federal Specification VV–L–791e(1953), with the exception that 75 ml of commercial toluene which has been previously dried over $CaCl_2$ and redistilled, is used as the diluent. The same results may be obtained by using the method for moisture detn under Ammonium Nitrate, Analytical Procedures

D)Specific Gravity(1.015 to 1.019 at 25°/4°). Det it by pycnometer method such as described in Federal Specification VV–L–791e (1953), Method 402.1 or in IEC, Anal Ed **16**, 55(1944)

Anilines, Nitrated, Analysis. Mononitroanilines can be detd by methods described in Refs 4–7 & 9–10

Dr H. Walter(Ref 15) used in his work the following method for detn of nitroanilines:

Weigh a sample, transfer it to a round-bottom flask equipped with a reflux condenser cooled with tap w and introduce through the inner tube of condenser, a quantity of acetic anhydride slightly in excess of that required for acetylation. After introducing through the condenser an amt of pyridine = to ca 10% of acetic anhydride, the reaction of acetylation starts spontaneously and then procedes at a much slower rate. The following reaction takes place:

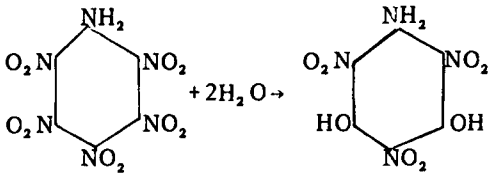
$$H_2N{\cdot}C_6H_4{\cdot}NO_2 + (CH_3CO)_2O \rightarrow$$
$$CH_3CO{\cdot}NH{\cdot}C_6H_4{\cdot}NO_2 + CH_3COOH$$

After allowing to stand for 1 hr, add distd w through the condenser in an amt sufficient to hydrolyze surplus anhydride to acetic acid. Transfer to a 1 l vol flask and dilute to the mark. Pipette an aliquot into a beaker and titrate with *N/10* NaOH soln in presence of an indicator, such as phenol-phthalein. Take the burette reading (R_1). Run a blanc by titrating with *N/10* NaOH the same quantity of acetic anhydride but without any nitroaniline, treated in exactly the same manner as above(burette reading R_2). The difference $R_2 - R_1$ is proportional to the amt of sample taken

Aniline, Polynitro Derivatives. As no info is available on the analysis of these compds, we asked the opinion of Mr. F. Pristera and Dr H. Walter, who had wide experience in analysis of polynitro derivs of toluene, phenol, etc. If the sample is a solid, its mp must be detd and if it is a liquid, its bp. Next comes the analysis by IR(if an apparatus is available). Experiments at PicArsn showed that IR spectrograms of various nitrated derivs of toluene permitted distinguishing, not only the degree of nitration but also the position of NO_2 groups(Refs 13 & 14). In later work spectrograms of o- and p-nitroanilines [See paper by F.Pristera et al published in Anal Chem **32**, 497(1960)] showed distinct differences between these two compds. If IR spectrograms do not give any definite info, it is advisable to det C and H contents by microcombustion method and also to det N content by micro-Dumas combustion method

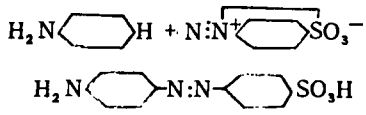
If the sample does not contain more than two (or three) NO_2 groups, the titanous chloride method(described above under detn of nitrobenzene in aniline) may be applicable. This method determines the nitro-nitrogen but not the amino-nitrogen. No info is available on application of the $TiCl_3$ method to tri-, tetra- and pentanitroanilines, but it definitely works with mono- and di-nitro derivs

In the opinion of Dr H. Walter, the application of the $TiCl_3$ method to aromatics contg more than 3 symmetrically arranged NO_2 groups might lead to inaccurate results, because this method exhibits a solvolytic effect on the 4th, 5th, etc groups. As these nitrogroups are very loose, they split off quantitatively by boiling with w(especially at pH>7) leaving a phenolic (–OH group) instead. This reaction may probably be used for analysis of compds, such as pentanitroaniline, as follows:



The resulting aminostyphnic acid can be identified by the mp and also in the form of its lead salt, obtained by the action of Pb acetate on aminostyphnic acid

Dr Walter stated also that diazobenzenesulfonic acid(prepd by diazotization of sulfanilic acid) readily couples with aromatic $-NH_2$ in acid medium or with $-OH$ group in alkaline medium, provided at least one ortho- or para- standing H-atom is available. The reaction proceeds as follows:

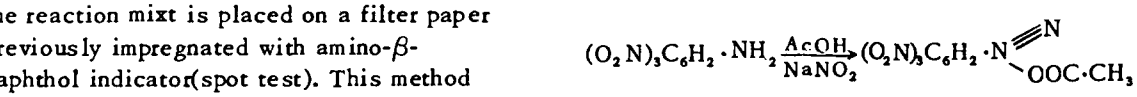


In this method, an alc soln of benzenesulfonic acid(previously standardized with phosgene by weighing the pptd diphenylcarbamide) is added from a burette to a sample of aniline(or its nitrated compd not higher than tetranitro-) until the appearance of a dirty-bluish coloration, when a drop of the reaction mixt is placed on a filter paper previously impregnated with amino-β-naphthol indicator(spot test). This method is not applicable for analysis of pentanitroaniline(Ref 15)

Refs: 1)W.F.Koppeschaar, ZAnalChem **15**, 233(1876) (Detn of aniline by bromination) 2)M.François, JPharmChim **9**,521(1899) & JCS **76 II**,713(1899) (Volumetric estimation of aniline by bromine w in presence of sulfate of indigo) 3)G.Dénigès, JPharmChim **10**,63(1899) & JCS **76 II**, 826(1899) (A claim for priority with respect to the method described by François) 4)F.L.English, IEC **12**,994(1920) (Application of $TiCl_3$ method to detn of nitro nitrogen in p-and m-MNA's) 5)B.M.Margosches & W.Kristen, Ber **56**, 1947(1923) (Kjeldahl method is applicable to m-MNA but not to o- & p-MNA or to 2,4-DNA) 6)I.M.Kolthoff & C.Robinson, Rec **45**, 171 & 174(1926) (Detn of nitro-nitrogen in p- & m-MNA by $TiCl_3$ method) 7)A.R.Day & W.T.Taggert, IEC **20**,545(1928) (Detn of m- and o-MNA by bromination method) 8)Kirk & Othmer **1**(1947),918(Small quantities of aniline can be detd colorimetrically by means of a purplish-violet color formed on treatment with hypochlorite soln or by conversion of this to a permanent blue dye on treatment with ammonia or phenol 9)W.Kemula et al RocznikiChem **29**,643 (1952) & CA **50**,3956(1956) (Separation of mixts of various isomers of nitrated aniline by the method or partition chromatography) 10)L.D.Johnson et al, AnalChem **27**,1494–8 (1955) and **28**,392(1956) (Bromination method is not always applicable in analysis of mixts of nitrated aniline and phenols) 11)Organic Analyses,Interscience,NY,vol 3(1956) 12)US Specification MIL–A–10450A 13)F. Pristera, ApplSpectroscopy **7**,115–21(1953) 14)F.Pristera & M.Halik, AnalChem **27**,217–22(1955) 15)Dr Hans Walter and Mr. Frank Pristera,PicArsn; private communication(1960)

Note: Dr Walter also suggested the following method for analysis of *trinitroaniline*.

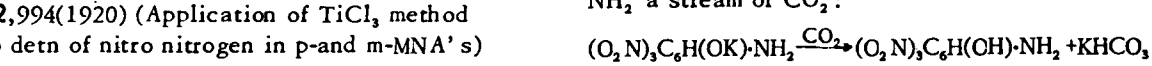
Dissolve a sample in AcOH, dilute with w and add Na nitrite:



Add an excess of dimethylaniline and transfer the resulting dark-red soln, contg $(O_2N)_3C_6H_2\cdot N{=}N\cdot C_6H_4\cdot N(CH_3)_2$, into a volumetric flask. Take an aliquot and test it colorimetrically

For analysis of *tetranitroaniline*, the following method was suggested:

Boil a sample with w to transform it to aminopicric acid $(O_2N)_3C_6H(OH)\cdot NH_2$. Add KOH and bubble through the resulting aq soln of potassium aminopicrate $(O_2N)_3C_6H(OK)\cdot NH_2$ a stream of CO_2:



Extract the resulting aminophenol with ether, evaporate and weigh

ANILINOACETIC ACID AND DERIVATIVES

Anilinoacetic Acid; Aminophenylacetic Acid; N-Phenylglycocoll or N-Phenylglycine (Anilinoessigsäure in Ger), $C_6H_5 \cdot NH \cdot CH_2 \cdot COOH$, is described in Beil **12**,468(263) & [249]

Anilinoacetic Acid, Azido–, $C_8H_8N_4O_2$ and *Diazido–* $C_8H_7N_7O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinoacetic Acid, $C_8H_8N_2O_4$. Its three isomers are described in Beil **12**,695, 709 & 725

2,4-Dinitroanilinoacetic Acid or N-(2,4-Dinitrophenyl)-glycine, $(O_2N)_2C_6H_3 \cdot NH \cdot CH_2 \cdot COOH$. Golden-yel scales(from aq alc), mp 112°(Ref 1); yel crysts(from aq methanol), mp 205°(Ref 2 & 4). Several methods of prepn are listed in Beil(Refs 1 & 2). It is evident that one value of the reported mp (115° and 205°) is wrong. Waldemann (Ref 5) did not clarify the point, although he also prepd the dinitroanilinoacetic acid. The mp of his compd is not given

Nearly all metallic salts of the dinitro compd, especially those of Pb, Ba & Ag are explosive(Refs 1 & 3)

Refs: 1)Beil **12**,756 2)Beil **12**,(363) 3)A.Sanna, Gazz **34** II, 222(1905) & JCS **88** I, 48–9(1905) 4)E.Abderhalden & P.Blumberg, ZPhysiolChem **65**,318(1910) & JCS **98** I, 371 (1910) 5)E.Waldmann, JPraktChem **91**,190 (1915) & JCS **108** I, 180(1915)

2,4,6-Trinitroanilinoacetic Acid, N-Picrylglycocoll or N-Picrylglycine, $(O_2N)_3C_6H_2 \cdot NH \cdot CH_2 \cdot COOH$, mw 286.16, N 19.58%. Yel ndls(from hot w), mp 161°; sl sol in w and sol in alc. Can be prepd by the interaction of glycine and picrylchloride. It is undoubtedly an expl and its salts even more so

Refs: 1)Beil **12**,770 2)K.Hirayama, ZPhysiolChem **59**,290(1909) & CA **4**,222 (1910)

Tetranitroanilinoacetic Acid, $C_8H_5N_5O_{10}$ – not found in Beil or CA through 1956

ANILINOAZOBENZENE AND DERIVATIVES

4-Anilino-azobenzene, Phenylazodiphenylamine or 4-Benzeneazo-diphenylamine, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot N{:}N \cdot C_6H_5$, is described in Beil **16**,314

Anilinoazobenzene, Azido–, $C_{18}H_{14}N_6$ and *Diazido–*, $C_{18}H_{13}N_9$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinoazobenzene, $C_{18}H_{14}N_4O_2$. Several isomers are described in Beil **16**,315, (311) & [153,178]

Dinitroanilinoazobenzene, $C_{18}H_{13}N_5O_4$. Several isomers are described in Beil **16**,314 & [148, 149,153]

Trinitroanilinoazobenzene, $C_{18}H_{12}N_6O_6$, mw 408.32, N 20.58%. Several isomers are described in Beil **16**,314 & [149,153]

Note: No higher nitrated derivs were found in Beil or CA through 1956, although the compds *hexanitroanilinoazobenzene*, $C_{18}H_9N_9O_{12}$, mw 543.32, N 23.20%, and higher nitrated derivs would be expected to be expl

ANILINOBENZALDEHYDE AND DERIVATIVES

Anilinobenzaldehyde, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot CHO$. One isomer is described in Beil **14**,37

Anilinobenzaldehyde, Azido–, $C_{13}H_{10}N_4O$ and *Diazido Derivatives*, $C_{13}H_9N_7O$ – were not found in Beil and in CA through 1956

Mononitroanilinobenzaldehyde, $C_{13}H_{10}N_2O_3$– not found in Beil

Dinitroanilinobenzaldehyde, $C_{13}H_9N_3O_5$. Two isomers are listed in Beil **14**,(357) & [22)

Trinitroanilinobenzaldehyde, $C_{13}H_8N_4O_7$ and higher nitrated derivs were not found in Beil or CA through 1956

Anilinobenzene. One of the names for Diphenylamine

ANILINOBENZENEDIAZONIUM HYDROXIDE AND DERIVATIVES

p-Anilinobenzenediazonium Hydroxide; Diphenylamine-4-diazonium Hydroxide or Phenylamine-4-diazonium Hydroxide, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot N_2 \cdot OH$, known only in the form of salts, some of which are explosive
Ref: Beil **16**,602-3,(371) & [307]

p-Anilinobenzenediazonium Azide, $C_{12}H_{10}N_6$ not found in Beil or CA through 1956

p-Anilinobenzenediazonium Nitrate or Diphenylamine-4-diazonium Nitrate, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot N_2 \cdot ONO_2$, mw 258.23, N 21.70%. Yel ndls, explg mildly on heating. Was prepd by treating N-phenyl-N-(4-nitrosophenyl)-hydroxylamine with nitrogen oxide
Refs: 1)Beil **16**,603 2)E.Bamberger et al, Ber **31**, 1515(1898)

p-Anilinobenzenediazonium Nitrate, Azido-, $C_{12}H_9N_7O_3$ and *Diazido-*, $C_{12}H_8N_{10}O_3$ *Derivatives* were not found in Beil or CA through 1956

Nitroanilinobenzenediazonium Nitrate, $C_{12}H_9N_5O_5$ – not found in Beil or CA through 1956

1-Anilino-2,6-dinitrobenzenediazonium Azide, $C_{12}H_8N_8O_4$ – not found in Beil and CA through 1956

1-Anilino-2,6-dinitrobenzene-4-diazonium Chloride, $C_6H_5 \cdot NH \cdot C_6H_2(NO_2)_2 \cdot N_2 \cdot Cl$; decomp at 175° when slowly heated and expl mildly when heated rapidly
Refs: Same as under Nitrate, described below

1-Anilino-2,6-dinitrobenzene-4-diazonium Nitrate; 2,6-Dinitrophenylamine-4-diazonium Nitrate, indexed in CA as **4-Anilino-3,5-Dinitrobenzenediazonium Nitrate**, $C_6H_5 \cdot NH \cdot C_6H_2(NO_2)_2 \cdot N_2 \cdot ONO_2$, mw 348.23, N 24.14%, OB to CO_2 -95.7%. Yel-brn ndls, mp decomp at 148° with emission of light on slow heating and detonates violently on rapid heating or when struck; sl sol in alc. Was prepd by treating 2,6-dinitro-4-aminodiphenylamine nitrate with iso-amylnitrite in alcohol
Refs: 1)Beil **16**, [307] 2)H.Lindemann & W.Wessel, Ber **58**,1227(1925) & CA **19**,2824 (1925)

1-Anilino-2,6-dinitrobenzene-4-diazonium Sulfate, $C_6H_5 \cdot NH \cdot C_6H_2(NO_2)_2 \cdot N_2 \cdot OSO_3H$, decomp at 180° when heated slowly and expl mildly when heated rapidly
Refs: Same as under Nitrate described above

1-(2,4-Dinitroanilino)-benzene-4'-diazonium Azide $C_{12}H_8N_8O_4$ – not found in Beil or CA through 1956

1-(2,4-Dinitroanilino)-benzene-4'-diazonium Chloride or 2,4-Dinitrophenylamine-4'-diazonium Chloride, $(O_2N)_2C_6H_3 \cdot NH \cdot C_6H_4 \cdot N_2 \cdot Cl$. Its monohydrate, in the form of golden-yel ndls, was prepd by diazotization of 2',4'-dinitro-4-aminodiphenylamine
Refs: 1)Beil **16**,603 2)G.T.Morgan & M.G.Micklethwait, JCS **91**,603(1908)

1-(2,4-Dinitroanilino)-benzene-4'-diazonium Nitrate or 2',4'-Dinitrodiphenylamine-4-diazonium Nitrate, $(O_2N)_2C_6H_3 \cdot NH \cdot C_6H_4 \cdot N_2 \cdot ONO_2$; yel crysts exploding on heating, comparable in sensitivity and stability to PA. Can be prepd in several steps starting from chloro-2,4-dinitrobenzene and acetyl-p-phenylene diamine (p-aminoacetanilide), as described in Ref 2. This compd is also listed in Refs 3 & 4 but no additional data are given
Refs: 1)Beil – not found 2)F.Steppes, GerP 291,156(1915) & CA **11**,889(1917) 3)A.H.Blatt & F.C.Whitmore, NDRC Div B Rept Serial No 442 and OSRD 1085(1942),23 4)A.H.Blatt, OSRD 2014(1944) (Under Diazonium Salts)
Note: No higher nitrated derivs of Anilinobenzenediazonium salts were found in Beil or CA through 1956

ANILINOBENZOIC ACID AND DERIVATIVES

Anilinobenzoic Acid; Diphenylaminocarboxylic Acid or N-Phenylanthranilic Acid, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot COOH$. Several isomers are described in Beil **14**,327,(533, 585) & [213]

Anilinobenzoic Acid, Azido–, $C_{13}H_{10}N_4O_2$ and *Diazido–*, $C_{13}H_9N_7O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinobenzoic Acid, $C_{13}H_{10}N_2O_4$. Several isomers are described in Beil **14**, 328–9, 374,377,442 & [214,233]

Dinitroanilinobenzoic Acid, $C_{13}H_9N_3O_6$. Several isomers are described in Beil **14**, 329,380,445,(560,572) & [214,235]

4-Anilino-3,5-dinitrobenzoylazide or 2,6-Dinitrodiphenylamino-4-carboxylazide, $C_6H_5 \cdot NH \cdot C_6H_2(NO_2)_2 \cdot CO \cdot N_3$, mw 328.24, N 25.61%. Pale red crysts(from benz), mp 135°; defl on rapid heating; easily sol in NB, sol in benz, diff sol in gasoline. Can be prepd by treating 4-anilino-3,5-dinitrobenzoylchloride with Na nitrite in AcOH
Refs: 1)Beil **14**,[274] 2)H.Lindemann & W. Wessel,Ber **58**,1228(1925) & CA **19**, 2824(1925)

Trinitroanilinobenzoic Acid, $C_{13}H_6N_4O_8$, mw 348.23, N 16.09%. Several isomers are described in Beil **14**, 329,393,429,(533,560, 572) & [236]

Tetranitroanilinobenzoic Acid, $C_{13}H_7N_5O_{10}$, mw 393.23, N 17.81% – not found in Beil or CA through 1956

ANILINOBENZONITRILE AND DERIVATIVES

Anilinobenzonitrile or Cyanodiphenylamine, $C_6H_5 \cdot NH \cdot C_6H_4 \cdot CN$ – not found in Beil or CA through 1956. It may be considered as the parent compd of the following derivs:

Anilinobenzonitrile, Azido–, $C_{13}N_9N_5$ and *Diazido*, $C_{13}H_8N_8$ *Derivatives* were not found in Beil or CA through 1956

Nitroanilinobenzonitrile or Nitrocyanodiphenylamine, $C_{13}H_9N_3O_2$. Several isomers are known
Refs: 1)Beil **14**,377,443,(584) & [235] 2)M.Schöpf, Ber **23**,3442(1890) 3)H.Goldstein & R.Voegeli, Helv **26**,1127(1943) & CA **38**,78(1944) 4)D.L.Vivian et al, JOC **20**,800(1955) & CA **50**,7813(1956)

m-(2,4-Dinitroanilino)-benzonitrile or 2,4-Dinitro-3'-cyanodiphenylamine, $(O_2N)_2C_6H_3 \cdot NH \cdot C_6H_4 \cdot CN$, mw 284.23, N 19.71%. Yel crysts, mp 190°. Was prepd by condensation of 3-aminobenzonitrile with 1-chloro-2,4-dinitrobenzene(Ref 2)
Refs: 1)Beil – not found 2)J.J.Blanksma & E.M.Petri,Rec **66**,358(1947) & CA **42**,148 (1948)

m-(2,4,6-Trinitroanilino)-benzonitrile or 2,4,6-Trinitro-3'-cyanodiphenylamine, $(O_2N)_3C_6H_2 \cdot NH \cdot C_6H_4 \cdot CN$, mw 329.23, N 21.27%. Golden-yel crysts(from alc); mp 180°; sl sol in w, insol in petr ether; moderately sol in alc & CCl_4; readily sol in warm NB and chlf and very sol in acetone. Was prepd by condensation of 3-aminobenzonitrile with picryl chloride(Ref 2,p 364). Its expl props were not examined
Refs: 1)Beil – not found 2)J.J.Blanksma & E.M.Petri,Rec **66**,364(1947) & CA **42**,148 (1948)

Tetranitroanilinobenzonitrile, $C_{13}H_6N_6O_8$ – not found in Beil or CA through 1956

Anilinobenzoylazide, Dinitro. See 4-Anilino-3,5-dinitrobenzoylazide under Anilinobenzoic Acid

Anilinobutane. Same as Butylaniline

ANILINOBUTANOL AND DERIVATIVES

Anilinobutanols; Butanolanilines, called also *Aminophenylbutanols and Phenylaminobutanols*, $C_{10}H_{15}NO$. Several isomers are described in the literature and they may be considered as parent compds of the following derivs:

Anilinobutanol, Azido–, $C_{10}H_{14}N_4O$ and *Diazido–*, $C_{10}H_{14}N_4O$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinobutanols, $C_{10}H_{14}N_2O_3$ were not found in Beil or CA through 1956

2-(2',4'-Dinitroanilino-N-)-1-butanol or 2-(2',4'-Dinitrophenylamino)-1-butanol,

$(O_2N)_2C_6H_3 \cdot NH \cdot CH\begin{matrix}\diagup CH_2OH \\ \diagdown CH_2 \cdot CH_3\end{matrix}$, mw 255.23,

N 16.47%. Col solid, mp 113–113.5°. Was prepd by Elderfield et al(Refs 2 & 5) by condensation of 2,4-dinitrochlorobenzene with 2-amino-1-butanol in alc soln and in the presence of NaOH. When nitrated it yields the explosive which may be named:

2-(N,2',4',6'-Tetranitroanilino)-1-butanol Nitrate; 2-(2',4',6'-Trinitro-N-nitranilino)-1-nitroxybutanol; 2-(N-Picryl-N-nitramino)-1-butanol Nitrate; 2-(2',4',6'-Trinitrophenylnitramino)-1-butanol Nitrate or 2-(N,2',4',6'-Tetranitrophenylamino)-1-butanol Nitrate. Elderfield called it(Ref 2): *2-(2',4',6'-Trinitrophenylamino)-butanol-N-nitramine Nitrate;* McGill(Ref 4): *2,4,6-Trinitrophenylisobutylolnitramine Nitrate* and Blatt(Ref 6): *2,4,6-Trinitrophenyl-(1-methylol)-propylnitramine Nitrate.*

$$(O_2N)_3C_6H_2N(NO_2)\cdot CH\begin{matrix}CH_2(ONO_2)\\ \diagup\\ \diagdown\\ CH_2\cdot CH_3\end{matrix},$$

mw 390.23, N 21.53%, OB to CO_2 −57.4%, OB to CO −16.4%. Yel crysts, d 1.39, mp 140–140.5°(decomp); insol in w & alc, sol in molten TNT and acetone. Was prepd by Elderfield et al by nitrating 2-(2',4'-dinitroanilino)-1-butanol with mixed nitric-sulfuric acids. A detailed description of the procedure is given in Ref 5

Tetranitroanilinobutanolnitrate is a powerful expl(Bal Mortar Test 117% TNT). When heated above its mp it deflagrated at 205–10° with a brilliant puff without deton, when heated to 216° it ignited in 5 secs. Its *Sensitivity to Impact* is comparable to that of tetryl(Ref 6). Fairly stable in storage and when heated to 100°; when heated in a closed tube at 135° for 30 mins, it exploded. Loss of wt in the 75° *International Test* 0.1%; *Hygroscopicity Test* at 90% RH and 25° gain in wt 0.06% and at 100% RH 0.19% (Refs 2 & 3). Its Q_c^v 1250.6 kcal/mol and Q_f −35.7 kcal/mol(Ref 3). Some other props are given in Ref 7, which is still classified

Refs: 1)Beil **12** – not found 2)R.C. Elderfield et al,OSRD Rept 158(1941), 7–10 & 15–17 3)G.B.Kistiakowsky, OSRD Rept 702(1942) (Table II) 4)R. McGill,OSRD Report 830(1942),65 5)R.C. Elderfield et al, OSRD Rept 907(1942), 5–6 6)A.H.Blatt,OSRD Rept 2014(1944) [See 2,4,6-Trinitrophenyl-(1-methylol)propylnitramine Nitrate] 7)A.D.Little,Inc, "Report on Study of Pure Explosive Compound," Cambridge,Mass, v 4(1952) Compd No 305, p 586(Part of rept is unclassified)

Note: No higher nitrated derivs were found in Beil or CA through 1956

ANILINOBUTYRIC ACID AND DERIVATIVES

Anilinobutyric Acids or Phenylaminobutyric Acids, $C_6H_5\cdot NH\cdot C_3H_6\cdot COOH$. Two isomers are described in Beil **12**,493,495 & [253], of which *α-anilino-isobutyric acid*, $C_6H_5\cdot NH\cdot C(CH_3)_2\cdot COOH$ is the parent compd of the following derivs:

Anilinobutyric Acid, Azido–, $C_{10}H_{12}N_4O_2$ and *Diazido–*, $C_{10}H_{11}N_7O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilino-iso-butyric Acid, $C_{10}H_{12}N_2O_4$ – not found in Beil

2-(2',4'-Dinitroanilino)-iso-butyric Acid or N-(2,4-Dinitrophenyl)-α-amino-iso-butyric Acid, called by Moulder: *L'acide 2,4-dinitroanilido-isobutyrique* and in Beil: *α-[2,4-Dinitroanilino]-iso-buttersäure*, $(O_2N)_2C_6H_3\cdot NH\cdot C(CH_3)_2\cdot COOH$, mw 269.21, N 15.61%. Lt yel leaflets(from dil AcOH), mp 190–1°(decomp beginning at 175°). Was prepd by Moulder(Refs 1 & 2) by the action of HCl(d 1.19) on the nitrile of α-(2,4-dinitroanilino)-iso-butyric acid.

Refs: 1)Beil **12**,756 2)A.Moulder,Rec **26**, 186–7(1907)

2-(2',4',6'-Trinitro-N-nitranilino)-iso-butyric Acid; N-(2,4,6-Trinitrophenyl)-N-nitro-α-amino-iso-butyric Acid; N,2,4,6-Tetranitro-N-iso-butyricacid -aniline or α-Picrylnitramino-iso-butyric Acid, called in CA, **α-Trinitropnenyl-nitraminoisobutyric Acid**, $(O_2N)_3C_6H_2\cdot N(NO_2)\cdot C(CH_3)_2\cdot COOH$, mw 359.12, N 19.50%, OB to CO_2 −64.6%, OB to CO 0%. Crysts, mp 152–3°(with complete decompn). Was prepd by Filbert of the duPont Co by nitrating N-(2',4'-dinitroanilino)-iso-butyric acid with nitric-sulfuric acid. Details of the method are given in Ref 2(See also Ref 3)

This acid as well as its heavy metal salts (such as those of Pb, Cu, Zn, Hg & Ag) are expl and were proposed as ignition components for electric blasting caps. Filbert also claimed that these heavy metal salts are suitable for use in some primary and

and initiating expl mixts as, for instance, with NS

Among the heavy metal salts, Filbert preferred the *basic lead salt*, $(O_2N)_3C_6H_2 \cdot N(PbOH) \cdot C(CH_3)_2 \cdot COO(PbOH)$ (probable formula), which was prepd by treating trinitronitranilinoisobutyric acid with lead nitrate in the presence of some NaOH. This salt is extremely sensitive to static electricity and fairly sensitive to shock and friction. It offers a marked advantage over lead styphnate in ease of ignition–in a loose condition it may be fired with a current of only 0.32 amp vs 0.38 amp required for lead styphnate

Filbert claimed that the basic lead salt is especially suitable for use as an ignition agent in fast electric blasting caps used in seismographic investigations

Note: In seismographic work it is desirable that there be no appreciable delay between the application of the current and the firing of the cap. Investigations by means of an oscillograph showed that caps contg the above basic lead salt as an ignition agent exhibited a time lag of 0.004 sec when fired by a current of 1 amp, compared with 0.006 sec when lead styphnate was used under the same conditions. In both cases, the time lag was greatly diminished by applying larger currents

Refs: 1)Beil – not found 2)W.F.Filbert, USP 2,115,066(1938) & CA **32**,4787(1938) 3)A.H.Blatt & F.C.Whitmore,OSRD 1085 (1942),115

Anilinodiazonium Hydroxide and Derivatives. See Aminobenzenediazonium Hydroxide and Derivatives

ANILINOETHANOL AND DERIVATIVES

2-(or β-)Anilinoethanol; Phenylaminoethanol; Phenylethanolamine; β-Ethanol-N-aniline; β-Anilinoethyl Alcohol; β-Hydroxyethylaniline or β-Hydroxyethylaminobenzene (called in Beil N-[β-Oxy-äthyl]-anilin), $C_6H_5NH \cdot CH_2 \cdot CH_2OH$, mw 137.18, N 10.21%. Col liq turning yel and then brn on standing, bp 167–70° at 19 mm, d 1.1129 at 25°/25°, n_D^{20} 1.5749. Was first obtained in 1873 by Ladenburg. Methods of prepn and other props are given in Beil **12**,182 & [106] (See also Aminophenylethanol)

2-Anilinoethanol, Azido–, $C_8H_{10}N_4O$ and *Diazido–*, $C_8H_9N_7O$ *Derivatives* were not found in Beil or CA through 1956

2-Anilinoethanol Nitrate, $C_6H_5NH \cdot CH_2 \cdot CH_2 \cdot ONO_2$, mw 182.18, N 15.38% – not found in Beil

Mononitroanilinoethanols, $(O_2N)C_6H_4 \cdot NH \cdot CH_2 \cdot CH_2 \cdot OH$. Several isomers were prepd and examined by Kremer from the point of view of their physiological props

Refs: 1)Beil – not found 2)C.B.Kremer, JACS **61**,1323(1939) & CA **33**,6259(1939)

Nitronitranilinoethanols, $(O_2N)C_6H_4 \cdot N(NO_2) \cdot C_2H_4OH$, mw 227.18, N 18.50% – not found in Beil

Nitroanilinoethanol Nitrates, $(O_2N)C_6H_4 \cdot NH \cdot C_2H_4 \cdot ONO_2$ – not found in Beil

Nitronitranilinoethanol Nitrate, $(O_2N)C_6H_4 \cdot N(NO_2) \cdot C_2H_4 \cdot ONO_2$ – not found in Beil

2–(2',4'-Dinitroanilino)-ethanol; N-(2,4-Dinitrophenylethanolamine; 2,4-Dinitrophenylaminoethanol; 2-4-Dinitro-hydroxyethylaniline or 2,4-Dinitro-1-(β-hydroxyethyl)-aminobenzene; $(O_2N)_2C_6H_3 \cdot NH \cdot CH_2 \cdot CH_2 \cdot OH$, mw 227.18, N 18.50%. Orange-yel crysts, mp 89.92°. Was prepd by Moran (Ref 2) from 2,4-dinitrochlorobenzene and aminoethanol. This method is described by Clark(Ref 3). Other methods of prepn are described in Refs 4 & 5. On nitration this compd yields the highly explosive 2-(2',4',6'-trinitro-N-nitranilino)-ethanol nitrate, designated as Pentryl(See p A425)

Refs: 1)Beil – not found 2)R.C.Moran, USP 560,427(1925) 3)L.R.V.Clark,IEC **25**, 1385(1933) 4)P. van Romburgh & C.W.Zahn, Rec **57**,444(1938) 5)K.F.Waldkötter, Ibid, 1295(1938) 6)J.C.Crawhall & D.F.Elliott, Biochem **61**,264(1955) & CA **50**,2694(1956) (A new method of prepn of 2,4-dinitrophenyl derivs of aminoalcohols, among them aminoethanols)

Dinitroanilinoethanol Nitrate, $(O_2N)_2C_6H_3NH\cdot CH_2\cdot CH_2ONO_2$, mw 272.18, N 20.59% – not found in Beil and CA through 1956

Dinitronitranilinoethanol, $(O_2N)_2C_6H_3\cdot N(NO_2)\cdot CH_2\cdot CH_2OH$, mw 272.18, N 20.59% – not found in Beil and CA through 1956

Dinitronitranilinoethanol Nitrate, $(O_2N)_2\cdot C_6H_3\cdot N(NO_2)\cdot CH_2\cdot CH_2(ONO_2)$, mw 317.18, N 22.08% – not found in Beil or CA through 1956

2-(2',4',6'-Trinitroanilino)-ethanol; 2,4,6-Trinitrophenylaminoethanol; 2,4,6-Trinitrohydroxyethylaniline; Hydroxyethylpicramide or 2,4,6-Trinitro-1-(β-hydroxyethylamino)-benzene, $(O_2N)_3C_6H_2\cdot NH\cdot CH_2\cdot CH_2\cdot OH$, mw 272.18, N 20.59%. Yel ndls, mp 109–10°, insol in eth & petr eth, sl sol in w. Can be prepd by the interaction of picryl chloride and aminoethanol. When nitrated it gives expl Pentryl(see below)

Refs: 1)Beil – not found 2)P.van Romburgh & C.W.Zahn,Rec **57**,444(1938) 3)K.F.Waldkötter, ibid,1297(1938)

2-(2',4',6'-TRINITRO-N-NITRANILINO)-ETHANOL NITRATE; 2-(N,2,4,6-Tetranitroanilino)-ethanol Nitrate; *2,4,6-Trinitro-1-(β-nitroxyethylnitramino)-benzene; sym-Trinitrophenylnitraminoethyl Nitrate; N-(2,4,6-Trinitrophenyl)-N-nitro-β-aminoethyl Nitrate; β-(2,4,6-Trinitrophenylnitramino)-ethyl Nitrate or Trinitrophenylethanolnitramine Nitrate*, designated as **Pentryl**[*Note:* According to A.Stettbacher,Protar **8**,91 (1942), the word "Pentryl" has been used to designate mixts of PETN and TNT, known also as Pentolites], $(O_2N)_3C_6H_2\cdot N(NO_2)\cdot CH_2\cdot CH_2(ONO_2)$, mw 362.18, N 23.22%, OB to CO_2 -35.3%, OB to CO 0%. Small cream-colored crysts, d 1.82(absol), d 1.73(maxim by compression), d 0.45 (apparent), d 0.74(compressed in detonators at 3400 psi = 239 kg(cm²); mp 126–9°(with sl decompn); expl at 235° when heated at the rate 20°/min from 100° on(Ref 6); ignites in 1.5 sec at 270–80°(Ref 8a). Was first prepd in 1925 by Moran(Ref 2) by nitrating 2,4-dinitrophenylaminoethanol with mixed nitric-sulfuric acid. E.von Herz(Ref 3) prepd pentryl by nitrating phenylaminoethanol, and patents were granted for its use as an explosive

Clark(Ref 4) gives the following method for the prepn of pentryl: a)Dissolve, with stirring, 100g of 2,4-dinitroanilinoethanol in 1000g of 95% sulfuric acid maintained at 20–30° by a cold water jacket, and pour this soln gradually over a period of 12–15 mins into stirred nitric acid(47° Bé = d 1.48 = 86% HNO_3), also maintained at 20–30°. b)Continue stirring for 30 mins at 30° then slowly raise the temp to 40° and maintain at this temp for 30 mins c)At the end of this second 30-min period raise the temp to 50° and hold at this temp for 30 mins d)Drown the mixt in a large amt of ice water and separate the resulting ppt of pentryl by filtration e)Rinse the ppt on the filter, first with cold water, then with cold dilute Na carbonate soln and finally with water (yield of the crude product is ca 90% of theoretical) f)Dry the product and purify it by recrystallization from benzene. A melting point of 128° was reported for the product

Waldkötter(Ref 7) prepd pentryl from the same ingredients as Clark and reported a mp of 129°. Desseigne(Ref 10) gives detailed description for the prepn of pentryl starting with the condensation of dinitrochlorobenzene and monoethanolamine, followed by nitration of the resulting product with mixed nitric-sulfuric acid. Pentryl was also prepd and investigated in Russia(Ref 5)

The explosive props of pentryl were detnd by Clark(Ref 4) at the USBurMines and by L.Médard(Ref 9) for the "Commission des Substances Explosives." Some of these props are also listed by Desseigne(Ref 10). A few props were detnd by van Romburgh (Ref 6). A brief description of pentryl is given by Davis (Ref 8)

Following are the properties of pentryl taken from various sources:

Action of Light. See Light, Action of

Ballistic Mortar Test. See Power, by Ballistic Mortar

Behavior Toward Flame. Whether confined or not, pentryl burns when ignited with a brilliant white flame without detonating. The same thing happens when pentryl is compressed at high pressure and confined in a detonator cap and then ignited by the spit of a fuse(Ref 4,p 1386)

Brisance by Fragmentation Test. When using a malleable iron grenade container with a 16.5g sample of explosive, the brisance was found to be greater than that of TNT and somewhat greater than that of PA (Ref 4,p 1390)

Brisance by Lead Block Compression (Crushing) Test. When using the procedure described in the USBurMines Bull **346**(1931), the compressions of 64.0g samples weighing 50.0g and of density 0.75 were: 14.8 mm for TNT(100%), 18.5 mm for Pentryl(125%) and 16.4 mm for PA(111%) (Ref 4,p 1389) (See also Ref 8a). This test serves also as a measure of **percussive force** of expls

Brisance by Lead Plate Cutting Test. When using the proc described in ChemMetEngrg **26**,1126–32(1922) and exploding a sample compressed at 2320 psi(163.1 kg/cm^2), pentryl cuts a hole in the plate equal in size to that made by tetryl and slightly larger than PA or TNT(Ref 4,pp 1387–8) (See also Ref 8a)

Brisance by Sand Bomb Test(Sand Crushing Test). When a No 2 USBurMines bomb was used with 0.50g samples compressed in a No 8 detonator shell at 3400 psi(239.0 kg/cm^2) and initiated by 0.30g of MF(compressed at 3400 psi), the amt of sand crushed by pentryl alone(after deducting ca 11.5g crushed by 0.30g MF) was 56.0g (129%) vs 43.6g(100%) for TNT, 54.2g(124%) for tetryl and 45.3g(104%) for PA(Ref 4, p 1387) (See also Ref 8a). The bomb and procedure are described in IEC **25**,664–5(1925)
Note: Some authorities believe that the sand test measures the *strength* of an explosive

Compressibility. When subjected to a pressure of 2500 kg/cm^2, pentryl attained a density of 1.73. At a pressure of 400 kg/cm^2 its d was ca 1.5, at 1000 ca 1.63, at 1500 ca 1.68 and at 2000 ca 1.70(Ref 9,p 52)

Dead-Pressing. Clark subjected pentryl to pressures as high as 5830 psi(409.8 kg/cm^2) and observed no dead-pressing(Ref 4,p 1387); Médard used pressures as high as 2500 kg/cm^2 and reported no dead-pressing(Ref 9, pp 51–2)

Detonation Equations. When pentryl is detonated in the absence of oxygen(or air), it does so approximately as indicated in the following reaction: $C_8H_6N_6O_{11} \rightarrow 8CO+3H_2O+3N_2$. When sufficient oxygen is present the reaction may be represented by: $C_8H_6N_6O_{11}+4O_2 \rightarrow 8CO_2+3H_2O+3N_2$

If K chlorate is used as the oxidizer, 47.4 parts of it are required for 52.6 parts of pentryl to give 0% oxygen balance to CO_2. This mixt, however, did not give a high brisance(by the sand test) and it is better to use a smaller amt of K chlorate. For instance, a mixt consisting of 20 p K chlorate and 80 p pentryl gives about a 2% higher sand test value than the corresponding amt of straight pentryl(Ref 4,p 1387)

Detonation Velocity(Rate of Detonation). When using the Mettagang method as described in USBurMines Bull **346**(1931), the average velocity for a sample compressed to d 0.80 in a light lead tube over 0.5 m long and 0.5"(1.27 cm) id was 5000 m/sec vs 4450 m/sec for TNT compressed to d 0.90(Ref 4,p 1389)
Note: The above velocities are not maximum because low densities were used. The Hercules Powder Co reported 5254 m/sec for a sample confined at d 1.0 in a $^3/_{16}$" glass tube(Ref 8a). Médard(Ref 9,p 51) reported the vel of deton detnd by the method of Dautriche for samples packed in cardboard cartridges of 30 mm diam and initiated by 1.5g MF, as 5330 m/sec at d 0.90, 5560 at d 0.99 and 7340 at d 1.65. In another series of tests, in which samples were initiated by MF and a PA booster, Médard reported (Ref 9,p 52): 6220 m/sec at d 1.20, 7020 at 1.40 and 7180 at 1.56. Desseigne(Ref 10,p 26)

reported 7300 m/sec at d 1.60 vs 7100 for PA

Drop Test. See Impact Sensitivity Test

Effect of Pressure on Performance in Detonators. See Pressure Effect, etc

Explosion(or Ignition) Temperature. When 0.02g charges of pentryl were dropped on molten Wood's metal preheated to various temps, no expln or ignition took place at 233°(5 trials), but at 235° the sample ignited in 3 secs after it touched the hot surface. The same time interval was observed at 240°, while at 250° it was 2 secs, at 260° 1.5 to 2 secs and at 270–280° 1.5 secs (Ref 4,p 1389)

Note: According to van Romburgh(Ref 6), pentryl exploded at 230° when heated from 100° at the rate of 20°/min

Flame Action. See Behavior Toward Flame

Fragmentation Test. See Brisance by Fragmentation Test

Friction Sensitivity. When using a Type A "Pendulum Friction Device" with a steel shoe and the procedure described in USBur Mines Bull **346**(1931) and in IEC **25**,664–5 (1925), pentryl proved to be somewhat more sensitive to friction than tetryl and very much more sensitive than PA or TNT(Ref 4, p 1386)

Gap Test(Propagation Test or Sympathetic Detonation Test). In order to ascertain the relative ability of the disturbance sent out by the explosion of a shielded detonator charged with pentryl as the base charge to transfer deton over an air gap to a receiving charge of an explosive(such as dynamite), Clark used the same method as he described for DDNP in IEC **25**,668(1933) When a shielded detonator contg a base charge of pentryl(0.50g) and a priming charge of MF(0.20g) (reinforced) was fired in a galvanized tube, it propagated deton to a charge of 40% straight dynamite located at a distance of 54 ft(16.5 m). This distance is 37% greater than that required for the Commercial No 8 tetryl detonators and about 4 times greater than that for a No 8 (80/20–MF/K chlorate) detonator(Ref 4,pp 1389–90)

Heat of Combustion, Q_c^v(calcd) 911.1 kcal/mol

Heat of Explosion, Q_e^v (calcd) 372.4 kcal/mol

Heat of Formation, Q_f^v (calcd from above heat of combustion) 43.4 kcal/mol(Ref 12)

Ignition Temperature. See Explosion(or Ignition) Temperature

Impact Sensitivity Test(Drop Test). When using the "Small Impact Device," described in USBurMines Bull **346**(1931) and in IEC **25**, 664–5(1925), the following max heights of drop in cm for a 2-kg hammer to produce "no explosion" in 5 trials were obtained: pentryl 30, tetryl 27.5 and TNT 100+. These tests show that the sensitivity to impact of pentryl is sl less than that of tetryl but considerably greater than that of TNT(Ref 4,p 1386). Blatt (Ref 8a) gives FI(figure of insensitiveness) 61% PA. Médard(Ref 9) reported that a 5-kg hammer falling from a height of 0.26 m (energy 1.30 kg m) on a sample of pentryl placed in a Bourges cap produced 56 explns out of 100 trials, while a 2-kg hammer falling from a height of 0.75 m(energy 1.50 kg m) produced 50 explns out of 100 as against 1.05 m(energy 2.10 kg-m) for tetryl . Desseigne(Ref 10,p 263) reported that PA required 5.0 kg m energy(more than three times that for Pentryl) for 50% detonations

Initiation, Sensitivity to(Sensitivity to Detonation by Initiating Agents). This may be expressed in terms of the minimum wt of an initiating agent which causes complete deton of an explosive used as a base charge. Complete deton is indicated when there is no further increase of sand crushed in the BurMines No 2 "sand bomb test" with an increased wt of initiator(except that resulting from the additional wt of the initiator itself). This proc is described in USBur Mines Tech Paper **125**(1916) and Rept Invest **2558**(1923)

Following are minimum amts of two primary explosives required to cause complete deton of 0.50g of pentryl loaded as a base charge into a No 8 detonator case and compressed at 3400 psi(239.0 kg/cm²): 0.150g of MF & 0.025g of LA. The corresponding values for PA are 0.225 & 0.12, for TNT – 0.240 & 0.16 and for tetryl–0.165 & 0.03g (Ref 8a)

The above values indicate that the sensitivity of pentryl to detonation is similar to that of tetryl and greater than that of TNT and PA(Ref 4, p 1387)

Médard(Ref 9) reported that 0.20g of MF assures complete detonation of pentryl at d's 1.50 to 1.65 and that 0.25g is required at d 1.73

Lead Block Compression(Crushing) Test. See Brisance by Lead Block Compression (Crushing) Test

Lead Block Expansion Test. See Power by Trauzl Test

Lead Cylinder Compression Test. Same as Lead Block Compression(Crushing) Test

Lead Plate Cutting Test. See Brisance by Lead Plate Cutting Test

Light, Action of. Slight decoloration after exposure to light for several months(Ref 4, p 1388)

Percussive Force. See Brisance by Lead Block Compression(Crushing) Test

Power by Ballistic Mortar Test. Was detnd at the USBurMines as 133% of TNT and by the Hercules Powder Co as 84% of blasting gelatin(Ref 8a)

Power(or Strength) by Trauzl Test(Lead Block Expansion Test). This test is supposed to represent the comparative "disruptive power" of explosives. When using 1g samples and a small lead block and the procedure described in the USBurMines Bull **346**(1931), the following values for expansion of the cavity in the block(in cc) and for relative strengths were obtained: pentryl 15.8cc(130%), TNT 12.2(100), tetryl 13.8(113), PA 12.4(104) and 80/20 pentryl/K chlorate 16.2cc(133%) (Ref 4,p 1388). Blatt (Ref 8a) gives 145% PA

Pressure Effect on Performance in Detonators. Results of the "Lead Plate Test," described in ChemMetEngrg **26**,1126–32(1922), indicated that excellent penetrations of the plate are obtained when pentryl is compressed at ca 2320 psi (163.1 kg/cm²) and that increasing the pressure to 5830 psi(409.8 kg/cm²) has no significant effect. When tested by the "Sand Bomb Test," pentryl showed nearly the same performance at pressures from 1160 to 5830 psi(81.5 to 409.8 kg/cm²). In these series of tests the amt of sand crushed was 56.4±0.6g (Ref 4,p 1386)

Propagation Test. See Gap Test

Rate of Detonation. See Detonation Velocity

Sand Bomb Test. See Brisance by Sand Bomb Test

Sensitivity to Detonation by Initiating Agents. See Initiation, Sensitivity to

Sensitivity to Friction. See Friction Sensitivity

Sensitivity to Impact. See Impact Sensitivity

Sensitivity to Initiation. See Initiation, Sensitivity to

Solubility of Pentryl (grams/100 grams of solvent)(Ref 4,p 1386)

Solvent	25°C	50°C
Benzene	0.70	v sol
Carbon tetrachloride	trace	trace
Chloroform	0.07	0.26
Ethanol	0.11	0.48
Ether	0.16	–
Ethylene dichloride	0.72	2.68
Methanol	0.67	2.14
Nitroglycerin	v sol	v sol
Toluene	0.63	1.70
Water	trace	trace

Specific Work(Travail Spécifique, in French). Médard(Ref 9,p 51) report–124 vs 100 for PA and Desseigne(Ref 10,p 263) gave 126 vs 100 for PA and 114 for tetryl

Stability, Thermal. When detnd by the 75° International Heat Test, described in USBur Mines Bull **96**(1916)–no loss of wt in 48 hrs. When the temp was raised to 120°, red fumes (nitrogen oxides) appeared in 4 hrs(Ref 4, p 1388) (See also Ref 8a)

Storage under Distilled Water for 15 days at RT resulted in no loss of brisance and strength, as detnd by the "sand bomb test." No decompn took place and the water remained neutral(Ref 4,pp 1387–8)

Strength. See Power by Trauzl Test and Brisance by Sand Bomb Test

Surveillance Test at 75°. Slight decomposition accompanied by a change of color from cream to yellow and a lowering of the mp from 128° to 127.5° took place after 40 days, but this did not affect its strength and brisance as detnd by the "sand bomb test" (Ref 4,p 1388)

Sympathetic Detonation Test. See Gap Test

Thermal Stability. See Stability, Thermal

Toxicity. According to Sax(Ref 11), its toxicity is unknown, but when heated to decompn it emits highly toxic fumes of nitrogen oxides. Hercules Co reported that it causes dermatatis(Ref 8a)

Trauzl Test. See Power(Strength) by Trauzl Test

Velocity of Detonation. See Detonation velocity

Work Specific. See Specific Work

Uses. Pentryl was proposed for use as a base charge in detonators in lieu of tetryl or nitromannite, as well as for some other purposes either alone or mixed with other explosives. Addition of an oxidizing agent(such as K chlorate) to pentryl greatly enhances its strength and is recommended by Clark as of distinct economic advantage(Refs 3,4,5,8, 9,10 & 11)

Refs: 1)Beil – not found 2)R.C.Moran, USP 1,560,427(1925) & CA **20**,112(1926) 3)E.von Herz,BritP 367,713(1930) & CA **27**, 2036(1933); GerP 530,704(1930) & CA **26**, 309(1932) 4)L.R.V.Clark,IEC **25**,1385–90 (1933) 5)M.S.Fishbein,Veyennaya Khimiya (Russia) **1933**,No 6,pp 3–8, Chem Ztr **1934**, II,1074–5 & CA **29**,7077(1935) 6)P.van Romburgh,ChemWeekblad **31**,728–9(1934) & CA **29**,3159(1935) (review of props of some explosive nitroamines and nitramines) 7)K.F.Waldkötter, Rec **57**,1296–8(1938) & CA **33**,1286–7(1939) 8)Davis(1943),229-32 8a)A.H.Blatt,OSRD Rept 2014(1944), under β-(2,4,6-Trinitrophenylnitramino)-ethyl Nitrate 8b)A.D.Little,Inc, Report on the Study of Pure Explosive Compounds, v 4 (1952),p 586(C) (not used) 9)L.Médard, MP **33**,51–2(1953) & CA **47**,5685(1953) 10)G.Desseigne,MP **33**,255–64(1953) & CA **47**,10229(1953) 11)Sax(1957),1223–4 (Trinitrophenylnitramine Ethyl Nitrate) 12)C.G.Dunkle,PicArsn,Dover,N.J.; private communication

Note: No higher nitrated derivs of anilinoethanol were found in Beil or CA through 1956

Pentryl Homologs. A series of explosive compds related to pentryl were prepd by den Otter(Ref 2) by nitrating the products of reaction obtained by reacting 1-amino-2,3-propanediol or 2-amino-1,3-propanediol with dinitrochlorobenzene, dinitrodichlorobenzene or dinitrochloronaphthalene. Most of these compds are listed in this work separately. Waldkötter(Ref 3) prepd a series of compds, some of them explosive, obtained by nitrating the products of the interaction of hydroxyethylamine with halo-benzene. Kremer & Meltsner(Ref 4) prepd a number of chloronitroanilino- and chloraminoaniline alkanols, intermediates of pentryl homologs

Refs: 1)Beil – not found 2)H.P.den Otter, Rec **57**,17–24(1938) & CA **32**,3354(1938) 3)K.F.Waldkötter,Rec **57**,1296–8(1938) & CA **33**,1286–7(1939) 4)C.B.Kremer & M. Meltsner,JACS **64**,1285–6(1942) & CA **36**, 4490(1942)

ANILINOETHANOL, HALOGEN DERIVATIVES

Halo-nitro-anilinoethanols; Halo-nitro-1-(β-hydroxyethylamino)-benzene, or *Halo-nitro-phenylaminoethanols* were prepd and examined by K.F.Waldkötter,Rec **57**,1298–1310 (1938). Among these compds, the *halo-dinitro-N-nitranilinoethanol nitrates* of general formula $(O_2N)_2 \cdot C_6H_2(X) \cdot N(NO_2) \cdot CH_2 \cdot CH_2(ONO_2)$ (where X is a halogen) proved to be explosive. They can be prepd by nitrating either halo-nitro-1-(β hydroxyethylamino)-benzene or halo-dinitro-1-(β-hydroxyethylamino)-benzene with absol nitric acid at −10 to −15°. Some *dihalo-nitro-N-nitranilinoethanol nitrates*, $(O_2N)C_6H_2(X_2) \cdot N(NO_2) \cdot CH_2 - CH_2(ONO_2)$, are also explosive but much weaker than the corresponding dinitro-compounds

2-(4'-Chloro-2'-nitroanilino)-ethanol and 2-(5'-Chloro-2'-nitroanilino)-ethanol, $(O_2N)C_6H_3(Cl) \cdot NH \cdot CH_2 \cdot CH_2OH$, N 12.93%, orange crysts, mp 107–107.5° and 116° resp, were prepd and examined by K.F. Waldkötter,Rec **57**,1298 & 1302(1938) and by C.B.Kremer & M.Meltsner,JACS **64**, 1285(1942)

2-(3'-Chloro-2'-nitroanilino)-ethanol and 2-(6'-Chloro-4'-nitroanilino)-ethanol, crysts, mps 78.5° and 120° resp, and *2-(6'-Chloro-2'-nitroanilino)-ethanol*, oil, bp 155–7° at 2 mm, were prepd and examined by C.B.Kremer & M.Meltsner, JACS **64**,1285(1942)

2-(4'-Bromo-2'-nitroanilino)-ethanol and 2-(5'-Bromo–2'-Bromo-2'-nitroanilino)-ethanol, $(O_2N) \cdot C_6H_3(Br) \cdot NH \cdot CH_2 \cdot CH_2OH$, orange and yel crysts, mps 106° and 126°, resp, were prepd and examined by K.F. Waldkötter,Rec **57**,1300 & 1304(1938)

2-(4',6'-Dichloro-2'-nitroanilino)-ethanol, $(O_2N)C_6H_2(Cl_2) \cdot NH \cdot CH_2 \cdot CH_2OH$, N 11.16% orange-red ndls, mp 51°, was prepd and examined by K.F.Waldkötter,Rec **57**, 1307–8(1938)

2-(4',6'-Dibromo-2'-nitroanilino)-ethanol, $(O_2N)C_6H_2(Br_2) \cdot NH \cdot CH_2 \cdot CH_2OH$, N 8.02%, orange-red ndls, mp 71°, was prepd and examined by K.F.Waldkötter, Rec **57**,1309 (1938)

2-(4'-Chloro-2',6'-dinitroanilino)-ethanol and 2-(5'-Chloro-2',6'-dinitroanilino)-ethanol, $(O_2N)_2C_6H_2(Cl) \cdot NH \cdot CH_2 \cdot CH_2OH$, N 16.06%, orange and yel ndls, mp 102° and 132° resp, were prepd and examined by K.F.Waldkötter, Rec **57**,1299 & 1303(1938). The 5'-chloro deriv seems to exist in two modifications, mps 132° and 116°

2-(4'-Bromo-2',6'-dinitroanilino)-ethanol, $(O_2N)_2C_6H_2(Br) \cdot NH \cdot CH_2 \cdot CH_2OH$, N 13.72%, orange ndls, mp 114°, was prepd and examined by K.F.Waldkötter, Rec **57**,1301(1938)

2-(4',6'-Dichloro-2'-nitro-N-nitranilino)-ethanol Nitrate, $(O_2N)C_6H_2(Cl_2) \cdot N(NO_2) \cdot CH_2 \cdot CH_2(ONO_2)$, N 16.42%. Nearly col crysts, mp– decomp at 187° and inflames at 305°. It is a weak explosive. Was prepd and examined by K.F.Waldkötter,Rec **57**,1307–8(1938)

2-(4',6'-Dibromo-2'-nitro-N-nitranilino-ethanol Nitrate, $(O_2N)C_6H_2(Br_2) \cdot N(NO_2) \cdot CH_2 \cdot CH_2(ONO_2)$, N 13.02%. Pale yel crysts, mp 69°, decomp at 178° and inflames at 305°. It is a weak explosive. Was prepd and examined by K.F.Waldkötter,Rec **57**,1309(1938)

2-(4'-Chloro-2',6'-dinitro-N-nitranilino)-ethanol Nitrate, $(O_2N)_2C_6H_2(Cl) \cdot N(NO_2) \cdot CH_2 \cdot CH_2(ONO_2)$. N 19.91%. Yel crysts, mp 90–92°. Seems to exist in a modification which melts at 81°, decomp at 105° and inflames at 296°. It is an explosive. Sol in chlf, insol in w, sl sol in eth and very sol in petr eth. Can be prepd by nitrating 2-(4'-chloro-2'-nitroanilino)-ethanol with absol nitric acid at −15°

Refs: 1)Beil – not found 2)K.F.Waldkötter, Rec **57**,1299(1938)

2-(5'-Chloro-2',4'-dinitro-N-nitranilino)-ethanol Nitrate, $(O_2N)_2C_6H_2(Cl) \cdot N(NO_2) \cdot CH_2 \cdot CH_2(ONO_2)$, N 19.91%. Yel crysts, mp– decomp at 180° and inflames at 253°; insol in w, very sl sol in petr eth, sl sol in eth and sol in chlf.

It is an explosive. Can be prepd by nitrating either 2-(5'-chloro-2'-nitro-anilino)-ethanol or 2-(5'-chloro-2',4'-dinitro-anilino)-ethanol with absolute nitric acid at −10°

Refs: 1)Beil – not found 2)K.F.Waldkötter, Rec **57**,1302–3(1938)

2-(4'-Bromo-2',6'-dinitro-N-nitranilino)-ethanol Nitrate, $(O_2N)_2C_6H_2(Br)\cdot N(NO_2)\cdot CH_2\cdot CH_2(ONO_2)$, N 17.67%. Col crysts, mp 95°, decomp at 180° and inflames at 256°; insol in w & petr eth, sl sol in eth and sol in chlf. It is an explosive. Can be prepd by nitrating either 2-(4'-bromo-2'-nitro-anilino)-ethanol or 2-(4'-bromo-2',6'-dinitro-anilino)-ethanol with absol nitric acid at −15°

Refs: 1)Beil – not found 2)K.F.Waldkötter, Rec **57**,1301(1938)

2-(5'-Bromo-2',4'-dinitro-N-nitranilino)-Ethanol Nitrate, $(O_2N)_2C_6H_2(Br)\cdot N(NO_2)\cdot CH_2\cdot CH_2(ONO_2)$, N 17.67%. Nearly col crysts, mp 114°, decomp at 173° and inflames at 262°; insol in w & petr eth, sl sol in eth and sol in chlf. It is an explosive. Can be prepd by nitrating 2-(5'-bromo-2'-nitro-anilino)-ethanol with absol nitric acid at −15°

Refs: 1)Beil – not found 2)K.F.Waldkötter, Rec **57**,1305(1938)

ANILINOETHYLAMINOETHANOL AND DERIVATIVES

Anilinoethylaminoethanol; Anilinoethylethanolamine or N-(β-Hydroxyethyl)-N'-phenyl-1,2-diaminoethane, $C_6H_5\cdot NH\cdot CH_2\cdot CH_2\cdot NH\cdot CH_2\cdot CH_2\cdot OH$, may be considered as the parent compd of derivs described below, although they were not prepd from it

Anilinoethylaminoethanol, Azido–, $C_{10}H_{15}N_5O$ and *Diazido–*, $C_{10}H_{14}N_8O$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinoethylaminoethanol, $C_{10}H_{15}N_3O_3$ was not found in Beil

N-[2-(2,4-Dinitroanilino)-ethyl]-aminoethanol or N-(β Hydroxyethyl)-N'-(2,4-dinitrophenyl)-1,2-diaminoethane, $(O_2N)_2C_6H_3\cdot NH\cdot CH_2\cdot CH_2\cdot NH\cdot CH_2\cdot CH_2\cdot OH$, mw 270.24, N 20.73%, OB to CO_2 −130.3%. Crysts, mp 121–2°(*from* alc). Was prepd in 75% yield by the condensation of 2,4-dinitrochlorobenzene with β-hydroxyethylethylenediamine, as described in Ref 2. Its Q_c^p is 1342 kcal/mol(Ref 3)

Refs: 1)Beil – not found 2)R.C.Elderfield,OSRD Rept 158(1941),7 & 9–10(PBL Rept 31094) 3)A.D.Little,Inc, "Report on Study of Pure Explosive Compounds," Pt 4(1952),547(Part of rept is unclassified)

N-[2-(2,4,6-Trinitroanilino)-ethyl-aminoethanol Nitrate or N-(β-Nitroxyethyl)-N'-(2,4,6-trinitrophenyl)-1,2-diaminoethane, $(O_2N)_3C_6H_2\cdot NH\cdot CH_2\cdot CH_2\cdot NH\cdot C_2H_4\cdot ONO_2$, mw 360.24, N 23.33%, OB to CO_2 −75.5%. Was not found in Beil and CA through 1956

Note: This product would be expected to appear on nitration of the above dinitro deriv. Instead, an indefinite compd was obtained by Elderfield, as reported in OSRD 158,p 8, and the work was abandoned

ANILINOGUANIDINE AND DERIVATIVES

Anilinoguanidine or Phenylaminoguanidine, $C_6H_5\cdot NH\cdot NH\cdot C(:NH)\cdot NH_2$, is described in Beil **15**,290,(71) & [106]

Anilinoguanidine, Azido–, $C_7H_9N_7$ and *Diazido–*, $C_7H_8N_{10}$ *Derivatives* were not found in Beil

Anilinoguanidine Nitrate, $C_6H_5\cdot NH\cdot NH\cdot C(:NH)\cdot NH_2\cdot HNO_3$, mw 213.20, N 32.85%. Pink crysts, mp 178°. Prepn and props are given in Ref 2

Refs: 1)Beil – not found 2)F.L.Scott et al, JACS **75**,4053–4(1953) & CA **48**,11395(1953)

1-Anilino-3-nitroguanidine or 1-Phenylamino-3-nitroguanidine, $C_6H_5\cdot NH\cdot NH\cdot C(:NH)\cdot NH\cdot NO_2$, mw 195.18, N 35.89%. Wh crysts, mp 164–172°. Methods of prepn are described in Refs 2–5

Refs: 1)Beil – not found 2)R.A.Henry, JACS **72**,5344(1950) & CA **46**,6088(1952) 3)F.L.Scott et al, JApplChem(London) **2**,

368(1952) & CA **48**,3354(1954) 4)F.L.Scott et al,JACS **75**,1296(1953) & CA **48**,5183(1954) 5)L.Fishbein & J.A.Gallaghan,JACS **76**,1878 (1954) & CA **49**,6838(1955)
Note: Isomers called *Aminophenylnitroguanidines* are described by R.A.Henry,JACS **72**, 5344(1950) & CA **46**(6088(1952)

N-Anilino-N'-nitroguanidine or 1-Phenylamino-3-nitroguanidine, $C_6H_5 \cdot NH \cdot NH \cdot C(:NH) \cdot NH \cdot NO_2$, mw 195.18, N 35.89%. Wh feathery ndls(from aq alc), mp 164°. Was prepd by adding 0.65 ml of phenylhydrazine, $C_6H_5 \cdot NH \cdot NH_2$, to a soln of 1g azidonitroamidine, $N_3 \cdot C(:NH) \cdot NH \cdot NO_2$, in 20 ml of water and heating the resulting mixt at 60° for about 10 mins in order to dissolve all the hydrazine. A yel ppt(mp 162°), obtained in cooling the soln, was purified by crystn from aq alc
Refs: 1)Beil – not found 2)F.L.Scott et al,JApplChem **2**,368 & 370(1952) & CA **48**, 3354(1954)

Dinitroanilinoguanidine, $C_7H_8N_6O_4$ and higher nitrated derivs were not found in Beil or CA through 1956

Anilinohydroxymethyldihydroxypropane and Derivatives. See Anilinotrimethylolmethane and Derivatives

Anilinohydroxymethylpropanediol and Derivatives. See Anilinotrimethylolmethane and Derivatives

ANILINOINDAZOLE AND DERIVATIVES

Anilinoindazole, $C_6H_5 \cdot NH \cdot C_7H_5N_2$, may be considered as the parent compd of derivs described below

Anilinoindazole, Azido–, $C_{13}H_{10}N_6$ and *Diazido–*, $C_{13}H_9N_9$ *Derivatives* were not found in Beil

Mononitroanilinoindazole, $(O_2N)C_6H_4 \cdot NH \cdot C_7H_5N_2$ – not found in Beil or CA through 1956

6-(2',4'-Dinitroanilino)-indazole, $(O_2N)_2C_6H_3 \cdot NH \cdot C_7H_5N_2$, mw 299.24, N 23.41%, is described in Beil **25**,317

5,7-Dinitro-6-anilino-indazole, $C_6H_5 \cdot NH \cdot C_7H_3(NO_2)_2N_2$, mw 299.24, N 23.41% is described in Beil **25**,318

6-(2',4',6'-Trinitroanilino)-indazole, called in Ref 2 "Trinitrophenyl-B-1-Aminoindazol," $(O_2N)_3C_6H_2 \cdot NH \cdot C_7H_5N_2$, mw 344.24, N 24.42%. Orange ndls(from benz+alc), mp 240–50°(decomp). Diff sol in most org solvents. Was prepd by treating 6-aminoindazole with 2-chloro-1,3,5-trinitrobenzene in alc soln and in the presence of Na acetate. Its expl props were not examined
Refs: 1)Beil **25**,317 2)E.Noelting,Ber **37**, 2582(1904)

5,7-Dinitro-6-(2',4',6'-trinitroanilino)-indazole, $(O_2N)_3C_6H_2 \cdot NH \cdot C_7H_3(NO_2)_2N_2$. This undoubtedly explosive compd is not described in Beil or CA through 1956. It may be possible to prepare it by careful nitration of either 5,7-dinitro-6-anilino-indazole or of 6-(2',4',6'-trinitroanilino)-indazole
Note: No higher nitrated derivs were found in Beil or CA through 1956

Anilinoisovalerianic Acid. See under Anilinovalerianic Acids

Anilinomethane or Phenylaminomethane. See Methylaniline

2-Anilino-2-methoxy-1,3-propanediol. See 2-Anilino-2-hydroxymethyl-1,3-dihydroxypropane

ANILINOMETHYLPROPANEDIOL AND DERIVATIVES

2-Anilino-2-methyl-1,3-propanediol or 2-Phenylamino-2-methyl-1,3-dihydroxy-propane, $C_6H_5 \cdot NH \cdot C(CH_3)(CH_2OH)_2$, may be considered as the parent compd of derivs listed below:

Anilinomethylpropanediol, Azido–, $C_{10}H_{14}N_4O_2$ and *Diazido*, $C_{10}H_{13}N_7O_2$ – not found in Beil or CA through 1956

Nitroanilinomethylpropanediol, $C_{10}H_{14}N_2O_4$ – not found in Beil or CA through 1956

2-(2',4'-Dinitroanilino)-2-methyl-1,3-propanediol) or 2-(2',4'-Dinitrophenylamino)-2-methyl-1,3-dihydroxy-propane, called by Elderfield 2,4-Dinitrophenyl-bis(hydroxymethyl)-methylamine, $(O_2N)_2C_6H_3 \cdot NH \cdot C(CH_3)(CH_2OH)_2$, mw 271.23, N 15.49%, OB to CO_2 −120.9%. Crysts(from alc), mp 162–3°. Was obtained in 35% yield by the condensation of chloro-2,4-dinitrobenzene with 2-amino-2-methylpropanediol in alc soln(Ref 2). Its Q_c^P is 1266.9 kcal/mol

Refs: 1)Beil – not found 2)R.C.Elderfield,OSRD Rept 158(1941),7 & 9–10(PBL Rept 31094) 3)A.D.Little,Inc, "Report on Study of Pure Explosive Compounds," Pt 4(1952),546(Part is unclassified)

2-(2',4',6'-Trinitro-N-nitroanilino)-2-methyl-1,3-propanediol Dinitrate or **2-(2',4',6'-Trinitrophenyl-N-nitramino)-2-methyl-1,3-dinitroxy-propane**, called by Elderfield, *2,4,6-Trinitrophenyl-(bishydroxy)-tertiary-butylnitramine Dinitrate*, $(O_2N)_3C_6H_2 \cdot N(NO_2) \cdot C(CH_3)(CH_2ONO_2)_2$, mw 451.23, N 21.73%, OB to CO_2 −37.2%, OB to CO −1.77%. Crysts, mp 159°; expl on heating in a test tube above the mp or when struck with a hammer. Was obtained in 45% yield by nitrating the above dinitro deriv with mixed nitric-sulfuric acid, as described in Ref 2,p 14. Its expl props were not reported

Refs: 1)Beil – not found 2)R.C.Elderfield, OSRD Rept 158(1941),8 & 14(PBL Rept 31094) 3)A.H. Blatt,OSRD Rept 2014(1944) – not found

Note: No higher nitrated derivs were found in Beil or CA through 1956

Anilinonaphthalene. See Phenylnaphthylamine or Phenylaminonaphthalene

N-Anilino-N'-nitroguanidine See Anilinoguanidine

ANILINOPHENOL AND DERIVATIVES

Anilinophenol or Hydroxydiphenylamine, called in Beil *Oxy-diphenylamin*, $C_6H_5NH \cdot C_6H_4OH$. Several isomers are described in Beil **13**,365,410,444,(131,150) & [213 & 231]

Anilinophenol, Azido–, $C_{12}H_{10}N_4O$ and *Diazido–*, $C_{12}H_9N_7O$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinophenols or Hydroxynitrodiphenylamines, $C_{12}H_{10}N_2O_3$, mw 230.22, N 12.71%. Two isomers are described in Beil **13**,421 & 444

Dinitroanilinophenols or Hydroxydinitrodiphenylamines, $C_{12}H_9N_3O_5$, mw 275.22, N 15.27%. Several isomers are described in Beil **13**,365,444,(138,150) & [169,216]

Dinitronitroanilinophenols or Hydroxydinitronitrodiphenylamines, $C_{12}H_8N_4O_7$, mw 320.22, N 17.50%. Two isomers are described by G.Leandri,AnnChim(Rome) **40**, 620(1950) & CA **46**,929(1952)

Trinitroanilinophenols or Hydroxytrinitrodiphenylamines, called in Beil, *Trinitrooxy-diphenylamine*, $C_{12}H_8N_4O_7$, mw 320.22, N 17.50%. The isomers contg NO_2 groups in the 2,4,6-positions are called *picrylaminophenols*. Several trinitroanilinophenols are described in Beil **13**,365,411, 425,445,(111,150,187) & [217,231], but none of them seems to be explosive

Tetranitroanilinophenols, $C_{12}H_7N_5O_9$, mw 365.25, N 19.18% and higher nitrated derivs were not found in Beil or CA through 1956

ANILINOPHENYLTETRAZOLE AND DERIVATIVES

5-Anilino-1-phenyl-1,2,3,4-tetrazole, called

in Ger, *1-Phenyliminophenyltetrazolon-(5)-anil*, $C_{13}H_{11}N_5$, mw 237.26, N 29.52%. Ndls (from alc), mp 162–3°, decomp >220°(Refs 1 & 2), leaflets, mp 159°(Ref 3), ndls mp 162° (Ref 4). Was prepd in 1900 from aminodiphenylguanidine and Na nitrite in HCl(Refs 1 & 2). Other methods of prepn are given in Refs 3 & 4
Refs: 1)Beil **26**,408 & [245] 2)M.Busch & P.Bauer,Ber **33**,1069(1900) 3)R.Stollé, Ber **55**,1292–3(1922) & CA **16**,3898(1922) 4)E. Oliveri-Mandalà,Gazz **52**,II,139(1922) & CA **17**, 1642(1923)

Anilinophenyltetrazole, Azido–, $C_{13}H_{10}N_8$ and *Diazido–*, $C_{13}H_9N_{11}$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinophenyltetrazole, $C_{13}H_{10}N_6O_2$, mw 282.26, N 29.78%; *Nitronitranilinophenyltetrazole*, $C_{13}H_9N_7O_4$, mw 327.26, N 29.96%; *Dinitroanilinophenyltetrazole*, $C_{13}H_9N_7O_4$ and higher nitrated derivs were not found in Beil or CA through 1956. If prepd, some would undoubtedly be explosive

ANILINOPHTHALIMIDE AND DERIVATIVES

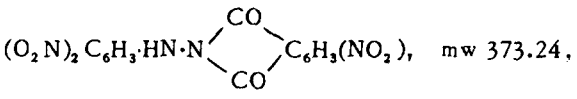
N-Anilinophthalimide or N-Phenyl-N',N'-phthalylhydrazine, $C_{14}H_{10}N_2O_2$, is described in Beil **21**,502,(388) & [371]

Anilinophthalimide, Azido–, $C_{14}H_9N_5O_2$ and *Diazido–*, $C_{14}H_8N_8O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinophthalimide, $C_{14}H_9N_3O_4$. One isomer is described in Beil **21**, 503 & (389)

Dinitroanilinophthalimide, $C_{14}H_8N_4O_6$ – not found in Beil or CA through 1956

N-(2',4'-Dinitroanilino)-3-nitrophthalimide or N-(2',4'-Dinitrophenyl-3-nitrophthalyl)-hydrazine,



$(O_2N)_2C_6H_3{\cdot}HN{\cdot}N(CO)_2C_6H_3(NO_2)$, mw 373.24, N 18.77%. Yel crysts, mp 249–50°. Can be prepd by the interaction of 2,4-dinitro-phenylhydrazide with 3-nitrophthalic acid
Refs: 1)Beil – not found 2)J.Cerezo & E. Olay,AnalesSocEspañQuím **32**,1090(1934) & CA **29**,2932(1935)

Dinitronitranilinonitrophthalimide, $C_{14}H_6N_6O_{10}$, mw 418.24, N 20.10%; *Trinitroanilinonitrophthalimide*, $C_{14}H_6N_6O_{10}$, and higher nitrated derivs were not found in Beil or CA through 1956. If prepd, some would undoubtedly be explosive

Anilinopropane or Phenylaminopropane. See Propylaniline

ANILINOPROPANEDIOL AND AND DERIVATIVES

Anilinopropanediol; Phenylaminopropanediol or Phenylaminodihydroxypropane, $C_9H_{13}NO_2$, exists in the following two forms:
1-Anilino-2,3-propanediol; 3-Anilino-1,2-propanediol; γ-Anilinopropyleneglycol; 3-Phenylamino-1,2-propanediol or 1-Phenylamino-2,3-dihydroxy-propane. Called in Beil [*β, γ-Dioxypropyl*]-*anilin*, C_6H_5-NH.CH_2.CH(OH).CH_2(OH). Solid, mp 52°, bp 249° at 50 mm (Refs 1 & 2), bp 175-85° at 0.5 mm (Ref 3). Methods for its prepn are given in Refs 2 & 3. On nitration, it gives the derivs described below
Refs: 1)Beil **12**,183 2)E.Bamberger & M.Kitschelt, Ber **27**,3425(1894) 3)R.C. Elderfield et al, OSRD Rept No 158(1941) & PBL Rept 31,094,pp 22-3)
2-Anilino-1,3-propanediol or 2-Phenylamino-1,3-propanediol, C_6H_5. NH.$CH(CH_2OH)_2$-not found in Beil. May be considered as the parent comp of derivs listed below.
Anilinopropanediol, Azido-, $C_9H_{12}N_4O_2$ and *Diazido-*, $C_9H_{11}N_7O_2$ *Derivatives* were not found in Beil or CA through 1956
1-Nitroanilino-2,3-propanediol,$(O_2N)C_6H_4$-NH.CH_2.CH(OH).CH_2OH–not found in Beil or CA through 1956
2-Nitroanilino-1,3-propanediol, $(O_2N)C_6H_4$.-NH.$CH(CH_2OH)_2$–not found in Beil

1-(2',4'-Dinitroanilino)-2,3-propanediol; 2',4'-Dinitroanilino2,3-propyleneglycol; 3-(2',4'-Dinitrophenylamino)-1,2-propanediol

or 1-(2',4'-Dinitrophenylamino)-2,3-dihydroxypropane, $(O_2N)_2C_6H_3.NH.CH_2.CH(OH).CH_2OH$, mw 257.20, N 16.34%. Yel solid, mp 95°; insol in w, petr eth, ether, chlf, CCl_4 & benz, sol in alcs, AcOH & benz. Can be prepd either by boiling an alc soln of 3-amino-1,2-propanediol with 2,4-dinitrochlorobenzene in the presence of Na acetate (Ref 2) or by nitrating 3-anilino-1,2-propanediol with fuming nitric acid(Ref 3)

Refs: 1)Beil–not found 2)H.P.den Otter, Rec **57,** 20 & 22 (1938) & CA **32,** 3354 (1938) 3)R.C.Elderfield et al, OSRD Rept 158(1941), PBL Rept 31,094,pp22-4

2-(2',4'-Dinitroanilino)-1,3-propanediol; 2-(2',4'-Dinitrophenylamino)-1,3-propanediol or 2-(2',4'-Dinitrophenylamino-1,3-dihydroxypropane, $(O_2N)_2C_6H_3.NH.CH(CH_2OH)_2$, mw 257.20, N 16.34%. Yel solid, mp 133-5°, same solubilities as for 1-(2',4'-dinitroanilino)-2,3-propanediol. Can be prepd by boiling an alc soln of 2-amino-1,3-propanediol with 2,4-dinitrochlorobenzene in the presence of Na acetate (Refs 2 & 3). This substance exploded weakly on heating in an open dish or test tube and upon being struck with a hammer on an iron surface (Ref 3, p 7)

Refs: 1)Beil–not found 2)H.P.den Otter, Rec **57,**15-17 (1938) & CA **32,**3354(1938) 3)R.C.Elderfield et al, OSRD Rept 907(1942), 6-7(PBL Rept 31085)

1-(2',4',6-Trinitroanilino)-2,3-propanediol; 3-(2',4',6'-Trinitrophenylamino)-1,2-propanediol or 1-(2',4',6'-Trinitrophenylamino)-2,3-propanediol, $(O_2N)_3C_6H_2.NH.CH_2.CH(OH).CH_2OH$, mw 302.20, N 18.54%. Yel solid, mp 136°, insol in w, eth, petr eth, chlf & CCl_4, sol in alc, acet, benz & NB. Can be prepd by boiling an alc soln of 3-amino-1,2-propanediol with 2,4,6-trinitrochlorobenzene in the presence of Na acetate. It exploded on heating or on impact

Refs: 1)Beil–not found 2)H.P.den Otter, Rec **57,**22-3(1938) & CA **32,** 3354(1938)

2-(2',4',6'-Trinitroanilino)-1,3-propanediol; 2-(2',4',6-Trinitrophenylamino)1,3-propanediol or 2-(2',4',6'-Trinitrophenylamino)-1,3-dihydroxy-propane; $(O_2N)_3C_6H_2.NH.CH(CH_2OH)_2$, mw 302.20, N 18.54%. Yel solid, mp 150°; insol in petr eth, chlf, CCl_4 & benz; sol in w, alc, acet & NB. Can be prepd by boiling an alc soln of 2-amino-1,3-propanediol with 2,4,6-trinitrochlorobenzene in the presence of Na acetate. It exploded on heating or on impact but milder than its higher nitrated product described below

Refs: 1)Beil–not found 2)H.P.den Otter, Rec **57,** 22-3(1938) & CA **32,** 3354(1938)

1-(2',4',6'-Trinitro-N-nitranilino)-2,3-propanediol Dinitrate; 1-(N,2',4',6'-Tetranitroanilino)-2,3-propanediol Dinitrate; 3-(2',4',6'-Trinitrophenylnitramino)-1,2-propanediol Dinitrate or 3-(2',4',6'-Trinitrophenylnitramino)-1,2-dinitroxypropane, $(O_2N)_3C_6H_2.N(NO_2).CH_2.CH(ONO_2).CH_2(ONO_2)$, mw 437.20, N 22.42%, OB to CO_2 −27.4%, OB to CO +5.5%. Lt yel crysts; mp- softens ca 67° and decomp ca 80°; insol in w, petr eth, CCl_4, sol in alc, ether, acet, chlf, benz & NB. Was first prepd in 1933 (Ref 2) by condensing glycerol with phenylamine and nitrating the resulting product. Later, it was prepd by nitrating 3-anilino-1,2-propanediol or its dinitrocompound with either fuming nitric acid (Ref 3) or mixed nitric sulfuric acid (Ref 4). It is a very powerful explosive which is unstable in storage

Refs: 1)Beil–not found 2)Westfälisch-Anhaltische Sprengstoff A-G, GerP 576, 152(1933) & CA **27,**3823(1933) 3H.P. den Otter, Rec **57,**20-23(1938) & CA **32,** 3354(1938) 4)R.C.Elderfield et al, OSRD Rept 158(1941), 22-4(PBL 31094) 5)A.H.Blatt, OSRD Rept 2014(1944), under 2,4,6-Trinitrophenyl(β, γ-dinitroxy)propylnitramine

2-(2',4',6'-Trinitro-N-nitranilino)-1,3-propanediol Dinitrate; 2-(N,2',4',6'-Tetranitroanilino)-1,3-propanediol Dinitrate or 2-(2',4',6'-Trinitrophenylnitramino-1,3-dinitroxy-propane; $(O_2N)_3C_6H_2.N(NO_2)CH(CH_2ONO_2)_2$, mw 437.20, N 22.42%, OB to CO_2 -27.4%, OB to CO +5.5%. Lt yel crysts, mp 142-3° (with decomp); insol in w, alcs, pet eth, chlf, CCl_4, benz & tol; sol in acet, AcOH, NB & pyridine. Can be prepd by nitrating either the corresponding dinitro- or trinitro- compds with fuming nitric acid (Ref 2). It is a powerful explosive

Note: According to Elderfield et al (Ref 3), the nitration of 2-(2';4'-dinitrophenylamino)-1,3-propanediol with mixed nitric-sulfuric acid yielded a product which melted at 146.5° and exploded on further heating or on being struck with a hammer on an iron surface. No analysis was made but it seems that this product was identical with the one prepd by den Otter

Refs: 1)Beil—not found 2)H.P.den Otter, Rec **57**,16-18(1938) & CA **32**,3354 (1938) 3)R.C.Elderfield et al, OSRD Rept 907(1942), 6-7(PBL Rept 31085)

ANILINOPROPANOL AND DERIVATIVES

Anilinopropanol; Anilinopropylalcohol; Phenylaminopropanol or Propanolaniline $C_9H_{13}NO$. One isomer, called in Ger *γ-Anilinopropylalkohol or [Oxy-propyl]-anilin*, $C_6H_5NH.C_3H_6OH$, is described in Beil **12**, [109]

Anilinopropanol, Azido-$C_9H_{12}N_4O$ and *Diazido-*$C_9H_{11}N_7O$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinopropanols, $C_9H_{12}N_2O_3$, mw 196.20, N 14.28%. Several isomers were prepd and examined by Kremer (Ref 2) from the point of view of their physiological action

Refs: 1)Beil—not found 2)C.B.Kremer, JACS **61**,1323(1939) & CA **33**,6259(1939)

Nitronitranilinopropanols, $C_9H_{11}N_3O_5$, mw 241.20, N 17.42%—not found in Beil or CA through 1956

Dinitroanilinopropanols, $C_9H_{11}N_3O_5$, mw 241.20, N 17.42%. The following isomer was found in the literature:

2-(2',4'-Dinitroanilino)-1-propanol or 2-Methyl-N-(2',4'-dinitrophenol)-ethanolamine. Listed in CA **50**, 2694d as *N-(2,4-Dinitrophenyl)-alaninol*, $(O_2N)_2C_6H_3.NH.CH.CH_2OH$, mp 94-5°. Was prepd by mixing 2,4-dinitrobenzenesulfonate with 2-amino-1-propanol (or its chloride or oxalate) and aq Na carbonate soln

Refs: 1)Beil—not found 2) J.C. Crawhall & D.F.Elliott, BiochemJ **61**,264 (1955) & CA **50**,2694(1956)

Dinitroanilinopropanol Nitrate, $(O_2N)_2C_6H_3.NH.C_3H_6ONO_2$, mw 286.20, N 19.58%; *Trinitroanilinopropanol* and higher nitrated compds were not found in Beil or CA through 1956

Chloronitroanilinopropanols, $C_9H_{11}N_2O_3Cl$, N 12.14%. In the course of a study of the condensation of substituted nitrobenzenes with aminoalcohols to produce intermediates from which analogs of *pentryl* (see under Anilinoethanol) might be obtained, several chloroanilinoalkanols, among them chloroanilinopropanols, were prepd and described by C.B.Kremer & M.Meltsner in JACS **64**, 1285(1942) & CA **36**,4490(1942)

ANILINOPROPIONIC ACID AND DERIVATIVES

Anilinopropionic Acid or Phenylalanine, $C_9H_{11}NO_2$. Two isomers are described in Beil **12**,488,492 & [253]

Anilinopropionic Acid, Azido-$C_9H_{10}N_4O_2$ and *Diazido-* $C_9H_9N_7O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinopropionic Acids, $C_9H_{10}N_2O_4$. One isomer is described in Beil **12**,725

Dinitroanilinopropionic Acids, $C_9H_9N_3O_6$, mw

255.19, N 16.47%. One isomer is described in Beil 12, (364).

Trinitroanilinopropionic Acid, $C_9H_8N_4O_8$, mw 300.19, N 18.67%—not found in Beil or CA through 1956

Anilinopropyl Alcohol. See Anilinopropanol

γ-Anilinopropyleneglycol. See 3-Anilino-1,2-propanedol or 1-Anilino-2,3-propandiol

ANILINOSUCCINIC ACID AND DERIVATIVES

Anilinosuccinic Acid, called in Ger *Anilino-bernsteinsäure* or *N-Phenyl-asparaginsäure*, $C_6H_5NH.CH(COOH).CH_2COOH$. Several isomers are described in Beil **12**, 508 & [262]

Anilinosuccinic Acid, Azido-$C_{10}H_{10}N_4O_4$ and *Diazido-*$C_{10}H_9N_7O_4$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinosuccinic Acid, $C_{10}H_{10}N_2O_6$—not found in Beil

Dinitroanilinosuccinic Acid, $C_{10}H_9N_3O_8$—not found in Beil

2,4,6-Trinitroanilinosuccinic Acid, called in Ger: *N-Pikryl-asparaginsäure*, $(O_2N)_3C_6H_2.NH.CH(COOH).CH_2.COOH$, mw 248.20, N 22.58%. Rhombic lflts, mp 137°, diff sol in w, easily sol in alc & ether. Was prepd by mixing an aq soln of asparaginic (amino-succinic) acid with an equivalent quantity of picryl chloride in toluene and, after making the mixt strongly basic (by adding NaOH), shaking it for 3 hours. Its expl props were not investigated

Refs: 1)Beil **12**,770 2)K.Hirayama, ZPhysChem **59**,291(1909 & CA **4**,222 (1910)

ANILINOTETRAZOLE AND DERIVATIVES

5-Anilino-α(or 1H)-tetrazole or 5-Phenyl-amino-tetrazole, called in Beil *Tetrazolon-(5)-anil*, $C_6H_5.HN.C(=N-NH-N=)$, mw 161.17, N 43.46%. Ndls, mp 206° (with vigorous evolution of gas); very sol in hot alc, sol in hot w, diff sol in eth. Was first prepd by Stollé et al (Refs 2 & 3) together with other products. A higher melting product(211-12°) was obtained by Finnegan et al by heating 1-phenyl-5-aminotetrazole, mp 160-1°, as described in Ref 5

Refs: 1)Beil **26**, [243] 2)R.Stollé et al, JPraktChem **124**,269,297-8(1930) & CA **24**, 2748(1930) 3)R.Stollé et al, JPraktChem **147**,286(1937) & CA **31**,1807(1937) 4)F.R. Benson,ChemRevs **41**,7(1947) 5)W.G.Finnegan, JOC **18**,790(1953) 6)R.A.Henry et al, JACS **76**,88-93(1954) & CA **49**,2427(1955) (Thermal isomerization of substituted 5-aminotetrazoles)

(See aslo Aminophenyltetrazole)

Anilinotetrazole, Azido-, $C_7H_6N_8$ and *Diazido-*$C_7H_5N_{11}$ *Derivatives* were not found in Beil or CA through 1956

5-Nitroanilino-α(or 1H)-tetrazole or 5-(nitrophenylamino)-tetrazole, $C_7H_6N_6O_2$, mw 206.17, N 40.77%. Not found in Beil but its o-, m- and p-isomers are described by W.L.Garbrecht & R.M.Herbst in JOC **18**,1278-82(1933) & CA **48**, 12092-3(1954). This paper also gives UV absorption curves for some of these compds

5-(2',4'-Dinitroanilino)-α(or 1H)-tetrazole or 5-(2',4'-Dinitrophenylamino)-α(or 1H)-tetrazole, $(O_2N)_2C_6H_3.NH.C(=N-NH-N=)$, mw 251.17, N 39.04%. Brn-red powd, mp 174° with decompn, diff sol in hot w, eth & hot alc. Was obtained by heating 2,4-dinitrochlorobenzene with Na aminotetrazole in toluene for 6 hrs on an air bath and under reflux

Refs: 1)Beil—not found 2)R.Stollé et al, JPraktChem **139**,64(1933) & CA **28**, 1345(1934)

5-(2',4',6'-Trinitroanilino-)-α(or 1H)-tetrazole; 5-(2',4',6'-Trinitrophenylamino)-α(or 1H)-tetrazole or 5-(Picrylamino)-α(or 1H)-tetrazole, $(O_2N)_3C_6H_2NH.C(=N-NH-N=)$, mw 296.17, N 37.79%,

OB to CO_2 -54.0%, OB to CO -16.2%. Yel pdr, mp 224°; expl at higher temps, easily sol in acet, more diff in alc & w, hardly sol in eth. Can be prepd by heating 5-aminotetrazole with picryl chloride in AcOH for 2 hrs (Ref 2, p 63).

Its **silver salt**, $C_7H_3N_8O_6Ag$, N 27.89% and Ag 26.98%, brownish-yel solid, is a powerful explosive (Ref 2, p 64). Its **copper salt** (greenish powder) and **lead salt** (golden-yel powd) are not as powerful explosives

Note: None of these salts was recommended by Stollé for use as igniting or initiating agents in blasting caps or detonators

Refs: 1)Beil–not found 2)R.Stollé et al, JPraktChem **139**,63-4(1933) & CA **28**,1345 (1934) 3)D.W.Moore & L.A.Burkhardt, Anal Chem **26**,1923(1954) & CA **49**,4363(1955)(X-ray powder diffraction pattern of 5-picrylaminotetrazole)

ANILINOTOLUENE; DIPHENYLMETHYLAMINES AND DERIVATIVES

Anilinotoluenes; Phenyltoluidines; Diphenylmethylamines and Methyldiphenylamines, $C_{13}H_{13}N$. All isomers are described in Beil **12**,180,787,857,905,(166) & [105,436,467,493]

Anilinotoluene, Azido-$C_{13}H_{12}N_4$ and *Diazido-*$C_{13}H_{11}N_7$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinotoluenes; Mononitrophenyltoluidines; Nitrodiphenylmethylamines and Mononitromethyldiphenylamines, $C_{13}H_{12}N_2O_2$, are described in Beil **12**,787,876,906 &[437].

Dinitroanilinotoluenes; Dinitrophenyltoluidines; Dinitrodiphenylmethylamines or Dinitromethyldiphenylamine, $C_{13}H_{11}N_3O_4$, mw 273.24, N 15.38%. Several isomers are described in Beil **12**,752,787,851,857,879,906, 1010 & [409,443]

Trinitroanilinotoluenes; Trinitrophenyltoluides; Trinitrodiphenylmethylamines and Trinitromethyldiphenylamines, $C_{13}H_{10}N_4O_6$, mw 318.24, N 17.61%. Several isomers are described in the literature:

Note: There seems to be confusion in the literature in regard to identification of some trinitro- and tetranitro- derivs. The compds are therefore listed according to their names and props as reported by the authors. The structural formulae of some compds cannot be given because they were not definitely established

2-(2',4', 6'-Trinitroanilino)-toluene; N-(2',-4', 6'-Trinitrophenyl)-o-toluidine; 2', 4',-6'-Trinitro-2-methyldiphenylamine or Picryl-o-toluidine. Called by Reverdin *o-Tolyl-2', 4', 6'-trinitrophenylamine*, $(O_2N)_3C_6H_2.NH.C_6H_4.CH_3$; orange-red crysts, mp 163-4°; easily sol in acet, chlf, benz & AcOH, diff sol in eth & cold alc, nearly insol in ligroin. Can be prepd by treating o-toluidine with picryl chloride or by other methods

Refs: 1)Beil **12**,787,(377) & [437] 2)F. Reverdin & P.Crépieux, Bull Fr [3]**29**,236 (1903) & Ber **36**,31(1903) 3)F.Kehrmann et al, Helv **4**,540(1921) & CA **15**,3449(1921)

3-(2',4',6'-Trinitroanilino)-toluene; N-(2', 4',6'-Trinitrophenyl)-m-toluidine; 2',4',6'-Trinitro-3-methyl-diphenylamine or Picryl-m-toluidine. Called by James et al *2,4,6-Trinitrophenyl-m-tolylamine*, $(O_2N)_3C_6H_2.NH.C_6H_4CH_3$; exists in stable yel form, mp 129° and in labile red form, mp 118.5° (Ref 3), mp 119° (Ref 4). Can be prepd by treating m-toluidine with picryl chloride in alc (Ref 2) or by other methods (Refs 1,3 & 4)

Refs: 1)Beil **12**,857,(399) & [467] 2)M. Busch & E.Pungs,JPraktChem *79*,549-50 (1909) 3)T.C.James et al, JCS **117**,1276 (1920) 4)B.Linke,Ber**56**,851(1923)

4-(2',4',6'-Trinitroanilino)-toluene; N-(2',4',6'-Trinitrophenyl)-p-toluidine; 2',-4',6,-Trinitro-4-methyl-diphenylamine or Picryl-p-toluidine. Called by James et al *2,4,6-Trinitrophenyl-p-tolylamine*, $(O_2N)_3C_6H_2.NH.C_6H_4CH_3$. Exists, according to Ref 2, as yel monocl ndls, mp 163° or as red rhombic ndls, mp 165-9°, and according to Ref 3 as orange-yel ndls, mp 163-4°, or as blood-red prisms, mp 165°. Can be prepd by treating p-toluidine with picryl chloride

in alc(Ref 2) or by other methods(Refs 1 & 3)

Refs: 1)Beil **12**,906,(414) & [494] 2)M. Busch & E.Pungs, JPraktChem **79**,547(1909) 3)T.C.James et al, JCS **117**,1276(1920)

2,2',6-Trinitro-N-methyl-diphenylamine or 1-(2'-Nitro-N-methylanilino)-(2,6-dinitrobenzene), $O_2N.C_6H_4.N(CH_3).C_6H_3(NO_2)_2$; yel ndls, mp 221-3°. Was obtained on prolonged heating of a mixt of 2,6-dinitrochlorobenzene, N-methyl-o-nitraniline, K_2SO_4, CuI and amyl alcohol

Refs: 1)Beil–not found 2)H.Hillemann, Ber **71B**, 50(1938) & CA **32**, 2134(1938)

4,2',4'-Trinitro-2-methyl-diphenylamine or N-(2',4'-Dinitrophenyl)-4-nitro-o-toluidine, $(O_2N)_2C_6H_3.NH.C_6H_3(CH_3).NO_2$; orange prisms, mp 159°. Was obtained after 2-methyl-2',4'-dinitrodiphenylamine had been shaken with glacial AcOH contg nitric acid (d1.42) and the mixt left at RT overnight

Refs: 1)Beil–not found 2)G.G.Coker et al, JCS **1951**,112 & CA **45**,8510(1951)

4,2',4'-Trinitro-3-methyl-diphenylamine or N-(2',4'-Dinitrophenyl)-4-nitro-m-toluidine, $(O_2N)_2C_6H_3.NH.C_6H_3(CH_3).NO_2$; red prisms mp 171-2°(from AcOH). Was obtained after a mixt of 1-chloro-2,4-dinitrobenzene, 6-nitro-m-toluidine and anhydrous AcONa had been heated for an hr at 190°

Refs: 1)Beil–not found 2)G.G.Coker et al, JCS **1951**,112 & CA **45**,8510(1951)

3,2',4'-Trinitro-4-methyldiphenylamine or N(2'4'-Dinitrophenyl)-3-nitro-p-toluidine, $(O_2N)_2.C_6H_3.NH.C_6H_3(NO_2).CH_3$; golden-brn plates, mp 207°. Can be prepd by heating a mixt of 1-chloro-2,4-dinitrobenzene , 2-nitro-p-toluidine and K_2CO_3 at 150-60° for an hour (Ref 2) or as described in Ref 3

Refs: 1)Beil–not found 2)A.F.Childs & S.G.P. Plant, JCS **1948**,1993 & CA **43**,2176 (1949)

2,2',4'-Trinitro-4-methyldiphenylamine or N-(2',4'-Dinitrophenyl)-2-nitro-p-toluidine, $(O_2N)_2C_6H_3.NH.C_6H_3(NO_2).CH_3$; orange-yel ndls, mp 221°. Can be prepd by heating 1-chloro-2,4-dinitrobenzene for an hr on a steam bath with nitric acid (d1.63) (Ref 2). This compd was considered by Reverdin & Crépieux to be the tetranitro with mp 219° (see below)

Refs: 1)Beil–not found 2)A.F.Childs & S.G.P.Plant, JCS **1948**,1993 & CA **43**,2176 (1949)

Tetranitroanilinotoluenes; Tetranitrophenyltoluidines; Tetranitrodiphenylmethylamines and Tetranitromethyldiphenylamines, $C_{13}H_9N_5O_8$, mw 363.24, N 19.28%. The following isomers are described in the literature:

2,4,2',4'-Tetranitro-N-methyl-diphenylamine or 1-(2',4'-Dinitro-N-methylanilino)-(2,4-dinitrobenzene), $(O_2N)_2C_6H_3.N(CH_3).C_6H_3(NO_2)_2$; yel leaflets(from alc or AcOH), mp 210°. Was prepd by nitrating 2,4-dinitro-N-methyl-diphenylamine as described in Ref 2. Its expl props were not examined

Refs: 1)Beil **12**,753 2)R.Nietzki & A. Raillard,Ber **31**,1461(1898)

2',4',x,x-Tetranitro-4-methyl-diphenylamine, called by Reverdin *Tetranitro-p-tolyl-phenylamine,* $(O_2N)_4C_{13}H_9N$, red-brn prisms(from alc), mp 219°. Was prepd by nitrating 2',4'-dinitro-4-methyl-diphenylamine(red ndls, mp 137°) with nitric acid (See 2,2'4'-Trinitro-4-methyldiphenylamine)

Refs: 1)Beil **12**,906 2)F.Reverdin & P. Crépieux,Bull Fr [3] **29**,237(1903) & Ber **36**, 31-2(1903)

Note: According to A.F.Childs & S.G.P. Plant, JCS **1948**,1993 & CA **43**,2176(1949), the product obtained by Reverdin & Crépieux is actually *2,2',4'-trinitro-4-methyldiphenylamine,* mp 221°(see above). Further nitration of this compd under more vigorous conditions gave *2,6,2',4'-tetranitro-4-methyldiphenylamine,* mp 169°(see below)

4,6,2',4'-Tetranitro-2-methyldiphenylamine or N-(2',4'-Dinitrophenyl)-4,6-dinitro-o-toluidine, $(O_2N)_2C_6H_3.NH.C_6H_2(CH_3)(NO_2)_2$; yel ndls, mp 190°. Can be prepd by heating, on a steam bath, 2',4'-dinitro-2-methyldiphenylamine with a mixt of glacial AcOH and nitric acid (d1.42) until a straw colored soln is obtained (ca ½ hr). Its expl props were not examined

Refs: 1)Beil—not found 2)G.G.Coker et al, JCS **1951**, 112 & CA **45**,8510(1951)

4,6,2',4'-Tetranitro-3-methyl-diphenyl-amine, or N-(2',4'-Dinitrophenyl)-4,6-dinitro-m-toluidine, $(O_2N)_2C_6H_3.NH.C_6H_2(NO_2)_2.CH_3$; yel plates (from AcOH), mp 208-9°. Was obtained on heating on a water bath 3-methyl-2',4'-dinitrodiphenylamine with concd H_2SO_4 and HNO_3(d 1.42) until the soln became pale yel (ca ½ hr). Its expl props were not examined

Refs: 1)Beil—not found 2)G.G.Coker et al, JCS **1951**, 113 & CA **45**, 8510(1951)

2,2',4',6'-Tetranitro-4-methyldiphenylamine or N-(2',4',6'-Trinitrophenyl)-2-nitro-p-toluidine, $(O_2N)_3C_6H_2.NH.C_6H_3(NO_2).CH_3$; golden-brn plates, mp 217-19°. Can be prepd by heating a mixt of picryl chloride and 3-nitro-p-toluidine to 160° (Ref 2). Its expl props were not examined

Refs: 1)Beil—not found 2)A.F.Childs & S.G.P.Plant, JCS **1948**, 1993 & CA **43**, 2176(1949)

2,6,2',4'-Tetranitro-4-methyldiphenylamine or N-(2',4'-Dinitrophenyl)-2,6-dinitro-p-toluidine- $(O_2N)_2C_6H_3.NH.C_6H_2(NO_2)_2.CH_3$; bright yel ndls (from alc), mp 169. Can be prepd by heating 2,2',4'-trinitro-4-methyldiphenylamine on a steam bath with concd HNO_3(d1.42) (Ref 2). Its expl props were not examined

Refs: 1)Beil—not found 2)A.F.Childs & S.G.P.Plant, JCS **1948**, 1993 & CA **43**, 2176 (1949)

Pentanitroanilinotoluenes, $C_{13}H_8N_6O_{10}$, mw 408.24, N 20.59% were not found in Beil or CA through 1956. There is, however, *Pentanitro-N-methyl-p-biphenylamine*, known also as *N-Methyl-N,2,2',4',6-pentanitroxenylamine*, which has the same empirical formula as above. This expl compd is described in this work under Methylbiphenylamine and Derivatives

Hexanitroanilinotoluenes; Hexanitrophenyltoluidines; Hexanitrodiphenylmethylamines and Hexanitromethyldiphenylamines, $C_{13}H_7N_7O_{12}$, mw 453.24, N 21.63%. The following isomer is described in the literature:

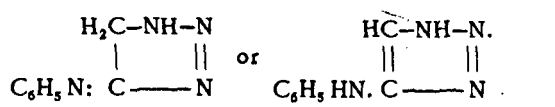
2,4,6,2',4',6'-Hexanitro-N-methyl-diphenylamine or N-Methyl-dipicrylamine, $(O_2N)_3C_6H_2.N(CH_3).C_6H_2(NO_2)_3$; yel leaflets (from alc); mp 236-7°; insol in w, v sl sol in eth & hot alc; sol in AcOH and v sol in acet. Was prepd by nitrating N-methyl-N-phenyl-2,4-dinitroaniline with nitric acid (d1.49) (Refs 1 & 2). Its expl props are: power by Trauzl Test 87% PA and FI (figure of insensitiveness) 92% PA (Ref 4). Hantzsch & Opolski (Ref 3) also prepd the compd as well as its *aci* isomer, $(O_2N)_3C_6H_2.N{:}C_6H_2(NO_2)_2{:}N.O.OCH_3$, violet crysts, mp 140-1° with decompn

Refs: 1)Beil **12**,766 2)A.Moulder, Rec **25**, 121-2(1906) 3)A.Hantzsch & St.Opolski, Ber **41**,1747-9(1908) 4)A.H.Blatt, OSRD, 2014(1944), listed as Hexanitrodiphenylmethylamine

ANILINOTRIAZOLE AND DERIVATIVES

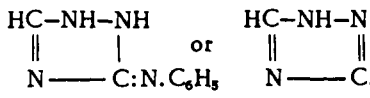
Anilinotriazoles, $C_8H_8N_4$, mw 160.18, N 34.98%. The following isomers are described in the literature:

4-Anilino-1,2,3-triazole, called in Beil *1.2.3-Triazolon-(4)-anil*,



Crysts, mp 139-40°. Methods of prepn are given in Beil **26**, 134 & [75]. Its constitution was discussed in JCS **123**,265(1923)

3-Anilino-1,2,4-triazole, called in Beil *1.2.4-Triazolon-(3)-anil,*



Crysts, mp 180°. Prepn is given in Beil **26**, [76]

(See also Aminophenyltriazoles and Derivatives, p A249L)

Anilinotriazole, Azido-$C_8H_7N_7$ and *Diazido-*$C_8H_6N_{10}$ *Derivatives* were not found in Beil or CA through 1956

Nitrosoanilinotriazole, $C_8H_7N_4.NO$, mw 189.18, N 37.02%. Yel crysts decompg ca 117-18°

Ref: Beil **26**, 134 & [75]

Note: This compd might be of interest on acct of its high nitrogen content

Mononitroanilinotriazole, $C_8H_7N_5O_2$, mw 205.18, N 34.12% and higher nitrated derivs were not found in Beil or CA through 1956

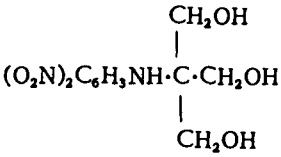
ANILINOTRIMETHYLOLMETHANE AND DERIVATIVES

Anilinotrimethylolmethane; N-(Trimethylolmethane)-aniline; Phenylaminotrimethylolmethane; N-Phenyl-(tris-hydroxymethyl)-methylamine; 2-Anilino-2-hydroxymethyl-1,3-dihydroxypropane; 2-Anilino-2-hydroxymethyl-1,3-propanediol or Phenyltrimethylolmethylamine, $C_6H_5.NH.C(CH_2OH)_3$. This may be considered as the parent compd of the derivs described below:

Anilinotrimethylolmethane, Azido-$C_{10}C_{14}N_4O_3$ and *Diazido-*$C_{10}H_{13}N_7O_3$ *Derivatives* were not found in Beil or CA through 1956

Mononitrotrimethylolmethane, $C_{10}H_{14}N_2O_5$ – not found in Beil or CA through 1956

2,4-Dinitroanilino-trimethylolmethane; *N-(2,4-Dinitrophenylamine)-trimethylolmethane; N-(2,4-Dinitrophenyl)-(tris-hydroxymethyl-methyl)-amine; 2-(2',4'-Dinitroanilino)-2-hydroxymethyl-1,3-propanediol; 2,4-Dinitrophenyltrimethylolmethylamine or 2,4-Dinitrophenyltrihydroxymethylmethylamine,*



N 14.63%, OB to CO_2 –108.6%. Golden-yel crysts from dil alc, mp 100°. Was obtained in 76% yield by condensation of chloro-2,4-dinitrobenzene with 2-amino-2-hydroxymethyl-1,3-propanediol, as described in Ref 2, p 12. Its Q_c^p is 1222.9 kcal/mol and Q_f^p –65.7(Ref 3)

Refs: 1)Beil – not found 2)R.C.Elderfield, OSRD Rept 158(1941),7 & 12(PBL Rept 31094) 3)G.B.Kistiakowsky, OSRD Rept 702 (1942)

N-(2,4,6-TRINITRO-N-NITRANILINO)-TRIMETHYLOLMETHANE TRINITRATE; N-(2,4,6 Trinitrophenyl-N-nitramino)-trimethylolmethane Trinitrate or N-(2,4,6-Trinitrophenyl)-(tris-nitroxymethyl-methyl)-nitramine, designated as **Heptryl.** Called by Kistiakowsky *N-Nitro-N-picryl-trimethylolmethylamine Trinitrate.* Also called: *2-(2',-4',6'-Trinitro-N-nitranilino)-2-nitroxymethyl-1,3-dinitroxypropane; 2-(N,2',4',6'-Tetranitroanilino)-2-nitroxymethyl-1,3-propanediol Dinitrate or 2,4,6-Trinitrophenyltrimethylolnitramine Trinitrate,* $(O_2N)_3C_6H_2.N(NO_2).C(CH_2.ONO_2)_3$, mw 512.23, N 21.87%, OB to CO_2 –21.9%, OB to CO +9.37%. Yel

crysts, mp 154-7° (decompn); ignites at 180° and explodes >360° with a purple flash. Was prepd by nitrating 2,4-dinitro-anilino-trimethylolmethane (see above) with mixed nitric-sulfuric acid, as described in Ref 2, pp 14-15. Heptryl was purified by dissolving the crude material in acetone and adding alcohol

Heptryl is a HE comparable in power, brisance and sensitivity to PETN (Refs 2,4 & 6)

Following are some props of Heptryl:

a)Brisance, comparable to PETN

b)Heat of combustion, Q_c^v 1160.7 kcal/mol (Refs 3,5 & 6)

c)Heat of formation, 57.3 kcal/mol (Ref 5)

d)Hygroscopicity at 25° & 90% RH, gained 0.07%; and at 100% RH, gained 0.2% (Ref 6)

e)Ignition temperature, ca 180° (Ref 6)

f)Impact sensitivity. A small sample wrapped in tin foil detonated when struck with a hammer on an iron anvil, and did not detonate when a concrete anvil was used (Ref 2)

g)International Test, 75° (thermal stability), loss of wt 0.5% (Ref 6)

h)Power by Ballistic Mortar Test, 143% TNT (Refs 4 & 6)

i)Stability (thermal) at 100°, not acid and no expln in 300 mins (Ref 4)

j)Stability (thermal) at 135°, exploded in 13 mins in a closed container (Refs 4 & 6)

Refs: 1)Beil– not found 2)R.C. Elderfield, OSRD Rept 158(1941), 8 & 14-16 (PBL Rept 31094) 3)G.B.Kistiakowsky, OSRD Rept 702(1942), Table I, Compd Gb2 4)R.McGill, OSRD Rept 830(1942), 29 5)A.H.Blatt & F.C.Whitmore, OSRD Rept 1085 (1942), 116 6)A.H.Blatt, OSRD Rept 2014(1944), listed as 2,4,6-Trinitrophenyl-trimethylolmethyl-nitramine Trinitrate

ANILINOVALERIC ACIDS AND DERIVATIVES

Anilinovaleric (Anilinovalerianic) and *Anilinoisovaleric* (Anilinoisovalerianic) *Acids,* $C_{11}H_{15}NO_2$ are described in Beil **12**,497

Anilinovaleric & Anilinoisovaleric Acids, Azido-, $C_{11}H_{14}N_4O_2$ and *Diazido–*$C_{11}H_{13}N_7O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanilinovaleric Acids, $C_{11}H_{14}N_2O_4$– not found in Beil

Dinitroanilinoisovaleric Acid, $C_{11}H_{13}N_3O_6$, is described in Beil **12**,(364). *α-(2,4,6-Trinitro-anilino)-isovaleric Acid*(called N-Pykrylvalin in Ger), $(O_2N)_3C_6H_2.NH.CH(COOH).CH(CH_3)_2$, mw 328.24, N 17.07%. Lt yel ndls, mp 171°, easily sol in alc or eth, sl sol in w (0.029% at RT). Was prepd by shaking 1 mol of α-aminoisovaleric acid, 1 mol NaOH and 1 mol picryl chloride for 3 hrs in toluene

Refs: 1)Beil **12**,770 2)K.Hirayama, ZPhysChem **59**,291(1909) & CA **4**,222(1910)

Note: No higher nitrated anilinovaleric acids were found in Beil or CA through 1956

Anilite. A liquid expl consisting of butane and liq nitrogen peroxide, N_2O_4. It is sensitive to mechanical action and equal in strength to 161% P A.

Ref: Anon, "Elements of Armament Engineering", US Military Academy, West Point, NY (1954), 71

Anilithe or Anilite Some small antipersonnel aerial bombs used by the French during WW I consisted of two separate compartments, one filled with liquid nitrogen peroxide, N_2O_4, the other with a liq combustible material

such as carbon disulfide or gasoline. When such a bomb was released from an airplane, a small propellant on the nose of the bomb actuated the mechanism which caused the two liquids to mix. During the flight, the bomb became filled with a very powerful and sensitive mixture (called anilithe or anilite) which detonated without any initiator upon impact with the target

Refs: 1)A.Holler,Nature **106**,831-4(1920) & CA **15**,1401(1921) 2)T.L.Davis, Army Ordn **20**,93(1939) 3)Davis(1955), 355 4)Bebie(1943),27-8

ANILINOXYLENE AND DERIVATIVES

Anilinoxylenes; Phenylxylidines; Ditolyl amines and Dimethyldiphenylamines, $C_{14}H_{15}N$. All isomers are described in Beil **12**, 787,858,907,1115,(377,414,415) & [437,467,494]

Anilinoxylene, Azido, $C_{14}H_{14}N_4$ and *Diazido-*$C_{13}H_{13}N_7$ *Derivatives* were not found in Beil or Ca through 1956

Mononitroanilinoxylenes, $C_{14}H_{14}N_2O_2$. Several isomers are described in Beil **12**,1001 & [530]

Dinitroanilinoxylenes, $C_{14}H_{13}N_3O_4$. Several isomers are described in Beil **12**, 787,1002 (443,479-481,483,488) & [462,479,481,494, 536,618]

Trinitroanilinoxylenes, $C_{14}H_{12}N_4O_6$, mw 332.27, N 16.86%. Several isomers are described in Beil **12**, 906,907,1109,1115,1133, (377,414) & [608]

Tetranitroanilinoxylenes, $C_{14}H_{11}N_5O_8$, mw 377.27, N 18.56%–not found in Beil or CA through 1956

Pentanitroanilinoxylenes etc, $C_{14}H_{10}N_6O_{10}$, mw 422.27, N 19.90%–not listed in Beil or CA through 1956

Hexanitroanilinoxylenes etc, $C_{14}H_9N_7O_{12}$, mw 467.27, N 20.98%. The following isomers are listed in the literature:

2,3,6(?),2',3',6'(?)-Hexanitro-4,4'-dimethyl-diphenylamine, called by Lehne *Hexanitroditolylamine,* $[CH_3C_6H(NO_2)_3]_2NH$, yel crysts, mp 285°, very diff sol in org solvents. Can be prepd by treating either p,p'-ditolylamine or N-nitroso-p,p-tolylamine with cold fuming nitric acid. It expl props were not investigated

Refs: 1)Beil **12**,1013 2)A.Lehne, Ber **13**,1545(1880)

2,4,6,2',4',6'-Hexanitro-3,3'-dimethyl-diphenylamine or Bis(3-methyl-2,4,6-trinitro-phenyl)-amine, $[CH_3.C_6H(NO_2)_3]_2NH$, brn tables (from alc), mp 60°. Can be prepd by warming 4,6-dinitro-3,3'-dimethyl-diphenylamine with nitric acid(d 1.5) on a water bath. Its expl props were not investigated

Refs: 1)Beil **12**,[482] 2)O.L.Brady et al, JCS **125**,2404(1924)

Animal and Vegetable Fats and Oils, Nitrated. See Fats and Oils, Nitrated

Anilit. A Ger expl contg at least 70% AN, not more than 5% sugar and the rest aniline copper sulfate

Ref: R.Escales, Ammonsprengstoffe (1909), 104

Anilite or Anilithe. The name given to some Panclastite type expls used by the French during WW I for filling some aeroplane bombs. These bombs contained two compartments divided by a horizontal partition. One compartment contained liquid nitrogen tetroxide, N_2O_4(called in Fr "peroxide d'azote), while the other contained CS_2,NB,MNT or gasoline. As long as the liquids were not mixed they could be transported without hazard in a plane. When the bomb was released, a small propeller on its nose, actuated by passage through the air, opened a valve which permitted the two liquids to mix. This transformed the contents into a very powerful expl mixt which was so sensitive that it required no fuze but exploded immediately

upon impact with the target. When it was desired to have these bombs explode in the air before they reached targets, they were provided with a time fuze

Anilite was cheap to prepare and so were the bombs using it. The power and brisance of these bombs was sl higher than those contg TNT or PA

The term "anilite" was extended after WWII to any liq expl contg N_2O_4 as an oxidizer, and a liq carbonaceous material (such as benzene, butane, etc) as a fuel

Several formulations of "anilite" consisting of N_2O_4 and butane were investigated before WWII at PicArsn(Ref 4). The mixts: N_2O_4/butane–70/30, 60/40 and 50/50, examined at PicArsn were extremely sensitive to rifle bullet test (using cal .30 rifle at a distance of 30 yards from the muzzle to an open bomb contg the sample). All samples were unstable (they were constantly fuming) and very hazardous to handle. The 70/30 mixt had a rate of deton ca 10% higher than TNT and brisance by fragmentation test ca 130% of TNT. In fragmentation test a 3-inch AA M42 shell loaded with 70/30–anilite gave 408 fragments vs 351 fragments for TNT

Refs: 1)A.Haller, BullSocEncourIndNat **119**,761-5(1920) & CA **15**,1401(1921) 2)Pascal(1930), 193-4 3)Pepin Lehalleur (1935), 360 4)C.J.Bain, "Tests of Liquid Nitrogen Peroxide Explosive", PATR 985 (1939) 5)Davis (1943), 355 6)Pérez-Ara(1945), 229 7)Kirk & Othmer **6**(1951),58

Anima (Ital) Bore (of a weapon)

Anima liscia (Ital) Smooth bore

Anima rigata (Ital) Rifled bore

Anisalcohol or Anisic Alcohol. Same as Anisyl Alcohol

Anisalanishydrazide Azide or Anisylidene-[*a*-azidoanisylidene]-hydrazine, $H_3CO.C_6H_4.C(N_3):N.N:CH.C_6H_4.OCH_3$ mw 309.22, N 22.64%. Ndls (from eth), mp 113° (decomp); defgr at higher temps; insol in w and fairly sol in eth or hot alc. Was prepd as described in Ref 2

Refs: 1)Beil **10**,[102] 2)R. Stollé, Ber **55** 1300 (1922)

ANISALDEHYDES AND DERIVATIVES

Anisaldehyde or Methoxybenzaldehyde, $H_3CO.C_6H_4.CHO$, mw 136.14

Note: Although the name "anisaldehyde" previously was reserved for the "p-methoxybenzaldehyde", current nomenclature extends the term "anisaldehyde" to all methoxybenzaldehydes, provided it is indicated which of the isomers is being described. Thus there will be o- (or 2-), m- (or 3-) and p- (or 4-) anisaldehydes

These compds are described as "methoxybenzaldehydes" in Beil **8**, 43,59,67,(519,525, 529) & [40,53,64]

Note: A.Albert & A.Hampton, JCS **1952**, 4985-93 & CA **48**,8231(1954) described prepn in 95% yield from m-$HO.C_6H_4.CHO$ with Me_2SO_4 & NaOH in MeOH and its nitration to 3,2-$MeO.(O_2N):C_6H_3.CHO$

Anisaldehyde, Azido-, $C_8H_7N_3O_2$ and *Diazido-* $C_8H_6N_6O_2$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanisaldehydes or Mononitromethoxybenzaldehyde, $H_3CO.C_6H_3$-$(NO_2)CHO$. Several isomers are described in Beil **8**, 56,57, 62,63,83,(523,527,533) & [58,77]

Dinitroanisaldehydes or Dinitromethoxybenzaldehydes, $H_3CO.C_6H_2(NO_2)_2.CHO$, mw 226.14, N 12.39%. The following isomers are described in the literature:

x,x-Dinitro-(m-anisaldehyde) or x,x-Dinitro-3-methoxy-benzaldehyde of mp 110°, first prepd by Tiemann & Ludwig(Ref 2), was later (Ref 3) identified as a mixt of 2,6- and 4,6-dinitro-3-methoxy-benzaldehydes

Refs: 1)Beil **8**,63 & [62] 2)F.Tiemann & R.Ludwig, Ber **15**, 2056 (1882) 3)H.H. Hodgson & H.G.Beard, JCS **1927**,2376

x,x-Dinitro-(m-anisaldehyde) or x,x-Dinitro-3-methoxy-benzaldehyde of mp 155°, prisms, first prepd by Tiemann & Ludwig(Ref 2), was later identified as 2,6-dinitro-3-methoxy-benzaldehyde, mp 157°(Ref 3)

Refs: Same as above and also J. Tröger & C.Eicker, JPraktChem **116**, 29(1927)

4,6-Dinitro-(m-anisaldehyde) or 4,6-Dinitro-3-methoxy-benzaldehyde, prisms, mp 131°. Other props & prepn are in Refs

Refs: 1)Beil **8**,[62] 2)H.H.Hodgson & H.G.Beard, JCS **1927**, 2381

3,5-Dinitro-(p-anisaldehyde) or 3,5-Dinitro-4-methoxy-benzaldehyde, yel ndls, mp 86°. Other props & prepn are in Refs

Refs: 1)Beil **8**,84 & [80] 2)E.Wörner, Ber **29**,157(1896) 3)M.P.de Lange, Rec **45**,47(1926) 4)H.H.Hodgson & H.G.Beard, JCS **1927**,2376

Trinitroanisaldehyde or Trinitromethoxy-benzaldehyde, $H_3CO.C_6H(NO_2)_3.CHO$, mw 271.14, N 15.50%—not found in Beil or CA through 1956

p-Anisaldehyde Perchlorate or 4-Methoxy-benzaldehyde Perchlorate, $2\ C_8H_8O_2 + HClO_4$; col deliq prisms or plates. Was prepd from ethereal soln of anisaldehyde and 70% perchloric acid as described in Ref 2. Its expl props were not investigated

Refs: 1)Beil **8**,(529) 2)K.A.Hofmann et al, Ber **43**, 2629(1910)

ANISALDEHYDEPHENYLHYDRAZONE AND DERIVATIVES

p-Anisaldehydephenylhydrazone; Methoxy-benzaldehyde-phenylhydrazone or Anisylidene-phenylhydrazine, $H_3CO.C_6H_4.CH{:}N{\cdot}NH{\cdot}C_6H_5$ is described in Beil **15**,192,(51) & [80]

Anisaldehydephenylhydrazone, Azido- $C_{14}H_{13}N_5O$ and *Diazido-*, $C_{14}H_{12}N_8O$ *Derivatives* were not found in Beil or CA through 1956

p-Anisaldehyde-(N-nitrosophenylhydrazone), $CH_3O.C_6H_4.CH{:}N.N(NO).C_6H_5$ is described in Beil **15**,417

p-Anisaldehyde-[2-(3- and 4-)nitrophenyl-hydrazones] $H_3CO{\cdot}C_6H_4{\cdot}CH{:}N{\cdot}NH{\cdot}C_6H_4(NO_2)$, are described in Beil **15**,(137) & [180,183,199]

p-Anisaldehyde-(2,4-dinitrophenylhydrazone), $H_3CO.C_6H_4.CH{:}N.NH.C_6H_3(NO_2)_2$, mw 316.27, N 17.72%, is described in Beil **15**,[218]

Anisaldehyde-trinitrophenylhydrazones, $H_3CO.C_6H_4.CH{:}N.NH.C_6H_2(NO_2)_3$, mw 361.27, N 19.39%, not found in Beil, but one isomer *anisaldehyde-picrylhydrazone or 4-methoxy-benzaldehyde-2,4,6-trinitrophenylhydrazone* is described by J.J.Blanksma & M.L.Wackers, Rec **55**, 665(1936)

p-Anisaldehyde-phenylhydrazone Peroxide, $H_3CO.C_6H_4{\cdot}CH\underset{O_2}{\overset{\frown}{\text{—}}}N.NH.C_6H_5$, mw 258.27, N 10.85%.

Yel ndls(from benz + petr eth), dec 83-4° and expl when heated, in a flame; v sol in eth, sol in alc or benz and diff sol in petr eth. Was prepd by treating anisaldehyde-phenyl-hydrazone with hydrogen peroxide. It turns brown by action of light but is stable in the dark

Refs: 1)Beil **15**,(51) 2)M.Busch & W.Dietz, Ber **47**,3287(1914)

3-Nitro-(p-anisaldehyde)-phenylhydrazone, $H_3CO.C_6H_3(NO_2).CH{:}N.NH.C_6H_5$, is described in Beil **15**, 193 & [80]

3-Nitro-(p-anisaldehyde)-(4'-nitrophenylhydrazone), $H_3CO.C_6H_3(NO_2).CH{:}N.NH.C_6H_4(NO_2)$ is described in Beil *15*,476 & [200]

Nitroanisaldehyde-dinitrophenylhydrazones, $H_3CO.C_6H_3(NO_2).CH{:}N.NH.C_6H_3(NO_2)_2$, mw 361.25, N 19.39%. They are not found in Beil, but the isomer, *2-nitro-(p-anisaldehyde)-2',4'-dinitrophenylhydrazone)* is described by W.R.Boon, JCS **1949**, Suppl Issue, p S230 and the isomer *4-nitro-o-anisaldehyde 2,4-dinitrophenylhydrazone* by W.Berends et al, Rec **74**,1341(1955)

Nitroanisaldehyde-trinitrophenyl hydrazones, $H_3CO.C_6H_3(NO_2).CH{:}N.NH.C_6H_2(NO_2)_3$ not found in Beil or CA through 1956

Dinitroanisaldehyde-phenylhydrazones,

$H_3CO.C_6H_2(NO_2)_2.CH:N.NH.C_6H_5$. Two isomers: *2,6-dinitro-3-methoxybenzaldehyde-phenylhydrazone*, mp 185° and *4,6-dinitro-3-methoxybenzaldehyde-phenylhydrazone*, mp 210°, are described by J. Troger & C.Eicker, JPraktChem, **116**,21 & 29(1927), of which only the first compd is listed in Beil **15**, [80]

Dinitroanisaldehyde-nitrophenylhydrazones, $H_3CO.C_6H_2(NO_2)_2.CH:N.NH.C_6H_4NO_2$, mw 361.25, N 19.39%. The following isomers are described in the literature:

2,6-Dinitro-(p-anisaldehyde)-4'-nitrophenylhydrazone or 2,6-Dinitro-3-methoxybenzaldehyde-4-nitrophenylhydrazone, lt orange plates exploding at ca 260°. Was obtained by treating 2,6-dinitro-3-methoxybenzaldehyde with p-nitrophenylhydrazone, as described in Ref 2

Refs: 1)Beil **15**,[199] 2)H.H.Hodgson & H.G.Beard, JCS **1927**, 2381

3,5-Dinitro-(p-anisaldehyde)-(4'-nitrophenylhydrazone), dk brn crysts, mp 275°. Was prepd by treating 3,5-dinitro-4-methoxybenzaldehyde with p-nitrophenylhydrazone as described in Ref 2

Refs: 1)Beil **15**, [201] 2)M.P.deLange, Rec **45**, 49(1926) & CA **20**, 1982(1926)

4,6-Dinitro-(m-anisaldehyde)-(4'-nitrophenylhydrazone), terra-cotta ndls mp >300°. Was prepd by treating 4,6-dinitro-3-methoxybenzaldehyde with p-nitrophenylhydrazone as described in Ref 2

Refs: 1)Beil **15**, [199] 2)H.H.Hodgson & H.G.Beard, JCS **1927**, 2381

Dinitroanisaldehyde-dinitrophenylhydrazones, $H_3CO{\cdot}C_6H_2(NO_2)_2{\cdot}CH:N{\cdot}NH{\cdot}C_6H_3(NO_2)_2$ not found in Beil or in CA through 1956

Dinitroanisaldehyde-trinitrophenylhydrazones, $H_3CO{\cdot}C_6H_2(NO_2)_2{\cdot}CH:N{\cdot}NH{\cdot}C_4H_2(NO_2)_3$ not found in Beil or CA through 1956

Trinitroanisaldehyde-trinitrophenylhydrazone, $H_3CO{\cdot}C_6H(NO_2)_3{\cdot}CH:N{\cdot}NH{\cdot}C_6H_2(NO_2)_3$ was not found in Beil or CA through 1956

Anisenyltetrazotic Acid. An old name for Anisyltetrazole

ANISIC ACIDS AND DERIVATIVES

Anisic or Methoxybenzoic Acid, $H_3CO{\cdot}C_6H_4{\cdot}COOH$, mw 152.14

Note: Although the name "anisic acid" previously was reserved for the "p-methoxybenzoic acid", current nomenclature extends the term "anisic acid" to all methoxybenzoic acids, provided it is indicated which of the isomers is being described. Thus there will be o- (or 2-), m- (or 3-) and p- (or 4-) anisic acids

These compds are described as *methoxybenzoic acids* in Beil **10**, 64, 137, 154, (27, 64, 69) & [39,80,91]

Note: According to Davis (1943), p 170, nitration of p-anisic acid yields trinitroanisole (see under Anisole and Derivatives)

Anisic Azide. See Anisoylazide

Anisic Acid, Azido-, $C_8H_7N_3O_3$ and *Diazido-*, $C_8H_6N_6O_3$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanisic or Nitromethoxybenzoic Acids, $H_3CO{\cdot}C_6H_3(NO_2){\cdot}CO_2H$ are described in Beil **10**, 117,146,147,181,(50,51,52,67,79) & [66, 67,68,84,85,106]

Dinitroanisic or Dinitromethoxybenzoic Acids, $CH_3O{\cdot}C_6H_2(NO_2)_2{\cdot}CO_2H$, mw 2421.4, N 11.57%. The following isomers are described in the literature:

2,4-Dinitro-(m-anisic) or 2,4-Dinitro-(3-methoxybenzoic) Acid, ndls (from w), mp 240-1° (dec). Was prepd by treatg 2,3,4-trinitrobenzoic acid with aq K methylate

Refs: 1)Beil **10**, (67) 2)M.Giua, Gazz **45** T, 355(1915) & CA **10**,599(1916)

2,6-Dinitro-(m-anisic) Acid, col ndls(from w), mp 195°(Ref 2), 199°(Ref 3). Can be prepd by oxidation of 2,6-dinitro-3-methoxybenzaldehyde with aq $KMnO_4$ soln(Refs 2 & 3)

Refs: 1)Beil **10**, [86] 2)J.Tröger & C.Eicker,

JPraktChem **116**, 31-2(1927) 3)H.H. Hodgson & H.G.Beard, JCS **1927**, 2381

4,6-Dinitro-(m-anisic) Acid, col ndls, mp 186-7°(Ref 2), 188°(Ref 3), 188-9° (Ref 4). Various methods of prepn are given in Refs 2-5

Refs: 1)Beil **10**, (67) & [86] 2)M.Giua, Gazz **45** I, 355(1915) & CA **10**, 599(1916) 3) J.Tröger & C.Eicker, JPraktChem **116**, 27 (1927) 4)H.H.Hodgson & H.G.Beard, JCS **1927**, 2381 5)H.Goldstein & R.Stamm, Helv **35**, 1332(1952) & CA **47**, 3269(1953)

2,3-Dinitro-(p-anisic) or 2,3-Dinitro-4-methoxybenzoic Acid, ndls, dec at 248-250°. Can be prepd by oxidation of 2,3-dinitro-4-methoxytoluene as described in Ref 2

Refs: 1)Beil **10**, 108 2)H.E.Dadswell & J.Kenner, JCS **1927**, 587

3,5-Dinitro-(o-anisic) or 3,5-Dinitro-2-methoxybenzoic Acid, called by Ullmann *3,5-Dinitromethylsalicylic Acid,* col ndls (from w), mp 165°. Was prepd by heating under reflux 2-chloro-3,5-dinitrobenzoic acid in methanol with Na methylate

Refs: 1)Beil **10**, 184 2)F.Ullmann, Ann **366**, 94(1909)

3,5-Dinitro-(o-anisic) or 3,5-Dinitro-4-methoxybenzoic Acid, col ndls, mp 181-2°. Was first prepd by nitration of 3-nitroanisic acid (Ref 2). Other methods of prepn are given in Refs 1,3,5

Refs: 1)Beil **10**, 184, (80) & [108] 2)H. Salkowsky & C.Rudolph, Ber **10**, 1255(1877) 3)F.Ullmann, Ann **366**, 94(1909) 4)M.P. deLange, Rec **45**, 45(1926) 5)J.van Alphen, Rec **48**, 1116(1929)

Trinitroanisic or Trinitromethoxybenzoic Acids, $CH_3{\cdot}O{\cdot}C_6H(NO_2)_3{\cdot}CO_2H$, mw 287.14, N 14.64% not found in Beil or CA through 1956

Anisic Alcohol or Anisalcohol. Same Anisyl Alcohol

Anisic Peroxide. See Dianisoylperoxide

Anisidine. Same as Aminoanisole

o-Anisidino-diazido-copper, $[(N_3)_2Cu{\cdot}C_7H_7ONH_2]$, crysts explg at ca 125°, but not on impact; insol in common org solvents, sol in dil sulfuric acid. Was prepd by mixing methanolic solns of Cu nitrate (or chloride) and o-anisidine with aq soln of Na azide, followed by cooling

Refs: 1)Beil – not found 2)A.Cirulis & M.Straumanis, JPraktChem **162**, 315(1943) & CA **38**, 1969(1944)

Anisidinotetrazole. Same as Anisylaminotetrazole

ANISOLE AND DERIVATIVES

Anisole (Ans), Methoxybenzene or Methylphenylether, $CH_3 \cdot O \cdot C_6H_5$, mw 108.13, OB to CO_2 -251.5% Col liq; fr p -37.3°, bp 154.5°, d 0.995 at 20/4°. Prepn & other props are in Beil **6**, 138, (79) & [139]

Anisole, Azidoderivatives. See Azidoanisole and Derivatives under Azides, Organic

Nitration of Anisole has been discussed in many papers such as:
1)C.A.Bunton et al, Nature **158**, 514(1946) 2)C.A.Bunton & G.J.Minkoff, JCS **1947**, 1416 3)R.M.Schramm & F.M.Westheimer, JACS **70**, 1782(1948) 4)N.C.Deno & R.Stein, JACS **78**, 578(1956)

Mononitroanisoles(MNAns) or Nitroanisoles, $CH_3 \cdot O \cdot C_6H_4(NO_2)$, mw 153.13, N 9.15%, OB to CO_2 -151.5%. Three isomers are known:

o-(or 2-) Nitroanisole, col liq, fr p 9.4°, bp 272-3, d 1.253 at 20/4°. Prepn & other props are given in Refs 1,4,5 & 6. It yields on nitration a mixt of 2,4- & 2,6-DNAns

m-(or 3-) Nitroanisole, col ndls (from alc), mp 38°, bp 258°, d 1.373 at 18°. Prepn & other props are given in Refs 2, 5 & 6. It yields on nitration a mixt of 2,5- 2,3- & 3,4-DNAns

p-(or 4-) Nitroanisole, col prisms, mp 54°, bp 274, d 1.233 at 20°. Prepn & other props are given in Ref 3. It yields on nitration 2,4-DNAns

Refs: (Mononitroanisoles) 1)Beil **6**, 217(114) [209] (o-MNAns) 2)Beil **6**, 224(116) [214] (m-MNAns) 3)Beil **6**, 230(119) [220] (p-MNAns) 4)E.H.Weltz, USP 1578943 (1926) & CA **20**, 1631(1926) 5)P.H.Griffiths et al, JCS **1934**, 631-3 & CA **28**, 4726 (1934) 6) R.M.Schramm & E.H.Westheimer, JACS **70**, 1782-4(1948) & CA **42**, 6337 (1948)

Dinitroanisoles (DNAns), $CH_3 \cdot O \cdot C_6H_3(NO_2)_2$, mw 198.13 N 14.14%, OB to CO_2 -97.0%. The following isomers (of which the 2,4-DNAns is the most important), are described in the literature:

2,3 – Dinitroanisole crysts, mp 118-19°, d 1.524 at 20°. Other props and prepn are given in Ref 1a. An improved method of its prepn is given in Ref 42

2,4-Dinitroanisole (2,4–DNAns) (Disol, in Ger). Wh prisms with amber tinge for pure material and yel crysts for tech grade. The monocl-prism form of a new modifn is described in Ref 10 and dimorphism in Ref 11. Tech grade DNAns has mp ca 89° and d 1.341 at 20°; mp of labile form 86.9° and stable form 94.6°(Ref 5); bp – sublimes (Ref 46); Q_c^p 820.2 kcal/mol(Ref 23), Q_f^p 45.0 kcal/mol (Ref 23) and Q_e 92.1 kcal/mol(Ref 45). Badoche (Ref 25) gives for Q_c^v the same value as for Q_c^p given above; Q_{fusion} 3.9 kcal/mol (Ref 17). It is sl sol in w; sol in alc or ether. Numerical values for the soly in w and in twelve org solvents are given in Ref 3. Chemical behavior of 2,4-DNAns is described in Refs 4,5,12,13,31,38 & 40. Binary systems of 2,4-DNAns with various compds are listed in Ref 43 and absorption spectra curves are given in Refs 21,27 & 37. Like other nitroaromatic compds it is toxic and this is discussed in Refs 16,30,33 & 44. A colorimetric method of detn of 2,4-DNAns is given in Ref 28

Various methods of prepn of 2,4-DNAns are discussed in Refs 2,3,4,6,19,20,24,31,32,35 & 41, but it seems that the best lab methods are: a) nitration of o- or p-MNAns and b) interaction of 4-chloro-1,3-dinitrobenzene with K or Na hydroxide in aq methanol. Yields as high as 95% were reported with the latter method

2.4-DNAns is an expl about 10% less powerful then TNT and less sensitive to impact (Refs 15 & 26); its sensitivity to initiation is such that it can be fired by No 8 detonator but not very well by No 6 detonator (Ref 26). Its calcd temp of expln is 1805°K(Ref 45)

It has been proposed as a component of some expl compn. Under the name of "Disol" it was used in Germany as a component of "Amatol 40": DNAns 50, AN35 & RDX 15% – an explosive employed for filling some warheads of V-1 (Ref 47, p 4). In the USA it has been used by military personnel as an insect repellant (Ref 38)

2,4–DNAns yields on nitration 2,4,6-TNAns, a powerful explosive

2,5–Dinitroanisole, col ndls (from benz + ligroin), mp 96.7°, d 1.476 at 18°. Other props and prepn are in Ref 1c

2,6–Dinitroanisole, col ndls (from alc), mp 116-18°, d 1.319 at 20°. Other props and prepn are in Ref 1d

3,4–Dinitroanisole, golden-yel ndls, mp ca 70°. Other props and prepn are in Refs 2a & 18

3,5–Dinitroanisole (3,5–DNAns), col crysts, mp 105-105.8°, bp - sublimes, d 1.558 at 20°/4°. Chemical reactions are described in Refs 12,13 & 29. Urbański (Ref 14) studied eutectic mixts of 3,5-DNAns with hexanitromannitol and with nitroerythritol. Methods of prepn are discussed in Refs 2b,8,9,22,31 & 34. The method of prepn from 1,3,5-trinitrobenzene and Na methylate in anhyd methanol is described in detail in Ref 8

3,5–DNAns is an expl less powerful than TNT and slightly less sensitive to impact than TNT (Ref 15)

Refs (Dinitroanisoles): 1a)Beil **6**, 251 [239] (2,3-DNAns) 1b)Beil **6**, 254 (126) [241] (2,4-DNAns) 1c)Beil **6**, 256 (127) (2,5-DNAns) 1d)Beil **6**, 257 (127) [245] (2,6-DNAns) 2a)Beil **6**, 258 (127) (3,4-DNAns) 2b)Beil **6**, 258 (128) [246] (3,5-DNAns) 3) L. Desvergnes, MP **19**, 269-99 (1922) & CA **17**, 469 (1923) 4)W. Borsche, Ber **56B**, 1488-93 (1923) & CA **17**, 3326-7 (1923) 5)L. Desvergnes, MonSci **14**, 249-57(1924) & CA **19**, 1700 (1925) 6)L. Raiford & J. Colbert, ProcIowa AcadSci **31**, 287-8(1924) & CA **20**, 2319(1926) 7)J.Tröger & C.Eicker, JPraktChem **116**, 17-33,(1927) & CA **21**, 2675(1927) 8)F.Reverdin, Org Synth **7**, 28-9(1927) & Col-Vol **2**(1944), pp 219-20 9)L. Desvergnes, RevChimInd **38**, 66-9(1929) & CA **23**, 4207 (1929) 10)M.Werther & J.Boak, ZKrist **73**, 572(1930) & CA **24**, 4440 (1930) 11) J. van Alphen, Ber **63B**, 94-5(1930) & CA **24**, 2441 (1930) 12)G.B.Kolhatkar & R.B. Ghaswalla, JIndian ChemSoc **8**, 511-6(1931) & CA **26**, 113-4(1932) 13)R.S.Cahn, JCS **1931**, 1121-3 & CA **25**, 4245 (1931) 14)T.Urbański, RoczChem **13**, 399-434 (1933) & CA **28**, 27(1934) 15)L.Wöhler & O.Wenzelberg, AngChem **46**, 175(1933) 16)R.Jonnard CR **117**, 618-9(1934) & CA **29**, 2596(1935) 17)J.Timmermans, BullBelg **44**, 17-40(1935) & CA **29**, 2433 (1935) 18)K.S.Topchiev, CR **4**, 201-6(1935) & CA **30**, 3820(1936) 19)Pepin Lehalleur (1935), p 256 20)C.W.Pohlman, Rec **55**, 737-52(1936) & CA **30**, 7110(1936) 21) H.Halban & B.Szigeti, Helv **20**, 746-61(1937) & CA **31**, 6557 (1937) 22)H.Degiorgi & E. Zappi, BullFr [5] **4**, 1636-42 (1937) & CA **32**, 519(1938) 23)E.Burlot et al, MP **29**, 257(1939) 24)B.M.Bogoslovskii & L.M. Tsil'man, PromOrgKhim **6**, 445-8(1939) & CA **34**, 2360(1940) 25)M.Badoche, Bull Fr **9**, 86-95(1942); Chem Ztr **1942** II, 2013 & CA **38**, 2558(1944) 26)NDRC Rpt, Sec B-1, Int Rpt PT-2, Sept to Oct 1942 27)H.Mohler, Helv **26**, 121-9(1943) & CA **38**, 299-300(1944) 28)M.S.Schechter & H.L.Haller, IEC (Anal Ed) **16**, 325-6(1944) & CA **38**, 3221(1944) 29) G.Zemplén et al, Ber **77**, 446-51 (1944) & CA **40**, 4374(1946) 30)P.Gavaudan, MSCE **32**, 418-42(1945) & CA **42**, 5601(1948) 31)P.E. Verkade et al, Rec **65**, 368(1946) 32)B.B. Dey et al, JSciIndResearch(India) **5B** No. 3, 37-40(1946) & CA **43**, 2599(1949) 33)J.R. Busvine, AnnApplBiol **33**, 271-9(1946) & CA **41**, 2850(1947) 34)P.E.Verkade & P.H. Witjens, Rec **65**, 631-9(1946) & CA **40**, 6435 (1946) 35)J. van Steenis, Rec **66**, 29-46 (1947) & CA **41**, 4787(1947) 36)Y. Ogata & M. Okano, JChemSoc (Japan), Pure Chem Sect **69**, 148-51(1948) & CA **46**, 4500(1952) 37)A.I.Shatenstein & Ya.M.Varshavskii, Zh-FizKhim **22**, 529-39(1948) & CA **42**, 6659 (1948) 38)J.H.Draize et al, JPharmExptl Therap **93**, 26-39(1948) & CA **42**, 6051(1948) 39)Y.Ogata & M.Okano, JACS **71**, 3211-2 (1949) & CA **44**, 2945(1950) 40)T.Canbäck, FarmRevy **48**, 217-24,234-41 & 249-58(1949) & CA **43**, 6174-5(1949) 41)W.B.Whalley, JCS **1950**, 2241-3(1950) & CA **45**, 3347(1951) 42)D.L.Vivian et al, JOC **16**, 6(1951) & CA **45**, 6642(1951) 43)H.Reiboldt & M.Perrier, Univ São Paulo, Faculdade Filosof Cienc e Letras, Bol No **129**, Química No 3, 75-97 & 127-9(1951) & CA **46**, 7552-4(1952) 44) E.W.Simon & G.E.Blackman, JExptl Bot 4, 235-50(1953) & CA **48**, 934(1954) 45) A.D.

Little Punch Cards (1954) 46)Sax (1957), p 628 47)PATR 2510 (1958), 4 & 36

5-Nitroso-2,4-dinitroanisole, $CH_3 \cdot O \cdot C_6H_2 \cdot (NO)-(NO_2)_2$, mw 227.13, N 18.50%, orange-yel crysts (from AcOH), mp 158° (dec). Was obtained by oxidation with chromic acid of N-(4,6-dinitro-3-methoxyphenyl)-hydroxylamine as described in Ref 2

Refs: 1)Beil **6**, [253] 2)W.Borsche & E. Feske, Ber **59**, 818 (1926)

Trinitroanisoles (TNAns), $CH_3 \cdot O \cdot C_6H_2(NO_2)_3$, mw 243.13, N 17.28%, OB to CO_2 -62.5%. The following five isomers are all expl:

2,3,4-Trinitroanisole, lt yel ndls, mp 155°; sol in alc, expl on heating. Prepd by Vermeulen by nitration at 80° of 2,3-DNAns with mixed nitric-sulfuric acid (Refs 3 & 9)

2,3,5-Trinitroanisole, leaflets or rhombic bipyramidal crysts, mp 104-106.8°, d 1.618 at 15°; sol in w, alc acet or pyridine. Quantitative soly in var org solvents given in Ref 30. Prepd by the nitration of 3,5-DNAns with mixed nitric-sulfuric acid (Ref 4)

2,4,5-Trinitranisole, almost colorless crysts from alc, mp 104-107°; sl sol in ligroin, sol in alc. Prepd according to Vermeulen by nitrating 2,5-DNAns with mixed nitric-sulfuric acid (Refs 4 & 9)

2,4,6-Trinitroanisole (TNAns) *(Nitrolit; Methyl Picrate or 2,4,6-Trinitrophenylmethyl Ether)* [Trinitroanisole in Fr; Trinitroanisol or Trisol in Ger; Trinitroanisol' in Rus; Trinitroanisolo in Italy; Trinitroanisol in Spain; Type 91 Explosive in Japan]. Pale-yel leaflets, mp 65-67° (coml) and 68.4° (pure); bp 310°, d (cast) 1.4, d at max loading press 1.7; Q_c^v 792.1 kcal/mol (av value from Refs 31,41,46 & 50), Q_c^p 784.4 kcal/mol (Ref 31), Q_f 42.5 kcal/mol (Ref 68), Q_e^v (calcd) 136.5 kcal/mol (Ref 68), Q vapn 15 kcal/mol (Ref 59), Q subln (Ref 63) appt dipole moment (Ref 64) and absorption spectra (Refs 44,52 & 66),

Solubilities of TNAns in Various Solvents, as Determined by Desvergnes (Ref 26) (g TNAns per 100 g solvent)

Solvent	15°	50°	100°
Water	0.02	0.14	0.39
Chloroform	25.6	334.5	–
Carbon tetrachloride	0.51	3.65	–
Benzene	95.	597.9	–
Toluene	86.5	421.5	–
Methanol	5.24	27.65	–
Abs alcohol	2.37	21.36	–
96% alcohol	2.31	17.79	–
Ether	4.18	7.86 (34°)	–
Acetone	194	813.2	–
Ethyl acetate	89.4	368.5	–
Carbon disulfide	0.43	1.11	–
Pyridine	40.4	221.	–

The toxicity of TNAns is discussed in Refs 16, 49,57,67 & 70a; its chem reactions in Refs 7,11,15,16,17,21,23,27,32,35,38,45, 47,55,65,69 & 70

TNAns forms eutectics with the following compds:

	%	mp	Refs
Picryl sulfide	13	62.5	25
Tetryl	70.5	22.8	29
o-Nitroaniline	33.3	30.2	33
Erythritol-tetranitrate	74	addition compd	42
TNB	37.5	45	42
TNT	40	42	42
TNT/TNB (26.8/27.6)	54.4	30	42
PA/TNB (3.8/34.2)	38.	37.5	43
TNT/PA (30/21)	51.	37	43

TNAns is hygroscopic and is decompd by hot water with the formation of iso-picric acid (qv) (Ref 26). TNAns was prepd in 1849 by Cahours by direct nitration of anisole (Ref 6). This method according to Davis (Ref 51) is dangerous as it may produce an

expln. He recommends for lab prepn the method of Jackson (Ref 7) in which a methanolic soln of picryl chloride is treated with an excess of sodium methylate or strong NaOH soln. Deposits of red crysts of $(O_2N)_3C_6H_2OCH_3NaOCH_3$ are obtained which on treatment with acid yield 2,4,6-TNAns. Davis (Ref 51) describes this lab method in detail. For plant scale prodn see Refs 9,24,34,36, 40 & 60. Additional info on the prepn and props is given in Refs 5,10,24,36 & 61. Damschroeder & Shriner (Ref 45) observed that 2,4,6-TNAns prepd from picryl chloride and MeONa in MeOH exists in 4 forms: sq plates-mp 50-1°, hexagonal plates - mp 56-7°, prisms - mp 58-9 and needles - mp 69-9° (stable form). Ovenston (Ref 58) reported the monotropic transformation in 2,4,6-TNAns. He prepd β-TNAns (mp 57.5°) by sealing α-TNAns (mp 68°) in glass, immersing it in boiling H_2O, supercooling and inducing crystn with broken glass. According to Ovenston, in a lab completely free of α-form, the β-form may be the sole product in the usual synthesis of TNAns

Explosive Properties. TNAns was developed as a substitute for PA which is high-melting and tends to react with metals. The dry TNAns does not attack metals since it contains no hydroxyl groups, and it therefore does not form dangerous metallic salts, as does PA. Its relatively low mp permits cast loading of the expl, but it will exude at tropical storage temps. TNAns is compatible with NC with which it forms colloidal mixts

The following expl props have been reported:

Booster Sensitivity, by Gap Test, 6.5 cm vs 13 cm for PA (Ref 62)

Brisance, by Sand Test, 43g vs 43 for TNT (Ref 57) and 110% TNT (Ref 28); by Copper Cylinder Crusher Test 92% PA or 100% TNT (Refs 18 & 52a)

Detonation Rate 6660 m/s at d 1.59 (Refs 18 & 68), 6900 m/s (Ref 57) and 7640 m/s vs 6880 for TNT (Refs 24 & 52a)

Explosion Temperature 165-296° (Ref 57), 225° (Ref 48) & 279-500° (Ref 53)

Impact Sensitivity 15-16" vs 14" for TNT by PicArsnApp using 2kg wt; max value for no expln with 5kg wt – 19 cm or 20% positive at 100 cm and 30% positive at 110 cm vs 50% positive at 30 cm for PA (Ref 24); FI 120-124% PA and by Rifle Bullet Test-no deton from impact of .30 cal bullet fired from 90 ft (Ref 57)(See also Ref 52a)

Impact Work for 50% explns with 2 kg wt: 10.1 m kg/cm² or 89% TNT (Ref 37)

Power by BalMort 106% of TNT (Refs 19 & 68) by Trauzl Test 98% PA (Refs 18 & 22) and 112% TNT (Ref 68); by *Manometric Bomb Test* – develops 2850 kg/cm² at loading d 0.25 vs 3230 for PA (Ref 24, p 282); by French Trauzl Test (CUP) 96% PA (Ref 62)(See also Ref 52a)

Pressure (maximum theoretical) if exploded in own volume and without loss of heat – 9235 kg/cm² at d 1.5 (Refs 20 & 52a)

Sensitivity to Initiation for 0.5 g sample of TNAns loaded in a detonator cap at 1100 kg/cm², a min of 0.37 g MF or 0.28 g LA is reqd (Refs 13 & 52a)

Specific Energy (f) 8232 vs 8080 for TNT or 102% TNT (Ref 18)

Stability – fairly stable in dry state, but in the presence of moisture it will hydrolyze to PA which reacts with metals to form sensitive picrates; in the presence of ammonia, TNAns gives picramide (Ref 36). Due to its low mp, TNAns will exude if stored at elevated temps

Temperature of Explosion or Detonation: 2366°K (calcd) (Ref 68)

Uses. Because of low mp (exudation) and poor stability in presence of moisture, TNAns was not used extensively for military purposes. It was used, however, as a substitute explosive and also when it was necessary to lower the mp of other expls, such as PA

Following are some uses of TNAns:

France. According to Pepin Lehalleur (Ref 40) TNAns was manufd at the Poudrerie de Saint-Fons by nitration of DNAns but he does not describe its uses

Germany. According to Marshall (Refs 12 & 34a) straight TNAns and its mixts with HE's and AN were used to some extent for filling bombs and shells. Straight TNAns was used in some boosters, and mixts of TNAns with hexanitrodiphenyl sulfide for filling bombs (Ref 51a). Straight TNAns (Trisol) was used in long range projectiles (Ferngeschütz-granaten), which were fired against Paris (Refs 60 & 72). It was also used in sea mines and torpedoes (Ref 57, p 110)

Great Britain – no information at our disposal

Italy – no info about its use for military purposes, but it was used in a mining expl constg of TNAns 20 & AN 80%. Belgrano(Ref 66a) analyzed the expl and reported its props as Trauzl test value 420 cc and gap test 2 cm

Japan. Straight TNAns, under the name of Type 91,was used during WW II in AP shells and bombs such as in jet-driven suicide planes (Refs 52a,53,54 & 57). Its mixtures with AN and some HE's were used by both Army & Navy. These included *A(ko) or Type A,* called also Otsu-B[TNAns(or TNT) 60 HNDPhA 24 & Al 16%) (Ref 52a & Ref 53, p 32) - it was intended to replace *Type 97H,* called also *Seigata* (TNT 60 & HNDPhA 40%)in torpedo warheads; B_4 or *Type 2* (incendiary expl contg TNAns 60 or 70 & Al powder 40 or 30%) (Ref 52a); "E" (Explosive),called also *Nitrolit* (TNAns 60 & AN 40%) (Refs 52a & 57, p 110); H_2 *Kongo* (H_2 Mixture) (TNAns 70 & HNDPhA 30%) - press-loaded in bomb auxiliary boosters (Ref 52b & Ref 53, p 31); same expl cast-loaded in bombs, sea mines and depth changes was called *Type 98* (Ref 53, p 32); *Type 98H* (Navy) (TNAns 60 & HNDPhA 40%) – cast-loaded in bombs, torpedoes, depth charges & other ammo, replacing PA & TNT(Ref 52a & Ref 57, p 109); *Type 94M* (TNAns 60&RDX 40%) – used in some shaped charges & booster surround; its use in torpedo warheads was discontinued due to its sensitivity to sympathetic detonations (Ref 52a & Ref 53, p 32)

Russia – no information at our disposal

Switzerland – used under the name of *Trisol* (Ref 60). Was manufd during WW II by the SSF A-G, Dottikon(Aargau)

United States of America. According to Colver(Ref 14) an Amer inventor Hudson Maxim patented in 1904-5 the use of TNAns in the manuf of smokeless propellants. In a later patent (Ref 8) is given the compn of such propellants as: TNAns 40-50 & pyrocellulose 60-40%. Recent investigation at the Armour Res Inst (Ref 71) has shown that TNAns is useful as an anticracking additive to cast TNT and Comp B. Optimum percentages are: 0.4-0.8% for TNT and 0.8-1.0% for Comp B. Exudation of samples contg 0.1-0.2% TNAns in Comp B and 0.1-0.4% in TNT was only slightly higher than that for straight TNT. At higher percentages of TNAns the extent of exudation was too great (Ref 71)

3,4,5-Trinitroanisole, sl yel needles, mp 119-120°, sl sol in ligroin, sol in AcOH. Can be prepd according to Vermeulen (Ref 9) by nitration of 2,5-DNAns with mixed nitric-sulfuric acid (Ref 2)

Refs (Trinitroanisoles): 1) Beil **6**, (129) 2) Beil **6**, (141) 3)Beil **6**, 264 & (129) 4)Beil **6**, 264, (129) & [253] 5)Beil **6**, 288, (140) & [280] 6)A.Cahours, Ann **69**, 236(1849) 7) C.Jackson et al, AmChem J **20**, 448(1898) & JCS **74**, I,517(1898); AmChemJ **23**, 294, 376-96 (1900) & JCS **78**,I,433(1900) 8)H.Maxim, USP 951,445(1910)&CA **4**, 1546(1910) 9) H.Vermeulen, Rec **31**, 101-4(1912) 10)A.L. Broadbent & F. Sparre, 8th Intl Cong Appl Chem **4**, 15(1912) & CA **35**, 3522(1941) 11) M.Kohn & F.Grauer, Monatsh **34**, 1751-5(1913) & CA **8**, 500(1914) 12)Marshall v1(1917) p 284 13)L.Wöhler & F.Martin, SS *12*, 19 (1917) 14)Colver(1918), 336-40, 342 & 702 15)M.Giua & F.Cherchi, Gazz **49** II, 152-7(1919) & CA **14**,1531(1920) 16)F. Koelsch, ZAngChem **33**,I,1-5(1920) & CA **14**, 2034(1921) 17)M.Giua et al, Gazz **50** II, 300-12(1920) & CA **15**, 2280(1921) 18)H.Kast, SS **15**, 173 & 184(1920) 19)W.Cope, IEC **12**, 870(1920) 20)J.Crawshaw, JFrankInst

189, 607(1920) 21)Michele Giua & Mario Giua, Gazz **51** I, 313-7(1921) & CA **16**, 77 (1922) 22)B.Flürscheim, JSCI **40**, I, 97 (1921) 23)M.Giua, Gazz **52** I, 182(1922) & CA **16**, 2493(1922) 24)L.Desvergnes, MP **19**, 270-84 (1922) & CA **17**, 469(1923) 25) C.T. Thomas & V.Thomas, CR **176**, 1323-5 (1923) & CA **17**, 2417(1923) 26)L.Desvergnes, MonScientQuesneville [5], **14**, 252-3(1925) & ChemZtr **1925** I,837 27)O.L.Brady & H.V. Horton, JCS **127**, 2230-3(1925) & CA **20**, 177 (1926) 27a)W.Borsche & E.Feske, Ber **59**, 818(1926) 28)W.Dehn & A.Wagner, ArOrdn **8**,35(1927) 29)N.N.Efremov & A.M.Tikhomirova, IzvInstFiz-KhimAnal **4**, 92-117(1928) & CA **23**, 3214(1929) 30)L.Desvergnes, RevChimInd **38**, 68(1929) & CA **23**, 4207(1929) 31)Wm.H.Rinkenbach, JACS **52**, 116(1930) 32) R.S.Cahn, JCS **1931**, 1121-3 & CA **25**, 4245 (1931) 33)M.Giua, AttiAccadTor **66**, 548 (1931) & CA **25**, 4452(1931) 34)Vennin, Burlot & Lécorché (1932)p 454 34a)Marshall, v 3(1932), 84 35)E.Hertel & J.Dressel, Z Physik Chem **B23**, 281-90(1933) & CA **28**, 958(1934) 36)G.Guastalla & G.Racciu, Ind-Chimica **8**, 1370-7(1933) & CA **28**, 2185(1934) 37)L.Wöhler & O.Wenzelberg, AngChem **46**, 173(1933) 38)E.Hertel & J.Dressel, ZPhysik Chem **B29**, 178-91(1935) & CA **29**, 6216(1935) 39)T.Urbański, RoczChem **15**, 191-7(1935) & CA **30**, 2834(1936) 40)Pepin Lehalleur(1935), p 257 41)Land-Börnst, **Eg III**, Tl 3 (1936), p 2914 42)T.Asahina & C.Shinomiya, JChem-SocJapan **57**, 732-42(1936) & CA **30**, 7434 (1936) 43)T.Asahina & C.Shinomiya, J ChemSocJapan **58**, 119-23(1937) & CA **31**, 2913(1937) 44)H.von Halban & B.Szigeti, Helv **20**, 746-61(1937) & CA **31**, 6557(1937) 45)R.E.Damschroeder & R.L. Shriner, JACS **59**, 931-3(1937) & CA **31**, 4291(1937) 46) E.Burlot & M.Thomas, MP **29**, 262(1939) & CA **34**, 1849(1940) 47)E.Hertel & H.Lührmann, ZElektrochem **45**, 405-8(1939) & CA **33**, 6267(1939) 48)A.F.Belyaev & E.Sanburskaya, DoklAkadN **30**, 632-4(1941) & CA **37**, 533 (1943) 49)Anon, US PubHealth Bull No 271 (1941), p 155 50)M.Badoche, BullFr **9**, 86-95(1942); ChemZtr **1942** II, 2013 & CA **38**, 2558(1944) 51)Davis(1943), p 169-73 51a) Bebie (1943), 152-3 52)M.Mohler, Helv **26**, 121-9(1943) & CA **38**, 300(1944) 52a)A.H. Blatt, OSRD Rept 2014(1944) 52b)R.A.Cooley et al, "Japanese Explosives," PBL Rept 53, 045(1945) 53)Anon,"Handbook of Japanese Explosive Ordnance," OPNAV 30-3M, US Govt Print Offc (1945), p 32 53a)Pérez-Ara(1945), 533-5 54)W.J.Osborn Jr, ARSJ **65**, 20-3 (1946) & CA **40**, 5253 (1946) 54a)Vivas, Feigenspan & Ladreda, v2 (1946), 258 55) Schweiz Sprengstoff-Fabrik AG, Swiss P 243,264(1946) & CA **43**, 5958(1949) 56) G.B.Tibbets et al, PB Rpt 50394(1946) pp 74-7 & 90 57)Anon, All & EnExpl (1946), pp 108-9(expls) & 157(toxicity) 58)T.C. Ovenston, Nature **159**, 437 (1947) & CA **41**, 4121(1947) 59)A.F.Belyaev, ZhFizKhim **22**, 91-101(1948)& CA **42**, 5227(1948) 60) Stettbacher (1948), pp 77 & 286 61)Thorpe (1949), p 484 62)E.Burlot & T.Tavernier, MP **31**, 125(1949) 63)I.Nitta et al, JChem-SocJapan, PureChemSect **71**, 378-82(1950) & CA **45**, 6448(1951) 64)C.G.LeFèvre & R.J.LeFèvre, JCS **1950**, 1829-33 & CA **45**, 2279 (1951) 65)H.Merwein, Ann **566**, 150-62(1950) & CA **44**, 5806(1950) 66)W.A. Schroeder, AnalChem **23**, 1742(1951) & CA **46**, 5434(1952) 66a)Belgrano(1952), 154-5 67)E.W.Simon & G.E.Blackman, JExptlBot **3**, 99(1952); **4**, 235-50(1953) & CA **48**, 934 (1954) 68)ADL Punch Cards(1954), Compd No 271 69)A.Langhans, Explosivst **1954**, 1/2,3-11 & CA **49**, 3046(1955) 70)J.B. Ainscough & E.F.Caldin, JCS **1956**, 2528-39 & CA **50**, 16310(1956) 70a)Sax(1957),1222 71)R.S.Spriggs & J.Krc, Jr, Armour Res Found Project C114, Rept No 5(Final Report) (1958) 72)PATR 2510(1958), 204

Trinitroanisole Complex, $CH_3 \cdot O \cdot C_6H_2(NO_2)_3$ - $3CHAcNaCO_2C_2H_5$, red amor ppt, expl on heating; sol in alc or acet. It was obtained on treating a benzolic soln of TNAns with a benzolic soln of ethyl sodium acetate. Another expl complex was obtained on treating TNAns with ethyl sodium malonate

Ref: C.L.Jackson & F.H.Gazzolo, Amer Chem-J **23**, 376(1900) & JCS **78**, I, 433-4(1900)

Tetranitroanisoles (TeNAns), $CH_3 \cdot O \cdot C_6H(NO_2)_4$, mw 288.13, N 19.45%, OB to CO_2 -38.9%. Three isomers are theoretically possible: 2,3,4,5-TeNAns, 2,3,4,6-TeNAns and 2,3,5,6-TeNAns,but only the latter two are described in the literature:

2,3,4,6-Tetranitroanisole(2,3,4,6-TeNAns), granular or plates, mp 94°, d 1.64; v sl sol in w, alc or eth; sl sol in cold chlf, readily sol in hot chlf. Prepd by nitrating m-nitroanisole (Refs 1 & 3). This expl is reported to be more powerful and brisant than TNT. It gelatinizes NC. Its sensitivity to impact is comparable to that of tetryl and it explodes on heating at about 300°. The nitro-group in the 3 position is readily replaced, for example, by a hydroxyl group on heating with water. 2,3,4,6-TeNAns was used in Germany in primary expl mixts and as an ingredient in HE compositions (Ref 5). This isomer is reported to be equal in expl props to the 2,3,5,6-isomer (described below) but because of its lower mp, the use of 2,3,4,6-TNAns is preferred to the higher melting compd (Ref 5)

2,3,5,6-Tetranitroanisole (2,3,5,6-TeNAns) exists in two cryst modifications: col with mp 153.5° and yel with mp 112°; on crystn from alc, col crysts appear first, then change to the yel modifn (Ref 2). It is non-hygroscopic. Desvergnes (Ref 6) reported the following solubility data:

g 2,3,5,6-TeNAns/100 g Solvent at 29°	
Chloroform	0.79
Carbon tetrachloride	0.07
Benzene	1.55
Toluene	1.02
Methanol	4.39
Abs alcohol	2.38
96% alcohol	1.88
Ether	1.66
Acetone	1.15
Ethyl acetate	37.0
Carbon disulfide	0.08
Pyridine	*

*Also dissolves in pyridine with evoln of nitrous gases, imparting a red coloration to the soln

2,3,5,6-TeNAns can be prepd by nitration, with mixed nitric-sulfuric acid, of either 2,3,5-TNAns or 3,5-DNAns (Ref 4)

Explosive Properties: Explosion Temperature 300° (Ref 4); *Impact Sensitivity* 20 to 35 cm vs 25 cm for tetryl & 110 cm for TNT, using 2 kg wt (Ref 4); *Power*, by Lead Block Expansion,380-400 cc vs 290 cc for TNT or 131-8% TNT (Ref 4); *Reactivity*- the nitro group in the 3 position is readily replaced; *Stability*- claimed to be as stable as TNT (Ref 4) but not found so by others. See also Ref 8
Uses: TeNAns was used in Germany in initiating and other expl mixts (Refs 3 & 7) but later was found to be too reactive and too sensitive for military or coml application. No information at our disposal about its uses in other countries
Refs (Tetranitroanisoles) 1)Beil **6**(142) 2) Beil **6**, 293,(142) & [284] 3)J.J.Blanksma, Rec **23**, 114-16(1904) 4)C.Claessen, GerP 288,655(1913) & CA **10**, 2800(1916) 5) C.Claessen, GerP 289,446(1914) & CA **10**, 3162(1916) 6)L.Desvergnes, Rev Chim Ind **38**, 69(1929) & CA **23**, 4207(1929) 7)A.H. Blatt & F.C. Whitmore, OSRD Report 1085 (1942), p 83 8)A.H.Blatt, OSRD 2014 (1944)

ANISOLE AND DERIVATIVES, ANALYTICAL PROCEDURES

A)Anisole gives with SeO_2 or with Na selenite in concd H_2SO_4 a dark green coloration (Ref 1). For quantitative estimation of anisole, Dr H. Walter (Ref 7) suggests the bromination method, simply by adding bromine water. This gives 2,4,6-tribromoanisole. Det its mp(87-8°) and mixed mp

B)Mononitroanisoles. The following method is suggested by Dr H. Walter: Hydrolyze the sample by boiling it with aq KOH in the presence of some alcohol:

$$H_3CO \cdot C_6H_4 \cdot NO_2 \xrightarrow{KOH} KO \cdot C_6H_4 \cdot NO_2$$

Acidify with aq HCl and extract the resulting nitrophenol with ether. Evap the ether,

recrystallize the residue from hot alc and identify the crysts by mp, mixed mp and/or IR spectra. When the sample contains a mixt of several isomers, the presence of o- and p-isomers is indicated by the intense yel coloration produced in aq KOH. The m-isomer does not produce this coloration in the presence of KOH

IR spectrograms of m- and p-nitroanisoles were prepd at PicArsn by Pristera et al (Ref 6)

C)Dinitroanisoles. The following methods are suggested by Dr H. Walter:

Method 1. Dissolve a sample in aq methanol, add KOH and reflux:

$$(O_2N)_2C_6H_3\cdot OCH_3 \xrightarrow{CH_3OK} (O_2N)_2C_6H_3\cdot OK$$

Evaporate the resulting soln of potassium dinitropnenolate to near dryness and dissolve the residue in concd H_2SO_4 (with cooling). Add gradually concd HNO_3(d 1.5) in order to obtain PA:

$$(O_2N)_3C_6H_3\cdot OK \xrightarrow{HNO_3} (O_2N)_3C_6H_2\cdot OH$$

Add an excess of Na acetate and then Pb acetate. Separate the resulting ppt of Pb picrate and weigh. PA content may also be detd colorimetrically

Method 2. Dissolve the sample in aq methanol, add KOH and gently reflux. Cool the soln and add aq HCl, followed by stannous chloride:

$$H_3CO\cdot C_6H_3(NO_2)_2 \xrightarrow{KOH} KO\cdot C_6H_3(NO_2)_2$$

$$\xrightarrow{HCl} HO\cdot C_6H_3(NO_2)_2 \xrightarrow[\text{with } SnCl_2]{\text{Reduction}} HO\cdot C_6H_3(NH_2)_2$$

Treat the resulting diaminophenol with diazotized sulfanilic acid to obtain the orange colored dye:

$$(H_2N)_2(HO)C_6H_3 + N\equiv\overset{+}{N}\cdot C_6H_4\cdot SO_3^- \longrightarrow$$

$$(H_2N)_2(HO)C_6H_2\text{–}N{=}N\text{–}C_6H_4\cdot SO_3H$$

Transfer the soln to a vol flask, take an aliquot and test it colorimetrically

Note: If additional identification is desired, det nitrogen content by titanous chloride method described on p A415

The IR spectrogram of 2,4-DNAns was made at PicArsn by Pristera et al (Ref 6)

A method of analysis of 2,4-DNAns samples contg 2,4-dinitrochlorobenzene was developed at PicArsn by S.M.Kaye(Ref 4). In this method the amt of 2,4-DNClB is estimated from the amt of chloride detd by means of a photoelectric turbidimeter

D)Trinitroanisole. The following method is suggested by Dr H.Walter:

Dissolve a sample in aq methanol, add KOH and reflux

$$(O_2N)_3C_6H_2\cdot OCH_3 \xrightarrow{CH_3OK} (O_2N)_3C_6H_2\cdot OK$$

Det the content of potassium picrate either colorimetrically or gravimetrically, such as by pptn with Pb acetate

IR spectrogram of 2,4,6-TNAns was prepd at PicArsn by Pristera et al (Ref 6)

Identification of TNAns when in mixt with HNDPhA, such as was encountered in the Japanese 45 mm rocket was done at PicArsn by Weissberger(Ref 2) as follows:

Procedure: a)0.4g sample of rocket chge was treated in a tared 100 ml beaker with 50 ml of hot benzene and the mixt allowed to stand on a hot bath for 10 mins with frequent stirring b)The beaker was removed, cooled to 10-12° and, by means of a filtering stick, the supernatant soln of a material, later identified as TNAns, was removed c)The residual material, later found to be HNDPhA, was washed 3 times with 5 ml of cold benzene (10°) and dried to const wt. The increase in wt of the beaker was considered as the amt of HNPhA d)After evaporating the benzene from soln (b) the residue was identified as TNAns by its mp (65-67°) and its equivalent wt of 13.5 was detnd by reduction with titanous sulfate e)The residue in beaker of proced (c) was identified as HNDPhA by its mp (242°) and its equiv wt of 12.6 f)The TNAns was confirmed, as such, by the mp of a mixt of the unknown material and pure TNAns (mixed mp), and the similarity of spectrophotometric curves and

the X-ray diffraction pattern of the unknown material and pure TNAns g)The HNDPhA was confirmed, as such, by the same tests as in proced (e)

Note: If additional identification is desired det nitrogen content by titanous chloride method, described on p A415

E)Tetranitroanisole. The following method is suggested by Dr H. Walter:

Reflux a sample with methanolic KOH in excess, whereby potassium styphnate is formed

$$H_3CO{\cdot}C_6H(NO_2)_4 \xrightarrow[CH_3OH]{KOH\ in}$$

$$KO{\cdot}C_6H(NO_2)_3{\cdot}OK$$

Weigh the sample and identify the styphnate by decompg a portion of it with aq HCl, extracting the free styphnic acid formed with ether, evapg the ether, and recrystallizing the residue from ethanol. Identify styphnic acid by detg its mp, mixed mp and/or IR spectra

Note: If additional identification is desired det nitrogen content by titanous chloride method, as described on p A415

Refs: 1)Beil **6**, [142] 2)S.Weisberger, Pic ArsnGenLabRept 117206(1946) 3)Kirk & Othmer – not found 4)S.M.Kaye, PicArsn GenLabRept 52-HI-3074(1952) 5)Organic Synthesis, Interscience, NY, vols 1-3(1953-6) – not found 6)F.Pristera et al, PATR 2254(1956) & Anal Chem **32**, 498(1960) 7) Dr H. Walter, PicArsn; private communication (1960)

Anisolebutane. Same as Butylanisole

Anisoleethane. Same as Ethylanisole

Anisolemethane. Same as Methylanisole

Anisolepropane. Same as Propylanisole

Anisoylazide or Methoxybenzoylazide *(Anisic Azide or Methoxybenzazide)* $H_3CO{\cdot}C_6H_4{\cdot}CO{\cdot}N_3$, mw 177.16, N23.72%. Following isomers are described in the literature

m-Anisoylazide or 3-Methoxybenzoylazide, mp 22.5°, dec 61°. Was prepd by treating 3-methoxybenzoylchloride in acetone with aq Na azide

Refs: 1)Beil – not found 2)C.Naegeli et al, Helv **21**, 1139(1938) & CA **33**, 540(1939)(not listed)

p-Anisoylazide or 4-Methoxybenzoylazide. mp 69°, dec 80° (Ref 3); dec explosively (Ref 2). Sah & Chang (Ref 2) prepd it in 95% yield by diazotization of p-methoxybenzoylhydrazide. Naegeli et al (Ref 3) prepd it by adding the calcd amt of Na azide to an acetonic soln of p-methoxybenzoylchloride. They also claimed that this compd was first prepd in 1933 by O.Brunner & R.Wohrl, Monatsh **63**, 376(1933)

Refs: 1)Beil – not found 2)P.P.T.Sah & K. Sh. Chang, Ber **69B**, 2764(1936) & CA **31**, 218 (1937) 3)C.Naegeli et al, Helv **21**, 1139 (1939)

Note: This compd is listed in CA **33**, 540 (1939), but the props are those of the 3-isomer

Anisoylperoxide. See Dianisoylperoxide

ANISYL ALCOHOL AND DERIVATIVES

Anisyl Alcohol or Methoxybenzyl Alcohol, $H_3CO{\cdot}C_6H_4{\cdot}CH_2OH$. All three isomers: o-, m- and p- are described in Beil **6**, 893, 896, 897, (439,440) & (878, 881 & 883)

Anisyl Alcohol, Azido –, $C_8H_9N_3O_2$ and *Diaziod–,* $C_8H_8N_6O_2$ *Derivatives* were not found in Beil or CA through 1956

Anisyl Alcohol, Nitrates, $H_3CO{\cdot}C_6H_4{\cdot}CH_2{\cdot}ONO_2$. All three isomers o-, m- and p- were prepd by J.W.Baker & T.G.Heggs, Chemistry & Industry **1954**, 464 from Ag nitrate and the corresponding chlorides. The p-isomer was too unstable to distil

Mononitroanisylalcohols, $C_8H_9NO_4$. Several isomers are described in Beil **6**, 901,(440) & [880, 884] and in various papers

Dinitroanisylalcohols, $C_8H_8N_2O_6$, mw 228.16,

N12.28%. Two isomers: *3-methoxy-2,6-dinitrobenzyl alcohol,* mp 139.5-40.5° and *3-methoxy-4,6-dinitrobenzyl alcohol,* mp 135-6° were prepd by E.L.Jackson, JACS **79**, 2912(1957) & CA **51**, 14615(1957)

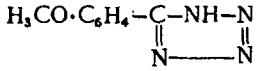
Dinitroanisylalcohol Nitrate, $C_8H_7N_3O_8$ and higher nitrated derivs were not found in Beil or CA through 1956

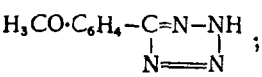
Anisylaminotetrazole and Derivatives. See Aminomethoxyphenyltetrazole and Derivatives

ANISLYTETRAZOLE AND DERIVATIVES

Anisyltetrazole or Methoxyphenyltetrazoles, $C_8H_8N_4O$, mw 176.18, N 31.80%. The following isomer is described in the literature:

5-[4'-*Methoxyphenyl*]-*tetrazole,* formerly called "Anisenyltetrazotic Acid",


or

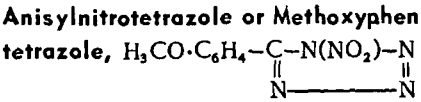

; tricl-pinacoidal

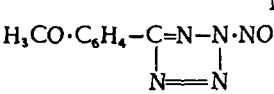
cryst, mp 228°(dec) and burns when heated on a Pt foil; easily sol in alc, insol in cold w, sl sol in hot w & in eth. Was prepd from ethyl ether of anisiminohydrazine hydrate as described in Ref 2

Refs: 1)Beil **26**, 395 2)W.Lossen & J. Colman, Ann **298**, 107(1897)

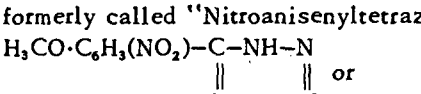
Anisyltetrazole, Azidoderivative, $C_8H_7N_7O$ – not found in Beil or CA through 1956

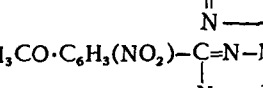
Anisylnitrotetrazole or Methoxyphenylnitrotetrazole,


or


, not found in Beil or CA through 1956

5-[3'-Nitro-4'-methoxyphenyl]-tetrazole, formerly called "Nitroanisenyltetrazotic Acid",


or


, mw 221.18, N 31.67%. Its monohydrate consists of yel ndls, mp 203°; insol in cold w, sl sol in hot w, and sol in eth, was prepd by nitrating the previous compd with nitric acid (d 1.4) (Refs 1 & 2)

Refs: 1)Beil **26**, 396 2)W.Lossen & J. Colman, Ann **298**, 115(1897)

Dinitroanisyltetrazole, $C_8H_6N_6O_5$ – not found in Beil or CA through 1956

Trinitroanisyltetrazole, $C_8H_5N_7O_7$ – not found in Beil or CA through 1956

ANJ. Cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 410

AN-M-69 Bomb. An incendiary bomb, weighing 6.2 lb used in WW II. It was filled with either Napalm Filling (Napalm 9, gasoline 91%) or IM Filling [isobutylmetacrylate polymer 5.0, fatty acids (such as stearic) 2.5, naphthenic acid 2.5, aq 40% NaOH soln 3.0 & gasoline 87%]

Ref: W.A.Noyes, Jr, "Science in WW II," OSRD, "Chemistry" Little, Brown & Co, Boston (1948), 389

ANP-512DS, ANP-514DD, ANP-528DV, ANP-2502EB, ANP-2512EE, ANP-2541CD, ANP-2566EN & ANP-2569EK are polyurethane fuel-oxidizer propellants; **ANP-2639AF & ANP-2655AF** are polyurethane propellants for rockets and **ANR** is a cast-double-base propellant

Ref: "Propellant Manual," SPIA/M2(1959), Unit Nos 480-4, 516-18, 568-9 & 381 (Conf)

ANS or Antisanzinate. A castable expl: AN 60, PETN 20, GuN 10 & DCDA 10%, developed by M. Tonegutti and used during WW II by the Italian Navy for filling some ammunition. Its comparatively low mp(104°) was due to the presence of GuN and DCDA. Incorporation of some Al increased the efficiency of ANS when used in underwater ammo, such as torpedoes, depth charges, and sea mines

Ref: Belgrano(1952), 96&187

Anschütz Thermometers. German-made thermometers known for their precision

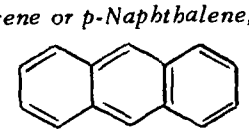
Ansonit Caps. See PATR 2510(1958), p Ger 7

ANT-623 & ANT-638BV are Aeroplex fuel-oxidizer propellants described in conf "Propellant Manual," SPIA/M2(1959), Unit Nos 486 & 487

Antacids. See Antiacids

ANTHRACENE AND DERIVATIVES

Anthracene or p-Naphthalene,



mw 178.22; col monocl crysts with blue fluorescence, mp 216.5-218°, bp 345°, d 1.25 at 27/4°, fl p 250°F(closed cup). Insol in w, sl sol in alc, eth, chlf & CS_2, sol in benz. It is one of the principal ingredients of coal tar, from which it is usually obtained commercially. It has been used as a flash-reducing agent in propellants and for producing gray smokes in trench warfare(Refs 2&3), but its principal use is in manuf of alizarin dyes (Ref 4). In BritP 23, 579(1893) (Ref 1a), anthracene was proposed in expl mixts with AN, K nitrate with or without K chlorate. US military requirements for technical grade anthracene are given in spec MIL-A-202A and its toxicity is discussed in Ref 5

Refs: 1)Beil **5**, 655, (321)&[569] 1a) Escales, Ammonsprengstoffe(1909), 59 2) Bebie(1943), 28 3)Davis(1943), 124, 129 & 327 4)Kirk & Othmer 1(1947), 941-3 5)Sax (1957), 304

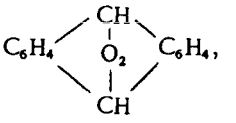
Anthracene, Azido-, $C_{14}H_9N_3$ and *Diazido-*, $C_{14}H_8N_6$ *Derivatives* were not found in Beil or CA through 1956

Note: Nitration of anthracene is discussed in the following papers:
1)P.P.Shorygin et al, ZhObshchKhim **8**, 981 (1938) & CA **33**, 3781(1939)(Anthracene reacts with N_2O_4 in $CHCl_3$ at 0° & 20-30°, yielding 40% 9,10-dinitro- and 4-8% 9-nitroanthracene)
2)R.Oda, JSocChemInd, Japan **42**, Suppl binding 414-18(1939) & CA **34**, 3259-60(1940) (Action of nitric acid in acetic acid produced various products, such as 9-nitroanthrone; 9,10-dinitroanthracene, 2,7-dinitroanthraquinone, etc)
3)R.Oda & M.Kotake, JapP 133052(1939) & CA **35**, 3270(1941)(Treatment of anthracene suspended in glacial AcOH + ⅜ its vol of w, with nitric acid yielded a mixt of 50-60% of 2,7-dinitroanthraquinone and 40/50% of anthraquinone)

Mononitroanthracenes, $C_{14}H_9NO_2$, mw 223.22, N 6.28%. One isomer, *9-nitroanthracene*, is described in Beil **5**, 666&[578]

Dinitroanthracenes, $C_{14}H_8(NO_2)_2$, mw 268.22, N 10.45%. One isomer, *9,10-dinitroanthracene*, is described in Beil **5**, 666, (327) & [579]
Trinitroanthracene, $C_{14}H_7(NO_2)_3$ and higher nitrated compds were not found in Beil

Anthracene Peroxide (Transannular Photoperoxide of Anthracene),



crysts, mp-expl ca 120°. Was prepd by treating anthracene (in dil CS_2 soln) with air under exposure to UV light

Refs: 1)Beil – not found 2)C.Dufraisse & M.Gerard, CR **201**, 428(1935) & **202**, 1859 (1936) 3)W.Bergmann & M.J.McLean, Chem Revs **28**, 367(1941) 4)Tobolsky & Mesrobian (1954), 27-8 5)J.W.Breitenbach & A.Kastell, Monatsh **85**, 676(1954) & CA **48**, 13372(1954)

ANTHRACHRYSONE AND DERIVATIVES

Anthrachrysone or Tetrahydroxyanthraquinone, $C_{14}H_8O_6$, mw 272.20. Several isomers are listed in Beil **8**, 551-2, (755) & [585]

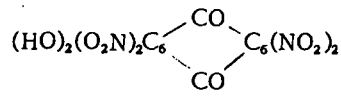
Anthrachrysone, Azido-, $C_{14}H_7N_3O_6$ and *Diazido-*, $C_{14}H_6N_6O_6$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanthrachrysone, $C_{14}H_7NO_8$, mw 317.20, N 4.42% – not found in Beil or CA through 1956

Dinitroanthrachrysone, $C_{14}H_6N_2O_{10}$, mw 362.20, N 7.73%. Several isomers are listed in Beil **8**, 753,(756) & [585]

Trinitroanthrachrysone, $C_{14}H_5N_3O_{12}$, mw 407.20, N 10.32% – not found in Beil

Tetranitroanthrachrysone, $C_{14}H_4N_4O_{14}$, mw 452.20, N 12.39%. One isomer: **2,4,6,8-tetranitro-1,3,5,7-tetrahydroxyanthraquinone**,



leaflets turning brown ca 200° and explg ca 285°; sol in w, alc & AcOH. Can be prepd by

nitrating anthrachrysone with mixed nitric-sulfuric acids

Refs: 1)Beil **8**, 553&586 2)G.Heller & P.Lindner, Ber **55**, 2675-6(1922) & CA **17**, 1012(1923)

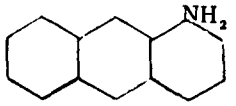
Anthracite. A nongelatinous permissible expl manufd by Independent Explosive Company of Pennsylvania

Ref: Bebie(1943),28

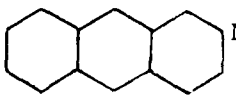
Anthracite Particles(Coal Dust). Their fire & expln hazard, as well as toxicity are discussed in Sax(1957),304

ANTHRAMINE AND DERIVATIVES

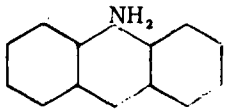
Anthramine or Aminoanthracene, $C_{14}H_9NH_2$, mw 193.24, N 7.25%, Exists in the form of three isomers; *1-aminoanthracene*



, yel crysts, mp 127-30°(Ref 1); *2-aminoanthracene*



, yel leaflets, mp 236-8° (Ref 2) and *9-aminoanthracene,*



, yel crysts, mp 145-50° (Ref 3)

Snelling & Wyler (Ref 4) proposed the use of anthramine (specific isomer not indicated and therefore presumably a commercial product which is a mixt of isomers) as a coating agent for AN crysts or to increase their sensitivity to initiation

Refs: 1)Beil **12**, 1335,(554-5) &[785] 2) Beil **12**, 1335,(555)&[786] 3)Beil **7**, 474, (257)&[416] 4)W.O.Snelling & J.Wyler, USP 1,827,675(1932) & CA **26**, 601(1932)

Anthramine, Azido-, $C_{14}H_{10}N_4$ and *Diazido-,* $C_{14}H_9N_7$ *Derivatives* were not found in Beil or CA through 1956

Mononitroanthramines, $C_{14}H_{10}N_2O_2$. One isomer *9-amino-10-nitroanthracene* is described by S.Hunig & K.Requardt, Ang Chem **68**, 152(1956) & CA **50**, 12944-5(1956)

Dinitroanthramine, $C_{14}H_9N_3O_4$ – not found in Beil or CA through 1956

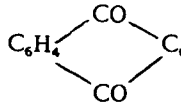
Trinitroanthramine, $C_{14}H_8N_4O_6$ – not found in Beil or CA through 1956

Anthranilic Acid. Same as Aminobenzoic Acid

Anthranoylazide. See Aminobenzazide under Aminobenzoic Acid and Derivatives, p A189

ANTHRAQUINONE AND DERIVATIVES

Anthraquinone or Dihydro-diketo-anthracene,



, mw 208.20 is described in the following refs: 1)Beil **7**, 780-5, (407-9)&[709-13] 2)Kirk & Othmer **1** (1947), 944-7 (several refs) 3)Faith, Keyes & Clark (1957), 122-125(manuf)

Anthraquinone Azides or Triazoanthraquinones also called **Azidoanthraquinones,** $C_{14}H_7N_3O_2$, mw 249.22, N 16.86%, OB to CO_2 -187.8% & to CO -99.5%. Following isomers are described in the literature:

1-Azido-Anthraquinone, bright yel ndls (from MeOH) (Ref 2). Its expl props were not examined. Prepn is described in Refs 1 & 2

2-Azido-Anthraquinone, bright yel ndls (from concd HCOOH), mp 160-162°, turning brown on exposure to light. Defl on heating above the mp. Prepn is described in Refs 1 & 3

Refs: 1)Beil **7**(416)&[722] 2)L.Gattermann & R.Ebert, Ber **49**, 2119(1916) 3)A. Schaarschmidt, Ber **49**, 1637(1916)

Anthraquinone Diazide or Ditriazoanthraquinone, also called **Diazidoanthraquinone** $C_{14}H_6N_6O_2$, mw 290.24, N 28.96%, OB to CO_2 -159.9% to CO -82.7%. Following isomers, most of them unstable, are described in the literature:

1,4-Diazidoanthraquinone, red brn, unstable (Refs 1 & 2)

1,5-Diazidoanthraquinone, yel-brn to red brn, unstable(Refs 1 & 2)

1,8-Diazidoanthraquinone, brn, unstable (Refs 1 & 2)

2,6-Diazidoanthraquinone, lt brn crysts light sensitive, defl 202° on rapid heating, expl when heated in a test tube (Refs 1 & 2)

Refs: 1)Beil **7**, [723] 2)K.Brass & F. Albrecht, Ber **61** 983-93(1928) & CA **22** 2938-9(1928)

Nitroazidoanthraquinone or Nitroanthraquinone Azide, $C_{14}H_6N_4O_4$, mw 294.22, N 19.04%, OB to CO_2 -146.8% & to CO -70.7%. Following isomers are described in the literature:

4-Nitro-1-azido-anthraquinone, yel ndls(from pyridine), mp 245° (dec), expl on rapid heating. Prepd by gentle warming of 4-Nitroanthraquinone-1-diazohydroxylamine with acetic anhydride and pyridine (Ref 2)

1-Nitro-2-azido-anthraquinone, Pale yel-grn crysts(from pyridine), mp 210° (dec), changing on exposure to light and in the air to pale orange-red (Ref 3). Prepd by rubbing β-anthraquinone-azide with concd nitric acid

Refs: 1)Beil **7**, [722] 2)L.Gattermann & H.Rolfes, Ann **425**, 147(1921) & CA **16**, 929 (1922) 3)F.Bayer & Co, GerPat 337,734 (1921) & ChemZtr **1921**, IV, 262

1,5-Dinitro-2,6-diazido-anthraquinone, $C_{14}H_4N_8O_6$, mw 380.24, N 29.47%, OB to CO_2 -101.0% & to CO -42.1% Bright yel ndls (from $C_6H_5NO_2$), mp 200-202°(dec). Prepd by treating 2,6-Diazoanthraquinone with fuming nitric or sulfuric acids

Refs: 1)Beil **7**, [723] 2)F.Bayer & Co, Ger Pat 337,734(1921) & Chem Ztr **1921**, IV, 262

Note: Nitration of anthraquinone is discussed in the following refs: 1)R.Oda & K.Tamura, BullInstPhysChemResearch (Tokyo) **16**, 950(1937) & CA **32**, 5631(1938) (Nitration of anthraquinone with mixed nitric-sulfuric acid gave 1-nitroanthraquinone) 2) Y.Ogata & R.Oda, Ibid **22**, 106-11(1943) & CA **42**, 7284(1948) (Nitration of anthraquinone with mixed nitric-sulfuric acid) 3)F.Ebel & W.Rupp, USP 2715131(1955) (Improvements in the production of nitrogenous anthraquinone compounds)

Nitranthraquinones or Mononitranthraquinones, $C_{14}H_7NO_4$, mw 253.20, N 5.53%. The 1- and 2-isomers are described in Beil **7**, 791-2, (415) & [719-20]. The 2-isomer is best formed by persulfate oxidation of 2-aminoanthraquinone [Kirk & Othmer **7** (1947), 955]

Dinitroanthraquinones, $C_{14}H_6N_2O_6$, mw 298.20, N 91.40%. Several isomers are described in Ref 1 and their purification in Ref 2. Nitration gives predominantly α-deriv (Ref 3)

Refs: 1)Beil **7**, 793-6, (415-6) & [721] 2) H.C.Olpin, C.S.Argyle & F.Brown, USP 2,309,708(1943) & CA **37**, 3769(1943) 3)Kirk & Othmer **1** (1947), 955 (Several refs) 4)J. Franc, ChemListy **49**, 872-5(1955) & CA **49**, 12836(1955) (Chromatography of dinitroanthraquinones)

Trinitro-, Tetranitro-, Pentanitro- and Hexanitroanthraquinones were not found in Beil or CA through 1956

β-Anthraquinonesulfonazide, $C_{14}H_7SO_2N_3$, mw 281.29, N 14.94%, OB to CO_2 (assuming S to SO_2) -179.2%. Yel plates, mp 153° and defl with copious evolution of smoke when heated on a spatula. Prepd by heating anthraquinonesulfonchloride with Na azide in alco soln

Refs: 1)Beil – not found 2)T.Curtius & H.Derlon, J.PraktChem **125**, 420-1(1930) & CA **24**, 3230(1930)

Antiacid Compounds or Antacids (Antazide or Säuerbindende Stoffe in Ger). Some explosives and propellants, especially those contg org nitrates (such as NG, NGc, NC, etc) may contain traces of mechanically entrapped acids, especially sulfuric acid. In other cases, acids (nitric and nitrous) might form on decompn of the above organic nitrates during storage, especially at high temps and in the presence of moisture. If these traces of acids are not immediately neutralized, they

might soon cause autocatalytic decompns of the expls or proplnts, thus rendering them useless for service. These decompns often develop into spontaneous combustion and even explns. Neutralization of the acids may be achieved by incorporating in the formulations of expls and proplnts some weakly basic substances, such as $CaCO_3$, ZnO, $MgCO_3$, urea, etc. These substances, "antiacids," may also be considered as "stabilizers" although this term usually applies to substances, such as diphenylamine, centralite, acardite, etc, which function in a different manner but achieve the same ultimate purpose

Refs: 1)Marshall **2** (1917), 640-1 2)Barnett (1919), 207-8 3)Davis(1943), 302 4)Kast-Metz (1944), 20

Addnl Refs: A)Olin-Mathieson Chem Corp, BritP 738441(1955) & CA **50**, 8148(1956) (Addn of metallic Mg, Ca, Zn & Al or their oxides or carbonates for stabilization of hydrazine) B)B.P.Enoksson, USP 2736742 (1956) & CA **50**, 6796(1956)(Addn to acid-contg nitrated products such as NG, NGc or NC of AN & ammonia and/or org bases, such as amines, amides, urea, etc) C)Nitroglycerin Aktiebolaget (Sweden), BritP 766588 (1957) & CA **51**, 8438(1957)(Same as in previous ref)

Antiaircraft (AA) refers to the weapons, ammunition and personnel used for defense against enemy aircraft

Refs: 1)Encyclopaedia Britannica **2**(1952), 59 2)Collier's Encyclopedia **2**(1957), 39-42

Antiaircraft Gunfire Control Systems are described in A.S.Locke et al, "Guidance," Van Nostrand, NY(1955), 22-33

Antiaircraft Projectile Charges of Willing contained a HE mixed with a material, such as emery capable of impairing the operation of internal combustion engines

Ref: M.S.Willing, USP 2,103,807(1935) & CA **32**, 1935(1938)

Anticaking Treatment of Explosives and Substances Used in Explosive Compositions. Substances such as AN have a tendency to cake in storage, thus rendering them difficult to load in cartridges, shells, etc. The problem of caking was recently investigated by LeRoux (Ref 2). Among the substances proposed to prevent caking ("anticaking agents") of substance, such as AN may be mentioned:

a)Coating compounds(matières d'enrobage, in Fr), such as petroleum oils, petroleum tars, paraffins & waxes (natural & synthetic) and resins. To these may be added organic substances which form gels in the presence of water, such as starches, dextrin, gum-arabic, Na-Al alginates, etc

b)Powdering agents(agents de poudrage, in Fr), such as finely powdered kaolin, bentonite, $CaCO_3$, $MgCO_3$, MgO, ZnO, SiO_2, Al_2O_3, talc (Mg silicate), Al, metallic soaps (such as Al stearate), etc. LeRoux proposed to incorporate ca 1% of Ca stearate in AN expls, such as "explosifs N" and "explosifs NR"

Whetstone (Ref 1) proposed to add "Acid Magenta" to a satd soln of AN, just before crystn. It has been claimed that this treatment modifies the form of crysts in such a manner that their agglomeration does not form cakes but leaves them as granules

Anticaking agents used with AN are described in this work on the following pages: A314(under "Continuous Process of Graining), A315 & A318 (under Crystallization Process), A318(under Preparation of FGAN), A334-5 (under Water Resistance and Prevention of Caking), A342-4(under AN Blasting Explosives) and A364(under FGAN)

Refs: 1)J.Whetstone, Industrial Chemist **25**, 401(1949) (Anticaking treatment of AN) 2)A. LeRoux, MP **33**, 265-82(1951) (Explosifs du type N résistant à l'eau)

Anticarro(Ital). Antitank

Antichar(Fr). Antitank

Anticoppering or Decoppering Agents. See under Fouling of Guns and Antifouling Agents

Anticracking Additives to Cast Explosives. The

problem of cracks in HE shells was investigated at Pic Arsn, by Heredia (Ref 1) and more recently by Johnson (Ref 3). According to Johnson, it has been reported that the problem of cracked cast chges has arisen when cooling w temps below 120°F (48.9°) for Comp B and below 65°F(18.3°) for TNT are utilized in an attempt to decrease solidification time. This condition has brought about the need for some compd(s) which, when added in small percentages to the melt, will prevent or minimize to a large extent cracked casts which are the result of thermal stresses. Previous work done at the Iowa Ordnance Plant, Burlington, Iowa and by the US Navy indicated that the addition of small amts of certain compds reduced the tendency of cast expls to crack when they were cooled at temps below 120°F for Comp B and 65°F for TNT

Additives used by the US Naval Ordnance Test Station, Inyokern, Calif included 0.5 to 1.0% α-MNN, 0.5% catechol, 0.25% anthracene and 0.25 to 0.75% Span 80A. In addition, small percentages of o- and p-nitrotoluene mixts, such as 3.36/1 were found to be effective. However, the high-melting eutectic-forming additives anthracene, α-MNN and cathechol were preferred because little exudation occurred at temps up to within ca 5° of the eutectic temp

Based on these earlier studies, a contract was entered into with the Armour Research Foundation, Chicago, Illinois for the purpose of finding compds that would overcome this tendency to crack. Optimum percentages of α-MNN, 2,4,6-TNAns and 1,2-dihydroxybenzene were investigated by the ARF (Ref 2) relative to prevention of cracks due to thermal shock and exudation when these compds were added to TNT and Comp B. Visual examination of laboratory casts (ca 3/16" diam by 3 5/8" length), when compared to control castings without crack preventing agents, showed progressive improvement in the casts with regard to the reduction in number of cracks as the percentage by wt of each additive was increased (such as from 0.1 to 1.5%) until the optimum percentage range was reached. Unfortunately, however, the amt of exudate increased as the percentage of each additive was increased. Lab test also have shown that the effectiveness of the additives as crack preventative agents was less for Comp B than for TNT

These rather optimistic results of lab tests, were not confirmed when the same additives were used at Picatinny Arsenal in cast-loaded 155 mm shell. The degree of cracking in the 155 mm shell casts was detnd by means of radiographs (which is a standard production inspection procedure), which showed that there was no apparent difference betw the degrees of cracking in an additive-contg cast and an ordinary cast. Exudation, as evaluated by subjecting the loaded shell to 160°F (71.1°) storage test for 30 days, showed that the casts contg additives exuded more than those without them

Further work on this subject is necessary

Refs: 1)R.J.Heredia, "Significance of Cracks in HE Shell and Effect of Interior Coating on Crack Formation," PATR 2269 (1956) 2) Armour Research Foundation Rept No 5 (Final Rept), "Industrial Engineering Study on the Determination of Additives to Eliminate Cracking of Cast Explosives," Chicago, Ill, Sept 8, 1958 3)D.H.Johnson, "Study of Crack Preventing Additives for Cast Explosives" TechRept DB-TR:6-60, I & MED, PicArsn, Dover, NJ, Feb 1960

Antidetonating or Antiknock Compounds (*Antidetonanti*, in Ital and Antidétonants, in Fr). Substances such as lead tetraethyl $Pb(C_2H_5)_4$ tin tetraethyl $Sn(C_2H_5)_4$, ferrocarbonyl $Fe(CO)_5$, nickel carbonyl $Ni(CO)_4$, aniline, etc possess the property of preventing knock in internal combustion engines and for this reason are also called "antiknock" substances. For their description see Refs 3,5,7 & 8

Several theories exist on the mechanism for antiknock action, such as outlined in Refs 1 & 5. Demougin(Ref 6, p 139), H.Moureu & C.Dufresse attribute the action of

"antidétonants" to their "antioxydizing power (pouvoir antioxygène, in Fr) which hinders the formation of peroxides

Some of the "antidétonants," such as tin tetraethyl, were proposed in France (Ref 6) as additives (in small quantities) to propellants as "flash reducing substances ("substances antilueurs," in Fr). The idea was based on the hypothesis that on the burning of a propellant such substances would evolve a dust of extremely small solid particles which when dispersed through the gases such as CO & H_2 would cool them thus preventing their combustion

Refs: 1)G.L.Wendt & F.V.Grimm, IEC **16**, 890-3(1924)(A suggested mechanism for antiknock action) 2)T.A.Boyd, IEC **16**, 893-5 (1924) (Relative effect of some nitrogen compds upon detonation in engines) 3)Wm.H.Charch et al, IEC **18**, 334-40(1926) (Antiknock materials) 4)H.L.Olin et al, IEC **18**, 1316-18 (1926)(The role of metallic colloids in the supression of detonation) 5)Pascal(1930), 303-9(Antidétonants) 6)P.Demougin, MP **25**, 130-41(1932-3) 7)Giua, "Dizionario" **1** (1948), 519-21(Antidetonanti) 8)Kirk & Othmer **6** (1951), 949-50 (Antiknock compds) 9)M.Taillandier & G.Touzeau, RevInd-Minérale **39**, 579-82(1957) & CA **51**, 17168 (1957) (Incorporation in expls some carbon or paraffin prevents rapid propagation of explns)

Antiflash Agents (Antiflash Compounds) (Antilueurs in Fr). See Flash-Reducing Agents

Antiflash Bags (Sachets antilueurs in Fr and Vorlage in Ger). See under Flash-Reducing Agents

Antiflash Pellets (Pastilles antilueurs in Fr). See under Flash-Reducing Agents

Antifoaming or Antifrothing Agents (Froth or Foam Preventing Compounds). For a general description of foams and antifoaming agents, see Refs 1,2,4 & 5. According to King (Ref 2), foaming is usually accompanied by a decrease in surface tension, but the converse is not necessarily true

Several instances of molten TNT becoming frothy when cast-loaded into shells were reported in the US during WWII. This resulted in undesirable cavitation at the interface between the solidified TNT and the upper part of the shell wall and also at the interface between the two layers of TNT when two pourings were made. Investigation conducted at PicArsn (Ref 3) showed that incorporation of small amts (such as 0.1%) of the surface-active agents Duponol C or Span 85 was sufficient to reduce frothing to the level of good grades of TNT. These additives did not reduce the stability of TNT and did not appreciably change its props such as setting point

The test for frothiness was conducted by melting ca 25g of TNT in a 6-inch test tube which was placed in a water-bath at ca 95°. The tube, about half filled with molten TNT, was stoppered, removed from the bath and vigorously shaken 5 times at the rate of one shake per 2 secs. The tube with contents was immediately placed in a thermostatic bath maintained at 86± 1°. The end point of frothing (duration of foam) was recorded as the time in seconds required for the froth to break until only 3 bubbles remained. The approximate amt of froth was also noted. Some standard grades of TNT showed duration of frothing as low as 50 secs, while TNT with abnormal frothing recorded 164 secs before addn of surface-active agents and as low as 59 secs after the addn

Further investigation of TNT with abnormal frothing showed that its surface tension (47 dynes/cm^2) was only slightly lower than that for a std TNT (51 dynes/cm^2), but there was also a sample of TNT with a low frothing value, which had an abnormally low surface tension (38 dynes/cm^2). The viscosity of frothy TNT was of the same order as that of the std lot of TNT. Chemical analysis of frothy TNT did not indicate any difference from std TNT. It was found, however, that incorporation in std TNT of small amts of impurities, such as wax or grease increased the frothing value, but incorporation of 0.06% acid-proof black paint or 0.07% tetranitromethane

had no adverse effect on the frothing value

Of the two surface-active agents investigated, the Duponol C, manufd by the duPont Co, is a solid and said to contain Na lauryl sulfate, while the Span 85, manufd by the Atlas Powder Co, is an oily liq and said to contain sorbitol trioleate. The use of Span 85 is preferred because, being purely organic, it does not contain any metallic ions, such as Na and also because, being a liquid, it dissolves quicker in molten TNT

Plant-scale tests at Iowa Ordnance Plant, Burlington, Iowa have shown that the addn of 0.1% of Span 85 to molten TNT is effective in preventing frothing and the consequent pithing and cavitation of the cooled charge. It has also been found that the presence of 0.1% of Span 85 has no significant adverse effect on the stability or impact sensitivity of TNT, and does not cause formation of TNT-water emulsion during the "streaming-out" of TNT chges. However, it causes significant reduction in sensitivity to initiation and a slight exudation in storage at 65°. TNT contg only 0.05% of Span 85 was found to have essentially normal sensitivity and brisance and to undergo no exudation in storage at 65°. It was recommended that authorization be granted for the addition of not more than 0.05% of Span 85 to TNT during loading operations

Refs: 1)S.Berkman & G.Egloff, "Emulsions and Foams," Reinhold, NY(1941) 2)F.G. King, JPhysChem **48**, 141-53(1944) (Foam formation in organic liquids) 3)A.J.Clear, PATR 1472(1944) (Cause and prevention of frothing of molten TNT) 3a)A.J.Clear, PATR 1553(1945) (Same title as above) 4) J.V.Robinson & W.W.Woods, "A General Method of Selecting Foam Inhibitors," Technical Note 1205, NACA, Washington,DC (1946) 5)Kirk & Othmer **5**(1950), 715-17

Antifouling Agents. See under Fouling of Guns

Antifreezes and Their Uses in Explosives and Propellants. For general description of various "antifreezes" used for general purposes see Refs 8

As some of the liq components of expls and propellants freeze at prevailing winter temps in Europe, Canada, US, etc, it is desirable to lower the fr p of such liqs by incorporating a substance exercizing an antifreezing effect. Such substance is called in Ger *"das gefrierpunktherabsetzendes Mittel"*

Widest uses of antifreezes are in NG contg expls, such as dynamites. Dynamites contg antifreezes are called *"low-freezing"* ("schwergefrierbare," in Ger) and *"non-freezing"* ("ungefrierbare," in Ger) dynamites ("Dynamite" in Ger)

Note: NG freezes at +13°, but when in compns, its crystn is rather slow. When an expl contg NG is stored in cold weather for a long time, it freezes to a mass as hard as a stone. Such a mass is unsuited for use because NG became insensitive. In order to make such an expl fit to use, it must be heated in order to melt NG. This operation, called thawing, is time-consuming and very dangerous, especially if conducted carelessly. It is possible that some accidents took place because on thawing part of the liquid NG escaped from the cartridges and was present in full strength at their surfaces. It is therefore highly desirable to prevent freezing of NG contg expls

According to Escales (Ref 2, pp 37-46) and Naoúm(Ref 3, pp 15-21 & 356-81), the first attempt to reduce fr p of NG was made by A.Rudberg(Swed P of April 30,1866) who added materials like benz or NB and later Nobel(Swed P, July 8,1876) proposed the addn of Me and Et nitrates,acetins, or NB. A.Wahlenberg & K.Sundström proposed in 1877 addn of o-MNT; K.Amark(1879) proposed nitrostarch, nitromannit and amyl nitrate and Liebert(1889) isoamyl nitrate. While Me & Et nitrates were the most suitable from the chem point of view, their volatility was too great. The other compds were effective only in such large quantities that they reduced the sensitiveness, strength and brisance of NG very appreciably

A more promising "antifreeze" was obtained in 1890 by A.Wohl(GerP 58947) who succeeded in polymerizing glycerin and nitrating to an expl oil, tetranitrodiglycerin, $[C_3H_5(ONO_2)_2]_2O$ very difficult to freeze. This process, however, attracted no attention at that time

A.Mikolajczak proposed in 1903 and patented the addn of dinitroglycerin (glycerin dinitrate), $C_3H_5(OH)(ONO_2)_2$, and developed a simple method for its prepn

S. Nauckhoff investigated the question of reducing the fr p of NG and showed in his paper (Ref 1) that all previously proposed agents could not completely achieve their purpose

The solution of the problem was more closely approached by the use of highly-nitrated aromatic hydrocarbons, especially the low-melting eutectic mixts of the isomers of di- and tri-nitrotoluenes (such as "TNT oil" or "drip oil" which dissolve readily in NG even at low temps and desensitize it much less than the mono-nitro compds. However, these compds did not produce dynamites which were absolutely non-freezing at the lowest winter temps reached in some countries such as in Canada or Siberia

Further research in this field showed that nitric esters homologous or related to NG, such as previously mentioned dinitroglycerin and tetranitrodiglycerin, as well as dinitrochlorohydrin, acetyldinitroglycerin and dinitroglycol, being misible in all proportions with NG and nearly equivalent in expl strength, were the most suitable antifreezes. Of these compds, the tetranitrodiglycerin (manuf patented by the "Zentalstelle fur Wissenschaftlich-technische Untersuchungen" in Neubabelsberg) found little use on acct of certain tech difficulties of manuf, which dinitrochlorohydrin does not present. Dinitroglycerin, although it is a satisfactory antifreeze, came into very little practical use on account of its solubility in w. Dinitrochlorohydrin, $C_3H_5Cl(ONO_2)_2$ was used quite extensively until the invention in 1904 (GerP 179879) of glycol dinitrate, $C_2H_4(ONO_2)_2$, or simply nitroglycol (abbreviated in our work as NGc). This substance has been widely used in the US since 1912 in "low-freezing dynamites" (qv). In actual practice a mixt of 20-30% glycol and 80-70% glycerine is nitrated in the same manner as straight NG

In addition to the foregoing antifreezes may be mentioned nitrosorbite (Ref 6, p 238) and nitrated sugar mixts (Ref 3, pp 251-8 & Ref 6, pp 239-40). A mixt of nitrosucrose and NG, prepd by nitrating a soln of 20 (or 25) parts of cane sugar and 80(or 75) parts of glycerin is known in the US as *nitrohydrene* and is suitable for use in non-freezing dynamites. Another low-freezing liq tried in the US dynamites was obtained by nitrating glycerin 60, glycol 20 and sugar 20%

According to E.Mohrenweiser of PicArsn, "gelatin-dynamites" freeze at a much slower rate than "straight-dynamites" and the higher the content of NC (which serves as a colloiding agent), the better is their resistance to low temps. The same applies to NG-contg propellants such as "ballistite," "cordite" and "solventless propellants." Most of these propellants do not contain any antifreezes. For instance, "double-base" rocket propellants manufd during WW II by the Hercules Powder Co at Radford, Va for shipment to Russia did not contain any antifreeze. There was added however, a small amt of liq plasticizer intended to facilitate the gelatinization of NC by NG. Some current US propellants contain DEGDN in lieu of NG and they are low-freezing. These propellants were invented in 1934 by the German Gen U. Gallwitz [See PATR 2510(1958), p Ger 70, as "G" Pulver]. The same inventor proposed in 1935, the use of nitrated polyglycol and TEGDN. Incorporation in "G" Pulver of large amt of NGu produced a propellant known as "Gudolpulver" [See PATR 2510 (1958), p Ger 81], proposed in 1937 by the Dynamite A-G

Refs: 1)S.Nauckhoff, ZAngewChem **18**, 11-22 & 53-60(1905) 2)R.Escales, "Nitroglycerine und Dynamit," Voigt, Leipzig (1908) 3)Naoúm, NG(1928), 15-21, 161-258 & 356-81 4) Stettbacher(1933), 166-72 233 5)Beyling &

Drekopf(1936),75-8, 92 & 316 6)Davis (1943), 154, 214-27 & 238-40 7)Stettbacher (1948), 61-2 & 83 8)Kirk & Othmer **2**(1948), 37-50(15 refs) 9)T.B.Wasserbach, USP 2,722,099(1955) & CA **50**, 2956(1956)(Tetra-, penta- and hexa-ethylene glycols when added to jet engine fuels, previously satd with w, prevented freezing even after 1 hour at –20° or –50°) 10)J.G.Tschinkel, IEC **48**, 732-5(1956) & CA **50**, 10369(1956) ($MeNO_2$ or NO_2 as antifreezes for rocket propellants contg TeNM as an oxidizer) 11)D.W.Riker, USP 2,768,888(1956) & CA **51**, 1589(1957) (Guanidine nitrate as an antifreeze for internal combustion and jet fuels contg hydrazine) 12)CalifResearchCorp, BritP 757916(1956) & CA **51**, 8413(1957) (Na nitrite solns as antifreezes for fuming nitric acid oxidizing compositions for hypergolic rocket propellants 13)E.Mohrenweiser, PicArsn, Dover, NJ; private communication(1960)

Antifrost Celladyne. An older Brit low-freezing dynamite which passed the Buxton test: NG+NGc 56.5, CC 3, $NaNO_3$ 6, NaCl 10.5 & borax 24%. Its ballistic pendulum swing was 2.51", vs 3.27" for 60% Gelignite

Ref: Marshall **1** (1917), 110

Note: The swing of 60% Gelignite, a std Brit expl is given by Barnett (1919), 184

Antifrost Gelamonite No 1. An older Brit "permitted" low-freezing dynamite: NG+NGc 24, CC 1, DNT + TNT 2, AN 36, WM 1 & NaCl 36%. Its ballistic pendulum swing was 2.30" vs 3.27" for 60% Gelignite

Ref: Marshall **3** (1932), 120

Antifrost Penryhn Powder. One of the older Brit "permitted" expls: AN 58.5-61.5, NG 7.5-9.5, WM 7.5-9.5, NaCl 20.5-22.5 & moisture 2.0%

Ref: Thorpe **4** (1940), 556

Antifrothing Agents. See Antifoaming Agents

Antifume Blasting Gelatin. An older SoAfrican (Transvaal) expl consisting of "blasting gelatin," to which a small quantity (ca 5%) of an oxidizer, such as AN, K nitrate, K chlorate or K perchlorate, was added. This expl was used in mines with poor ventillating facilities, where the formation of even small quantities of the highly poisonous CO (as often occurs on expln of ordinary "blasting gelatin") is very undesirable

Ref: Naoúm, NG(1928), 321

Antigel de Sûreté. One of the older Belgian permissible expls of the type "explosifs SGP":NG 25, Na nitrate 20, DNT 15, Amm sulfate 5, cellulose and/or WM 35%

Ref: Marshall **1** (1917), 376

Antigrisou D'Arendonck. One of the oldest Belgian permissible expls: AN 72, NG 27 & CC 1%

Refs: 1)Daniel(1902), 28 2)Gody(1907), 701

Antigrisou(Explosifs) are expls safe for use in coal mines in the presence of firedamp ("grisou", in French). The terms "antigrisou," "antigrisouteux" and "grisoutine" have been used in France, but in Belgium such expls are known as "explosifs, SGP" ("explosifs Securité-Grisou-Poussière") (See tables on pp 419-21 of Ref 4). All these expls contain large amts AN. The following "antigrisou" expls called also antigrisous Favier or grisounites, were introduced (before WWI by Favier (Ref 2, pp 593-4) and manufd for some time by the SA des Explosifs Favier at Vilvorde (Troix Fontaines, near Bruxelles):

Names of Explosives	Components			
	AN	DNN	TNN	NH_4Cl
Antigrisou No 1	87.6	12.4	–	–
Antigrisou No 2	87.6	12.4	–	8.0(added)
Antigrisou No 3	81.0	–	6.0	13.0
Antigrisou II	80.9	11.7	–	7.4
Grisounite roche	92.0	8.0	–	–
Grisonite couche	95.5	–	4.5	–

Note: Pérez-Ara (Ref 5) gives for No 1: AN 81, DNN 6 & NH_4Cl 13% and for No 2: AN 81, TNN 6 & NH_4Cl 13%

The calcd temp of expln of No 1 is ca 2000°, of No 2 1878°, and of No 3 1400°

According to Pepin Lehalleur(Ref 4) the Antigrisou No 1 was used during WWI for filling some HE shells. It was safe to handle, inexpensive and insensitive, but not as powerful and brisant as TNT

Pepin Lehalleur (Ref 4, p 343) gave also the following compns of "antigrisou" explosives reported in 1935 to be in use in France at that time

	Antigrisou (Explosifs)		
Components	Roche	Couche	Couche salpêtrée
NG	29.1	11.7	12.0
CC	0.9	0.3	0.5
KNO_3	–	–	5.0
AN	70.0	88.0	82.5

Médard (Ref 6) gives the following compns of current "explosifs antigrisouteux."

	Explosifs antigrisouteux		
Components	N no 7	N no 9	N no 62
AN	76.0	48.2	12.0
DNN	7.0	–	–
TNT	–	9.15	–
PETN	–	–	23.0
WF*	2.0	1.65	–
NaCl	15.0	41.0	65.0

*WF = wood flour

The first of these expls, belonging to the type "l'explosif-couche nitraté," was proposed in 1933 by Burlot & Schwob. Its CUP value (coefficient d'utilisation pratique) is 87(PA = 100) and gap test value (coefficient de self-excitation) 6cm [See also C(Explosifs), CSE(Explosifs), Dynamites, Grisou-dynamites, Grisou-dynamites chlorourées, Grisounites, Grisoutines, Grisoutites, N(Explosifs), etc]

Refs: 1)Daniel(1902), 28 2)Gody(1907), 593,701 & 705 3)Pascal(1930), 219-20 4) Pepin Lehalleur(1935), 343 & 419-21 5) Pérez-Ara(1945),240 6)L.Médard, MP **32**, 219-22(1950)

Antiknock Compounds. See Antidetonating Compounds

Antilueur (Fr). Antiflash

ANTIMONY(Stibium in Lat; Antimon, in Ger; Antimoine, in Fr; Antimonio, in Ital & Spanish; Soor'ma, in Russian), Sb, at wt 121.76. Lt greyish metal, mp 630.5°, bp 1380°, d 6.684 at 25/4°, sp heat 0.049, latent heat of fusion 38.3 cal/g, Moh's hardness 3-3½. Temp of ignition of powder in air >600° (Ref 9, p 37). It is insol in w; sol in aqua regia & in hot concd H_2SO_4

Native Sb element is known, but it is too rare to be commercially important. It exists in nature in the form of several ores of which the most important is *stibnite,* which is sulfide, Sb_2S_3. Commercial method for obtaining free Sb consists of fusing stibnite with iron turnings or shavings, followed by slagging off the resulting iron sulfide with NaCl or Na sulfate. Detailed descriptions of methods of Sb recovery from ores are given in Ref 5 pp 64-9

According to Kirk & Othmer (Ref 5, p 51), metallic Sb is non-toxic, although many of its compds are toxic. According to Sax (Ref 10) antimony is highly toxic, but because it is usually associated in industry with lead and arsenic, it is often difficult to assess the toxicity of Sb and its compds. According to Elkins (Ref 7, p 67) "occasionally workers exposed to the dust of antimony or its compounds exhibit symptons of gastrointestinal upset, usually accute rather than chronic in character". Elkins gives MAC for Sb dust or fume in air ca 1 mg/m³ (Ref 7, p 225), while Jacobs (Ref 6, p 253) gives 0.1 mg/m³. The same author gives on p 766, 0.5 mg/m³ as "probable safe concn limits of exposure for toxic dusts, fumes and mixts," as recommended by the US Govt hygienists. Physiological action of Sb and its compds is described in Mellor (Ref 1, p 385)

In addn to the above common metallic form (rhombohedral crysts), Sb also exists in *yellow, black* and the so-called *explosive* forms.

The *yellow* form is non-metallic and corresponds to yel phosphorous or yel arsenic.

It is obtained by adding oxygen to Sb hydride at -90°. It is unstable and changes to the black form at temps above -90°. According to Krebs et al (Addnl Ref C), the yel form is a mixed polymer contg chemically bonded H atoms

The *black*, amorphous, form is produced by sudden cooling of Sb vapors. The black form is more active and more easily volatilized than the metallic form. It is said (Ref 5, p 51), that if Sb is distilled in high vacuum, it deposits as an amorphous powder which might explode on heating or scratching

The *explosive antimony*, prepd either electrolytically or by other methods, is described below under the title "Antimony, Explosive."

Antimony is used in many alloys, chiefly for its props of hardening the softer metals, such as Pb. The best known of these alloys are: type metal, bearing metal, hard lead and pewter. The powdered Sb is used in some pyrotechnic compns, as a fuel (Ref 9, p 32) and as a source of white light (Refs 3a & 8)

Following are some pyrotechnic compns listed by Davis (Ref 3a), expressed in parts by wt:

Components	White Lights		White Stars	
Sb, metal	12	5	5	40
Amm picrate	–	5	–	–
K nitrate	32	–	28	180
Ba nitrate	–	30	–	–
Sulfur	15	–	8	50
Pb_3O_4	10	–	–	–
Sb_2S_3	–	–	–	10
Dextrin	1	–	1	6
Lampblack	–	1	–	–
Paraffin	–	1	–	–
Charcoal dust	–	–	–	3

Izzo (Ref 8) lists quite a number of pyrotechnic compns contg Sb powder, of which the following were selected as examples:

Components	White Lights				
K nitrate	49	50	60	31.5	–
Sulfur	15	16.5	20	5	–
Charcoal	–	–	–	16	14.5
Pulverin	18	–	–	31.5	43
Lampblack	–	–	–	–	28
Antimony	18	16.5	10	16	14.5
Sb_2S_3	–	17	–	–	–
As_2S_2	–	–	10	–	–

One of the white light compns listed in Izzo (Ref 8, p 217) contained black antimony 20, K nitrate 54, sulfur 20, charcoal 2.5 & lampblack 3.5%

Antimony was also proposed as a component of expl compns (Addnl Refs A & B)

According to Weingarten (Ref 11), metallic antimony is not used in any current US military pyrotechnic compns, but the US Military Specification MIL-A-10841B, 10 Sept 1958, deals with Antimony, Technical, intended for use in pyrotechnics (See Antimony, Analytical Procedure

Refs: 1)Mellor **9** (1929), 339-90 2)Thorpe **1** (1937), 439-46 3)Gmelin, Syst Nr 18, Teil 1 (1942-3) 3a)Davis(1943), 64,70-1 & 83 4) C.Y.Wang & G.C.Riddell, "Antimony," in Liddel's "Handbook of Nonferrous Metallurgy," McGraw-Hill, NY, **2**(1945), 104-38 5)Kirk & Othmer **2**(1948), 50-69 6)Jacobs(1949), 253-63 & 766 7)Elkins(1950), 67-8 & 225 8)A. Izzo, "Pirotecnia e Fuochi Artificiali," Hoepli, Milano(1950), 216-19 & 225 9)A.A. Shidlovskii, "Osnovy Pirotekhniki," Gosizdat Oboronprom, Moscow(1954), 32, 37 10)Sax (1957), 305-6 11)G.Weingarten, PicArsn, private communication (1960)

Addnl Refs: A)L.Carta, CanP 378158(1938) & CA **33**, 2339(1939) (Expls contg K chlorate, antimony, sulfur & Na bicarbonate with suitable binding and combustion controlling agents, such as flour, coal, wood, grease & petroleum) B)S.Kinoshita & T.Sakamaki, JapP 2498(1953) & CA **48**, 6700(1954) (Use of antimony powder in electric detonator compns. Eg: Sb 80, Pb mononitroresorcinate 10 & K chlorate 10% with a suitable binder) C)H.Krebs, et al,

ZAnorgAllgemChem **288**, 177(1955) & CA **50**, 9816(1956) (Allotropy of antimony)

Antimony, Explosive. According to Mellor (Ref 1), it was first prepd in 1855 by G.Gore and then in 1858 by R.Böttger. It is a black powder usually obtained by electrolysis of solns contg antimony trichloride, tribromide or triiodide using Pt, Cu, Zn, Hg or graphite as cathodes. A low temp or high current density favors production of an expl deposit but if the current exceeds a certain limiting value the deposit might explode during the electrolysis. The deposited material is a black, inhomogeneous, amorphous mass, which always contains varying amts (such as 4-15%) of occluded or absorbed halide. It explodes when subjected to a mechanical action (such as impact, grinding or scratching) or when rapidly heated to 110-125°. The heat of expln of a sample contg 4% of $SbCl_3$ is, according to Coffin & Hubley (Ref 14), ca 22.2 cal/g. Sidgewick (Ref 13) gives for a sample, contg 10-15% of a halide ca 2.4 kcal/atom. According to theory of Coffin (Ref 6), expln of Sb is due to heat evolved when the amorphous structure (probably gel-like) is changed to crystalline. According to Glazunov & Lazarev (Ref 8), the expl props of Sb depend upon the quantity of Cl in the space lattice of the Sb. The groups $SbCl_2$ & SbCl are formed in the space lattice by the reactions $SbCl_4^- \rightleftharpoons SbCl_2^+ + 2Cl$ and $SbCl_4^- \rightleftharpoons SbCl^{++} + 3Cl^-$. Within limits, the greater the current density, the more undecomposed $SbCl_2^+$ & $SbCl^{++}$ remain and the more abundantly they pass into the cathode metal. These groups force apart the space lattice of Sb and thus produce a lability and a tendency to transformation. According to Krebs et al (Ref 15), the expl form of Sb is a mixed polymer contg chemically bonded Cl atoms. According to Frongia & Ladu (Ref 11), the expl Sb is inhomogeneous, and crystn to normal Sb can begin simultaneously at various points throughout the mass. The heat thus liberated propagates the reaction. Application of heat, or electricity renders the reaction explosive

Refs: 1)Mellor **9**(1929),359 2)E.Cohen & C.C. Coffin, ZPhysikChem **149A**,417(1930) & CA **24**,5553(1930) (Prepn and physico chem study of expl Sb) 3)H.von Steinwehr & A. Schulze, ZPhysik **63**,815(1930) & CA **24**, 5553(1930) (The nature of expl Sb) 4)J.A. Prins, Nature **131**,760(1933) & CA **27**,4160 (1933) (Electron diffraction patterns of amorphous and cryst Sb) 5)C.C.Coffin & S.Johnston, ProcRoySoc **A146**,564(1934) & CA **29**,974(1935) (Microscopic examination of expl Sb) 6)C.C.Coffin, ProcRoySoc **A152**, 47(1935) & CA **30**,924(1936) (Structure, elec conductivity & rate of crystn of expl Sb) 7)C.C.Coffin, CanadJRes **13A**,120(1935) & CA **30**,2061(1936) (Magnetic susceptibility of expl Sb) 8)A.Glazunov & N.Lazarev, ChemListy **34**,89–90(1940) & CA **38**,5741 (1944) (Mechanism of formation and structure of expl Sb) 9)R.Glocker & H.Hendus, ZElektrochem **48**,327(1942) & CA **37**,5633 (1943) (Diffraction diagram of expl Sb) 10)H.Hendus, ZPhysik **119**,265(1942) & CA **37**,6514(1943) (Structure of expl Sb detd by X-rays) 11)G.Frongia & M.Ladu, Rend-SeminarFacoltaSciUnivCagliari **15**, No 2/3, 19pp(Separate) & CA **45**,6843(1951) (Structure of expl Sb and mechanism of transformation to cryst form 12)Kirk & Othmer **2** (1948),51 13)Sidgwick, ChemElems **1**(1950), 759 14)C.C.Coffin & C.E.Hubley, Canad JRes**28B**,644–7(1950) & CA **45**,3703(1951) (Detn of the heat of transformation of expl Sb to cryst form) 15)H.Krebs et al, ZAnorg AllgemChem **288**,177(1955) & CA **50**,9816 (1956) (Structure of expl Sb)

Antimony, Analytical Procedures. Various qualitative and quantitative procedures for detn of Sb including its detection in air are given in Refs 1,2 & 3. Specification requirements for refined antimony are given in Ref 4 and the US Military Specification requirements for technical antimony are given

The military requirements and tests are given as follows:

A)Color and Appearance. The material shall be a grayish-black powder when visually examined

B) Purity. The material shall contain not less than 98.0%, by wt, of Sb, when tested as described below:

Transfer ca 0.2 g sample weighed to 0.1 mg to a 500 ml Erlen flask contg 5g $NaHSO_4$ and 20 ml concd H_2SO_4. Heat until soln is complete and if any sulfur is visible, volatilize it by heating the flask over an open flame. Cool, add 10 ml H_2O and cool again. Add cautiously 5 ml of concd H_3PO_4 and 1 g Na_2SO_3. Heat for 20 mins to expel the excess of SO_2 and cool again. Add 100 ml H_2O and 15 ml concd HCl. Cool in an ice bath to 5–10° and titrate with 0.1N $KMnO_4$ to the appearance of a pink color which persists for 5 secs. Run a blank and calc as follows:

$$\%Sb = \frac{6.088 \times N \times (V_1 - V_2)}{W}, \text{ where}$$

N = normality of $KMnO_4$ soln; V_1 = ml of $KMnO_4$ soln used for sample; V_2 = ml of $KMnO_4$ soln used for blank and W = weight of sample

C) Moisture (max 0.1% by wt). Dry a glass-stoppered weighing bottle in a vacuum oven at 50–55° and 25" Hg for an hr, cool in a desiccator, and tare it to 0.1 mg. Transfer ca 10 g of the sample to the bottle and weigh. Heat unstoppered in the same vacuum oven as above for 2 hrs, stopper the bottle, cool it in vacuum desiccator and reweigh. Calculate as follows:

$$\% \text{ Moisture} = \frac{100 \times (W_2 - W_3)}{W_2 - W_1}, \text{where}$$

W_1 = weight of empty bottle; W_2 = wt of bottle with sample before removal of moisture and W_3 = wt of bottle with sample after removal of moisture

D) Granulation of the material shall be such that not less than 99.5% will pass through a No 80 US Std sieve(177-micron), not less than 90% through a No 140 sieve(105-micron) and not less than 75.0% through a No 270 sieve(53-micron), when tested as described below:

Weigh to the nearest 0.1 g clean, dry sieves and assemble them in order of increasing fineness, with the coarest sieve on top, so that the material passing through a sieve is transferred directly to the next one in the series. Place under the bottom sieve a pan. Weigh a 200 g sample to the nearest 0.5 g and place it on the upper(No 80) sieve, cover it and attach the assembly by means of clamps to a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Shake for 3 mins and weigh each sieve to the nearest 0.1 g. Calculate the percentage passing through each sieve as follows: % through No 80 = $\frac{100 \times (W - A)}{W}$; % through No 140 = $\frac{100 \times (W - A - B)}{W}$ and % through No 270 = $\frac{100 \times (W - A - B - C)}{W}$, where W = wt of sample used (200 g); A = wt of material retained on No 80 sieve; B = wt of material retained on No 140 sieve and C = wt of sample retained on No 270 sieve

Refs: 1)W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis," Van Nostrand,NY(1939),63–86 2)Jacobs(1949), 254–9 3)Elkins(1950,279 4)ASTM Standards 1955,Part 2,pp 522–3, ASTM Designation B237–52(reapproved in 1955) 5)US Military Specification MIL–A–10841B, 10 Sept 1958(Antimony, Technical) (For use in pyrotechnics)

Antimony Azide. See under Azides, Inorganic

Antimony Chloride. See under Chlorides

Antimonyl Potassium Tartrate. See under Tartrates

Antimony Pentasulfide. See under Sulfides

Antimony Selenide. See under Selenides

Antimony Sulfides. See under Sulfides

Antimony Telluride. See under Tellurides

Antimony Trichloride. See under Chlorides

Antimony Triethyl. Same as Triethylstibine

Antimony Trimethyl. Same as Trimethylstibine

Antimony Trisulfide. See under Sulfides

Anti-Motor-Torpedoboat (AMTB) was used during WWII to combat Ital motor-torpedoboats, each equipped with a powerful gun and running close under the heights along the coast. AMTB's were equipped with

90 mm guns on the special mount M3 which permitted abnormal depression of the gun tube *Ref:* F.W.F.Gleason, "A Glossary of Ordnance Terms," AmyOrdn **29**,368(1947)

Antioxidants. In general, an antioxidant is a substance which prevents or retards oxidation of various constituents of materials. For some substances, for example rubber, an antioxidant means an age-resistor; and substances like tannic acid, anthraquinone or aniline are used. It is said that antioxidants possess "antioxidant power" ("pouvoir antioxygène," in French). A fairly comprehensive description of antioxidants is given in Kirk & Othmer (Ref 5)

In case of propellants, several French investigators(Refs 1,2,3), called some substances, which prevent oxidation of CO to CO_2, "antioxidants." These substances are usually incorporated in propellants to suppress the flash and are called in Fr "antilueurs." These investigators found that when small amts of certain substances, particularly KCl, K hydrogen tartrate or powdered tin, which are volatile at high temps, are vaporized in an atm of mixed CO and air, the temp of ignition of CO is raised to such an extent that the gas exiting from a weapon does not ignite at the muzzle. For instance, for a mixt contg 24.8% CO and 75.2% air, the regular temp of ignition, 656° was raised to 1010° by a simple addn of 3.5% KCl to the propellant. For a propellant producing a CO–air mixt contg 44.1% CO, 2.5% KCl was sufficient to raise the ignition temp to 1000°. It was also found that KCl has no effect upon the ignition temp of hydrogen–air mixt

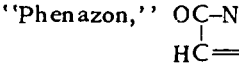
In another series of experiments it was found that addn of small quantities of so-called "antidetonating substances" ("antidétonants," in Fr), such as tin tetraethyl(Ref 1), reduced the flash, because these "antidétonants" acted as "antioxidants" since they prevented the formation of peroxides

Davis(Ref 4) gives a short resume of work of these French investigators(Refs 1,2,3)

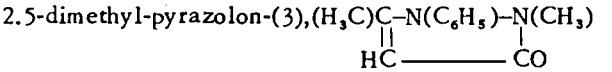
(See also under Flash Reducing Compounds)

Refs: 1)P.Demougin, MP **25**,139–41(1932–3) 2)J.Fauvau & (?)LePaire,MP **25**,142–59 (1932–3) 3)M.Prettre,MP **25**,160–7(1932–3) 4)Davis(1943),323 5)Kirk & Othmer **2**(1948), 69–75(12 refs) and 1st Suppl(1957),77–88 (68 refs)

ANTIPYRINE AND DERIVATIVES

Antipyrine; 2,3-Dimethyl-1-phenyl-3-pyrazolin-5-one or Phenazone, listed in Beil as 1-Phenyl-2.3 -dimethyl-pyrazolon-(5) or "Phenazon,"  , is described in Beil **24**,27,(194) & [11]

There is also an isomer called:

3-Antipyrine or 2,5-Dimethyl-1-phenyl-4-pyrazoline-3-one, listed in Beil as 1-Phenyl-2.5-dimethyl-pyrazolon-(3), 

Ref: Beil **24**,34,(198) & [17]

4-Azidoantipyrine, $C_{11}H_{11}N_5O$, mw 229.24, N 30.55%. Lt yel crysts(from benz by petr eth), mp 74°(dec); easily sol in alc, benz or acet; insol in petr eth. Can be prepd by diazotization of 4-aminoantipyrinesulfate, followed by addn of NaN_3. Its expl props were not examined

Refs: 1)Beil **24**,56 2)M.O.Forster & R. Müller,JCS **95**,2075(1909)

Diazidoantipyrine, $C_{11}H_{10}N_8O$ – not found in Beil or CA through 1956

Antipyrine Nitrate, $C_{11}H_{12}N_2O{\cdot}HNO_3$ – not found in Beil

Antipyrine Picrate, $C_{11}H_{12}N_2O + C_6H_3N_3O_7$. Two isomers; yel ndls, mp ca 188°(not sharp) and lt yel ndls, mp 168°(not sharp) are listed in Beil **24**,31,34

Antipyrine Complexes with Nitrates of Some Rare Earth Metals are mild expls

Ref: Beil **24**,(196)

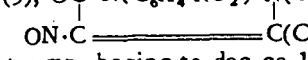
Antipyrine Complexes with Perchlorates of Some Rare Earth Metals and of some other metals are mild expls

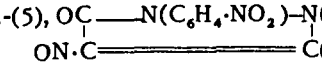
Ref: Beil **24**,[14–15]

Mononitroantipyrines, $C_{11}H_{11}N_3O_3$. Several isomers, none of them expl, are described in Beil **24**,55,(197–8) & [27]

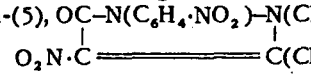
Nitroantipyrine Nitrates, $C_{11}H_{11}N_3O_3 + HNO_3$. One isomer *3-nitroantipyrine nitrate*, mp 143°, is described in Beil **24**,(198)
Nitroantipyrine Picrates, $C_{11}H_{11}N_3O_3 + C_6H_3N_3O_7$. Two isomers: yel crysts, mp 165° and yel crysts, mp 101° are listed in Beil **24**,(198)
Nitrosoantipyrine, $C_{11}H_{11}N_3O_2$. One isomer *4-nitrosoantipyrine* is described in Beil **24**,(217)

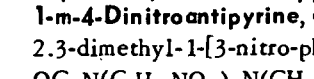
Nitronitrosoantipyrines, $C_{11}H_{10}N_4O_4$, mw 262.22, N 21.37%. The following isomers are described in the literature:

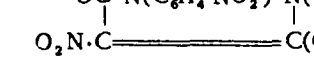
1-(m-Nitro)-4-nitrosoantipyrine, called in Ger 4-Nitroso-2.3-dimethyl-1-[3-nitrophenyl]-pyrazolon-(5),  . Green crysts; mp–begins to dec ca 165° and deflagrates at 188–90°; insol in common solvents. Was obtained by treating 1-m-nitroantipyrine with calcd amt of Na nitrite in AcOH, and cooling
Refs: 1)Beil **24**,(217) 2)A.Michaelis et al, Ann **378**,302(1911) & CA **5**,1274(1911)

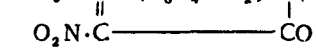
1-(p-Nitro)-4-nitrosoantipyrine, called in Ger 4-Nitroso-2.3-dimethyl-1-[4-nitro-phenyl]-pyrazolon-(5),  . Green crysts; mp– turns yel & brn at ca 176° and melts at 188–9°; sol in acet; sl sol in w, alc & AcOH; insol in petr eth. Its expl props were not investigated
Refs: 1)Beil **24**,(217) 2)A.Michaelis et al, Ann **378**,332(1911) & CA **5**,1274(1911)

Dinitroantipyrines, $C_{11}H_{10}N_4O_5$, mw 278.22, N 20.14%. The following isomers are described in the literature:

1-(o-Nitro)-4-nitro-antipyrine or 1-o-4-Dinitroantipyrine, called in Ger 4-Nitro-2.3-dimethyl-1-[2-nitro-phenyl]-pyrazolon-(5),  . Ndls, mp 244°; sl sol in w, alc or chlf; more sol in AcOH; insol in ligroin. Was prepd by treating 1-o-nitroantipyrine with concd nitric acid at temp below 60°. Its expl props were not investigated
Refs: 1)Beil **24**,(220) 2)A.Michaelis et al, Ann **378**,321(1911) & CA **5**, 1275(1911)

1-(m-Nitro)-4-nitro-antipyrine or 1-m-4-Dinitroantipyrine, called in Ger 4-Nitro-2.3-dimethyl-1-[3-nitro-phenyl]-pyrazolon-(5),  . Wh shiny ndls, mp 203°(with expl decompn); insol in alc, eth & w; sol in AcOH. Was prepd by treating 1-m-nitroantipyrine with concd nitric acid, with sl warming
Refs: 1)Beil **24**,(221) 2)A.Michaelis et al, Ann **378**,302(1911) & CA **5**,1274(1911)

1-(p-Nitro)-4-nitro-antipyrine or 1-p-4-Dinitroantipyrine, called in Ger 4-Nitro-2,3-dimethyl-1-[4-nitro-phenyl]-pyrazolon-(5),  . Col crysts, mp 276°; insol in w or alc; sl sol in AcOH. Can be prepd by treating 1-p-nitroantipyrine with concd nitric acid or by treating antipyrine with mixed nitric-sulfuric acid. Its expl props were not examined
Refs: 1)Beil **24**,(221) 2)A.Michaelis et al, Ann **378**,333–4(1911) & CA **5**,1275(1911)

1-(m-Nitro)-4-nitro-3-antipyrine or 1,4-Dinitro-3-antipyrine, called in Ger 4-Nitro-2.5-dimethyl-1-[3-nitro-phenyl]-pyrazolon-(3),  . Yel crysts, decompg explosively at ca 271°; diff sol in alc, more sol in AcOH. Was obtained by treating 1-m-nitro-3-antipyrine with concd nitric acid, with cooling
Refs: 1)Beil **24**,55 2)A.Michaelis & A. Stiegler, Ann **358**,155(1908)
Trinitroantipyrine, $C_{11}H_9N_5O_7$ and *Tetranitroantipyrine*, $C_{11}H_8N_6O_9$ were not found in Beil or CA through 1956

Antisanzionite(Ital). Same as ANS(Esplosivo)

Antisubmarine Weapons include: guns, mines, nets & booms, projector charges, bombs, depth charges & depth bombs, torpedoes(aircraft, surface and submarine launched), rockets and guided missiles
Ref: H.P.Cooper,Ordnance **36**,583–5(1952)

Antitank(A/T) Ammunition and Weapons include: A/T grenades, guns, incendiaries, mines, rifle grenades and rockets

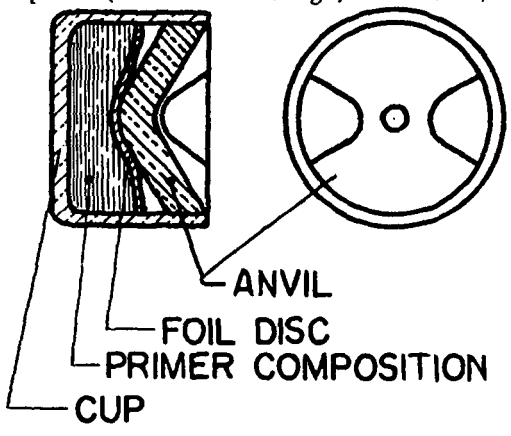
Refs: 1)Ohart(1946),4,9,139,179,354,361, 363–7 2)G.E.Rogers, "Antitank Mines and Fuzes," Lecture delivered at PicArsn,Dover, NJ, on 6 Feb 1948 3)ArmamentEngrg(1954), 343–6 4)Anon,"Ammunition General," Dept of the Army Manual TM 9–1900(1945),109, 162,215–26,243–55

Antonite Cava. An Ital mining expl manufd by the Società Vulcania di Brescia
Ref: A.Izzo, "Manuale del Minatore Esplosivista," Hoepli, Milano(1953),32

Antonite Galleria Extra. An Ital mining expl consisting of AN & TNT in propn to give an oxygen balance of ca +2.5%. Its props are reported as *Trauzl test* 400 cc, *vel of deton* (by Dautriche method) 4300 m/sec, *impact sensitivity* with 2 kg wt 100 cm, *expln temp* >180°, *temp of expln* 2570°, *expln pressure* 8290 kg/cm². It was manufd by the Società Vulcania di Brescia
Ref: Same as above,pp 17 & 32

ANU. A cast double-base propellant described in conf Propellant Manual SPIA/MZ(1959), Unit No 412

Anvil of a Percussion Primer is a metallic item of a special shape, which is pressed into the open end of a metallic cup contg primer compn(such as one consisting of MF, K chlorate, Sb sulfide & ground glass). A paper disc is placed over the compn, prior to insertion of anvil, to provide a moisture seal. The ensemble is pressed into an ammunition component(such as a cartridge) and fired by striking the bottom of the cup with a firing pin which crushes the primer compn against the anvil



Refs: 1)Davis(1943),455 2)Ohart(1946),47 3)"Ammunition General," TM 9–1900(1956), 72–3 4)A.B.Schilling, PicArsn; private communition(1960)

AOE;AOK;AOR;AOV. Cast double-base propellants described in conf "Propellant Manual," SPIA/M2(1959),UnitNos 414,415,416 & 417

AP. See Armor-piercing

Apache Coal Powder. A nongelatinous permissible expl manufd by the Apache Powder Co
Ref: Bebie(1943),28

Apache Powder Co, located in Benson, Arizona was established in 1921 by W.W. Edwards(1876–1922), formerly of Aetna Explosives Co. Apache Co has been manufg various grades of dynamites and its principal trade is in copper mines of S.Arizona(such as Bisbee) and adjacent territory
Ref: Van Gelder & Schlatter(1927),634

APC. See Armor-piercing Capped

Äpfelsäure(Ger). Malic Acid

Aphosite. An older Brit "permitted" expl: AN 58–62, K nitrate 28–31, charcoal 3.5–4.5, WM 3.5–4.5, sulfur 2–3 & moisture 0–1.5%
Refs: 1)Daniel(1902),29 2)Escales, Ammonspr(1909),188

APIGENIN AND DERIVATIVES

Apigenin; 4',5,7-Trihydroxyflavone or 4',5,7-Trihydroxy-2-phenylchromone, called in Ger 5.7.4'-Trioxy-flavon or 5.7-Dioxy-2-[4'-oxy-phenyl]-chromon

, is described in Beil **18**,181,(396) & [172]
Apigenin, Azido-, $C_{15}H_9N_3O_5$ and *Diazido-*, $C_5H_8N_6O_5$, *Derivatives* were not found in Beil or CA through 1956
Mononitroapigenins, $C_{15}H_9NO_7$. One isomer is listed in Beil **18**,183
Dinitroapigenins, $C_{15}H_8N_2O_9$ – not listed in Beil

Trinitroapigenins, $C_{15}H_7N_3O_{11}$. Two isomers: yel ndls, mp 296° and orange leaflets, mp 245–6° are listed in Beil **18**, 183

Tetranitroapigenins, $C_{15}H_6O_5(NO_2)_4$, mw 450.23, N 12.45%. One isomer *x,x,x,x-tetranitro-4',5,7-trihydroxyflavone*, nearly col ndls, mp 243–4° (dec), diff sol in common solvents, is described in Refs 1–3. Can be prepd by nitrating apigenin with mixed nitricsulfuric acid. Its expl props were not investigated

A compd of mp 258°, described in Ref 4, was prepd by heating with 20% HNO_3 the brn-red vitexin (Ref 4), $C_{15}H_{14}O_7$, obtd from air-dried and powdered bark of Vitex peduncularis

Refs: 1)Beil **18**, 184 2)A.G.Perkin, JCS **73**, 1025(1898) 3)Ibid, **77**, 420(1900) 4)G.Barger, JCS 89II, 1222(1906) 5)V.N.Sharma, JSciIndResearch(India) **14B**, 267(1955) & CA **50**, 5649(1956)

Aplastic Anemia and Toxic Hepatitis can be caused by exposure to TNT or other aromatic polynitro compds and to polynitramino compds

Refs: 1)R.L.Stump et al, US PubHealthBull No **291**, 85–98(1945) (included are 70 refs) 2)R.F.Sievers et al, Occupational Medicine **1**, 351–62(1946)

Apparatus for Blasting Coal, patented by Davis et al, consists of a pressure-resistant metal tube, the end of which is sealed by a rupture disk for releasing the gas pressure at the desired value. The tube contains a chge of gas-generating expl, such as AN + starch, with a nichrome elec heater embedded in it. The material is heated until decompd and sufficient gas pressure is generated to rupture the disk

Ref: C.O.Davis et al, USP 2,736,261(1956) & CA **50**, 10412(1956)

Apparatus for Indicating the Presence of Explosive or Flammable Vapors or Gases was developed by H.T.Ringrose, BritP 711,133 (1954) & CA **48**, 8544(1954)

Apparatus(Laboratory) for Continuous Preparations(Including Nitrations) of Explosives is described by F.Tabouis & J.Vaganay, MP **37**, 413–16(1955)

Apparechio(Ital). Apparatus

Apparechio nebbiogeno(Ital). Smoke generator

Appareil(Fr). Apparatus

Appareil d'écoute(Fr). Sound locator

Appareil de pointage(Fr). Sighting gear

Apparent Density. See under Density

Apparent Ignition Temperature in Air is the same as Spontaneous Ignition Temperature (SIT) described in this work under Flammability of Gases, etc

Applications of Explosives for Non-Explosive Purposes. See Explosives, Applications of Non-Explosive Nature

Applied Analysis. Title of the book by Cornelius Lanczos, Prentice Hall, Englewood Cliffs, NJ(1956). It deals with mathematics and must not be confused with Applied Chemical Analysis

Applied Chemistry and Applied Chemical Analysis deal with application of chemistry to practical purposes such as manuf and analyses of glass, ceramics, pigments, metals, expls, plastics, etc

Following are some refs on these subjects: 1)J.R.Partington, "Origins and Development of Applied Chemistry," Longmans-Green, London(1935) 2)M.D.Curwen, Edit, "Chemistry in Commerce," G.Newnes, London, 4 vols (ca 1937) 3)J.F.Thorpe et al, "Thorpe's Dictionary of Applied Chemistry," Longmans-Green, London, 4th edn; vols 1 to 11(1937 to 1954); vol 12(General Index) (1956) 4)S.R. Wilson & M.R.Mullins, "Applied Chemistry," Holt,NY(1947) 5)R.E.Kirk & D.F.Othmer, eds, "Encyclopedia of Chemical Technology," Interscience,NY, Vols 1 to 15(1947 to 1956); 1st Supplement(1957) 6)M.Giua & C.Giua-Lollini, "Dizionario di Chimica," UTET, Torino, Vols 1–3(1948–50) 7)K.Winnacker & E.Weingaertner, "Chemische Technologie," C.Hanser, München, Vols 1 to 5(1950 to 1954) 8)E.S.Gyngell, "Applied Chemistry for Engineers," StMartins,NY(1951) 9)F.Ullmann,

"Enzyklopädie der Technischen Chemie," Urban & Schwarzenberg, Berlin, 3rd edn, Vols 1 to 10(1951 to 1958) 10)C.K.Tinkler & H.Masters, "Applied Chemistry," Lockwood, London(1953) 11)W.F.Willebrand & C.E.F. Lundell, "Applied Inorganic Analysis," Wiley,NY(1953) 12)Coll, "Reports on Progress of Applied Chemistry," Yearly publications by the Society of Chemical Industry, London; the last rept printed is Vol 43 of 1958 (See also Refs under Chemical Engineering and under Industrial Chemistry)

Appoint(Fr). See Increment

APQ. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 419

Aprotic Solvents are those which have no tendency either to lose or to gain a proton. To these belong the hydrocarbons and their halogen derivs, such as benzene, pentane, toluene, chloroform, carbon tetrachloride, chlorobenzene, etc. Because of the inert nature of these solvents, no dissociation or other reaction can take place when a single acid or base is dissolved

Ref: P.B.Bell, "Acids and Bases," Methuen, London(1952),36 & 81

Aptitude à la Déflagration(Fr). Sensitivity to Deflagration

Aptitude à la Déflagration par Choc Mécanique(Fr). Sensitivity to Deflagration by Impact(See under Impact Sensitivity Tests)

Aptitude à la Détonation(Fr). Sensitivity to Detonation(See under Sensitivity to Initiation by Detonators and Boosters)

Aptitude à l' Inflammation, Épreuve(Fr). Sensitivity to Inflammation, Test. This French test, also called *épreuve de combustion; combustion en gouttière* or *combustion en tas conique*, is described here under Flame Tests

APU. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 421

APX Explosives. A series of experimental expls developed and tested in the US during WW II, such as **APX-5A**, which contained EDNA 87, wax 5 & Al 8%

Ref: All & EnExpls(1946),144

Note: There was also **APX-4A** which contained Comp A 92 & Al 8%

Apyrite. A single-base smokeless propellant invented at the end of the last century by Skoglund of Sweden and manufd for many years by the Grakrut Co. It was used by the Swedish Navy

Ref: Daniel(1902),29

AQG;AQL;AQN;AQO. Cast double-base propellants described in conf "Propellant Manual," SPIA/M2(1959),Unit Nos 422,423,425 & 426

Aquadag. A colloidal suspension of graphite in w used as a lubricant. The Acheson Graphite Co manuf it by grinding graphite and tannin in w and then adding dil ammonia. If a mineral oil is used instead of ammonia, the resulting product is called **Oildag**

Refs: 1)Hackh(1944),70 2)Kirk & Othmer 4 (1949),226

Aquametry is a generic term for detn of water content in various substances. Following is a partial list of methods used in aquametry: a)Oven drying method b)Karl Fischer reagent methods c)Distillation methods, which, in general, depend on a heterogeneous azeotropic distn with hydrocarbons(such as benzene) or org halides(such as CCl_4) in which the water separating as a phase in the distillate is measured volumetrically d)Absorption methods are usually based on the evoln of w at elevated temps into a stream of inert gas by which it is carried into a tared tower contg an active desiccant, such as Dehydrite[anhyd $Mg(ClO_4)_2$], Drierite (anhyd $CaSO_4$), $CaCl_2$, P_2O_5, etc. e)Evaporation method in which w remains as a residue(see under Ammonia, Analytical Procedures) f)Specific gravity method, such as used in estimating the strength of alcohols, some acids, etc. g)Refractive index method h)Viscosity method i)Electric conductivity method j)Dew point test k)Cloud point test l)Heat of dilution test(such as used in detn of sulfuric acid strength) m)Boiling point test n)Polarimetric tests o)Calcium carbide test, based on measuring the vol of

acetylene evolved on contact of CaC_2 with w (See also determinations of moisture under individual substances)
Ref: J.Mitchell, Jr & D.M.Smith, "Aquametry." Application of the Karl Fischer Reagent to Quantitative Analysis Involving Water," Interscience, NY(1948)

Aqua Regia or Aqua Regis(Nitrohydrochloric or Nitromuriatic Acid) (L'eau régale, in Fr; Königwasser or Goldscheidenwasser, in Ger; Tsarskaya Vodka, in Rus; Aqua Regia, in Span; Acqua Regia, in Ital). The name aqua regia(or regis) was given to a liq dissolving gold. It was prepd by dissolving sal ammoniac(NH_4Cl) in strong nitric acid. E. Davy prepd in 1831 a similar solvent by mixing HCl with HNO_3. It is now usually prepd by mixing 1 vol of concd HNO_3 with 3–4 vols of concd HCl. The liq is at first colorless, but it gradually becomes orange-yel due to the formation of nitrosyl chloride and free chlorine:
$HNO_3+3HCl \longrightarrow Cl_2+2H_2O+NOCl$(nitrosylchloride)

This reaction is believed to proceed in two steps: $HNO_3+HCl \longrightarrow H_2O+NO_2Cl$(nitrochloride) and $NO_2Cl+2HCl \longrightarrow Cl_2+H_2O+NOCl$

Aqua regia dissolves not only gold,but also other noble metals(such as platinum) as well as sulfur

It is not advisable to bottle and store aqua regia(Ref 6)
Refs: 1)W.C.Moore,JACS **33**, 1091(1911) & **35**,333(1913) 2)Gmelin,Syst Nr 6(Chlorine) (1927),439–41(Königwasser) 3)Mellor **8** (1928),618 4)Thorpe **1**(1937),453 5)Kirk & Othmer **2**(1948),108–9 6)H.Faucett, C & EN **33**,897(1955) & CA **49**,5843(1955)

ARA. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2 (1959), Unit No 429

Arabic, Gum. See under Gums

ARABINOSE AND DERIVATIVES

Arabinoses, $C_4H_5 \cdot CHO(OH)_4$, mw 150.13. Colorless monosaccharides of which *α*-arabinose or L-arabinose is the common form; rhombic crysts, mp 159.5°, d 1.585 at 20/4°. It is usually obtained from plant gums by hydrolysis with mineral acids. There is also D-arabinose which is produced by the degradation of D-glucose
Refs: 1)Beil **1**,859,(435) & [900] 2)Kirk & Othmer **2**(1948),869

Arabinose, Azido-, $C_5H_9N_3O_5$ and *Diazido-*, $C_5H_8N_6O_5$ *Derivatives* were not found in Beil or CA through 1956

Arabinose, Mononitrate, $C_5H_9NO_7$, *Dinitrate*, $C_5H_8N_2O_9$ and *Trinitrate*, $C_5H_7N_3O_{11}$ – were not found in Beil or CA through 1956

Arabinose Tetranitrate, $C_4H_5 \cdot CHO(ONO_2)_4$, mw 330.13, N 16.97%, OB to CO_2 ± 0%, OB to CO +24.2%. Col monocl crysts; mp 85°, dec ca 120° and expl at higher temps; sol in acet, alc & AcOH; insol in w & ligroin. Was prepd by nitrating D-arabinose as described in detail by Will & Lenze(Ref 2). It explodes also by shock; not stable in storage even at temps as low as 50°
Refs: 1)Beil **1**,863 2)W.Will & F.Lenze, Ber **31**,72(1898) 3)Davis(1943),240
Note: No refs were found in CA through 1956

ARABITOL AND DERIVATIVES

Arabitol(Arabit in Ger), $C_5H_7(OH)_5$, mw 152.15, exists in several forms, of which D-arabitol is most common; col prismatic crysts, mp 103°; can be prepd by reduction of D-lyxose or D-arabinose
Refs: 1)Beil **1**,531,(281) & [604] 2)O.Ruff, Ber **32**,555(1899) & **33**,1802(1900)

Arabitol, Azido, $C_5H_{11}N_3O_5$ and *Diazido*, $C_5H_{10}N_6O_5$ *Derivatives* were not found in Beil or CA through 1956

Arabitol, Mononitrate-, $C_5H_{11}NO_7$, *Dinitrate*, $C_5H_{10}N_2O_9$, *Trinitrate-*, $C_5H_9N_3O_{11}$ and *Tetranitrate*, $C_5H_8N_4O_{13}$ – were not found in Beil or CA through 1956

Arabitol Pentanitrate, $C_5H_7(ONO_2)_5$, mw 377.15, N 18.57%, OB to CO_2 +6.36%, OB to CO +27.5%. Wh syrupy mass; sol in alc, eth & acet. Can be prepd by nitrating D-arabitol with fuming nitric acid at –5°, followed by addn of concd sulfuric acid. Its expl props were not investigated

Refs: 1)Beil **1**,531 2)L.Vignon & F.Gerin, CR **133**,641(1901) & JCS **82** I,9–10(1902)
Note: No refs were found in CA through 1956

Araldite is an epoxy resin bonding agent, made by Bond Master Rubber & Adhesive Co, Irvington, NJ, used in some US elec detonators, such as the T44. In this item, the pin and the inside of the plug are coated with Formvar for insulation and then bonded together with Araldite
Refs: 1)P.B.Tweed,Ordnance **44**,654(1960) 2)P.B.Tweed, PicArsn; private communication(1960)

Aranaz, Ricardo. A Spanish general specializing in Ordnance, who died in 1932. In 1906 he introduced in Spain the modern progressive smokeless propellants and did considerable work on their improvement. He also did some work on HE's, such as tetryl, and was the author of several publications on expls and propellants
Refs: 1)Pérez Ara(1945),426 & 429 2)Vivas, Feigenspan & Ladreda, vol 3 (1948), 86(footnote) & 212

ARB. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 430

Arbalest or Crossbow. A weapon, invented in the 4th century, consisted of a stock (arbier) and a short powerful bow fixed transversely near the end of the stock facing the target. The stock contained a groove to guide the missile(such as an arrow, stone, dart or bullet), a notch to hold the string of the bow, and a trigger to release it. The end of the stock opposite the bow was placed against the shoulder, gun fashion, and the string was released by trigger

This weapon was used successfully for over 10 centuries(including Norman invasion of England in the 11th century and battle of Crécy in the 14th century) and even competed for some time with early firearms, which were not as efficient as arbalests of the 14th and 15th centuries
Refs: 1)Webster's Unabridged Dictionary (1951),137 2)Encyclopedia Britannica, vol 6(1952),755(Crossbow) 3)Collier's Encyclopedia, vol 2(1957), under Archery

Archery. The term derived from the Latin "arcus"(a bow), covers the equipment and procedures of shooting with all types of bows and arrow for war, hunting and sport. History of development and a comprehensive survey of various types of bows and arrows are given in Collier's Encyclopedia, NY, vol 2(1957) 135–41 (See also Arbalest)

Arcites are rocket propellants developed recently in the US by Atlantic Research Corp, Alexandria, Virginia. They usually contain oxidizers(such as AN or NH_4ClO_4), binders (such as polyvinyl chlorides) and plasticizers(such as dibutyl sebacate)
Ref: Warren(1958),11

Arcites 251,309,358,362 & 368 are described in conf "Propellant Manual," SPIA/M2(1959), Unit Nos 460,462,547,546 & 524

Ardeer Cordite. A solventless cordite developed in 1919 at the Ardeer plant of Nobel Co: NC(12.2%N) 50, NG 42 & phenylbenzylurethane 8%. Its stability did not meet Brit military requirements, mainly on account of acid products which developed in the presence of moisture
Ref: J.N.Pring,"Modern Propellants Employed in British Ordnance," Paper read at a Meeting of the Chemical Engineering Group, London, SWI, May 4, 1948, p 5

Ardeer Plant of Nobel's Explosives Co, Ltd is located at Stevenston, Scotland

Ardeer Powder. An older Brit "permitted" expl: NG 31–34, kieselguhr 11–14, Mg sulfate 47–51, K nitrate 4–6 Na carbonate 0.5 & Amm carbonate 0.5%
Refs: 1)Daniel(1902),30 2)CondChemDict (1942),287(not listed in newer editions)

Argarit. A Swiss expl contg PETN. Its compn is based on Stettbacher's patents which are now expired
Ref: Dr A.Stettbacher, Zürich; private communication, June 25, 1958

Argent fulminant de Berthollet(Fr). Fulminating Silver of Berthollet

ARGENTINE ARMAMENT. Argentina is capable of producing all explosives required for

their industrial and military purposes, as well as for most of their ammunition and small arms. It still depends on foreign countries (such as Sweden, France, GtBritain, Italy and USA) for cannons, howitzers, mortars, some machine guns and rockets

According to Capitán de Navío López (see Ref), most of their small arms come from foreign countries; a great variety of them exist, but it is expected that some standardization will be achieved when all (or most) of the arms will be manufd in Argentina. One of the first small arms manufd in Argentina was the pistol Colt C/45. The production of weapons was gradually extended to the manuf of rifles and machine-guns. Larger caliber weapons up to 12.7 mm and even 20 mm and 40 mm are also produced but on a small scale. Most ammo and some rockets (such as 57 mm) are also manufd

The current armament of the Argentine *Army* includes:

a)7.65 mm rifles and carbines (such as the Mauser, previously manufd in Germany & Spain, but now to a limited extent in Argentina)

b)7.65 & 12.7 mm machine guns

c)20 & 40 mm automatic guns

d)80 mm antiaircraft gun

e)105 mm howitzer and

f)75,105 and 155 mm cannons

The current armament of the Argentine *Navy* includes:

a)20 and 40 mm automatic guns

b)75,80,120,127.5,150 & 190 mm cannons

The following explosives and propellants are used by the Argentine Army and Navy:

a)TNT (Trotil) is used as a HE filling for most shells, bombs, grenades, mines, torpedoes & rockets. Some of the shells imported from the US are filled with ammonium picrate. The possibility of using PETN (Pentril) and RDX (Hexogeno) has also been investigated

b)Tetryl is used for boosters

c)Mercuric fulminate (fulminato de mercurio) and lead azide (nitruro de plomo) are used in primers

d)Mercuric fulminate and lead styphnate (trinitroresorcinato) are used in detonators

e)Single-base propellants (NC, DNT & DPhA), multiperforated, are used in various cannons

f)Single-base propellants (NC with DPhA), single perforated, are used in rifles, pistols and machine guns

g)Double-base propellants (NC, NG & centralite), tubular solventless and tubular using acet as a solvent, are used in various cannons

h)Triple-base propellants (NC, NG & NGu), laminated, are used in mortars

i)Propellants consisting of NC, NG, DNT & TNT, are used in rockets

Following is a list of Argentine plants manufg explosives, ammunition and weapons:

A. Government Owned Plants

a)Fábrica Militar de Pólvoras y Explosivos, Villa María, Córdoba (NC, NG, NC propellant, NC-NG rocket propellant, TNT, RDX and dynamites)

b)*Fábrica Naval de Explosivos Azul,* Azul, Provincia Buenos Aires (NC, NG, NC & NC-NG propellants, TNT and dynamites)

Note: This modern plant (1955) may be considered among the best in the Americas. It was constructed by Bofors AB(Sweden) and by other European firms. Manuf of TNT is continuous (by the method of Bofors) and so is the manuf of NG (method of Meissner). Stainless steel is used for all apparatus. Its laboratory is well equipped not only for analytical work, but also for research. The sr author (BTF) of this book had the priviledge of visiting this plant in 1955

c)Fábrica Militar José de la Quintana, Córdoba (Ball powder for small caliber weapons, PETN, RDX, TNT, LA and LSt)

Note: Ball powder is called in Argentine "pólvora W", where "W" stands for Western Powder Co

d)Fábrica Material Pirotecnico, Pilas, Pcio Buenos Aires (Primacord, fuses, detonators MF, LA, LSt and various pyrotechnic mixtures and items)

e)Fábrica Militar de Municiones, San Lorenzo, Santa Fe (MF and ammo for small arms)

f)Fábrica Militar de Municiones, San Francisco, Córdoba (Ammo for small arms)

g)Fábrica Militar, Río Tercero, Córdoba (Artillery ammo)

h)Fábrica Militar de Armas Portatiles, Rosario, Santa Fe (Pistols, rifles and machine guns)

B. *Privately Owned Plants*

1)DESA, Sierras Bayas, Pcia Bs Aires (NG, RDX & dynamites)

2)Delbene y Serris, Olavarría, Pcia Bs Aires (Black powder & chlorate expls)

3)FADEX, San Vicente, Pcia Bs Aires (NG & dynamites)

4)FAPOL, Tandil, Pcia Bs Aires (Black powder)

5)FOTI, Rafaela, Santa Fe (Black powder)

6)LG-PCM, Haedo, Pcia Bs Aires (Various pyrotechnic compns & items)

7)COASA, Florencio Varela, Pcia Bs Aires (MF, LSt and ammo for pistols, rifles & shotguns)

8)Scorzatto Hnos y Cía, Luján, Pcia Bs Aires (Ammo for shotguns)

9)Spreafico SAIC, Florencio Varela, Pcia Bs Aires (MF, detonators for hand grenades, signal cartridges and ammo for shotguns)

10)La Bengala, Moreno, Pcia Bs Aires (Pyrotechnic items)

11)Imaz y Ryser, Miramar, Córdoba (MF, initiating compns and percussion caps)

12)Armotor SA, Bs Aires (Pistols)

13)Belenda, Scapusio y Cía, SRL (Machine guns 7.65 & 12.7 mm, hand grenades and pyrotechnic pistols)

14)CATI-TA, Bs Aires (Shells for 120 & 150 mm cannons)

15)Dillon Ernesto Pablo, San Martín, Pcia Bs Aires (Revolvers)

16)DeBoer y Barbieri SRL, Bs Aires (Shotguns)

17)Establecimento "Klockner" SAIC, Bs Aires (Rifles, rocket launchers and shell bodies)

18)Establecimento Metalurgico "Guerrino Venturini," San Isidro, Pcia Bs Aires (Revolvers)

19)Fábrica de Armas "Centauro", Lanús, Pcia Bs Aires (Automatic carbines and compressed air rifles & pistols)

20)Fábrica de Armas "Mabely", Lanús, Bs Aires (Same as above)

21)Fábrica de Armas "Halcon", SRL, Avellaneda, Pcia Bs Aires (Machine pistols, automatic rifles & carbins and shotguns)

22)FADA, Chascomús, Pcia Bs Aires (Shotguns)

23)FAPESA, Bs Aires (Fuzes)

24)Garb Monetti y Cía, Mar del Plata, Pcia Bs Aires (Automatic pistols)

25)GUK Metal SRL, Haedo, Pcia Bs Aires (Signal pistols and launching equipment)

26)HAFDASA, Bs Aires (Carbines, pistols and automatic pistols)

27)Infantino Hnos y Cía, San Antonio de Padua, Pcia Bs Aires (Pistols)

28)Indurgica Argentina SRL, Ramos Mejía, Pcia Bs Aires (AA gun carriages and gun sights)

29)Industrias "Marcati", Avellaneda, Pcia Bs Aires (Compressed air and automatic rifles)

30)Krahmer, Pfeffer y Cía, Bs Aires (Compressed air rifles and Cal .22 pistols)

31)Lambda SRL, Bs Aires (Semi-automatic carbines)

32)Luan, Comercial e Industrial, Bs Aires (Machine pistols and pistols Cal .22)

33)Martin Bass y Cía, Bs Aires (Pistols)

34)Metalurgica Industrial Argentina, Bs Aires

(Carbines & pistols syst Mauser and shotguns)

35)Metalurgica "Jaguar" SRL, Bs Aires (Rifles and shotguns)

36)OTME SRL, Córdoba (Machine pistols, semi-automatic carbines, shotguns and other items)

37)Pasper SRL, Avellaneda, Pcia Bs Aires (Shotguns and compressed air rifles)

38)Saboy e Hijo, Alfredo V., Lanús, Pcia Bs Aires (Rifles)

39)Silva Antonio María, Bs Aires (Pistols)

40)Sole Nelson G., San Francisco, Córdoba (Shotguns)

41)TALA SRL, Bs Aires (Pistols)

42)Televel SA, Bs Aires (Shotguns)

Abbreviations: Bs Aires – Buenos Aires; **CATI-TA** – Compañía Argentina de Talleres Industriales, Transportes y Annexos; **Cía** – Compania; **COASA** – Cartuchería Orbea Argentina Sociedad Anónima; **DESA** – Dieterle Explosivos Sociedad Anónima; **FADA** – Fábrica Argentina de Armas; **FADEX** – Fábrica Argentina de Explosivos; **FAPESA** – Fábrica Argentina de Productos Eléctricos Sociedad Anónima; **FAPOL** – Fábrica Argentina de Pólvoras; **FOTI** – trade name for Manufactura Argentina de Pólvoras; **HAFDASA** – Hispano Argentina Fábrica de Armas Sociedad Anónima; **Hnos** – Hermanos (Brothers); **LG-PCM** – Laboratorios Giorgi – Pirotecnia Civil y Militar; **OTMESRL** – Organización Técnica de Mécanica Especializada Sociedad de Responsabilidad Limitada; **Pcia** – Provincia; **SA** – Sociedad Anónima; **SAIC** – Sociedad Anónima Industrial y Comercial; **SRL** – Sociedad de Responsabilidad Limitada; **TALA** – Talleres de Armas Livianas Argentinas

Ref: Capitán de Navio Adolfo E. López, IngEsp (RE), Director de Industrias Químicas, Buenos Aires, RA; private communications 15 July 1959, 4 Sept 1959 and 1 Oct 1959

Argol (Argal or Argil). A grayish or reddish crust deposited in wine casks during fermentation of grape juice. It consists of ca 70% potassium hydrogen tartrate and was used during WWI by the French as a flash reducing agent. It was packed in flat, circular cotton bags (*sachets antilueurs)*, which were assembled along with the smokeless propellant and black powder igniter in silk cartridge bags to make up a complete propellant charge. Since the antiflash material tended to reduce the ballistic effect of the chge, it was necessary to add an additional quantity *(appoint)* of smokeless propellant. Thus, for ordinary firing of the 155 mm gun, the chge consisted of 10 kg of *poudre BM7* along with an igniter system contg a total of 115 g of black powder. For a flashless round, 3 bags each contg 500 g of argol were used with an additional 305 g of smokeless propellant to restore the ballistics to normal

Refs: 1)Davis (1943), 325-6 2)Hackh(1944), 73

Argonit. A plastic HE manufd at Dottikon, Switzerland: PETN 70-80 & liq waste TNT 30-20%

Ref: Dr A. Stettbacher, Zürich; private communication, 23 Aug 1958

Argus. A type of older Brit blasting powder: K nitrate 87-82, charcoal 17-20 & sulfur 0.5-1%

Ref: Daniel (1902), 30

Arkite. An older Brit "permitted" expl, similar to duxite: NG 31-3, CC 0.5-1, K nitrate 26-8, WM 8-10, & Amm oxalate 29-31%. An expln of arkite during its manuf is described in Ref 1

Refs: 1)A.P.Desborough, HMInspExplos SpecRept **193** (1910) & CA **4**, 2879(1910) 2)Marshall **1** (1917), 374 3)Barnett(1919), 136

Arlberg Dynamite. An older dynamite prepd by mixing 65 parts of NG with 35 ps of absorbent, a mixt of kieselguhr, Ba nitrate & charcoal

Ref: Cundill, MP **5**, 288(1892)

Arma (Ital). Arm or weapon

Arma bianca (Baionetta) (Ital). White arm (bayonet)

Armada (Span). Fleet, Navy

Arma da fuoco (Ital). Firearm; gun

Armament is the aggregate of a nation's military strength, which includes all the items used by the Army, Navy and Air Force (ships, aircraft weapons, ammunition, expls, tanks, transportation, etc), as well as the personnel to man them. It also includes all military installations (fortifications, barracks, ammo magazines, etc) and all industries working for war purposes

Ref: Webster's Unabbridged Dictionary(1951), 149

Note: In connection with armored vehicles such as tanks, the word **armament** indicates the protective plate (see also Armor)

Ref: A.B.Schilling, Pic Arsn; private communication (1960)

Armament Engineering. This subject is discussed in the following book:
Anon, "Elements of Armament Engineering", US Military Academy, West Point, NY (1954) (722 pp) (It contains the following parts: a) military expls, b)fundamentals of ballistics, c)ammunition and weapons, including atomic weapons & guided missiles and d)a brief glossary of armament engineering terms)

Armament Research Establishment (ARE) is a Brit military institution located at Halstead, Kent, England

Armament Research Establishment, Canada (AREC) is located at Valcartier, Québec

Armament Research Establishment, Royal Arsenal (ARERA) is located at Woolwich, England
(See also Woolwich Research Department)

Armamento (Ital & Span). Arming

Arme (Fr). Arm; rifle

Armé (Fr). Cocked; armed

Arme à chargement par la culasse (Fr). Breech-loading weapon

Armed Forces (Wehrmacht in Ger; Armée, in Fr). This term includes in the US: Army, Navy & Air Forces; in Germany Heere, Kriegsmarine & Luftwaffe and in France: Armée de Terre, Armée de Mer and Armée de l'Air

Armée (Fr). Armed Forces; Army

Armée de l'Air (Fr). Air Force

Armée de Mer (Fr). Navy; Fleet (lit Sea Force)

Armée de Teree (Fr). Army (lit Land Force)

Armée Mécanique (Fr). Tank Corps (lit Mechanized Army)

Arme à feu (Fr). Firearm

Arme à feu automatique (Fr). Automatic firearm

Arme portative (Fr). Small Arm (lit portable arm)

Armeria (Ital). Armory

Arming. This term applies to fitting or equipping an item to be ready for action. Arming corresponds in a rough way to cocking in a small arm. A fuze may be armed (set for detonation) by utilizing forces exerted within or outside the gun. Various methods of fuze and booster arming are described by Ohart (1946), 127 & 166

Armor is a covering intended to protect a person, ship, tank, aircraft combat vehicle, etc from the destructive effects of various types of missiles. For individual protection of a person a flexible fabric of interlinked metal rings (called mail) is commonly used. For protection of other items, the most common material is steel, but research indicates the possibility of aluminum,titanium and even of some non-metallic material

A comprehensive description of various

types of armor is given in Refs 1,2,3, especially in Ref 2

Refs: 1)Encyclopedia Britannia **2** (1952), 392-4 2)Armament Engineering (1954), 227-49 (Armor protection against ballistic attacks) 3)Collier's Encyclopedia **2** (1957), 259-68

Armored Car (Military) may be defined as a wheeled motor-vehicle (such as truck, jeep, station wagon), protected by a light armor and provided with one or more machine-guns, rocket launchers, hand & rifle grenades and, in some cases, with light guns (such as 20 mm or 37 mm). The first armored motor car was designed in 1898 by R.P.Davidson, Colonel in the Illinois National Guard (Ref 5). This car was actually semi-armored (Ref 1). Soon after this, armored cars were built in England (the Pennington) and in France (the Charron). Italians employed a number of armored cars in their campaigns in Africa (1913) (Ref 5). Russians used some armored cars (as well as some armored trains) in WWI, Russian Civil War (1918-21) and WWII. Some idea about German armored cars of WWII may be obtained from Refs 4,6 & 7

US armored cars have been primarily employed for reconnaissance, although in some cases, they were used against tanks. Newman (Ref 2) gives photographs of US armored scout car with AA machine-gun mounts and of US armored half-track scout cars. Barnes (Ref 3) gives photographs of US armored car M8 (used during WWII in all theaters) and of US armored car M38, 6 × 6

Historical development of armored cars is described in Refs 1 & 5

Refs: 1)R.J.Icks Army Ordn **17**, 331-3 (1937) (Four Decades of Mechanization; Our Record of Combat-Vehicle Development) 2) J.R.Newman, "The Tools of War," Doubleday, Doran & Co, NY (1943), 198-9 3)G.M.Barnes, "Weapons of World War II," Van Nostrand, NY (1947), 286-7 4)G.B.Jarrett, "Achtung Panzer," Great Oaks, Aberdeen, Md(1948) 5)Encycl Britannica **2** (1952), 388 6) D.F. von Senger u Etterlin, "Taschenbuch der Panzer," Lehmann, München (1954) 7) B.T.Fedoroff et al, PATR 2510 (1958), 123-6

Armored Ships. The necessity of protecting ships by some kind of armor was realized after the introduction of HE shells in sea warfare (ca 1849), and introduction of AP shells at a later date. The first ship protected by armor was "La Gloire" the French wooden ship, dressed in 1859 in iron plates. Two years later, the British launched the 9000-ton Warrior with 4½" thick iron plates all around and 6 ft below the waterline

The advantage of armor was shown during the American Civil War in the battle (1862) between Merrimac (South) and Monitor (North). Both ships withstood numerous hits by HE shells and were not incapacitated

From that time on, the warships of all nations were protected by armor. A more perfect protection against artillery was achieved in the 20th century when the construction of wooden ships was abandoned in favor of steel ships

Ref: J.R.Newman, "The Tools of War," Doubleday, Doran & Co, NY (1943), 204-67

Armored Train is one in which locomotive and cars are protected by a light armor capable of withstanding the impact of bullets. Such trains can carry artillery (artillery train) or troops provided with weapons, such as small arms, rockets, etc. In case of cars carrying artillery, each gun can be provided with a shield

Before the appearance of motor cars and construction of good highways, armored trains were considered very useful. They were employed successfully in American Civil War (1861-5), Franco-Prussian War (1870-1) and South-African War (1899-1900) (Ref 1). In Russia, where the roads are generally in a poor condition, armored trains were used in WWI and in Russian Civil War (1918-20). Some of the Russian "armored" trains were makeshift affairs consisting of an ordinary locomotive protected by sandbags and several flat cars with sandsbags laid along the sides (Ref 2)

(See also Artillery Train)

Refs: 1)Encyclopedia Britannica **2** (1952), 388 2)M.M.Kostevitch, Buenos Aires, Argentina; private communication (1955)

ARMOR-PIERCING (AP) PROJECTILE

(Panzergeschoss, in Ger; Projectile perforant in Fr; Proietto perforante, in Ital; Proyectil perforante, in Span; Broneprobivayoushchii snariad, in Rus). AP projectiles are designed to penetrate the armor plate of ships, tanks, etc and may be divided into the following types:

A)A solid projectile contg no explosive, such as *AP Bullet*, which usually contains a core of hardened steel, a gilding metal jacket and a base (Ref 5, p 185; Ref 6, p 76 & Ref 12, p 76) and *AP Shot*, called in Ger *Panzergranate*, which usually consists of a solid steel cylindrical block pointed at the nose, provided with a rotating band and tracer. AP shots are used now mostly for target practice and are provided with tracers (Ref 6, pp 108-9; Ref 10, p 8 & Ref 14)

There is also the so-called HVAP (hypervelocity armor-piercing) shot which consists of a tungsten carbide core surrounded by a steel body and provided with a cone-shaped steel nose and a pointed windshield (See below) (Ref 6, pp 108 & 111; Ref 10, p 8)

B)A hollow projectile (shell) contg a HE charge and a base-detonating fuze are designed to penetrate the armor (or concrete) without exploding until they are inside the target. They are usually made of high-carbon, heat-treated alloy steel (in contrast with the common forged steel of the HE shell) and are fashioned with an extremely hard nose and a relatively soft, tough body with thick walls, especially in the forward section

AP shell intended to penetrate face-hardened armor is usually provided with a slightly blunted nose which is fitted with a cone-shaped cap made of soft and tough forged alloy steel, face hardened. Such shell is called **APC** (armor-piercing capped). The cap of this shell is fitted with a conical cup, called *windshield or ballistic cap*, (also called *false ogive)* the purpose of which is to improve streamlining of projectile as it speeds toward the target. Windshield, usually made of thin Al, shatters on impact with armor, leaving the projectile with only its steel capped nose. The tough steel nose strikes into the armor and weakens it. The projectile then starts to penetrate the softened-up armor spot, while the nose cap breaks away from the remaining part of the projectile. This remaining portion then penetrates the armor and explodes inside the target by the action of base fuze (Ref 6, p 107-9)

It is important that the HE charge be so insensitive that it will not be exploded by the tremendous shock caused by the impact of the shell on the armor. However, it must be sensitive enough to be detonated by the action of the fuze. Black powder fulfills this condition and was used until the end of the last century. It was replaced later in some countries by some AN expls, such as ammonals. These expls were more powerful than black powder, but inferior to ammonium picrate (Expl D) and guanidine picrate (Gu P) commonly used now

An interesting AP shell was patented during WWI by Quartieri & E.Molinari (Ref 1). In order to render such shell insensitive to shock, the forward part was partly filled with HE (such as TNT or PA) desensitized by 1 to 10% camphor or paraffin. This served as a cushion to take up the stress of impact. A thin felt was placed over the chge and the middle and base part of the shells were filled with a straight HE, such as TNT. Then the booster and the base fuze were inserted. A schematic view of such shell is given in CA **10**, 694(1916)

An ingenious complex filling for AP and A/C shells was used by the German during WWII. It had in the forward section an insensitive charge (KCl, followed by KCl/wax/TNT), in the middle section a moderately sensitive chge of TNT/wax in different proportions and in the base section a fairly sensitive chge of straight TNT (Ref 12, p Ger 48)

Because of the thickness of the projectile

walls, the bursting chge of AP and APC projectiles is comparatively small, representing only 5 to 25% of the total wt of the shell

The so-called **API** (armor-piercing incendiary) projectile contains an incendiary mixture and the **API-T** (armor-piercing incendiary tracer) is similar to API but has a tracer in the rear of the bullet (Ref 6, p 65)

Various **AP** and **SAP** (semi armor-piercing) bombs are described by Ohart (Ref 6, pp 232-4)

C)A projectile designed to utilize the principle of the Munroe-Neumann Effect (qv) is called *Shaped Charge Projectile* **(SCP)** *or Hollow Charge Projectile*. They are similar in appearance to conventional projectiles, except that the forward part of the bursting chge is cast with an indentation in the shape of a cone. The cone surface is usually provided with a metallic liner which increases the penetrating effect (Ref 6, pp 41-3). These projectiles function immediately on impact with armor or concrete, making a relatively small hole through it and hurling hot fragments at a very high velocity inside the target. These fragments are very effective as A/P (antipersound) missiles especially in small enclosures, such as the interior of tanks, pill-boxes, etc. In addition to the damage caused by the flying fragments, a greater damage is probably caused by the blast effect of the chge and by the high temperature (ca 2000°C) and suffocating effect of the gases developed on expln. Combinations of these effects are usually 100% fatal whereas the fragments alone are not

These projectiles are filled with cast HE, such as cyclotol, pentolite, TNT and are provided with a base fuze and a cap designed to provide the necessary stand-off required for proper formation of the jet

As most of the US recent shaped charge projectiles are classified, the reference here is made to unclassified WWII items described by Ohart (Ref 6), such as: bombs (p 240), HEAT (high-explosive antitank) shells (pp 108, 110, 112 & 138), HEAT rockets such as 4.5-in (p 347). A greater variety of shaped charge ammunition was used during WW II by the Germans, as can be seen from the following examples: Faustpatrone (shaped charge A/T rocket) (p 46); 75 mm SC shells (pp 74 & 76); 88 mm, 100 mm & 105 mm SC shells (p 77); Hafthohlladung (adhering shaped charge) used for destroying tanks (p 85); shaped charge handgrenade (p 86); Panzerhandmine and Haftmine (shaped charge adhering mines) used for destroying tanks (p 87); shaped charge bombs DS (p 92); 105 mm & 75 mm SC projectiles (p 92); Panzerfaust & Panzergranate (shaped charge A/T rocket grenades) (p 126); Panzerschreck (shaped charge A/T rocket) (p 127); Panzerwurfmine (shaped charge A/T hand grenade) (p 127) shaped charge pistol grenades (p 133); shaped charge rifle grenades (p 152); and shaped charge rockets (pp 161 & 168)

Refs: 1)F.Quartieri & E.Molinari, BritP 19547 (1914) & CA **10**, 694(1916) (An AP shell insensitive to shock) 2)L.Gabeaud, MAF **14**, 85-92(1935) (Essai sur la théorie de la perforation des blindages) 3)Ibid, **14**, 399-414(1935) (Sur la loi de variation de la poussée dynamique dans la perforation des blindages) 4)P.Regnault, MAF **14**, 379-97 (1935) (Note sur la pénétration des projectiles et la perforation des blindages) 5)Anon, "Ammunition Inspection Guide", War Department Technical Manual, TM9-1904(1944), 16,179 & 185 6)T.C.Ohart "Elements of Ammunition," Wiley, NY (1946) (see Index p 400) 7)F.Gleason, Ordnance, **31**, 368-9 (1947) (A glossary of Ordnance terms) 8) L.Gabeaud, MAF **21**, 97-112(1947) (Sur la perforation des blindages) 9)F.Primus, MAF **22**, 971-80 (1948) (Perforation des blindages) (Translated from Polish) 10)Anon, "Artillery Ammunition," Dept of the Army Tech Manual TM9-1901(1950), 8 & 14 11)Armament Engineering (1954), pp 199-225 (Defeat of armor and concrete); 254(AP bullets); 283 & 286 (AP & APC shells); 285 (HEAT shell); 287 (HVAP shot); 308 (3.5-in HEAT rocket,

M28); 343 (HEAT, T41 rifle grenade, Energa) 12)Anon, Ammunition General," TM9-1900(1956), 76 13)B.T.Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons" (German Section), PATR 2510 (1958) 14)A.B.Schilling, PicArsn; private communication (1960)

Armor Testing. The purpose of testing is to det the effect produced on armor (such as plates, castings, weldments, etc) on impact of various projectiles, such as HE, AP, APC, HEAT, HVAT, etc. The tests are described in Ordnance Proof Manual No. 22-10 (1950)

Note: Discussion on evaluation of weld-cracking tests on armor steel was given by S.Weiss et al, Welding J (NY), **35**, 348_s–56_s (1956) (20 refs) & CA **50**, 13705-6 (1956)

Armour Research Foundation (ARF), of the Illinois Institute of Technology, Chicago, Illinois, is an organization doing work on explosives, propellants and related items under US Govt contracts. Their reports are listed, when used as refs, under indvdl compds

Armory. This term usually means a Government establishment where arms and other military items are stored for distribution to troops. Some US armories (such as Springfield Armory, Springfield Mass) are also engaged in manuf arms and other items, while others serve as a drilling place for troops

Ref: Webster's Unabridged Dictionary (1951), 151

Arms. See under Ammunition and Weapons

Armstrong Airbreaker is a blasting device activated by compressed air, used for breaking down coal in fiery mines. For its description see J.Taylor & P.F.Gay, "British Coal Mining," Newnes, London (1958), 137 & 140-2

Armstrong Gun. A rifled gun, constructed according to the system of built-up, wrought-iron ordnance, invented ca 1855 by Sir W.G. Armstrong of England

Ref: Webster's Unabridged Dictionary (1951), 151

Armstrong's Explosive or Mixture is a red solid substance prepd by blending under a volatile liq (such as alc or acet) 75 parts of powdered K chlorate with 25 ps of red phosphorus and then allowing the volatile liq to evaporate. The mixt is very explosive and sensitive. Extreme care must be exercized in handling it, because it may detonate on a slight shock, touch or when brought into contact with a drop of concd H_2SO_4. The mixt is known for over 100 years and it has caused many mishaps

Germans used this mixt for loading the so-called *"Hinterhaltsminen"* the land mines left by them on retreat

Stettbacher calcd the heat generated by the reaction: $5KClO_3 + 6P_{(red)} \rightarrow 3P_2O_5 + 5KCl$ as equal to 1417 kcal/kg

Ref: A. Stettbacher, Protar **10**, 159-60 (1944)

Army (Heere or Armee, in Ger; Armée de terre, in Fr; Esercito, in Ital; Ejército, in Span; Armiya, in Rus). A large organized body of men, armed for war and designed for land service. It is a part of the Armed Forces (qv)

According to the definition given in ORDP 50-13, p 84 (Ref 4), the Department of the Army (US) is charged with the responsibility of providing support for national and international policy and the security of the US by planning, directing, and reviewing the military and civil operations of the Army Establishment, to include the organization, training and equipping of land forces of the US for the conduct of prompt and sustained combat operations on land in accordance with plans for national security

Refs: 1)Webster's Unabridged Dictionary (1951), 115 2)Encyclopedia Britannica **2** (1952), 397-416 3)Collier's Encyclopedia **2** (1957), 272-7 4)Ordnance Pamphlet, "Basic Training Course for Training Offices" (1958)

Army Ordnance Corps (US) is an organization which is responsible for procurement, storage

and distribution (to the Army, Navy, Air Force, Marine Corps, National Guard, etc) of small arms, automatic weapons, artillery, fire control equippment, ammunition & explosives, bombs & mines, transport & combat vehicles, rockets & guided missiles, tanks, and other

Up until 1950 it was known as *Army Ordnance Department.* The Dept was created by an Act of Congress, 14 May 1812. Prior to this, in colonial days, the colonists used ammunition and weapons seized from the British either on land or seas; and originally the Quartermasters Corps handled supply problems. Some muskets were produced by locksmiths imported from France and West Indies. Colonial government storage depots were established, but no provisions were made for producing new weapons until 1794 when Congress authorized the establishment of Springfield Armory (constructed in 1794-5) and Harper's Ferry, W Va, (1796). Springfield Armory has been an arms-making center for many years and also served as a military storehouse. The armory at Harper's Ferry was destroyed by Federal troops early in the Civil War so that Confederate troops could not use it

After establishing the Army Ordnance Department, the construction of *several* arsenals (qv) was authorized by the Congress. For testing of weapons and ammunition, the so-called Sandy Hook Proving Ground, New York, was established. This was replaced in 1917 by the Aberdeen Proving Ground, Maryland

During WWI the Ordnance Dept expanded until it had on payroll 6000 officers, 60000 enlisted men and 73000 civilians. After WWI the Dept was rapidly demobilized and munition plants and machinery were converted to peacetime production. The Dept personnel, both military and civilian, was drastically reduced in numbers. It average yearly budget was reduced to ca 10 million dollars

The greatest expansion of the Dept took place just prior and during WWII when an average yearly budget was boosted to ca 7 billion dollars. Total of ca 34 billion dollars was assigned to the Dept during WWII

After WWII the activities of the Dept were greatly reduced, but were again increased when the Korean War started (1950). At this time the Army Ordnance Department was renamed the Army Ordnance Corps

Still greater expansion of Ordnance Corps took place in the last 10 years

Refs: 1)Anon, "Supervisor Development Program," Training Branch, PicArsn, Dover, NJ, Sept 1953 (Revised), 21-4 2)A.B. Schilling, PicArsn; private communication (1960)

Arnoudts' Explosive. A blasting expl contg K chlorate 60 & sugar 40% with added turpentine 2, vegetable tar 2 & K permanganate .00125 parts

Ref: Ch. Arnoudts (of Guatemala), USP 964,365 (1910) & CA **4**, 2733 (1910)

ARO. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2 (1959), Unit No 433

Aroclor. Trade name for a series of polychlorinated polyphenyls manufd by the Monsanto Chem Co of St Louis as liquids, resins or solids. The use of one of the Aroclors as an ingredient of propellants contg no NC is discussed in Ref 1. Other uses of Aroclors are listed in Ref 2

Following are some examples of propellants listed in Ref 1:

	1	2	3
Amm picrate	54.6	71.6	38.5
K nitrate	36.4	17.9	31.5
Et cellulose	3.6	6.0	5.0
Aroclor 1254	5.4	4.0	5.0
Ca stearate	–	0.5	–
Zn stearate	–	–	0.5(added)

Refs: 1)W.E.Campbell et al, Aerojet Engineering Corp, Azuza, Calif, Reports 194 & 199 (1946) 2)Cond Chem Dict (1956), 109

Aromatic-Aliphatic Nitrocompounds and Nitrate Esters. Title of OSRD Report No 176, Nov 1941, by L.F. Fieser

Aromatic Alkylamino Alcohols. Their nitration

in two stages, first with 36% and then with 99% nitric acid, both nitrations in the cold, is described by J.Barbière, BullFr **7**, 621-6 (1940) & CA **36**, 1913 (1942)

Aromatic Compounds are substances characterized by benzene-type structures (ring or cyclic), and in many cases having pleasant odors. The compounds which are of interest in ordnance are described in this work individually, such as benzene, phenol, toluene, etc

For more info on this subject see textbooks on organic chemistry (such as Karrer's, Fieser & Fieser's, etc) and also Kirk & Othmer **2** (1948), 109-12

Aromatic Compounds, Nitration. See under Nitration

Aromatic Diazo Compounds and Their Technical Application. Title of the book by K.H. Saunders, Arnold, London (1949)

Aromatic Hydrocarbons, Detection in Aqueous Solutions. Presence of small amts of aromatic hydrocarbons in water (1 to 500 ppm) may be detected by means of formaldehyde-sulfuric acid reagent as described by H.E.Morris et al, IEC, AnalEd **18**, 294-5(1946)

Aromatic, Nitrated Derivatives. *Aromatic nitrocompounds* are ring type substances, which contain one or more NO_2 groups attached to carbons (such as nitrobenzene, trinitrotoluene, etc). *Aromatic nitroamino compds* or *C-nitroamino compds* are those contg NO_2 groups attached to carbons (as 2,4,6-trinitroaniline) and should not be confused with *nitramino* compds which are *N-nitroamino* derivs and which have the NO_2 group attached to the amino nitrogen to give an $NH-NO_2$ group. If the NO_2 group replaces a H atom of an imino group, :NH, to give $:N.NO_2$, the deriv is called a *nitrimino* compd, but if the $:N.NO_2$ is not attached through the double bond but joins two other groups through single bonds [as in $\cdot CH_2\cdot N(NO_2)\cdot CH_2\cdot$], the compd is still called nitramino-, as in cyclotrimethylenetrinitramine. *Nitronitramino* compds have NO_2 groups attached to both the carbon and nitrogen of an amino compd, as in 2,4,6-trinitrophenylmethylnitramine

Throughout this work care has been taken to differentiate clearly between nitroamines, nitramines, nitronitramimines and nitrimines. In the literature, especially in Brit, this differentation is not always made

In aromatic compds contg aliphatic alcoholic groups, such as anilinoethanol, benzyl alcohol, etc, the NO_2 group can be introduced into both the aromatic and aliphatic portions of the molecule as in N, 2,4,6-tetranitroanilinoethanol nitrate, $(O_2N)_3C_6H_2\cdot\cdot N(NO_2)\cdot CH_2\cdot CH_2\cdot ONO_2$. In this compd the NO_2 group replaces the H of the aliphatic OH group. The resulting ONO_2 group is called a *nitrate* or an *oxynitro* derivative. The term nitrate is also used when HNO_3 combines directly with an organic molecule without replacing any H, as in aniline nitrate

Many nitrated aromatic and aromatic-aliphatic derivs have been prepd and theoretically many more can be prepd. Numerous compds of these types, already prepd are expl, but relatively few possess a combination of props which make them acceptable for use as military or industrial expls

The individual nitrated compds are described in this work under what may be considered their parent compds; eg TNT under toluene, PA (2,4,6-trinitrophenol) under phenol, etc

Aromatic, Nitrited Derivatives. These derivs are similar to nitrated derivs except that they contain *nitroso-* groups, ·NO, in lieu of nitro groups. If the NO group is attached directly to C, the deriv is called *nitroso-* and if to the nitrogen of an amino group, the deriv is called *nitrosamino-*. There are also *nitronitroso-*, *nitronitrosamino-* and *nitrosonitroamino-* derivs

Many of these compds are known but only few of them are explosives suitable for military or industrial purposes. They are described in this work under their parent compds; eg 2,4 dinitrosoresorcinol under

resorcinol, trinitrodinitroso-β-naphthol under naphthol, etc

Aromatic Peracids. See under Peroxides, Organic, with the Structure RC(:O)OOH

Aromatic Silanes. See under Silanes

Aromatic Substitution-Nitration and Halogenation. Title of a book by P.B.D. DeLaMare & J.H.Ridd, Academic Press, NY(1959)

Aromatic Triazenes. Compds of general formula Ar. NH·N:N·Ar were investigated by Campbell in regard to their reactions with nitric oxide. Some of the compds prepd by him proved to be expl

Eg: When 1,3-di-p-tolyltriazene, $H_3C{\cdot}C_6H_4{\cdot}NH{\cdot}N{:}N{\cdot}C_6H_4{\cdot}CH_3$, in dry toluene at 0°, was treated with NO at 23° and 758 mm pressure, a solid compd was obtained. It was filtered off, dissolved in anhyd isopropanol and pptd by adding ether as wh hygroscopic ndls which exploded when struck with a hammer or when heated to ca 75°. Its empirical formula was reported as $C_7H_7N_3O_2$ (which corresponds to mw 165.15 and N25.45%), but the structure was not detd

Ref: T.W.Campbell, JACS **73**, 4019-20(1951) & CA **46**, 7573(1952)

ARP. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2 (1959), Unit No 434

Arquebus or Harquebus was the first practical firearm (invented in 15th century). It had a bent stock and the touchhole was at the side of the barrel with a little pan for priming. Its range was 400 to 500 paces. Later models were equipped with matchlock which was also used in the early **musket** which replaced the arquebus, late in the 16th century. But before the replacement took place, the caliber of the arquebus was standardized and the resulting weapon was called in England **caliver** and in France **arquebus de calibre**

Refs: 1)J.R.Newman, "Tools of War", Doubleday & Doran, NY (1943), 36 2)Webster's Unabridged Dictionary (1951) 1141(under Harquebus)

Arrested Burning of Gun Propellants. A convenient apparatus for recovery partially burned gun propellant grains as result of arrested burning was described by J.F. Kincaid & B.P.Dailey in OSRD Rept 1836 (1943)

Arrosage des poudres (Fr). Spraying of propellants with gelatinizers or phlegmatizers in order to make them progressive-burning can be condusted by various methods. For example, Fauveau & Delpy describe a procedure in which the propellant grains were sprayed with an alcoholic soln of camphor and centralite using a painter's spray-gun

Ref: J.Fauveau & R.Delpy, MP **31**, 162(1949)

Arrow is a slender shaft with a pointed head used as a missile which is propelled by releasing the tension of the string of a long bow. A bow consists of a strip of wood or other elastic material with a tension cord connecting both end. The bow and arrow constituted one of the earliest weapons. A later development of the long bow was the crossbow, also known as arbalest (qv). Eventually both were replaced by firearms

Ref: Webster's Unabridged Dictionary (1951), 154 & 318

Arrow Projectile (Pfeilstabiles Geschoss, in Ger), also called *Needle Shell* (Ref 5), is a slender, very long, fin-stabilized, projectile, fired from a smooth-bore gun at supersonic velocity. Its development, described in Refs 1 & 4, may be considered as one of the outstanding Ger achievements of WWII. Some info on these projectiles is given in Ref 3, pp 9-10

It seems that slender projectiles developed during WWII by Dr Otto Gessner of Peenemünde and described by Dornberger (Ref 2, pp 22-3) and also in Ref 3, p 69, under *Gessner Projectile* are also arrow projectiles.

The so-called *Röchling Anticoncrete Projectile* (Röchlingsgranate 42, Beton) resembled in appearance the arrow projectile, except that instead of the fin assembly of the arrow projectile it had a discarding flange serving as a driving band. It was manufd by the firm Röchling at Saarbrücken, Saar (Ref 4, p 160 & Ref 6)

Refs: 1)H.Kurzweg, "Die grundsätzlichen aerodynamischen Untersuchungen zur Entwicklung pfeilstabiler Geschosse," Schriften der Deutschen Akademie der Luftfahrtforschung, Nr 1059/43(1943), pp 33-71 2)W.Dornberger, "V-2," Viking, NY (1954) 3)B.T.Fedoroff et al, PATR 2510(1958), 9-10, 69 & 160 4) R.H.Riel, "Arrow Projectile Development in Germany," Aberdeen Proving Ground, Md, April 1958 5)H.H.Bullock, PicArsn Museum; private communition (1958) 6)R.Lusar, "Die deutschen Waffen und Geheimwaffen des 2 Weltkrieges und ihre Weiterentwicklung," Lehmanns Verlag, München (1958), 213 & 217

Arrowroot is a starch obtained from the roots of some varieties of plants belonging to the genus Maranta. It is a light wh powder, usually imported from Bermuda (Refs 2-4)

According to MacDonald (Ref 1), an explosive similar to *xyloidine* was prepd in 1847 by Gladstone in England by nitrating dry arrowroot

Refs: 1)G.W.MacDonald, "Historical Papers on Modern Explosives," London (1912), 36 2)Thorpe 1 (1937), 468 3)Hackh's(1944), 75 4)CondChemDict (1956), 110

Arrowhead Projectiles (Needle Point Projectiles) were AP projectiles used by the Germans during WWII against the tanks. They consisted of a pointed tungsten carbide core cemented to a steel body which had forward and rearward flanges, a plastic arrowhead shaped head covered with a sheet steel ballistic cap and a tracer assembly. The forward flange acted as a rotating band, while the rear flange acted as a bourrelet. It could be fired from an ordinary gun. The wt of an arrowhead projectile was about half that of the conventional HEAP projectile. More info is given in Refs 1 & 2

Refs: 1)Anon "German Explosive Ordnance," Dept of the Army Tech Manual TM 9-1985-3" (1953), 373 & 376-7 2)B.T.Fedoroff et al, PATR 2510(1958), p Ger 9

Note: According to Mr. A.B.Schilling, PicArsn, similar projectiles are called hypervelocity, armor-piercing (HVAP)

Arsanilic Acid. See Aminophenylarsonic Acid

ARS. A cast double-base propellant described in conf "Propellant Manual", SPIA/M2 (1959), Unit No 435

ARSENAL. A US Arsenal is a military installation of the Army Ordnance Corps (qv), primarily involved with the development, manufacture, loading, storage and issue of materiel used by the armed forces for the conduct of war. Arsenals with primary function of storage are usually called Depots

As it is mentioned under Army Ordnance Corps, the oldest US military installation, which may be considered as a manufacturing arsenal, is Springfield Armory, Springfield, Mass, established in 1794-5. The first US military establishment named "Arsenal" was the Watervliet Arsenal, Watervliet, NY. It was authorized by Congress in 1812, built in 1813, but known as "the arsenal near Troy" or Gibbonsville Arsenal, until named Watervliet in 1817. Watertown Arsenal, Watertown, Mass was established in 1816, but it's history dates back to 1800 when an arsenal was created at Charlestown, Mass. Frankford Arsenal, Philadelphia, Pa was authorized by Congress in 1815, constructed in 1816. The Augusta Arsenal, Georgia was authorized in 1816-17, but a new site was chosen in 1827. All the above arsenals were called "manufacturing arsenals," to distinguish them from "storage and repair arsenals," also called "depots."

New arsenals were established as the nation grew to the West, particularly during and after the Mexican War (1846-8). Two arsenals (which are actually depots) which continued to the present, are Benicia Arsenal (Depot), Benicia, Calif (established in 1851) and San Antonio Arsenal (Depot), Texas (1858)

During the Civil War, Rock Island Arsenal was established in 1863 at Rock Island, Ill to supply the Union troops in the Mississippi Valley

Although Picatinny Arsenal, Dover, NJ, was established in 1879-80 (as a powder depot), the history of arms-making at Picatinny

goes back beyond the Revolution, when cannon balls were made at what was known as Middle Forge. Its present name "Picatinny Arsenal" dates from 1907 when the propellant plant was built

During the WW I, the activities of arsenals were greatly expanded and a permanent storage arsenal, Raritan Arsenal, Metuchen, NJ was built (1918). In 1920 the Ogden Arsenal (for storage and repair) was built

Other US Arsenals, include in alphabetical order: Detroit Arsenal, Detroit, Mich; Edgewood Arsenal, Maryland (Army Chemical Center), near Aberdeen; Indiana Arsenal (Ordnance Works) (Hoosier Ordnance Plant, Charlestown, Ind; Joliet Arsenal (Elmwood Ordnance Plant and Kankakee Ordnance Works), Joliet, Ill; Lake City Arsenal, Independence, Mo; Radford Arsenal (Ordnance Works), Radford, Va; Raritan Arsenal (Depot), Metuchen, NJ; Ravenna Arsenal, Apco, Ohio; Redstone Arsenal, Huntsville, Alabama; Red River Arsenal (Depot), Texarkana, Tex; Rocky Mountain Arsenal, Denver, Colo and Schuylkill Arsenal, Philadelphia, Pa

The name "arsenal" is not used for the Ordnance establishments of the US Navy or Air Force

Refs: 1)Anon, "Supervisor Development Program," PicArsn Training Branch, Dover, NJ, Sept 1953 (Revised), pp 21-4 2)Anon, "Organization, History and Mission of Office, Chief of Ordnance and US Ordnance Installations and Activities," Special Text ST 9-175, The Ordnance School, Aberdeen Proving Ground, Md, July 1955 3)Ibid, ST 9-184, Dec 1955 4)Anon, Ordnance **40**, 69 (1955) 5)Anon, "US Army Installations," Dept of the Army Pamphlet, PAM 210-1, Nov 1956 6)Map of the Major Army, Navy, and Air Force Installations in the United States," AMS8205, AMSCE, Washington, DC(1958) 7)A.B.Schilling, PicArsn, Dover, NJ; private communication (1960)

Arsenic. As, at wt 74.91; exists in three modifications all corresponding to the formula As_4, mw 299.64. The most common form is the crystalline or α-form, known also as metallic arsenic. Its mp is 814° at 36 atm press, sublimation point 615°, d 5.72° at RT (5.6-5.9 for commercial grade) and hardness 3.5 Mohs. Prepn & props are given in Refs 1-6; toxicity, fire & expln hazards of As dust are discussed in Ref 7. Arsenic is used in some alloys and for hardening lead shot

US military requirements for As metal intended for use in the manuf of Mg arsenide are covered by spec MIL-A-10852B

Arsenic compds have been used in some pyrotechnic compns (see Arsenic Disulfide and Arsenic Trisulfide described under Sulfides) and as chemical warfare agents (see Arsenic Tribromide described under Bromides, Arsenic Trichloride described under Chloride and Arsine and Derivatives listed under Arsine)

Refs: 1)Mellor **9** (1929), 1-48 2)Thorpe **1** (1937), 468-72 3)Kirk & Othmer **2** (1948), 113-38 4)Gmelin Syst Nr 17(1952), 1-194 5)Ullmann **3** (1953), 828-35 6)CondChem Dict (1956), 111 7)Sax(1957), 315

Arsenic Azide. See under Azides, Inorganic

Arsenic Disulfide. See under Sulfides

Arsenic Hydride or Hydrogen Arsenide. See Arsine and Derivatives

Arsenic Pentasulfide. See under Sulfides

Arsenic Sulfides. See under Sulfides

Arsenic Tribromide. See under Bromides

Arsenic Trichloride. See under Chlorides

Arsenic Trioxide. See under Oxides

Arsenic Trisulfide. See under Sulfides

ARSENIDES are metallic derivs of arsenic. As they can be prepd by heating of some metals with arsenic, they may be called *arsenic alloys*. Ullmann calls them *Metall-Arsenide*. A number of arsenides occur in

nature; some of them are definite compds, while others are mixts. The compd of hydrogen and arsenic, hydrogen arsenide or arsenic hydride is described below as *arsine*

Several arsenides are described in Refs 1,2,4&5

US military specification MIL-M-12057A covers requirements and tests for technical grade **magnesium arsenide.*** Purified grade, Mg_3As_2 is a chocolate-colored solid, mp ca 800° & d 3.148 at 25°/4°. It can be prepd by strongly heating (to red heat) a mixt of powdered Mg with As in atm of hydrogen. Sand may be used as a diluent in order to slow down the reaction (Ref 1, p 66; Ref 3, p 413 & Ref 4, p 122)

**Note*: Mg arsenide is intended for use in the manuf of CWA's: arsine, diphenylaminechloroarsine, and diphenylchloroarsine

Refs: 1)Mellor **9** (1929), 61-90 2)Thorpe **1** (1937), 472 3)Gmelin, Syst Nr 27(1938), 413-14 (Magnesiumarsenid) 4)Kirk & Othmer **2** (1948), 122 5)Ullmann **3** (1953), 851

ARSINE AND DERIVATIVES

Arsine (Hydrogen Arsenide; Arseniuretted Hydrogen; Arsenic Hydride or Trihydride) (Arsenwasserstoff in Ger), AsH_3, mw 77.93; col gas; with offensive odor resembling that of garlic; sp gr 2.695 (Air = 1.0); fr p −113.5°; bp −55°; dec ca 230°; moderately sol in w; sl sol in alc; insol in eth. It decomposes with heat and is inflammable

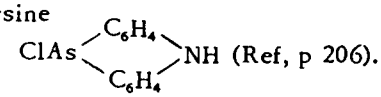
Arsine is a nerve and blood poison and a concn of 500 ppm is lethal for a man after exposure for a few minutes. MAC (max allowable concn) in air is 0.05 ppm (Refs 4, 5 & 9)

It can be prepd by the action of H_2SO_4 on metallic zinc mixed with arsenic compds (Ref 8) or by other methods (Refs 1-3 & 6 & 7)

Detection and detn of arsine in air, urine etc is discussed in Ref 4

Refs: 1)Mellor **9** (1929) 2)Thorpe **1** (1937), 472-3 3)Kirk & Othmer **2** (1948), 121-2 4) Jacobs (1949), 246-7 5)Elkins(1950), 66-7 & 227 6)Gmelin Syst Nr 17(1952), 195-233 7)Ullmann **3** (1953), 851 8)CondChemDict (1956), 113 9)Sax (1957), 321

Arsine Derivatives, Organic. Many arsine derivs were proposed as CWA's. More than 60 of such derivs are listed by Wachtel (Ref, pp 189-92). The most known of these compds is *Lewisite or M1*, which is *β-chlorovinyldichloroarsine*, $Cl{\cdot}CH{:}CH{\cdot}AsCl_2$, first isolated in 1917 by Dr W. Lee Lewis and developed as a war gas by the US Chemical Warfare Service (Ref, pp 202-6). Another important arsine CWA is *Adamsite* (Brit) (designated in the US as *DM)* or diphenylaminechloroarsine



(Ref, p 206).

Arsine derivs used by the Germans were not as effective as Lewisite. They included: a) *Dick*, US designation *ED*, *ethyldichloroarsine* $C_2H_5{\cdot}AsCl_2$ (Ref, p 194); b)*Clark I* (US designation *DA*) biphenylchloroarsine, $(C_6H_5)_2AsCl$ (Ref, p 196) and *Clark II*, biphenylcyanoarsine, $(C_6H_5)_2As{\cdot}CN$ (Ref, p 199). Another Ger arsine deriv was phenyldibromoarsine, $C_6H_5{\cdot}AsBr_2$,

Ref: C.Wachtel, "Chemical Warfare," ChemPubgCo, Brooklyn (1941), 184-206

Arsol One of the names for cyclotrimethylenetrinitrosamine (R-Salz, in Ger), described in this work under Cyclotrimethylenetriamine

ART. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 436

Artifice (Fr). Pyrotechnic device or composition

Artifices (Fr). Fireworks

Artifices à fumées colorées (Fr). Colored smoke devices used for signalling during daytime

Artifices de guerre (Pyrotechnie militaire) (Fr). Military pyrotechnics

Artifices incendiaires (Fr). Incendiary pyrotechnic devices

Artifices lumineux (Fr). Illuminating devices used for nighttime signalling

Artifices produisant un sifflement (Fr). Whistle-producing devices, designed for signalling by sound. They were usually made

by loading long, narrow tubes ca 10 mm diam and made from reed, cardboard or plastic, with a mixt of K picrate 87 & K nitrate 13%

Ref: Pepin Lehalleur (1935), 478

Artifices de signalisation(Fr). Pyrotechnic signals

Artifices pour signaux(Fr). Same as pétards pour signaux

Artificial Barricade means an artificial mound or revetted wall of earth of a minimum thickness of 3ft, erected as protection around places storing expls, propellants or ammo

Ref: Cook(1958), 355

Artificial Silica can be prepd by decompg silicon fluoride with water. It possesses high absorptive value and was used by M.Berthelot in France for prepg some dynamites

Ref: Marshall 1 (1917), 360

Artifizi da guerra(Ital). Military pyrotechnics

Artifizi pirotecnici per usi bellici(Ital). Pyrotechnic devices for use in war

Artiglieria(Ital). Artillery; ordnance

Artillería(Span). Artillery; ordnance

Artillerie(Fr & Ger). Artillery; ordnance

Artilléríya(Rus). Artillery; ordnance

Artillery is the branch of the armed forces which uses weapons of caliber greater than firearms. This includes cannons, howitzers, mortars and rocket launchers, which may be mounted on wheels and towed by horses or motor vehicles, or mounted on tanks, motor vehicles, boats, ships etc

The calibers of US artillery weapons are listed under Ammunition and Weapons

Refs: 1)Merriam-Webster's (1951), 157 2) EncyclBritannica 2 (1952), 463-78 3)Collier's Encycl 2 (1957), 294-310

Artillery Ammunition. See under Ammunition and Weapons

Artillery Ammunition Components Tests. The purpose of these tests is to determine if the components of a round of artillery ammo (such as cartridge cases, powder bags, propelling & bursting charges, projectiles, boosters, igniters and fuzes) function according to the requirements of the US Armed Forces Specifications. These tests may be conducted also in order to obtain data for further research and development

Descriptions of testing methods for various components are given in the following Ordnance Proof Manuals:
a)OPM No 8-10(1947) (General) b)OPM No 8-11(1943) (Projectiles) c)OPM No 8-12 (1937) (Propellants) d)OPM No 8-13(1948) (Fuzes) e)OPM No 8-14(1943) (Primers for cannons) f)OPM No 8-15(1942) (Boosters) and g)OPM No 8-16(1942) (Cartridge cases and bags)

Artillery Cannon and Cannon Equipment. General specification for manuf and inspection is in MIL-A-13931(Ord)

Artillery Carriages & Mounts; Recoil Mechanisms; Rocket Launchers; Auxiliary Equipment and Parts. General specifications for these items are in MIL-A-13917A(Ord)

Artillery Materiel and Its Testing. Artillery materiel includes items such as cannons, mounts & carriages, recoil systems, sighting systems, subcaliber guns, rocket launchers, aircraft armament and recoilless weapons. The purpose of tests is to determine whether or not the submitted materiel meets the requirements of the US Armed Forces Specifications

The following Ordnance Proof Manuals describe these tests:
a)OPA No 16-10(1947) (Artillery materiel; general) b)OPA No 16-10-75(1944) (Pilot units) c)OPA No 16-10-75A(1952) (Winterization testing of artillery materiel) d) OPA No 16-11(1943) (Cannons) e)OPA No 16-12(1943) (Carrages & mounts) f)OPA No 16-13(1942) (Recoil systems) g)OPA No 16-14(1943) (Telescopic sights) h)OPA No 16-15(1942) (Subcaliber guns) i)OPA

No 16-16(1944) (Aircraft armament) j)OPA No 16-17(1948) (Erosion and service life tests) k)OPA No 16-20(1951) (Recoilless weapon materiel development tests)

Artillery Propellants. See under Propellants

Artillery Rockets. See under Rockets

Artillery, Railway. This term covers artillery, mostly of large caliber, mounted on special carriages attached to heavy duty platforms (flat cars) of special trains (mostly armored trains). Protection of gun crews is usually achieved by means of steel shields

A comprehensive description of British & French, Italian and US railway artillery used during WWI is given in Ref 1

Newman (Ref 2) describes three pieces of US railway artillery: a)8-inch Railway Gun b)12-inch Railway Mortar and c)14-inch Railway Gun. US Railway Artillery which was at the beginning of WWII a part of the Coast Artillery, was disbanded in March 1942

Two examples of Ger railway artillery of WWII, used on the Russian Front are given in Ref 3, p 263: a)310 mm Glött Gun and b) 510 mm Self-Propelled Mortar Karl Gerät

Refs: 1)H.W.Miller, "Railway Artillery," Ordnance Department Document No 2034, Washington, DC (1922) 2)J.R.Newman, "Tools of War," Doubleday & Doran & Co, NY (1943), 164-6 3)B.T.Fedoroff et al, PATR 2510(1958), 263

Artillery Train comprises a number of pieces of artillery (ordnance) mounted on carriages equipped for marching, together with their munitions and the vehicles transporting them

Ref: Merriam-Webster's(1951), 157

Arukōru(Jap). Alcohol

Aruminyūmu(Jap). Aluminum

Arylamines and Their Explosive Derivatives are described individually, such as aniline, anilinoethanol, etc

Arylamines, Qualitative Reactions were discussed by S.I.Burmistrov, ZhAnalKhim **1**, 265(1946) & CA **43**, 5344(1949)

Arylaminoguanidines; Arylaminotetrazoles and Arylaminotriazoles and Their Explosive Derivatives are described individually

Arylazides. See under Alkyl- and Arylazides

Arylparaffins. Theory of nitration of side chains of arylparaffins was discussed by A.I.Titov, UspKhim **21**, 881-913 (1952)

ARZ. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 437

ASA (Azide-Styphnate-Aluminum). British initiating mixts of LA, LSt & Al (flake powder) for detonators using tetryl as a base charge. Brit "Service ASA" contains the crystalline "Service Azide," while commercial ASA contains dextrinated LA

The No 6 commercial detonator consists of an Al cap containing 0.35 g of ASA as an upper layer and 0.25 g of tetryl as a lower charge. The larger and stronger detonator, No 8, contains 0.35 g ASA & 0.55 g tetryl. PETN can be used as an alternative base charge for tetryl to obtain detonators of increased initiating power or to reduce the detonator charge required. The corresponding charge of ASA & PETN are 0.20 & 0.22 g respectively for the No 6 cap and 0.20 & 0.45 g for the No 8

Still stronger detonators may be produced by using three layers: ASA, an intermediate charge of PETN or tetryl slightly compressed and a base charge of PETN or tetryl very highly compressed

Refs: 1)A.R.Ubbelohde, Trans Roy Soc (London), **241A**, 215 & 217(1948) 2)Taylor & Gay (1958), 54-5

Asbestos(Earth-Flax; Stone-Flax; Mountain Cork) (Asbest in Ger; Amianthe in Fr). Asbestos is a class name for several native fibrous materials, but commercial asbestos is mainly the fibrous form of serpentine known as chrysolite, which is a hydrous

magnesium silicate, 3 Mg $O.2SiO_2 \cdot 2H_2O$. Other types of asbestos often contain silicates of magnesium, iron and aluminum as well as of magnesium. Commercial forms of asbestos can be white, grayish, bluish or greenish in color. Asbestos is acid- and heat-resistant and may be spun or woven (Refs 2-4)

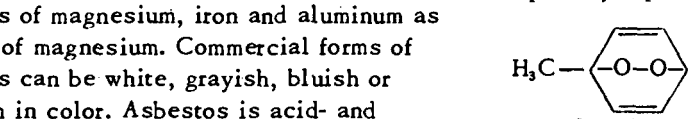
Uses of asbestos include: acid- & fire-proof cord, gloves, clothing, pads, paper, carton, gaskets & filtering disks, acidproof cements & putties and as filter bed in Gooch crucibles and funnels (Refs 2-4). Powdered asbestos was used as an absorbent for NG in some older dynamites (Ref 1). Fibrous asbestos has been used in *Ascarite* and in *Aitch-Tu-Ess*. Ascarite is an absorbent for CO_2 made of asbestos with NaOH and Aitch-Tu-Ess is a solid generator of H_2S made by compressing asbestos with a sulfide decompg on heating. Platinized asbestos has been used as a catalyst both in laboratory and plant. Hutchison (Ref 5) proposed to use asbestos in solid gas generating units employed for actuation of pressure-operated mech devices in blasting operations, propulsion of rockets, etc. In these units, self-sustained, exothermic, nondetonating, gas-evolving,decompn reactions are obtained by igniting local areas of NGu and/or GuN compns contg 0.25-1% by wt of asbestos fiber & 0.2 – 10% $H_2MoO_4 \cdot CeO_2$ or $H_2MoO_4 \cdot V_2O_5$

Asbestos dust in air can be trapped with an impinger dust-sampling apparatus using 25% aq ethanol as the collecting medium (Ref 4). The safe threshold value for asbestos dust exposure is considered 5 million particles per cubic foot

US military requirements for crude asbestos are given in specification MIL-A-13651 and for asbestos sheet, compressed, MIL-A-7021A

Refs: 1)Daniel (1902), 31 2)Hackh(1944), 78 3)Kirk & Othmer **2**(1948), 134-42(Asbestos) (13 refs); 142-50(Asbestos-Cement Products) (49 refs) 4)Jacobs(1949), 188 5)A.C. Hutchison, USP 2,710,793(1955) & CA **49**, 13652(1955)

Ascaridol(α-Terpinene Peroxide or 1,4-Epidioxy-2-p-menthene),



H_3C- —$CH(CH_3)_2$, mw 166.21. Liq bp 83° at 4-5 mm & 96-7° at 8 mm,dec explosively >130° at atm pressure; n_D 1.4769 at 25°. A naturally occuring (in ethereal oil of Chenopodium seeds) transannular peroxide, which may also be obtained from α-terpinene and other substances. It is toxic

Refs: 1)Beil **19**, 17,(611) & [18] 2)O. Wallach, Ann **392**, 59(1912) 3)E.K.Nelson, JACS **33**, 1404(1911) & **35**, 84(1913) 4)K. Bodendorf, ArchivPharmazie **271**, 1-35(1933) 5)H.Hock & F.Depke, Ber **84**, 122 & 349 (1951) 6)Tobolsky & Mesrobian(1954) 24-6, 166 & 178

Ascarite. See under Asbestos

ASG. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 449

Ash is a solid left after a combustible material is thoroughly burned at not too high temp. Ashes vary in compn , both qualitatively and quantitatively, but in most cases they consist of oxides and non-volatile salts of Na, K, Ca, Mg & Fe. Some sand and silicates may also be present. The amt of ash is sometimes taken as a measure of the "mineral matter" of the original material

Refs: 1)Thorpe **1**(1937), 503-11 2)Kirk & Othmer **4**(1949),93-4 3)Merriam-Webster's (1951), 161

Ash Determination. This is one of the tests for the detn of purity of substances consisting primarily of one or more organic compds

A)If the material is normally slowburning (such as coal, wood, paper, etc) and not an explosive nor readily ignited (such as NC or propellants), weigh accurately a sample (1g or more depending on the amt of expected ash) in an accurately tared crucible (porcelain or metallic) and heat in an oven at ca 105° until all the moisture is removed (1-2 hrs). Cool in a desiccator and weigh. The loss in wt, divided by wt of sample and

multiplied by 100 gives % of volatiles. Place the crucible in a muffle furnace (or use Bunsen burner or an electric heater) and heat gently at first (until most of the smoke disappears) and then increase the heat gradually to a dull red (700-750°) in order to destroy all the carbonaceous material. Avoid heating to higher temps to prevent fusing of ash to the walls of the crucible. Occasionally stir the ash with a platinum wire. Cool the crucible in a desiccator and weigh. Heat again for 30 mins, cool and reweigh. Repeat until a const wt is obtained (Ref 3, p 598)

B)If the substances is an explosive, the direct heating in a muffle furnace or in a flame is not advisable because all the ash would be blown out by the expln. In order to avoid this, the sample must be treated prior to combustion with some liquid, such as H_2SO_4, oil, isopropanol, etc, preventing the expln and slowing down the combustion

For example, when analyzing PA, weigh ca 5 g in a tared crucible and add few drops of concd H_2SO_4. This will decomp PA with formn of carbonaceous material. Heat carefully to remove the excess H_2SO_4 and to burn the bulk of carbon. Cool, add few drops of concd H_2SO_4 + HNO_3 and heat at below dull red to eliminate all carbonaceous material. Cool in a desiccator and weigh. The difference betw this wt and the wt of empty crucible gives ash as sulfates

During WWII in analyzing TNT for sodium content in the ash, the following method for combustion of TNT was used at Keystone Ordnance Works, Meadville, Pa:

Weigh on a rough balance in a tared platinum dish a 25 g sample and moisten it with a little isopropanol. Heat the dish, under the hood, with the colorless flame of a Bunsen burner until all the TNT is melted. Care must be taken not to heat platinum in a yellow flame as the incandescent carbon of such a flame attacks platinum, forming carbide which is extremely brittle. If the material has not already ignited, ignite it, remove the burner, close the hood and allow the combustion to proceed by itself. Do not be alarmed if the material flashes at the end of burning. Transfer the dish into a muffle furnace preheated to 700-750° and leave there to burn off all the carbon (ca 5 mins). Cool the dish in a desiccator and weigh

If it is desired to transform the carbonates and oxides to sulfates, add a few drops of dil H_2SO_4 (ca 10%) and with the aid of a rubber policeman rub down the sides of the dish moistening them with the liquid in the dish. Evaporate the liquid and heat the dish in the muffle furnace at below dull red (Ref 1)

C)If the sample is a propellant, treat 1-2 g in a tared crucible (porcelain or platinum) with a few drops of concd nitric acid and heat on a steam bath, under the hood, until the evolution of nitrogen oxides subsides. Continue heating until the liquid fraction evaporates, thus leaving a gummy mass. Transfer the crucible to a triangle, and using a low flame heat carefully until most of the carbonaceous matter has been burned off. Finish the combustion at ca 600° for ca 1 hr, cool in a desiccator and weigh (Refs 1 & 2)

D)If the material is a liquid or a syrup consisting of water with some dissolved combustible substance (such as a red water or thick liquor of TNT manuf), pipette out 10 ml to a tared, low form, procelain crucible, and evaporate the water with stirring, on a steam bath. Transfer to oven at 135° and heat for 4 hrs. Cool in a desiccator and weigh. This gives *total solids*

Moisten the residue with ca 10 ml of acetone, slant the dish at ca 45° angle and ignite. Remove the gas burner, cool the dish, and repeat the above treatment. Heat the crucible on a gas burner or in a muffle furnace until the disappearance of carbonaceous matter, cool in a desiccator and weigh (Ref 1)

(See also under individual compounds)

Refs: 1)Clift & Fedoroff, **1**(1942) & **3**(1943)

2)Olsen & Greene(1943), 37&64 3)Snell & Biffen(1944), 598

ASN. An expl compn developed by CPV (Ref 1): AN 70, PETN 20 & dicyanodiamide 10%. It was used during WWII both in Germany and Italy as an underwater explosive. Its props were: rate of deton at d1.55, 5500 m/sec and equation of expl decompn:

$$14\,NH_4NO_3 + C(CH_2ONO_2)_4 + 2(CN{\cdot}NH_2)_2 \rightarrow$$

$$9\,CO_2 + 36\,H_2O + 20\,N_2$$

Vol of gases evolved according to the above reaction is 972 l/kg at NTP and heat evolved 938 kcal with H_2O in vapor phase

Brandimarte (Ref 3) describes the prepn of ASN, as follows: PETN (20 parts) was added at 115-18° to a binary mixt of 70 ps AN and 10 ps dicyanodiamide, which had been previously fused for 6-8 hrs. The mixt was analyzed and found to contain ca 2% of GuN and ca 6% of biguanide nitrate, which were formed by interaction betw dicyanodiamide and AN

An improved underwater expl compn was prepd by mixing 90 parts of ASN with 10 ps Al powder

Refs: 1)Report of the Chemisch-physikalische Versuchsanstalt, Berlin(1943) 2)Mangini (1947), 225 3)E.Brandimarte, Chim e Ind (Milano) **35**, 553-5(1953) & CA **48**, 4219-20 (1954) 4)PATR 2510(1958), 212

Asphalines. Brit blasting expls used at the end of the 19th century: a)Asphaline No 1 was prepd by thoroughly mixing 54 parts of $KClO_3$ with 42 ps of bran and/or wheat, barley, etc & 4 ps of K sulfate, in presence of small amt of w. After drying the mixt could be bound and waterproofed by incorporating a small amt of mineral oil, paraffin, soap or ozocerite b)Asphaline No 2 consisted of 75 parts asphaline No 1 and 25 ps K nitrate

Refs: 1)Cundill's Dictionary, MP **5**, 287 (1892) 2)Gody (1907), 263

Asphalt; Asphaltum or Mineral Pitch (Earth Pitch; Jew's Pitch or Trinidad Pitch) is a solid or semisolid black or dark brown amorphous material found native in many parts of the world. One of the greatest known deposits is in Trinidad ("Pitch Lake"). A soft variety, which is found on the surface of Dead Sea, has been known since Biblical times

Asphalt is one of the *bitumens,* to which also belong *tar* and *pitch.* Bitumen, as well as asphalt, is a mixt of hydrocarbons associated sometimes with mineral matter. The org constituents are sol in CS_2 and may be roughly divided into *asphaltines* and *carbenes.* The former are insol in ether and mineral oils, whereas the latter are insol in CCl_4 or in $CHCl_3$

The common form of asphalt is a black, compact, amorphous, brittle mass of dull luster, which breaks showing a polished surface and fuses at 188-90°F (ca 87°), d 1.40-1.42 at 77°F (25°C) and Moh's hardness 1 to 2

Large amt of asphalt used in the US is artifical. It is obtained as the residue from distillation of asphaltic and mixed base crude petroleum oils

Another variety of artificial asphalt is one of the by-products of manuf of coal gas. This asphalt is the residue left in the retorts after removal from coal tar (by distillation) of aromatic hydrocarbons, phenols, cresols, etc

The mixt of asphalt with sand and limestone used in roofing and for paving roads is commonly called "asphalt"

The purest variety of natural bitumens is *gilsonite* or *mineral rubber.* It belongs to the group of *asphaltites.* Gilsonite is easily distinguished from the other asphalts by its brown streak, lower d, fixed carbon and low sulfur content. Its d is 1.03-1.10 at 77°F (25°), softening point (ring & ball) 270-400°F (132-204°) and soly in CS_2 >98%. There are three commercial grades: "selects," "seconds" (standard) and "jets" (Ref 3, p 166)

Uses: Asphalt is used for paving streets, surfacing floors & roofs; as an ingredient of paints, varnishes, cements; for impregnating

belting material and as a bonding and waterproofing agent. Davis (Ref 1) lists uses of asphalt in some commercial pyrotechnic compns and Sutton (Ref 7), Warren (Ref 8) and Herrick & Burgess (Ref 9) describe uses of asphalt in rocket propellants. US military specification MIL-A-3029(1) deals with asphalt used in waterproofing fiber ammunition containers

Powdered gilsonite has been used as a component of some US pyrotechnic and ignition compns

The following requirements for gilsonite are given in specification JAN-A-356: a)Granulation – through No 100 US Std Sieve 99% (min) b)Sp gr at 25/25° 1.05 ± 0.05 c) Softening point 125° (min) d)Mineral matter 1.0% (max) e)Grit-none f)Volatile matter 1.0% (max) g)Reaction of water extract – alkaline to methyl orange h)Solubility, %, min – in CCl_4 99, in petr ether 30 and in CS_2 99

For description of tests see the above specifications

Refs: 1)Davis(1943),121 2)Hackh(1944), 80 3)Kirk & Othmer 2(1948),164-199(68 refs) 4)P.Horwink, Edit, "Elastometers and Plastometers–Manufacture, Properties and Application," vol 2, Elsevier Amsterdam (1949), Chap 13, "Asphalt," by R.N.Traxler 5)EncyclBritannica 2(1952),549-50 6)Cond ChemDict(1956),116 7)Sutton(1956),313 & 337 8)Warren(1958),37 9)J.W.Herrick & E.Burgess, Edits, "Rocket Encyclopedia," AeroPublishers, Los Angeles 26, Calif(1959), 29

Asphalt-Perchlorate Castable Propellants for use in JATO's were developed at GALCIT (Guggenheim Aeronautical Laboratory, California Institute of Technology) and by the Aerojet Corp, Azuza, Calif. The original propellant was called *Galcit* and it consisted of K perchlorate and asphalt

Refs: 1)J.E.Burchard, "Rockets, Guns & Targets," Little, Brown & Co, Boston(1948), 19 2)W.A.Noyes, Jr, "Science in WWII, Chemistry," Little, Brown & Co, Boston (1942), 112

Asphaltenes. See under Asphalt

Aspirin and Derivatives. See Acetylsalicylic Acid and Derivatives

Asplund Process of Pulping. See under Pulp and Pulping

Assisted Take-off (Units), abbreviated to **ATO.** This term usually refers to an auxiliary rocket engine specifically used for providing extra thrust to a heavily loaded aircraft during the take-off run and initial climb. It is generally understood that an **ATO** is a liquid propellant engine to distinquish it from **RATO** (rocket-assisted take-off) which is operated by a solid propellant. ATO and RATO are designed to operate only during take-off, and all or any part of powerplant system can be jettisoned after completion of take-off. The term **JATO** (jet-assisted take-off) was coined during the early part of WWII for a solid-propellant rocket power plant that would give assistance to the take-off of heavily-laden airplanes and seaplanes. The term JATO is discontinued in favor of RATO

Refs: 1)C.M.Bolster, "The Assisted Take-off of Aircraft," Norwich Univ, Northfield, Vermont (J.J.Cabot Fund Publication No 9) (1950) (It covers the whole range of ATO including catapult take-off) 2)W.W.Holler, Edit, "Glossary of Ordnance Terms," OEHO, Duke University, Durham,NC(1959),163 3) J.W.Herrick & E.Burgess, Edits, "Rocket Encyclopedia Illustrated," Aero Publishers, Los Angeles 26 Calif (1959), 29-31(ATO); 240-2(JATO) and 375(RATO)

AST. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 442

Aston, Francis W. (1877-1945). A Brit physicist, professor at Cambridge Univ, noted for studies of isotopes and for the development of mass-spectra

Refs: 1)Hackh(1944),81 2)Guia, Dizionario 1(1948),582

Astralits are AN mining expls contg as sensitizers TNT or DNT and sometimes a small amt of NG. Compn of five Ger Astralits are listed in Ref 3. One of the Ger Astralits: AN 84.5, NG 4.0, TNT 7.0, WM 1.0, charcoal

1.0 & paraffin oil 2.5% was used during WW as a filling for trench mortars and hand grenades (Ref 1)

According to Izzo (Ref 2) some Astralits were manufd in Italy by the Società Dinamite

According to Antonelli (Ref 4), the Ger *Astralit IA* contained AN 68.3, TNT 25.0, NG 4.0 & WM 2.7% and the corresponding Brit Astralit contained GC instead of WM

Refs: 1)Marshall 1 (1917),397 2)Izzo, Minatore(1953),53 3)PATR 2510(1958),10 4)R.P.Antonelli, OTIA, Arlington,Va(1960)

Astrodyne Synthetic Rubber Propellant, a solid rocket propellant made from synthetic gum stock mixed with AN in rubber-making machinery. After mixing, the material is extruded at low pressing into shapes with the desired cross section and then cured by heating for 3 days at ca 180°F

Ref: B.Kit & D.S.Evered, "Rocket Propellant Handbook," Macmillan, NY(1960),33

Astronautics or Space Travel (Astronavigatsia, in Russian) is the study of the physical possibilities of voyaging through space to other celestial bodies, including stars

The subject considered for many years as fiction, became a reality when the Russians launched their "Spootnik" in 1956

Although space travel is outside of the scope of our work, it would be appropriate to give a partial list of books on this subject: 1)N.A.Rynin et al "Mezhplanetnyiye Soöbshcheniya (Astronavigatsia)," Gosizdat, vols 1-9 (1928-1939) (Vol 9 contains a comprehensive bibliography covering nearly every publication in any language up to 1931) 2) A.Ananoff, "L'Astronautique," Librairie Arthème Fayerd, Paris(1949) 3)H.H.Koelle & H.J.Kaeppeler, "Literature Index of Astronautics" (German & English), W.Pustet Verlag, Tittmoning (Bayern) (1954) (A recent bibliography on Astronautics) 4)G.Portel, "Dizionario di Tecnica dei Razzi e d'Astronautica," Associazione Italiana Razzi, Roma (1955) (Dictionary in Ital, Ger, Engl & Fr on rockets & astronautics) 5)A.Zaehringer, "Solid Propellants and Astronautics," 5th Congress International Astronautical Federation, Innsbruck, Austria, Aug 1955 & CA **49**, 10627(1955) 6)Willy Ley, Rockets, Missiles and Space Travel," The Viking Press, NY(1957), 489-520 (An extensive bibliography in all languages on space travel) 7)M.T.Bizoni & R.Griffin, Edits, "The Space Encyclopaedia," Dutton, NY (1957) 8)E.Burgess, "Satellites and Spaceflight, Macmillan, NY(1957) 9)A.Fritz, "Start in die dritte Dimension," Herold-Verlag, Stuttgart (1958) 10)A.G.Haley, "Rocketry and Space Exploration," VanNostrand, NY(1958) 11)F.G.Grieger, "Behind the Sputniks," Public Affairs Press, Washington,DC(1958) 12)E.Bialborski, "Raketen, Satelliten, Raumschiffe," Urania-Verlag, Leipzig(1958) 13)H.S.Seifert, "Space Technology," Wiley, NY(1959) 14) Journals: Aerospace Engineering, ARS Journal, Astronautica Acta, Astronautics, Interavia, Journal of the British Interplanetary Society, Missiles and Rockets, Raketen und Raumfahrtforschung, Space Aeronautics, Spaceflight, Space Technology, Thiokol Astronaut & Weltraumfahrt 15)Russian Journals translated into English may be obtained from Consultants Bureau, Inc, New York 11, NY

AT. Rus abbrn for amatol

ATJ. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2 (1959), Unit No 444

Atlas. One of the numerous US missiles

Ref: J.L.Chapman, "Atlas: The Story of a Missile," Harper,NY(1960)

Atlas Dynamite, patented in 1883 by Kalk contained NG and,as an absorbent,a mixt of NC, NS, nitromamite and powdered glass

Ref: Daniel(1902), 32

Atlas Powder Company (APC), Wilmington, Delaware, was organized in 1912 to comply with the decree of US Federal Court dissolving the E.I. du Pont de Nemours Powder Co into three separate units: E.I. du Pont de Nemours & Co, Atlas Powder Co and Hercules Powder Co. The name "Atlas" was chosen to represent the brand of dynamite which had been assigned to the new Company

Ref: Van Gelder & Schlatter(1927), 211 & 465-78

Atlas Powders were blasting expls manufd

by the Atlas Powder Co before WWI. Some of these powders were used in building the Panama Canal

Following are two examples of Atlas Powders given in Marshall 1(1917), 364: a)**No 1**:NG 61.1, woodpulp 14.1, KNO_3 21.6, MgO (or $CaCO_3$) 3.0 & moisture 0.2% b)**No 2**: NG 45.7, woodpulp 10.5, KNO_3 40.9, $CaCO_3$ 1.9 & moisture 1.0%

Note: According to Mr. G.D.Clift, none of the Atlas Powders analyzed by him while working before WWI at the duPont laboratory, contained KNO_3, but only $NaNO_3$

Atmospheric Pollution. See Air Pollution

ATN. A cast double-base propellant described in conf "Propellant Manual" SPIA/M2(1959), Unit No 447

ATO. See Assisted Take-off

Atomic Ammunition. See Atomic Weapons and Ammunition

Atomic Artillery. See Atomic Weapons and Ammunition

Atomic (or Nuclear) Bomb.* A weapon invented during WWII and developed in the United States as a joint effort with the British and Canadian governments. It utilizes for its destructive effect the energy of an *Atomic or Nuclear Explosion* (qv). Since atomic explosions are of two types, fission and fusion, atomic bombs are of corresponding types. However, it has been necessary to first initiate an atomic explosion with a nuclear fission reaction in order to bring about the conditions under which a nuclear fusion(thermonuclear) reaction can occur. Thus a *Fusion Bomb, (Hydrogen or H Bomb, a Thermonuclear Bomb)* must contain means of initiating both types of atomic explosion

The three bombs exploded during WWII – in the New Mexico desert on July 16, 1945, over Hiroshima on Aug 6, 1945, and over Nagasaki on Aug 9, 1945 – were all of the fission type and of tens of kiloton(thousands of tons of TNT equivalent) yield. Efforts since the war have had two aims. One is the development of fusion bombs. The first one was tested at Eniwetok in the spring of 1948. The bomb of several megaton (millions of tons of TNT equivalent) yield reported in 1955 seem to be of a three-stage fission-fusion-fission type. An ordinary fission bomb at the center is surrounded by lithium deuteride, the fusion component, which on initiation emits fast neutrons. These induce fission in the outer component, U238. Efforts are in progress to minimize or eliminate the fission reaction and thus produce a relatively *"Clean" Bomb* of fusion type, with reduced output of poisonous radioactive isotopes of extremely long half-life. Problems here are similar to those involved in the attempt to generate power for industrial purposes by fusion reactions. The second aim was the development of atomic(presumably fission) bombs of smaller size and yield, adaptable to missions of military units in the field,*Atomic Artillery*

It is necessary, in the construction of an atomic bomb, to utilize the energy released by the nuclear reaction or reactions in such a way that an explosion of the desired yield takes place. This requires control, so that the explosion occurs when wanted and not before (usually obtained by keeping the fissionable material in units smaller than critical size) and then effecting a chain reaction which takes place so quickly that a large amt of material undergoes nuclear reaction before the bomb flies apart. The latter is done by bringing the parts together quickly, for instance by shooting one part as a projectile against the other part as a target. A suitable tamper of dense material can be used to delay the break-up

Refs: 1)H.D.W.Smyth, "Atomic Energy for Military Purposes," Princeton Univ Press, Princeton,NJ(1945) [Comprehensive resumé in French is given in MAF **20**, 217-35(1946)] 2)J.K.Robertson, "Atomic Artillery and the Atomic Bomb," VanNostrand, NY(1945) 3) H.Sabatier, MAF **20**, 437-59(1946) (Note sur la bombe-uranium) 4)A.K.Solomon, Fortune **33**, No 5, 115-22, 173-4 & 176(1946); CA **40**, 6969(1946)(Physics of the atomic bomb) 5) W.L.Lawrence, "Dawn over Zero; The Story of the Atomic Bomb," Knopf, NY (1947)

*Written by C.G.Dunkle

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(Other refs are given under Atomic Energy)

Atomic (or Nuclear) Energy; Atomic (or Nuclear) Reactions; Atomic (or Nuclear) Explosions.* In chemical reactions the atomic nuclei maintain their charges, masses, and individual identities. These all change in atomic or nuclear reactions, first revealed in the discovery of radioactivity by Becquerel in 1895 and of radium by the Curies in 1898. The natural radioactive disintegrations evolved energies per atom almost a million times those evolved in chemical reactions, but resisted all attempts to change their rates or control them by any chemical means. Therefore physicists began bombarding various nuclei, first with alpha- and beta-particles and gamma rays from natural sources; and later with protons, deuterons,etc from artificial sources. In 1939, discoveries made almost simultaneously by E.Fermi in Italy, by O.Hahn, F.Strassmann et al in Germany, the Joliots and others in France, and J.Chadwick et al in England, gave a new dimension to nuclear research. Uranium nuclei, bombarded by neutrons, were found to split into two other nuclei of roughly equal masses, forming such pairs as Kr and Ba or Xe and Sr

The energy of the process, about 200 mev (million electron volts) is out of all proportion to the energies of only 5 mev or so given by nuclear transmutations known up to that time. For comparison, since 23 kcal/mole or per gram atom is equivalent to only 1 ev (electron volt) per particle, a heat of explosion of 240-250 kcal/mole (TNT) is less than 11 ev per molecule

The evolution of energy in a nuclear reaction follows from the Einstein relation between mass m and energy: $E = mc^2$ (where E = energy in ergs, m = the mass in grams and c = velocity of light in cm/sec). The measured mass of a nucleus is not exactly the sum of the masses of the nucleons (protons and neutrons) present, but somewhat less. The small discrepancy represents the loss of mass due to the association or binding together of these particles. The energy equivalent to this loss, by the Einstein relation, is called the *binding energy*

Nuclear forces between any two nucleons are very much alike, whether they are neutrons or protons, and arrangements contg equal numbers of each tend to be most stable in light nuclei. As the size and weight of the nucleus increase, however, Coulomb repulsion gains in relative importance because it falls off only with the square of the distance, much more slowly than the nuclear force. Thus, with increasing atomic weight the number of neutrons increases more and more, relative to the number of protons

The binding energy per nucleon, if plotted against the mass number (total number of nucleons) of the nucleus, jumps sharply from its minimum of 1.0 mev for hydrogen, rises above 8.5 mev to a level stretch for mass numbers from 40 to 80, and then falls off slowly, dropping below 8.0 mev for mass numbers over 175. The atoms near the center of the sequence therefore have the largest binding energies and hence the greatest stability against nuclear distintegration. The lighter elements and the much heavier ones are less firmly bound aggregates of nucleons. Reactions which either convert heavy elements into those near the center of the

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table by *fission,* or combine lighter nuclei to make heavier ones by *fusion,* evolve large amounts of energy. These are the two types of *Nuclear Reactions* applicable to production of *Atomic Explosions*

Three nuclei known to undergo fission are U^{233}, U^{235} and Pu^{239}. One of these on capturing a neutron can apparently oscillate violently and then, like an oversized raindrop, split into approximately equal halves along with smaller droplets or neutrons. The splitting is a statistical process rather than one that always occurs in the same way; sometimes one pair of product nuclei results and sometimes another; sometimes two neutrons are produced and sometimes three. A typical fission reaction producing two neutrons is:

$$_{92}U^{235} + {}_0n^1 \rightarrow {}_{57}La^{147} + {}_{35}Br^{87} + 2{}_0n^1$$

(Subscripts indicate atomic number or positive charge on the nucleus, superscripts indicate mass number)

The product nuclei as initially formed are highly unstable isotopes and emit *delayed* neutrons as well as electrons and gamma photons while settling down into their stable configurations, which are usually isotopes of different elements from those first formed. The neutrons, both *prompt* and *delayed*, continue the reaction by encountering other fissionable nuclei

Thus one neutron suffices to start the reaction while 2 or 3 are produced by it, and are available to initiate fissions of other nuclei. This makes a *chain reaction* possible, and in fact inevitable if neutrons are generated more rapidly than they escape or are otherwise lost, for any excess if present grows exponentially with extreme rapidity. Since the rate of escape depends on surface area and is thus proportional to the square of the length whereas the rate of production depends on the mass or volume and is thus proportional to the cube of the length, there is for each fissionable material a *critical mass.* Smaller masses remain unchanged, but if brought together quickly enough to form a mass exceeding the critical size, undergo the fission reaction at once. The initiating neutron can come from a cosmic ray or from an artificial source

Fusion or thermonuclear reactions produce more energy than fission. Furthermore, the reactants are cheap and easily available, and the products are harmless and thus give hope for developing a "clean" nuclear bomb. Reactions of this general type supply the energy of the sun and other stars, where gravitational forces hold the reactants together despite temperatures of several million degrees K. Since strong Coulomb repulsion must be overcome for the nuclei to collide and hold together long enough to react, it is likely that the only reactions practically realizable on an industrial scale are those between nuclei having small charges, such as the following:

$$_1D^2 + {}_1D^2 \rightarrow {}_2He^3 + {}_0n^1 + 3.2 \text{ mev}$$

$$_1D^2 + {}_1D^2 \rightarrow {}_1T^3 + {}_1H^1 + 4.0 \text{ mev}$$

$$_1D^2 + {}_1T^3 \rightarrow {}_2He^4 + {}_0n^1 + 17.6 \text{ mev}$$

$$_1H^1 + {}_3Li^7 \rightarrow {}_4Be^8 + 17.2 \text{ mev}$$

Temperatures high enough to induce transitory fusion reaction can be reached with a fission reaction. Despite the short duration of the fusion, thermonuclear bombs have yields in the megaton (TNT equivalent) range as compared to kilotons from fission bombs. Problems involved in starting the fusion without a preliminary fission, or of holding the reactants together in a steady fusion reaction for power generation, have not been solved

The fission reaction has been successfully applied to *industrial power production;* here the reaction is conducted not explosively, but in such a way as to provide a steady source of energy for power generation by conventional heat engines. This method is suitable also for large vehicles such as *atomic energy submarines* and *atomic energy aircraft* (See also Atomic Weapons and Ammunition)

Atomic or Nuclear Explosions evolve quantities of energy, per unit mass of reactant, from

a million to a billion times those available from chemical explosives. Therefore,the shock waves, although basically of the same nature as those from HE, have much higher pressures in the positive pulse, lower pressures in the negative phase, and much longer duration

The atomic explosion not only produces high temperatures and a great shock wave but emits intense IR and UV radiation, with burning effects over several square miles, intense enough to char wood and cause third-degree burns. The heavy flux of neutrons emitted would suffice in itself to be deadly to individuals close to the explosion. The great amounts of gamma radiation can be deadly even to those who are some distance away from the explosion and indoors. Besides the radiation emitted at the instant of explosion, the blast scatters radioactive materials as *fallout* over wide areas, and much of it is carried hundreds of miles downwind. These materials decay, and this decay also yields radiation which is harmful. Alpha- and beta-particles emitted in the blast itself have only short ranges and present no problems, but those emitted by the fallout material make it a very serious hazard. They can do great damage if the material is breathed or ingested as dust, or introduced into wounds. Gamma radiation may also be dangerous over a wide area after the explosion

In view of the foregoing, considerable study has been devoted to effects of nuclear blasts on houses, industrial structures, underground piping and vaults, and to design of blast-resistant construction. For both military and civil defense against nuclear as well as chemical and biological weapons, much effort has been applied to developing means of detecting nuclear radiations and other effects of these weapons, design and construction of fallout shelters, and other defense measures

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Atomic Explosions. See under Atomic Energy

Atomic Gun (A-Gun) Shell. See under Atomic Weapons and Ammunition

Atomic Reactions. See under Atomic Energy

Atomic Rifle (Dave Crockett). See under Atomic Weapons and Ammunition

Atomic Rockets. See under Atomic Weapons and Ammunition

Atomic Shell. See under Atomic Weapons and Ammunition

Atomic Submarine. See under Atomic Weapons and Ammunition

Atomic Weapons and Ammunition* are a natural consequence of the tremendous power and destructiveness of nuclear explosions. *Atomic artillery* has been developed by adaptation of the fission process to projectile warheads. Development of an *atomic shell* for the 280-mm gun was announced in 1953 in newspaper releases. The process has been adapted for local tactical application to smaller shell fired from guns of regulation caliber, though with some loss of efficiency in the use of the fissionable material. Development of an *atomic rifle* called the "Davy Crockett", a weapon for use by the foot soldier, was developed about 1958, according to newspaper reports. This weapon is sufficiently portable to be mounted on a jeep or carried by several men. The warhead has considerable destructive force but the effective radius is so small that infantrymen firing it will not be endangered by the blast. Danger from radioactive fallout is minimized

Atomic rockets are obtainable through application of the nuclear reaction to propulsion rather than explosion. In 1952, construction of a *nuclear fission rocket* within ten years was predicted. A small atomic pile would heat hydrogen to a temperature of several thousand degrees and expel it through a nozzle. A chunk of fissionable uranium would serve as the fuel and, because the only gas in the exhaust stream would be hydrogen, the lightest possible molecule, the thrust of a nuclear rocket would be very large. The specific impulse might be around 900 sec, from three to four times that available from the most powerful chemical rocket fuels

Energy from nuclear fusion might also be used for propelling jets and rockets. Formidable difficulties would be encountered, however. For reactions occurring at ca 10^8 °K, such as those between the hydrogen isotopes, chamber pressures of around 100 atm could give rates of energy production comparable to those from the familiar chemical fuels. The rates would be much lower for heavier atoms, and increase with the square of the combustion pressure. The half-life of burnup in the most rapid reactions may drop to periods as short as those of chemical fuels. Despite the high temperatures, the radiated

*Written by C.G.Dunkle

energy can fall short of that produced, particulary with an inefficiently radiating material such as a gas like hydrogen which is easily ionized completely

On the other hand, the fraction of completely ionized particles in the gases undergoing nuclear combustion must not exceed a certain upper limit because the corresponding densities lie in the range of high vacua. Only if a considerable proportion of the particles remain un-ionized can the densities be kept high enough to make chamber diameters of several dozen meters sufficient. At temperatures and pressures of interest here, the mean free path is about 10^7 cm in a fully ionized plasma. Sänger discusses three types of nuclear rocket propulsion: (1)direct expansion of the gases in a pure nuclear rocket, (2)admixture of the gases with surrounding air in turbojets, ramjets etc, or with other gases in thermal atomic rockets, and (3) conversion of energy into photon gas in the photon rocket. He concludes the combustion pressures available to attain the required chamber loading in practice seem inadequate for the last two methods, except possibly in central power stations, marine propulsion plants, or in pure nuclear rockets of very large dimensions

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Atomization is the reduction of a substance to very small particles often approaching the size of atoms or molecules. In case of liquids, atomization is done by spraying. The same method may be applied to solids when they melt without decompn. Spraying may also be applied to solns of solids in volatile liquids, such as acetone, alcohol, etc. Solids may also be diminuted to very small size by the methods used for prepn of colloids such as by using a colloid mill or a homogenizer, as described in Ref 1. Another method for prepn of small solid particles is *electric atomization,* which is achieved by passing an electric arc between electrodes of pure metal in distilled water contained in a vessel made of practically insol material. This method produces a stable colloidal soln of the metal used as the electrodes (Ref 3)

Some atomized metals are used in expl and pyrotechnic compns. The following US specifications give tests and requirements for such metals:

a)JAN-A-289[Aluminum powder, flaked, grained or atomized (for use in ammunition)]
b)JAN-A-512 [Aluminum, powdered (grained or atomized) (from secondary metal)]
c)JAN-M-454 (Magnesium-aluminum alloy, powdered)
d)JAN-M-382A (Magnesium powder, for use in ammunition
e)JAN-M-476A (Manganese, powder, for use in ammunition)

Refs: 1)Kirk & Othmer **5** (1950), 705-6 2) Perry (1950), 839-40 3)Encycl Britannica **2** (1952), 651 3)E.Giffen & A.Muraszew, "The Atomization of Liquid Fuels," Wiley, NY(1953) 4)P.Barret, "Étude de Mécanisme de la Pulverisation des Solutions Electrolytiques par l'Étincelle Anodique," Magasin CTO, Paris (1953) 5)Kirk & Othmer **11** (1954), 714-21(Sprays) 6)W.R.Marshall, Jr, "Atomization and Spray Drying," AmInst-ChemEngrs, NY(1954) 7)W.R.Marshall, Jr, ChemEngProgr, Monograph Ser, No 2, 122 pp (1954) (Atomization and spray drying; a review) 8)R.Baillères & A.Avy, MSCE **39**, 113-18(1954) & CA **50**, 16195(1956) (A new method for fine atomizing of liquids) 9)C.C. Miesse, IEC **47**, 1690-1701(1955) & CA **50**, 9069(1956) (Correlation of experimental data on the disintegration of liquid jets) 10)R.D. Ingebo, "Atomization, Acceleration and Vaporization of Liquid Fuels," a paper reported in the "6th Symposium on Combustion," Reinhold, NY(1957), pp 684-7(11 refs) 11) R.P.Fraser, "Liquid Fuel Atomization," a paper reported in the 6th Symposium on Combustion," Reinhold, NY(1957), pp 687-701(29 refs)

Atoxylic Acid. See p-Aminophenylarsonic Acid, p A245

ATT (Attenuated) Ballistite. A ballistite, claimed to be flashless, was made in France from a mixt of CP_1 (NC with ca 13% N) & CP_2 (NC with ca 12% N) treated with NG and a non-volatile solvent (Ref 2, p A259)

An improved variety, called *superattenuated ballistite* and used during WWI by the French and Italians, contained CP_1 30, CP_2 30, NG 25 & DNT 15%. It was made without volatile solvent (Ref 1 & Ref 2, p 327)

Refs: 1)Pascal(1930), 227 2)Davis(1943), 259 & 327

Attasorb. A light-weight, free-flowing, highly adsorptive powd derived from the mineral attapulgite, a hydrated Mg-Al silicate. It is manufd by the Minerals & Chemicals Corp of America (Ref 1). It has been used as an anticaking agent for substances, such as AN(eg Attasorb 0.5, ZnO 0.2 and the rest AN) (Ref 2)

Refs: 1)O.T.Zimmerman & I.Lavine, "Supplement II to the 1953 Edition of Handbook of Materials Trade Names" Industrial Research Service Inc, Dover, New Hampshire (1957), 26 2)R.F.Muraca et al, "Analysis of Ammonium Nitrate," Jet Propulsion Lab Progress Rept No 20-311 (1957), 4

Attenuated Ballistite. See ATT

Attenuating Materials. Effects of Detonation Induction Distances in Gases is discussed by M.W.Evans et al, JApplPhys **26**, 1111-13 (1955) & CA **49**, 16436(1955)

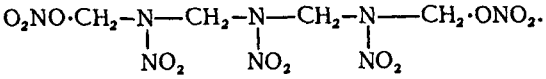
Attenuation is, in most general sense, reduction in concentration, density, effectiveness, etc. It is also a general term for the decrease in amplitude of waves of various kinds as they progress

Attenuation of shock waves in air as a function of distance was detd by R.G.Stoner & W.Bleakney, JApplPhys **19**, 670-8(1948) & CA **42**, 8475(1948). They measured the velocity of propagation produced on expln in air of chges TNT or 50/50 Pentolite 1.45 to 8 lb (either spherical or cylindrical in shape) and then calcd peak pressures by applying the velocity-pressure relation derived from the Rankine-Hugoniot equations

Attrition Mill is an apparatus contg two-sided knives which grind the material very fine by friction. Attrition mills are used at some expls plants, such as Wabash Ordnance Works. For drawings and description, see

Perry (1950), pp 1123 (Fig 24) and 1143-4 (Figs 48 & 49)

ATX or NBSX. Code letters designating 1,7-Dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane or 1,7-Dinitroxy-1,3,5,7-tetramethylene-2,4,6-trinitramine,



See under 1,7-Dihydroxy-1,3,5,7-tetramethylene-2,4,6-triamine and Derivatives

ATZ. See Aminotetrazole and Derivatives

Audemars Explosives, patented in 1855 in England, were prepd by nitrating purified barks of mulberry or of some other trees

Refs: 1)Cundill's Dictionary, MP **5**, 288 (1892) 2)Daniel (1902), 33

Aufschläger, Gustav (1853-1934). Ger engineer specializing in expls. He established in 1882 the Dynamitfabrik Muldenhütten bei Freiberg i S and in 1884 the Dresdener Dynamitfabrik. Since 1889, he was the Generaldirektor of the Dynamit AG, Hamburg. Numerous publications on expls; one of the editors of the Zeitschrift für das gesamte Schiess- und Sprengstoffwesen, abbreviated in our work as SS

Refs: 1)Anon, Angew Chem **36**, 65(1923) 2)F.Ebeling, SS **28**, 1-2(1933) 3)P.Müller, SS **29**, 127-8(1934)

Augendre Powder, also called **American Powder or White German Powder** (Poudre blanche allemande or Teutonite, in Fr). The original mixt patented in France in 1849 contained $KClO_3$ 48-50, K_4FeCy_6 25-29, & sugar 23-25%. It was intended as a replacement for black powder as a propellant, but proved to be too sensitive for this purpose. If found use, however, in primer compns and percussion caps. The modified compn: $KClO_3$ 41.7, K_4FeCy_6 25.0, sulfur (or sugar) 20.8, & charcoal 12.5% was proposed for use in electric primers. Its modifications were also used in Germany

Refs: 1)Cundill's Dictionary, MP **5**, 288-9 (1892) 2)Daniel (1902), 33 & 74 3)Marshall **1** (1917), 358 4)Davis (1943), 358

AUN. A cast double-base propellant described in conf "Propellant Manual," SPIA/M2(1959), Unit No 449

"Aunt Jemina" Explosive was a white powdery material developed in the US during WWII consisting of a mixt of flour with a white finely powdered HE which was not sufficiently toxic to hurt a person tasting it. The mixt was insensitive to handling and could be heated (baked) without explg it. It was intended to be shipped ostensibly as flour to neutral countries, where it could be used for purposes of sabotage

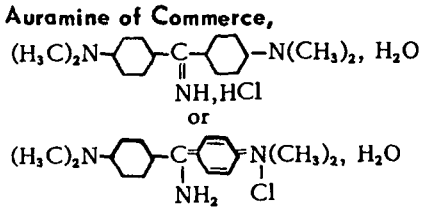
Ref: W.A.Noyes, "Chemistry" (Science in WWII, OSRD), Little, Brown & Co, Boston (1948), 51

AURAMINE AND DERIVATIVES

Auramine; Auraminebase or Tetramethyl-p_2-diaminoimino-benzophenone (called in Beil 4,4'-Bis-dimethylamino-benzophenon), $(CH_3)_2N{\cdot}C_6H_4{\cdot}C(:NH){\cdot}C_6H_4{\cdot}N(CH_3)_2$, mw 267.36, N 15.72 %. Col lfts mp 136°; insol in w; sol in alc & in eth. Can be prepd by treating its hydrochloride (see below) in aq alc with dil NaOH, as described in Ref 3, or by heating 4,4'-bis-dimethylamino-diphenylmethane with sulfur followed by treatment with ammonia (Ref 1, p 92)

Refs: 1)Beil **14**, 91, (392) & [58] 2) C.Graebe, Ber **20**, 3260-9(1887) 3)L.Semper, Ann **381**, 235(1911) 4)H.E.Fierz-David & L.Blangey, "Grundlegende Operationen der Farbenchemie," Springer, Wien(1943), 282 5)Hackh's(1944), p 87

Auramine Hydrochloride; Auramine O or Auramine of Commerce,



Yel powd; mp 267° for anhyd salt (Ref 2, p 3264); sol in alc; diffc sol in w. Can be prepd by heating tetramethyldiaminobenzophenone with NH_4Cl in presence of Zn(Ref 2)

Shidlovskii (Ref 5, pp 243-245) gives for Auramine O: mp 216-18°, d ~1.3, latent heat of vaporization 26.8 kcal/mol or 110 kcal/kg and vapor pressure 2.0 mm Hg at 260°. Its vapor is yel with a definite grn tinge. If it is desired to obtain a smoke of pure yel color, some Chrysoidine (brn dye) is mixed with Auramine O

Auramine hydrochloride (Auramine O) in various colored smoke pyrotechnic compns. Davis (Ref 3) gives yel and green smoke-signal grenade compns used by the US planes during WWI and Izzo (Ref 4) gives yel and orange smoke compns used by the US CWS and by some European countries. Shidlovskii (Ref 5, p 252) lists several colored smoke compns contg Auramine O, which seem to be of Russian origin

TABLE

Some US Smoke Compositions with Auramine O

Auramine O	34	15	38.0	16.4	11.7
$KClO_3$	33	33	24.4	25.9	25.9
$NaHCO_3$	–	–	28.5	23.0	24.0
Lactose	24	26	–		
Sulfur	–	–	9.1	10.1	10.1
Chrysoidine	9	–	–	–	–
Indigo	–	26	–	–	–
α-Amino-anthraquinone	–	–	–	24.6	–
1,4-Di-p-tolui-dineanthraquinone	–	–	–	–	28.3
Color of smoke	Yel	Grn	Yel	Orn	Grn
Refs	3	3	4	4	4

TABLE

Some European Smoke Compositions with Auramine O

Auramine O	40	41	34	27	10	15
$KClO_3$	30	34	33	34	30	33
Lactose	20	25	24	25	20	26
Chrysoidine	10	–	9	–	–	–
Indigo	–	–	–	14	20	26
Oxalate of malachite-green	–	–	–	–	20	–
Color of smoke	Yel	Yel	Yel	Grn	Grn	Grn
Refs	4&5	5	4	5	4	4

Auramine hydrochloride intended for use in some US colored smoke compns must be tested according to specification MIL-A-3664 and comply with the following requirements:
a)Purity – not less than 86.0% when tested by both chemical and spectrophotometric analysis specified in 4.4.2
b)Moisture – not more than 3.5% when tested as specified in 4.4.3
c)Particle size – not less than 99% through No 60 US Std Sieve not less than 90% through No 100 sieve, and not less than 40% through No 200 sieve when tested as specified in 4.4.4
d)Apparent density – 0.35 ± 0.15 when tested as specified in 4.4.5
e)Performance. The material fired in yellow smoke grenades shall burn without flaming and shall emit a continuous cloud of yel smoke for a period of 70 ± 20 secs when tested as specified in 4.4.6. The smoke shall be distinguishable as to color against a contrasting color background, at a distance of 10000 ft on a clear day

Methods of detection of auramine are described in Ref 6

Refs: 1)Beil **14**, 92-3, (392) & [58] 2)C. Graebe, Ber **20**, 3261-2 & 3264(1887) 3) Davis (1943), 123 4)Izzo, Pirotecnia(1950), 235-6 5)Shidlovskii, Pirotekhnika(1954), 243 & 247 6)OrgAnalysis, Interscience, v 3(1956), 190

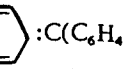
Auramine, Azido– $C_{17}H_{20}N_6$ and *Diazido-* $C_{17}H_{19}N_9$ *Derivatives* were not found in Beil or CA through 1956

Mono-, Di-, Tri- and Tetranitroauramines were not found in Beil or CA through 1956

Aurantia. See 2,2',4,4',6,6'-Hexanitrodiphenylamine, Ammonium Salt, under Diphenylamine and Derivatives

AURINE AND DERIVATIVES

Aurine;Corallin or 4,4'-Dihydroxyfuchsone, called in CA **4-[Bis(p-hydroxyphenyl) methylene]-2,5-cyclohexadien-1-one** and in Beil 4,4'-Dioxy-fuchson; Pararosolsäure or p-Chinon-mono-[bis-(4-oxy-phenyl)-metid],

O:  :C(C_6H_4·OH)$_2$, mw 290.30; deep red (garnet-like), rhmb crysts; mp 308-10°(dec); insol in w or benz; sol in alc or eth. Was first prepd in 1878 by heating a mixt of phenol oxalic and sulfuric acids (Ref 2). Many other methods of prepn are listed in Beil (Ref 1) and in Merck Index (Ref 3)

Aurine is used in small amt (ca 0.25%) as a component of EC Propellant for Blank Cartridges and Fragmentation Hand Grenades. A gravimetric method of aurine detn in EC propellant is described in US Army specification No 50-13-8B, while a colorimetric method is given in US Military Standard MIL-STD-286A(1960)

A historical discussion on the nomenclature of aurine is given in Beil **8**, 361

Refs: 1)Beil **8**, 361, (671) & [417] 2)C. Zulkowsky, Ann **194**, 109 & 122 (1878); **202**, 179(1880); Monatsh **16**, 358(1895) 3) Merck Index (1952), 109

Aurine Perchlorate, $C_{19}H_{14}O_3 + HClO_4 + H_2O$; red crysts, forming on prolonged heating an anhydrous salt. Was first prepd by Hofmann (Ref 2) by treating aurin in AcOH with concd $HClO_4$. It was also obtained by Pfeiffer (Ref 3)

Refs: 1)Beil **8**, (671) & 418 2)K.A.Hofmann et al, Ber **43**, 184(1910) 3)P.Pfeiffer, Ann **412**, 333 (1917)

Aurine, Azido- $C_{19}H_{13}N_3O_3$ and *Diazido-* $C_{19}H_{12}N_6O_3$ *Derivatives* were not found in Beil or CA through 1956

Mono-, Di- and Trinitroaurines were not found in Beil or CA through 1956

Tetranitroaurine, $C_{19}H_{10}(NO_2)_4O_3$, mw 470.30, N 11.91%; brownish microscopic needles; mp ca 140° when carefully heated. It is a mild explosive, sol in alc, nearly insol in w, eth, chlf or benz. Was prepd by treating 1 part of powdered aurin with 4 ps nitric acid (d 1.51) in the cold. The positions of the nitro groups are not given

Its **silver salt,** $C_{19}H_9(NO_2)_4O_3Ag$ is a mild explosive

Refs: 1)Beil **8**, 365 2)E.Ackermann, Ber **17**, 1625-6(1884) 3)No later data was found in Beil or CA through 1956

Aurous Acetylide. See Gold Acetylides under Acetylides

Aurum Fulminans or Aurum Tonitruans. Old Latin names for Fulminating Gold (qv). Ger names,Knallgold and obsolete "Donnergewaltig Gold"; Or fulminant, in Fr

Austin Powder Co, Cleveland, Ohio was established in 1883 under the name of Austin Black Powder Mills by L. Austin(1817-1887) and his brothers (Ref 1). The plant manufactures mining expls among them "Austin Red-D-Gel" (gelatinous permissible expls) and "Austin Red Diamond" (nongelatinous permissible expl) (Ref 2)

Refs: 1)VanGelder & Schlatter (1927), 265 2)Bebie(1943), 30

Australian Ammunition, Explosives and Weapons. No information at our disposal

Australian Warplants. No information

Austrian Ammonal or Austrian Military Explosive. See under Ammonals

Austrian Ammunition, Explosives and Weapons. The items used during WWII were practically the same as used by the Germans [See PATR 2510(1958)]. Some of the Austrian weapons were of WWI vintage, as for example: 47/32 mm Antitank Gun Boeler; 7.65 cm FK 5/8(ö); 7.65 cm FK 17(ö); 8 cm lFK 18(ö); 10 cm lFH 14(ö); 10 cm lFH 14/37(ö) and 10 cm GebH 16(ö). No information about current items

Abbreviations: **F** – Feld(field); **Geb** – Gebirg(mountain); **H** – Haubitze(howitzer); **K** – Kanone(cannon); **l** – leicht(light); **(ö)** – österreichisch(Austrian)

Austrian Propellants of WWI. Compositions of the following two propellants were detd in Russia by Dr M.M.Kostevich: a)NC (ca 13.25% N) 42, NG 40, Ba nitrate 16 & vaselin 2% b)NC (ca 12.1% N) 67 NG 26 & vaselin with diethylphthalate and volatiles 7.0%

Ref: M.M.Kostevitch, Buenos Aires; private communication (1955)

Austrian Warplants, Arsenals, etc. Dr W.J. Lohninger, formerly of Austria and now at Picatinny Arsenal, remembers the following war establishments operating in Austria during WWII: a)Govt Explosives and Ammunition Plant at Wöllersdorf b)Govt Arsenal, Wien c)Govt Proving Ground Bruck und Leitha d)Govt Proving Ground Steinfeld (Wiener Neustadt) e)AG Dynamit Nobel, Wien, Plant at St Lambrecht f)Donnau Chemie AG, Moosbierbaum g)Eisenwerke Oberdonnau, Linz h)Gebrüder Böhler AG with Plants at Deutschendorf, Kopfenberg, St Egidy, St Marein, Waidhofen and Wien i)Krupp Metallware Werk, Berndorf (Wien) j)Metallwerke Plansee Reutte, Tyrol k) Nibelungenwerke, St Valentin l)Steyr-Daimler-Puch AG, Wien with Plants at Wien, Steyr, Linz and Graz

No information at our disposal about the current war establishments

Authorized or Acceptable Explosives are those which conform to certain regulations of transport, safety in handling, etc. They are to be distinguished from permissible (Brit permitted) expls. Both kinds authorized and permissible expls belong to the class of safety expls

Ref: Davis(1943), 347

Autoclaves are thick-walled steel cylindrical vessels, designed to withstand very high pressures. They are used for prepn of some products and for the study of reactions at high pressure and high temp. The contents of autoclaves may be agitated either by rocking the autoclave or by a mechanical agitator, such as turbine type, placed inside the autoclave. Heating or cooling may be accomplished by a jacket (with circulating liquid), by internal coils, or by electrical resistance wires (or strips)

Refs: 1)Thorpe 1(1937), 550-8 2)D.M. Newitt, "Design of High Pressure Plant and Properties of Fluids under High Pressures," Clarendon Press, Oxford (1940) 3)D.B. Gooch, IEC **35**, 927-46(1943) (Description of various autoclaves for high-pressure reactions) 4)Riegel, Chem Machinery (1944), 484-94 5)Giua, Dizionario (1948), 592-3 6)Perry (1950), 1256-7 7)A.H.Thomas Catalog (1950), 56-61 & 231 8)K.Thormann, ChemIngTech **24**, 689-92(1952) (Pressure and vacuum technique) 9)Kirk & Othmer **11** (1953), 102 10)E.Kuss ChemIngTech **28**, 141-52(1956) (Review on autoclaves, etc) (62 refs)

Autofrettage; Self-Hooping or Cold-Working (Autofrettage in Fr; Kaltrecken in Ger; Samoskrepleniye Stvola, in Rus). Autofrettage is a process for manufg gun barrels, in which the inner surface layers of a plain tube are initially stressed by expansion using high hydraulic pressure beyond the elastic limit which would be reached by the explosion of any charge to be used subsequently in the gun. In this process, the inside diam of the gun can be permanently enlarged ca 6% while the outside diam enlarges ca 1%

Autofrettage is also used for strengthening the walls of tubes, cylinders, pressure vessels, etc and it is claimed that it nearly doubles the elastic strength of the walls

Refs: 1)A.E.Macrae, "Overstrain of Metals and Its Application to the Autofrettage Process of Cylinder and Gun Construction," HMSO, London(1932) 2)L.Gentil, MAF **15**, 313-45(1936) 3)G.de la Chaise, MAF **15**, 811-940(1936) 4)E.Bergeron, MAF **15**, 941-48(1936) 5)P.Malavel, MAF **15**, 347-64 & 1003-1061(1915) 6)T.J.Hayes, "Elements of Ordnance," Wiley, NY(1938), 164-7 7) T.Lyman, Edit, "Metals Handbook," Amer Soc for Metals, Cleveland, Ohio(1948), 2 & 250 8)Perry(1950), 1241 9)Merriam-Webster's Unabridged Dictionary(1951), 186 10)J.G.Henderson & J.M.Bates, "Metallurgical Dictionary," Reinhold,NY(1953), 28

Autogenous Ignition; Autoignition; Self-

ignition;Spontaneous Ignition. See under Ignition

Autoignition Temperatures of Organic and Inorganic Powders in Air was determined by D.Costa et al, Chimica e Industria(Milano), **34**, 645-54(1952); CA **47**, 12817(1953)

Automatic Arms. See under Automatic Weapons

Automatic Computors and Calculators. Computing mechanisms are of two distinct types: (a)arithmetical or *digital* computors and (b) continuously acting (geometrical) or *analog* computors that range from simple cams and levers to enormously complex devices. The latter have been used for the direction of naval and antiaircraft gunfire (Ref 1, p 1), and both types of computers assist materially in ballistic and thermochemical calculations and other means of evaluating effects of propellants and explosives

Following is a partial list on automatic computing machines:
1)A.Svoboda & H.M.James, "Computing Mechanisms and Linkages," McGraw-Hill, NY(1948) 2)A.D.Booth & K.H.Booth, "Automatic Digital Calculators," Academic Press, NY(1953) 3)W.Soroka, "Analog Methods in Computation and Simulation," McGraw-Hill, NY(1954) 4)C.A.A.Wacs, "Introduction to Electronic Analogue Computers," Pergamon Press, London(1955) 5)N.Chapin, "An Introduction to Automatic Computers," Technology Center,Chicago, Ill(1955) 6)US National Bureau of Standards, "Computer Development(SEAC & DYSEAC) at the NBS," Govt PrntgOff, Washington (1955) 7)E.C.Berkeley & L.Wainwright, "Computers, Their Operation and Application," Reinhold, NY(1956) 8)C.L. Johnson, "Analog Computer Techniques," McGraw-Hill, NY(1956) 9)G.A.Korn & Th. M.Korn, "Electronic Analog Computers," McGraw-Hill,NY(1956) 10)M.V.Wilkes, "Automatic Digital Computers," Wiley,NY (1956) 11)R.K.Livesley, "An Introduction to Automatic Digital Computers," UnivPress, Cambridge, Mass(1957) 12)W.J.Karplus, "Analog Simulation Solution of Field Problems," McGraw-Hill, NY(1958) 13)F.L. Alt, "Electronic Digital Computers," Acadamic Press, NY(1958) 14)E.M. McCormick "Digital Computer Primer," McGraw-Hill, NY(1959) 15)J.N.Warfield, "Introduction to Electronic Analog Computer," Prentice-Hall, Englewood Cliffs, NJ(1959) 16)S.Williams, "Digital Computing Systems," McGraw-Hill,NY(1959) 17)S.H.Hollingdale, "High Speed Computing," The English Universities Press, London (1959)

Automatic Control. See under Automation

Automatic Feed Mechanism. A mechanical arrangement in an automatic weapon which repeatedly inserts fresh cartridges in position for firing

Ref: Same as under Automatic Weapon(qv)

Automatic (Self-Acting) Weapon. A weapon that acts by itself without application of power from an outside source. This may be accomplished either by employing propellent gas pressure, or force of recoil and mechanical spring action for ejecting the empty cartridge case after the first shot, loading the next cartridge from the magazine, firing and ejecting this cartridge and repeating the above cycle as long as the firing mechanism is held in the proper position and there is ammunition in the magazine. Machine-gun, heavy and light are typical examples of automatic weapons

The *semi-automatic* is similar to the automatic but the trigger must be pulled for each round fired. Many automatic weapons are designed to permit semi-automatic fire

Besides machine-guns there exist automatic and semi-automatic small arms, such as machine-rifles and machine-pistols

The so-called *contact mine* belongs also to the class of automatic weapons

Refs: 1)Hayes(1938),630 2)M.M.Johnson, Jr & Ch.T.Haven, "Automatic Weapons, Their History, Development and Use," W.Morrow,

NY(1943) 3)M.M.Johnson, Jr "Rifles and Machine Guns," W.Morrow,NY(1944), 118-223 4)M.M.Johnson, Jr & Ch.T.Haven," Automatic Weapons of the World," W.Morrow, NY(1946) 5)G.M.Chinn, "The Machine Gun," Bur of Ordn, Dept of the Navy, US Govt Prtg Off, Washington, DC (vol 1(1951)) (Vols 2 & 3 are conf) 6)G.M.Chinn, "The Machine Gun," US GPO, Washington, vol 4(1955) 7) W.H.B.Smith, "Small Arms of the World," MilServicePubgCo, Harrisburg, Pa(1955), 81,193,196,204 & 208 8)G.Baillard, MAF **30**, 383-509(1956) Cinématique des armes automatiques) 9)A.B.Shilling, PicArsn; private communication(1960)

Automation; Automatic Control; Automatic Process Control. Automation is, according to definition given in Ref 19, the technique of improving human productivity in the processing of materials energy and information in utilizing in various degrees, elements of automatic control and of automatically executed product programming. Automatic control consists, according to the definition given by Perry (Ref 6), of maintaining within limits, or altering in predetermined manner, the energy and sometimes the material balance of matter undergoing treatment in a process. The process is controlled automatically by measuring the state of a selected process variable, either continuously or at frequent intervals, and then correcting the imput of energy or material to maintain the value of the variable within acceptable limits

For more information on this subject, consult some of the following references

1)J.C.Peters & Th.R.Olive,ChemMetEngrg **50**, 98-107 (May 1943) (Fundamental principles of automatic control) 2)Editorial Staff Review, Ibid **50**, 108-24 (May 1943) (Instruments for measuring and controlling process variables) 3)Editorial Staff Review, Ibid **50**, 125(May 1943) (Automatic control therminology) 4)E.S.Smith, "Automatic Control Engineering," McGraw-Hill,NY(1944) 5) D.P.Eckman, "Principles of Industrial Process Control," Wiley,NY(1945) 6)Perry (1950), 1309-40(Fundamentals of automatic control) 6)P.E.Nixon, "Principles of Automatic Controls," Prentice-Hall, NY (1953) 7)I.Flügge-Lotz, "Discontinuous Automatic Control." PrincetonUnivPress, Princeton,NJ(1953) 8)M.H.LaJoy, "Industrial Automatic Controls," Prentice-Hall,NY(1954) 9)S.Furman, "A Selected Bibliograph on Automation," Special Libraries Association,NY(1954) 10)A.J.Young, "An Introduction to Process Control System Design," Longmans, Green, London(1955) 11)W.F.Wade & E.N.Kemler, "Automatic Control Bibliography" Spring Park, Minnesota(1955) 12)D.O.Woodbury, "The Full Story of Automation," Harcourt,Brace,NY (1956) 13)N.H.Ceglske, "Automatic Process Control for Chemical Engineers," Wiley, NY(1956) 14)Magnus Pyke "Automation, Its Purpose and Future," Philosophical Library, NY(1957) 15)Kirk & Othmer, 1st Supplement (1957), 88-103(20 refs) 16)W.G. Holzbock, Automatic Control: Principles and Practice," Reinhold,NY(1958) 17)R.L. Cosgriff, "Nonlinear Control System," McGraw-Hill,NY(1958) 18)D.P.Eckman, "Automatic Process Control," Wiley,NY (1958) 19)G.Merrill, Edit, "Dictionary of Guided Missiles and Space Flight," VanNostrand,NY(1959),69 20)Russian Publications on Automation and Automatic Control, translated into English, may be obtained from the Consultants Bureau, Inc, 227 W 17th St, New York 11, NY

Autopropulsion or Propulsion par réaction. French term indicating reaction engines carrying as a source of energy not only a combustible substance (carburant) but also an oxidizer(comburant). The fuels used in such engines are called *propergols* and the devices that utilize the principles of autopropulsion are called "engins autopropulsés." The Ger weapon V-2 was driven by such a motor (See also Jet Propulsion and Reaction Motors)

Refs (French): 1)P.Blanc,MAF **20**, 877-1004 (1946) & **21**, 885-1006(1947) 2)J.J.Barré, MAF **22**, 323-76(1948) 3)J.Oudin, MAF **22**, 379-412(1948) 4)J.Fauveau, MP **31**, 287-305

(1949) 5)P.Blanc, MAF **25**, 103-16(1951) 6)P.Carrière, MAF **25**, 253-360(1951) 7) E.Roth, MAF **30**, 551-5(1956) 8)H.Moreu, MAF **32**, 405-35(1958)

Autopropulsive Devices. See Jet Propulsion and Reaction Motors

Autoxidation is a low-temperature oxidation of a substance (usually a liquid) by the atmosphere without the aid of other oxidizing agents, but requiring, in many cases, the presence of inductors for initiation of oxidation

For detailed description of mechanism of autoxidation see Refs 1,3,5&6

The process of autoxidation was used during WWII by the Germans for manuf of some chemicals, among them hydrogen peroxide (Ref 2)

Evans (Ref 4) discussed expln hazards of autoxidized solvents

Refs: 1)J.L.Bolland, Quarterly Revs **3**, 1-21(1949) 2)Kirk & Othmer **7**(1951), 735 and **9**(1952),677 3)L.Bateman, Quarterly Revs **8**, 147-67(1954) 4)A.G.Evans, JRoyInst-Chem **80**, 386-9(1956) 5)H.E.De La Mare & W.A.Vaughan, JChemEduc **34**, 64-70(1957) 6)G.A.Russell, JChemEduc **36**, 111-18(1959)

Autoxygen Company Process of Nitration. The Autoxygen Company of New York, NY, during WW II, proposed a method of nitration of substances which it claimed would eliminate the use of sulfuric acid, reduce the proportion of nitric acid to material nitrated, and eliminate the necessity for reworking spent acid

In this process, the material to be nitrated is dissolved or suspended in an inert solvent which form azeotropes with water (such as petr eth, CCl_4, etc) and treated with concd nitric acid under such conditions that water formed during nitration is removed as an azeotrope. Dilution of nitric acid is thus avoided. The azeotrope which distills at the temp of nitration is cooled by condensation and the solvent minus the water is continuously returned to the nitrating vessel. After the nitration,the solvent is removed by distillation, leaving as residue the nitrated product plus unused nitric acid

This method was investigated at PicArsn, Dover, NJ and found to have only a very limited application (Ref 1). The azeotropic method seems to be suitable for nitration of benzene (Ref 2)

Refs: 1)H.A.Aaronson,PATR 1164 & 1209 (1942) 2)D.F.Othmer et al, IEC **34**, 286-91 (1942)

Autozone. G.M.Schwab, Umschau **1922**, 538-9 & CA **17**, 468(1923) coined this word for an isomer of ozone, which does not seem to exist. The word "autozone" could not be found in Mellor's, Kirk & Othmer's,Ulmann's, Hackh's, CondChemDictionary nor CA's indices except the one referring to **17**, p 468

AUV. A cast double-base propellant. It is described in conf "Propellants Manual," SPIA/M2, (1959), Unit No 451

Auxiliary Booster. See under Booster

Auxoexplose or Auxoplosophore; Explosophore or Plosophore. According to Lothrop & Handrick (Ref 3), Pletz (Ref 1) proposed the theory of *explosophores* and *auxoexploses* analogous to the Witt theory of chromophores & auxochromes and to the Ehrlich theory of toxophores and autotoxes

According to the Pletz theory the expl props of any given substance depend upon the presence of definite structural groupings called explosophores, while the auxo-exploses modify or fortify the expl props brought about by explosophores

On the basis of this theory Pletz examined eleven classes of organic compds and subdivided all expls into the following eight classes contg explosophores:

a)$-NO_2$ and $-ONO_2$ groups connected to inorg or org radicals [eg: HNO_3, $C(NO_2)_2$, $C_3H_5(ONO_2)_3$ & $C_6H_3(NO_2)_3$]

b)$-N=N-$ and $-N-N$ [eg: $Pb(N_3)_2$ & CH_3N_3]

c)$-NX_2$ group (eg: NCl_3 & $RNCl_2$)

d)C=N–group[eg:HONC & $Hg(ONC)_2$]
e)$-OClO_2$ & $-OClO_3$ groups(eg:$KClO_3$, $KClO_4$ and org chlorates and perchlorates)
f)–O–O– & –O–O–O–groups(eg: peroxides & ozonides)
g)–C≡C–group(eg: acetylene or its derivs)
h)A metal atom connected by an unstable bond to the carbon of certain org radicals (eg: org compds of Hg, Tl & Pb)

While the arrangement of Pletz embraced the whole expl field in a purely empirical fashion the distinction betw the terms explosophore and auxoexplose was not clearly defined. This was done later in the US, mostly under the direction of Dr A.H.Blatt as a wartime project. The object of this work has been the collation and classification of information through relationships which exist betw org structure and the expl props of HE's which are of interest in military applications

In the course of this work the term *plosophore* was coined (Ref 2 & Ref 3, pp 423-4) for a group of atoms which on substitution into a hydrocarbon is capable of forming an expl compd. Inspection of the groups which can function in this way indicated that there are two classes of plosophores, differing sharply in effectiveness and consistency in producing power and hence they were called *primary* and *secondary plosophores*

Primary plosophores include the following groups: nitrate ester, aromatic nitro, aliphatic nitro, and nitramines; while the secondary plosophores include the remainder, such as azo, azido, nitroso, peroxide, fulminate, chlorate, bromate, perchlorate, perbromate, etc groups

It was also established that primary plosophores are responsible for high power and brisance of expls and these are at the maximum in compds whose oxygen balance to CO_2 (see under Available Oxygen) is favorable (close to zero). The relationship betw power and oxygen balance vanishes when one considers *secondary plosphores* and further, with few exceptions, secondary plosophores do not, as a rule, exceed primary plosophores as power-producing groups. Although the secondary plosophores cannot be recommended in the synthesis of powerful HE's, they often impart desirable qualities of another kind, for example, in forming imitiating expls [eg $Pb(N_3)_2$, $Hg(ONC)_2$]

When two or more different primary plosophores are present in a single molecule, the compd is called *hybrid* (eg: nitrate & aliphatic nitro, nitrate & aromatic nitro, nitramine & aromatic nitro). It was established that hybrids are as powerful (or more) as pure types, although they tend to exhibit somewhat greater variability. This property of hybrids is of importance, because it extends enormously the synthetic possibilities for expls

A very common feature of many expls is the presence in them of a large variety of substituent groups which are not plosophoric, since they do not in themselves produce expl molecules, but which may be expected to alter the expl props in the same way as an auxochromic group is found to vary the intensity or shade of a dye. Such substituents are designated *auxoplosive*. To these belong hydroxyl, carbonyl, chloride, sulfide, ether, amino, etc groups. Very often their presence affects the oxygen balance favorably, but with hardly an exception auxoplosive groups are deterimental to power whether or not they improve oxygen balance. The chief justification for the presence of such groups in powerful expls is expediency in synthesis

Refs: 1)V.Pletz,ZhurObshchKhim **5**, 173 (1935) 2)S.R.Brinkley & E.B.Wilson, Jr, OSRD Rept No 905(1942) 3)W.C.Lothrop & G.R.Handrick, ChemRevs **44**, 419-45(1949)

Auxoplosive Group, Same as Auxoexplose or Auxoexplosophore

"Available Diphenylamine" is the total percentage of DPhA and derivs available for stabilization of NC propellants. The term is confined to the product obtained by the

soda-distillation method (see Available Stabilizer) and consists of residual DPhA of the propellant, plus the N-nitrosodiphenylamine, converted by the soda-distillation treatment to DPhA, and a small amt of 2-nitrodiphenylamine

Ref: E.F.Reese, private communication(1960)

Available Energy of Explosives. See under Power of Explosives

Available Ethyl Centralite. See under Available Stabilizer

Available HNO_3 in mixed acids is equal to actual nitric plus half the HNO_3 equivalent of the nitrogen oxides, calcd as NO_2 (see also pA81, under Acidity of Acids)

Available Oxygen; Active Oxygen; Effective Oxygen; Oxygen Balance to CO_2 and to CO. *Available oxygen* is oxygen which can be utilized as an oxidizer. The amt of such oxygen depends on the conditions of reaction. For instance, the amt of available oxygen in $KMnO_4$ is five for each $2KMnO_4$ in an acidic medium and only three in an alkaline medium. Methods of detn of available oxygen are given in Refs 1 & 5

Active oxygen is oxygen which is generally liberated in the free state, expecially in the presence of small amts of alkali, heavy metals, etc. Such oxygen is found in all peroxy compds. Two methods for detn of active oxygen in peroxides are given in Refs 2 & 3(See also p A101)

In many cases available and active oxygen are identical

Oxygen balance to CO_2 (OB to CO_2) for an organic compd, is the percentage of oxygen required for complete conversion of the carbon to CO_2, the hydrogen to H_2O and the nitrogen to N_2. For any compd contg x atoms of C, y atoms of H, and z atoms of O the OB to CO_2 = $-[1600(2x+y/2-z)]/\text{MolWt}$. Thus an expl having perfect OB to CO_2 has zero balance (eg NGc), one lacking sufficient O has a negative balance (eg TNT), and one contg excess O has a positive balance (eg Amm nitroform). It is noted that with very few exceptions expls have decidedly negative OB to CO_2

The above method of computation makes no distinction betw oxygen already bound to carbon or hydrogen and that bound to nitrogen. The latter is termed in Ref 4, the *effective oxygen.* It differs from other oxygens in that it is not yet reduced but is still available for combustion

Oxygen balance to CO (OB to CO) of an organic compd, is the percentage of oxygen required for complete conversion of C to CO, H to H_2O, and N to N_2. It is calcd from the formula $-[1600(x + y/2 - z)]/\text{MolWt}$. This calcn is required for compds used or intended for use in propellants or in industrial expls of low brisance, acting by heaving action

Refs: 1)W.W.Scott & N.H.Furman, "Standard Methods of Chemical Analysis," Van Nostrand, NY(1939), 675 [The detn of available oxygen in metal peroxides is sometimes required on account of their use as oxidizing agents in various processes, such as the use of MnO_2 in manuf of chlorine from HCl. There are two methods for such analysis: a)Direct method consists of treating a weighed amt of a peroxide by a measured amt of a std reducing agent, such as ferrous sulfate: $MnO_2 + 2FeSO_4 + 2H_2SO_4 \rightarrow MnSO_4 + Fe_2(SO_4)_3 + 2H_2O$, followed by titrating with std $KMnO_4$ the excess of reducing agent. This gives the exact amt of reducing agent required by the peroxide b) Indirect method consists of treating a peroxide with HCl and KI, followed by titration of liberated iodine with std thiosulfate: $MnO_2 + 4HCl \rightarrow MnCl_2 + 2H_2O + Cl_2^\circ$; $Cl_2^\circ + 2KI \rightarrow 2KCl + I_2^\circ$ and $I_2^\circ + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$] 2)Lucidol Division, Novadel – Agene Corp, Buffalo, NY, Bulletin No 9 (1948) (Active oxygen in a peroxide is detd by treating its acetonic soln with aq KI, followed by titration of liberated iodine with N/10 $Na_2S_2O_3$) 3)R.Cliegee et al, Ann **565**, 16(1949) (Active oxygen in a peroxide is

detd by adding it to a soln of NaI in glacial AcOH, previously freed from oxygen by bubbling pure CO_2 gas. After allowing to stand for 30 mins, the soln is dild with O_2-free water and the liberated iodine is titrated with N/10 $Na_2S_2O_3$) 4)W.G.Lothrop & G.R.Handrick, ChemRevs **44**, 421(1949) (The relationship between performance and constitution of pure organic compounds) 5)H.H.Willard, N.H.Furman & C.E.Bricken, "Elements of Quantitative Analysis," Van Nostrand,NY(1956) 234-5(Available oxygen in MnO_2 is detd by heating a sample in dil sulfuric acid soln with a known wt of Na oxalate until the dioxide has dissolved: $MnO_2 + H_2C_2O_4 + 2H^+ \rightarrow Mn^{++} + 2CO_2 + 2H_2O$. The excess of oxalate is then titrated with 0.1N $KMnO_4$ soln)

Available Stabilizer (Diphenylamine and/or Ethyl Centralite) in Aged or Stored Propellants is determined by Method 217.2(T) listed in specification MIL-STD-286

In this method ca 5g sample of propellant (previously cut into small pieces as described in Method 509.3 of MIL-STD-286) is placed in a 1000 ml balloon flask contg 100 ml of distd w and 100 ml of 30% NaOH soln. After connecting the flask to a condenser, adapter, receiver (a 750 ml Erlen fl contg 25 ml distd w), and a steam generator, the mixture is steam distilled until 350-400 ml of distillate is collected in the receiver. After adding to the contents of receiver 5g NaCl they are transferred to a separatory funnel where the stabilizers are extracted with ether

If only DPhA or only Et centralite is present the ether is evaporated with a current of dry air and the contents of stabilizer detd by a standard volumetric bromination described in Method 201.1 (in case of DPhA) or in Method 202.2 (in case of Et Cent) of spec MIL-STD-286

If both stabilizers are present, their contents are detd by the volumetric bromination procedure described in Method 217.2(T) of spec MIL-STD-286

Average Particle Size Measurements. See under Particle Size Measurements

Aviation Gasoline-Explosive Characteristics of. Studies of the explosiveness of gasoline air mixts showed that: a)Ignition temps increased and expl press decreased as the fuel/air ratio varied towards the limits of flammability b)Ign temp increased slightly and the expl press decreased as RH increased c)Ign temp increased with increasing air vel & increasing altitude and the expl press decreased rapidly at alts above 20000 ft d) Both ign temp and expl press decreased rapidly at low ambient temp

Ref: D.J.Babic & H.G.White, Petroleum Engr **28**, No 7, C41-4(1956) & CA **50**, 15055 (1956)

Avigliana 3 or Nitramite. An ammonal-type expl contg AN 71-72, Al 22 & paraffin or pitch 7-6%

Refs: 1)Allied & Enemy Explosives(1946), 84 2)Giua, Dizionario **2**(1949), 165

Avigliana Dynamite Factory, located at Avigliana, near Torino, Italy is one of the largest and best equipped factories in Europe. It was founded in 1872. See Nobel Società Generale di Esplosivi e Munizioni (Nobel-SGEM) under Italian Warplants

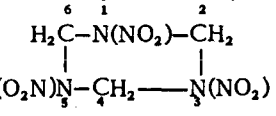
Axite. A Brit double-base smokeless, sporting propellant manufd by Kynoch Ltd. It is essentially *Cordite MD II* (GC 65, NG 30 & MJ 5%) to which 2% of K nitrate has been added (Ref 2). Marshall (Ref 1) gives its compn as: GC 63.1, NG 29.7, MJ with oil 5.1, K nitrate 1.9 & volatile matter 0.2%

Refs: 1)Marshall **1** (1917), 308 2)Barnett (1919), 78

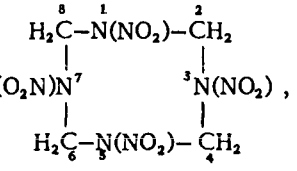
Az. Fr abbrn for azote (nitrogen)

Aza(Nomenclature). The name *aza* is now applied to hetero nitrogen atoms occurring in a ring. In this system of nomenclature a hetero oxygen is called *oxa* and sulfur *thia*. The compd commonly known as cyclonite,

RDX or cyclotrimethylenetrinitramine,

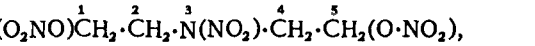


may be called 1,3,5-trinitro-1,3,5-triazacyclohexane, and the compd known as cyclotetramethylenetetranitramine or HMX,

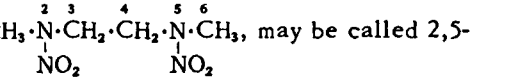


may be called 1,3,5,7-tetranitro-1,3,5,7-tetrazacycloöctane

The aza nomenclature is also applied by some to linear nitrogen compds. For example, the compd,



known as diethanolnitramine dinitrate (DINA), may be called 1,5-dinitroxy-3-nitro-azapentane and the compd,

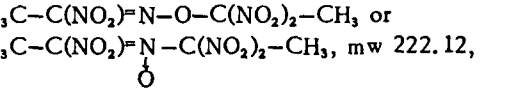


may be called 2,5-dinitro-2,5-diazahexane

Refs: 1)A.M.Patterson, JACS **55**, 3912 (1933) 2)N.Jones & G.D.Thorn, Can JRes **27B**, 832(footnote) (1949)

Azacyclo-. A prefix indicating the presence of a hetero nitrogen in a saturated carbon ring [See Aza (Nomenclature)]

3-Aza-4-oxa-2-hexene; 2,5,5-Trinitro or 2,4,4-Trinitro-3-aza-2-pentene 3-oxide,



mw 222.12, N 25.23%. Sl yel crysts, mp 121.2-121.6°. Was prepd by Belew et al from ammonium 1-nitroethanenitronate and 1,1-dinitroethane and previous to this, it was prepd by A.D. Little labs using aq K salt of 1,1-dinitroethane

Its expl props were not investigated

Refs: 1)Beil – not found 2)J.S.Belew et al, JACS **77**, 1112-13(1955) & CA **50**, 1648-9 (1956)

AZAUROLIC ACIDS

Azaurolic Acids are compds represented by the general formula ON·C(R):N·NH·C(R):NOH

The following azaurolic acids or their derivs might find application in the expl industry

Methylazaurolic Acid, ON·CH:N·NH·CH:NOH, mw 116.08, N 48.27%. Dk yel prisms (from methanol), mp ca 138° with deton; sl sol in w or alc. Was first prepd by Wieland & Hess (Refs 1 & 2) on passing bromine vapor through hydrazoformaldoxime suspended in cold w

Its metallic salts are expl (Refs 1 & 2) especially the *lead salt* which was patented by Rathsburg (Ref 3) for use in detonators. The lead salt is more sensitive to friction than MF (Ref 4)

Refs: 1)Beil **2**, 94 & [89] 2)Wieland & H.Hess, Ber **42**, 4187-8(1909) 3)H.Rathsburg, Ger P 447,459(1916) 4)H.Rathsburg, ZAngew-Chem **41**, 1285(1928)

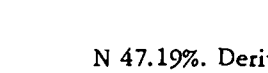
Note: No later refs through 1956 were found

Ethylazaurolic Acid, ON·C(CH_3):N·NH·C(CH_3):NOH, mw 144.14, N 38.87%. Orn-red ndls (from methanol), mp 142°(dec); sl sol in w; appreciably sol in eth & in hot alc; nearly insol in chlf, benz & ligroin. Can be prepd by reducing ethylnitrolic acid with sodium amalgam (Refs 1 & 2) or by other methods (Refs 1 & 3)

Its expl props were not investigated

Refs: 1)Beil **2**, 192-3 2)V.Meyer & E.J. Constam, Ann **214**, 330-1(1882) 3)H.Wieland, Ann **353**, 83-5(1907) & CA **1**, 2238(1907)

Note: No later refs through 1956 were found

"Azdioxdiazin,"  , mw 89.06, N 47.19%. Derivs of this hypothetical high nitrogen compd were claimed to be prepd by Jovitschitsch (Refs 1 & 2), but the existance of such compds was denied by Semper & Lichtenstadt (Ref 3)

Refs: 1)Beil **27**, 789 2)M.Z.Jovitschitsch, Ber **30**, 2426(1897); **31**, 3036(1898)

3)L.Semper & L.Lichtenstadt, Ann **400**, 302 (1913) & CA **8**, 63(1914)

Azeotrope. A liq mixt which exhibits a max or minim boiling point

Azeotropic Distillation. See under Azeotropy

Azeotropy (l'Azéotropisme, in Fr). Azeotropy may be defined as the capability of a liquid to form with other liquids, some mixts, which possess constant boiling points (max or minim). An azeotropic mixt resembles a chemical individual in boiling without undergoing change in compn, but differs from it in losing this characteristic as soon as the pressure is altered

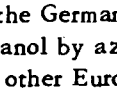
According to Timmermans (Ref 2), M.Berthelot discoved in 1863 that some org liqs (such as pinene-ethanol) form const boiling point mixts, but this phenomenon was already observed for some inorganic liqs in about 1859 when H.E.Roscoe et al distilled some aq inorganic acids. Many azeotropic mixts were discovered since then in the 2nd half of the 19th and the beginning of the 20th century, but no industrial application was made until 1917, when the Germans started to manuf absolute ethanol by azeotropic distn

After WWI other European countries, than Germany, and also US started to use azeotropic distn in prepn of abs ethanol, as well as of some other anhydrous substances (such as abs AcOH) and by the middle thirties the method became well established

Azeotropic distn can be employed in the explosives industry as, for instance, for removal of water formed during nitration (See Autogen Company Nitration) and also for detn of water in various liquids (See under Aquametry)

Refs: 1)V.Grignard, "Traité de Chimie Organique," Masson, Paris, tome I(1935), 154,156 & 165 2)J.Timmermans, "Les Solutions Concentrées" (Théorie et application aux mélanges binaires de composés organiques), Masson, Paris(1936) (Included are numerous Tables of azeotropic mixts, as for instance on pp 127,171,214,245,287,377, 418,454,495 & 543) 3)R.H.Ewell et al, IEC **36**, 871-5(1944) (Azeotropic distn) (14 refs) 4)R.H.Ewell & L.M.Welch, IEC **37**, 1224-31(1945)(Rectification in ternary systems contg binary azeotropes) (8 refs) 5)L.H. Horsley, Anal Chem **19**, 508-600(1947) (Tables of azeotropes and non-azetropes) (172 refs) 6)H.S.Nutting & L.H.Horsley, AnalChem **19**, 602-3(1947) (Graphical methods for predicting effect of pressure on azeotropic systems 7)M.Lécat, "Tables Azéotropiques," tome I (Azéotropes binaires orthobares), Bruxelles (1949); may be obtained from the author 29, rue Auguste Danse, Ucele-Bruxelles, Belgium(Data for 230 azeotropes of nitrocompds with other org compds) 8)Kirk & Othmer **5**(1950), 175-9 (under Distillation by E.G.Scheibel) 9)A.Weissberger, "Physical Methods of Organic Analysis," Interscience, NY, **4**(1951), 356-85(Azeotropic Distillation by C.S.Carlson) 10) Ullmann **1**(1951), 431ff (Destillation) 11) M.G.Larian, "Fundamentals of Chemical Engineering Operations," Prentice-Hall, Englewood Cliffs, NJ(1958), 415-21

Azete (Azacyclobutadiene or Pyriculine),



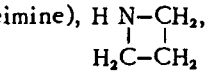
heterocyclic compd was reported to be obtained by Abderhalden & Paquin (Ref 2). According to Gensler (Ref 3) the cmpd obtd by A&P was not azete but probably allylamine

Elderfield (Ref 4) lists azetidine, 1-azetine and azetidinones as derivs of azete

Refs: 1)Beil – not found 2)E.Abderhalden & M.Paquin, Ber **53**, 1137(1920) & CA **14**, 3656(1920) 3)W.J.Gensler, JACS **69**, 1966 (1947) & CA **41**, 6873(1947) 4)R.C.Elderfield, Edit, "Heterocyclic Compounds," Wiley, NY (1950), Chapter 3 "Derivatives of Azete" by S.A.Ballard & D.S.Melstrom, pp 80 & 116-18

AZETIDINE AND DERIVATIVES

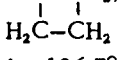
Azetidine (Trimethyleneimine or

Cyclotrimethyleneimine), 

mw 57.09, N 24.53%. Liq, bp 63° at 748 mm; miscible with w & alc. Was first described in 1888 (Ref 1,2&4), but was not isolated in pure state until 1899 (Refs 1,3&4). An improved method of prepn of azetidine is reported in Ref 5

Refs: 1)Beil **20**, 2-3 2)S.Gabriel & J. Weiner, Ber **21**, 2676-7(1888) 3)C.C.Howard & W.Marchwald, Ber **32**, 2032-4(1899) 4) R.C.Elderfield, Edit, "Heterocyclic Compounds," Wiley, NY, vol 1 (1950) 5)F.C. Schaefer, JACS **77**, 5929(1955) & CA **50**, 8669(1956)

Azido-, $C_3H_6N_4$ and *Diazido-*, $C_3H_5N_7$ *Derivatives of Azetidine* were not found in Beil or CA through 1956

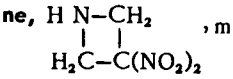
1-Nitrosoazetidine or N-Nitrosotrimethylenimine,  , mw 86.09, N 32.54%. Lt yel oil; bp 196-7°; miscible with w. Was prepd by heating azetidine with $NaNO_2$ in dil AcOH. Its expl props were not examined

Refs: 1)Beil **20**, 3 2)C.C.Howard & W.W. Marckwald, Ber **32**, 2035(1899)

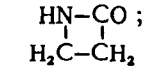
Note: No later refs were found in CA through 1956

Nitroazetidine, $C_3H_6N_2O_2$ – was not found in Beil or CA through 1956

Dinitroazetidine or Dinitrotrimethyleneimine, $C_3H_5N_3O_4$, mw 147.09, N 28.57%. The following isomer is found in the literature

3,3-Dinitroazetidine,  , mw 149.09, N28.57%; was obtained from 2,2-dinitro-1,3-propanediol and aq ammonia

Refs: 1)Beil – not found 2)R.Schenck & G.A.Wetterholm, SwedP 148217(1954) & CA **50**, 1893(1956) 3)Ibid, USP 2,731,460(1956)

2-Azetidinone (2-Ketotrimethyleneimine),  ; **2,4-Azetidinedione**, and **1-Azetine**, are derivs of *azete* and are known only in the form of derivs

Ref: R.C.Elderfield, Edit, "Heterocyclic Compounds," Wiley, NY(1950), 98-115

AZIDES, INORGANIC

Azides; Trinitrides or Triazoates (formerly called Azoimides) are the salts of hydrazoic acid, HN_3. This acid is a little stronger than acetic acid. The $-N_3$ radical in HN_3 resembles the chloride ion (Cl^-) in its chemical behavior. Hydrazoic acid was first prepd by Curtius (Ref 1) by the reaction of nitrous acid with hydrazine. For addnl info see **Hydrazoic Acid.** Azides may be divided into two main classes; inorganic and organic. The org azides and the complex azide compds are listed under their indvdl names as given in CA. Inorg azides are described below. Due to their extreme sensitivity only a few azides are suitable for use in the expl industry (Ref 3). The prepn and props of most known azides are described in the general Refs 2,4,5,6,8,9,10,11,12,14,15,17,18,19,25 &27; their structure and stability (Refs 13, 14a,25&26); their sensitivity to initiation by impact and heat (Refs 7,8,20&21); their thermochemisty (Refs 23&24); the theory of decompn (Refs 16&16a);Raman spectra (Ref 18a);and their analytical determn (Ref 22) are described in the references indicated

Refs: 1)T.Curtius, Ber **24**, 3341(1891) 2) W.Wislicenus, Ber **25**, 2084(1892) 3)M. Berthelot & P.Vielle, MP **8**, 7-16)1895-6); L.Chenel, MP **8**, 17-24(1895-6) 4)T.Curtius & J.Rissom, JPraktChem **58**, 269&295(1898) & JCS **76** II, 90-2(1899) 5)L.Dennis & H.Isham, Ber **40**, 458(1907) 6)L.Wöhler & W.Krupko, Ber **46**, 2045-57(1913) 7)L.Wöhler & F.Martin, ZAngChem **30** I, 33-9(1917) & JSCI **36**, 570(1917); CA **11**, 3432(1917) 8) L.Wöhler & F.Martin, Ber **50**, 586-96(1917) & JCS **112** I, 383-4(1917); CA **11**, 2900(1917) 9) L.Wöhler & F.Martin, SS **12**, 1-3,18-21, 39-42, 54-7& 74-6(1917) & CA **12**, 629(1918) 10) Marshall **2**(1917), 687 11)Mellor **8**(1928), 344-56 12)A.Haid et al, Jahresber CTR **8**, 102-8(1931)&CA **26**, 3669(1932) 13)A.Hantzsch, Ber **66**, 1349(1933) & CA **28**, 985(1934) 14) L.F.Audrieth, ChemRevs **15**, 169-224(1934)& CA **29**, 700(1935) 14a)J.H.deBoer, Chem Weekblad **31**, 729-32(1934) & CA **29**, 2826(1935) 15)Thorpe **1**, (1937), 562 16)N.F.Mott, Pr RoySoc **172A**, 325-35(1939) & CA **33**, 8502 (1939) 16a)N.F.Mott, PrRoySoc **172A**, 325-35(1939) 17)P.Sah et al, JChineseChemSoc **13**, 22-76(1946) & CA **42**, 148(1948) 18)P. Sah et al, JChineseChemSoc **14**, 52-64(1946) & CA **43**, 7446(1949) 18a)L.Kahovec & K.W. Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666(1948) 19)Kirk & Othmer **2** (1948), 213-4 20)F.P.Bowden & H.T.Williams, Pr RoySoc **208A**, 176-88(1951) & CA **46**, 5844 (1952) 21)A.D.Yoffe, PrRoySoc **208A**, 188-99(1951) & CA **46**, 5845(1952) 22)L.P. Pepkowitz, AnalChem **24**, 900-1(1952) & CA **46**, 7940(1952) 23)P.Gray & T.Waddington, PrRoySoc **235A**, 106-19(1956) & CA **50**, 12627 (1956); PrRoySoc **235A**, 481-95(1956) & CA **50**, 15203(1956) 24)T.Waddington & P.Gray, "ComptRend 27e CongInternlChimInd", Bruxelles(1954), **3** & IndChimBelge **20**, Spec NO, 327-30(1955); CA **50**, 16328(1956) 25) B.L.Evans & A.D.Yoffe, PrRoySoc **238A**, 568-74(1957) & CA **51**, 15129(1957) 26)B.L. Evans, ProcRoySoc **246A**, 199-203 (1958) & CA **52**, 21106(1958) 27)H.Rosenwasser, US ArmyEngrRes & DevelopLabsRpt 1551-TR, 72pp (1958), "Hydrazoic Acid and the Metal Azides" (a literature survey) 28)B.L.Evans A.D.Yoffe & P.Gray, "Physics and Chemistry of Inorganic Azides," Chem Reviews, **59**, 515-68(1959) (160 refs) 29)J.Krc, Jr & T.A. Erickson, "Investigation of Crystallographic Properties of Primary Explosives," Armour Research Foundation Quarterly Progress Rept Nos 1-3 and Final Rept No 3130-4, May 1, 1958 through May 1, 1959 (Study of crystn of LA under various conditions of manuf) 30)G.Todd & E.Parry, "The X-Ray Decomposition of Alpha Lead Azide," ARDE Rept No (MX)17/59, July 1959 (Complete destruction of LA can be achieved by exposing it to strong radiation, such as a dose of 3.35×10^8 r)

LIST OF INORGANIC AZIDES

Aluminum Triazide, (formerly called Aluminum Azoimide) $Al(N_3)_3$, mw 153.04, N 82.38%; Wh crysts, sol in tetrahydrofuran, insol in eth or benz, hydrolyzed by w(Ref 3). Prepd in quant yield by adding an eth soln of excess HN_3 to a frozen ether soln of AlH_3 and thawing(Ref 3). A tetrahydrofuran soln of $Al(N_3)_3$ is prepd by reaction of $AlCl_3$ and NaN_3 in benz, and extraction with tetrahydrofuran. When the tetrahydrofuran soln of $Al(N_3)_3$ was boiled under reflux with phenyl cyanide for 25 hrs, the product after decompn with HCl gave 76.5% yield of 5-phenyltetrazole (qv). Reaction of "nascent" $Al(N_3)_3$ with phenyl cyanide gave an almost quant yield. Similar reaction of the "nascent" azide with thioacetamide gave 55% yield of 5-methyltetrazole and a 65% yield of pentamethylenetetrazole (Cardiazole) from thiocaprolactam (Ref 3). According to Mellor (Ref 2) when a soln of ammonia-alum is treated with an alkaline azide, $Al(OH)_3$ is pptd and not $Al(N_3)_3$ (Ref 1)

$Al(N_3)_3$ is sensitive to percussion and decrepitates in a flame (Ref 3)

See also *Methylaluminum Diazide* [$CH_3Al(N_3)_2$] which reacts with sulfuric acid, occasionally igniting with expln.

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 261-309(1898) & JCS **76** II, 92(1899) 2) Mellor **8** (1928), 352 3)E.Wiber & H.Michaud, ZNaturforsch **9b**, 495-7(1954) & CA **49**, 767 (1955) 4)H.Rosenwasser, USArmyEngrRes &DevelopLabsRpt 1551-TR, 48(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Ammonium Azide (formerly called Ammonium Trinitride or Ammonium Azoimide), NH_4N_3, mw 60.06, N 93.29%; col, nonhygr, rhmb plates; mp expl 160° (Ref 5), starts to sublime at 133-4°, bp expl above temp limit for slow decompn ca 300°, d 1.346 at 20°, Q^P_f −26.0 kcal/mol (Refs 17&21) Q^v_f −19 kcal/mol (Ref 9) $Q^{P \text{ or } v}_c$ approx 98 kcal/mol (Ref 6) & Q_e 31.2 kcal/mol (Refs 6&9); sol in w, sl sol in alc or NH_4OH, insol in eth. It forms with ammonia a diammonate $NH_4N_3 \cdot 2NH_3$, in clear, col elongated plates, which is stable at −33°, unstable at 0° and undergoes transition at −9° (Ref 7). One g liq NH_3 dissolves 0.7g Amm azide at −33° and 1g at 0° (Ref 7), forms a eutectic with NH_3 at −87° and 76% NH_3 concn (Ref 14). Addnl soly data and other props given in Ref 7). Amm azide was first prepd in 1890 by Curtius (Ref 1) by the action of NH_3 on hydrazoic acid. Detailed descrpns of methods of prepn can be found in Refs 4,10,11,13,15,16&18.

While it sublimes below 250° at press 0 to 150 mm Hg, it shows slow decompn between 250-450° at 70 mm and betw 250°-310° at 150 mm. Amm azide is one of the more stable azides (Ref 20). According to Gray & Waddington it vaporizes and dissociates into NH_3 & HN_3 and then the HN_3 explodes. A hot wire causes Amm azide to burn quietly in air rather than detonate (Ref 20). This azide detonates violently when properly initiated, heated rapidly or heated under confinement (Refs 2 & 10). Temp of expln is 1400° and specific energy 7102 kg/l (Ref 9). It is considered a non-brisant expl as it decomposes in an ideal manner producing only innocuous gases (1148 l/kg at 0° and 760 mm) (Ref 6). The toxicity of Amm azide is unknown (Ref 22). Ephraim (Ref 19) states that this salt may be regarded as a polymer of imide$(NH)_X$. Infrared spectra studies of solid Amm azide are presented by Dows et al (Ref 19a) and thé cryst structure detd by Frevel (Ref 17a)

By mixing ammonium azide and hydrochloroplatinic acid and concg the soln by evapn, a very expl residue was obtained (Ref 10, p 355)

Ammonium Azide Ammonates. Ammonium azide forms with ammonia addition products: a)*Monoammonate,* $NH_4N_3 \cdot NH_3$, its existence was established from a study of the system ammonium azide-ammonia (Refs 7&8) b) *Diammonate,* $NH_4N_3 \cdot 2NH_3$, clear, col elongated plates stable at −33°, but incapable of existence at 0° (Ref 7) c)*Tetrammonate,*

$NH_4N_3 \cdot 4NH_3$, found to exist at low temp (Refs 8 & 12) and d) *Pentammonate*, $NH_4N_3 \cdot 5NH_3$, wh cryst solid which undergoes transition into the diammonate at −71°, with the eutectic located at −87° and 76% ammonia (Ref 14)

The expl props of these solvates have not been studied. See discussion under Hydrazine Azide

Refs: 1)T.Curtius, Ber **23**, 3023(1890) & **29**, 759(1896) 2)T.Curtius, Ber **24**, 3342 & 3347-8 (1891) 3)M.Berthelot & C.Matignon, Ann ChimPhys [7] **2**, 144(1894) & BullFr[3] **11**, 744(1894) 4)M.Berthelot & P.Vieille, MP **8**, 7-12 & 19(1895-6) 5)T.Curtius & J.Rissom, JPraktChem **58**, 261-309(1898) & JCS **76** II, 91(1899) 6)A.Darapsky, SS **2**, 41-2 & 65-7 (1907) & CA **1**, 1059 (1907) 7)A.Browne & A.Houlehan, JACS **35**, 657-8(1913) & CA **7**, 3261(1913) 8)A.W.Browne & O.R.Overman, JACS **38**, 288(1916) 9)H.Kast, SS **21**, 206-7 (1926) & **22**, 6(1927) & CA **21**, 3745(1927) 10)Mellor V**8**(1928) 344 11)W.Frost et al, JACS **55**, 3516-8(1933) & CA **27**, 5019(1933) 12)A.L.Dresser et al, JACS **55**, 1964(1933) 13)L.F.Audrieth, ChemRevs **15**, 169(1934); CA **29**, 700(1935) & InorgSynth **2** (1946) 136-8 14)D.H.Howard, Jr et al, JACS **56**, 2332-40 (1934) & CA **29**, 700(1935) 15)W.J.Frierson & A.W.Browne, JACS **56**, 2384(1934) & CA **29**, 699(1935) 16)Pepin Lehalleur (1935), 151 17)Bichowsky & Rossini (1936) 17a) L.K.Frevel, ZKrist**94A**, 197(1936) 18)Gmelin, System Nr **23**, Lieferung 1 (1936) 80-83 19) Ephraim (1943), 662 19a)D.A.Dows et al, JChemPhys **23**, 1475(1955) & CA **49**, 15486 (1955) 20)P.Gray & T.Waddington, Research Correspondence, Suppl to Research **8**, No. 11, 556-7 (1955) & CA **50**, 4692(1956) 21)P.Gray & T.Waddington, ProcRoySoc **235A**, 106-19 & 489(1956) & CA **50**, 12627(1956) 22)Sax (1957), 274 23)H.Rosenwasser, USArmy EngrRes & DevelopLabs Rpt 1551-TR, 25 (1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)
Note: N.W.Luft, IndChemist **31**, 502-4(1955) & CA **50**, 5388(1956), gives latent heat of sublimation at 25° 38.9 kcal/mole

Ammono-basic Mercuriazid. See under Mercuric Azide

Ammono-basic Ferric Azide. See under Ferric Azide

Ammono-basic Nickel Azide. See under Nickel Azide

Antimony Triazide (formerly called Antimony Trinitride), $Sb(N_3)_3$, mw 247.83, N 50.87%; yel solid which exploded on heating. Was obtained by Browne et al using an antimony anode in the electrolysis of a soln of ammonium azide in liq NH_3 at −67° (Refs 3 & 5). An electrical discharge through a mixt of N_2 and Sb vapors results in the formation of antimony nitride, SbN, which when heated decomp with a mild expln (Ref 2). The nitride prepn and props are also discussed in Refs 6,7,8&9. SbN is extremely sensitive to moisture and decomp on being heated to 550° (Refs 6&10) [Also see Refs 1 & 4 for unsuccessful efforts to isolate $Sb(N_3)_3$]

Refs: 1)T.Curtius & A.Darapski, JPrakt Chem **62**, 419(1900) & JCS **78** II, 475(1900) 2)F.Fischer & F.Schröter, Ber **43**, 1465-79 (1910) & CA **4**, 2075(1910) 3)A.W.Browne et al, JACS **41**, 1769(1919) & CA **14**, 28(1920) 4)A.C.Vournazos, ZAnorgChem **164**, 263(1927) 5)Mellor **8** (1928), 354 6)R.Schwarz & A.Jean-Marie, Ber **65B**, 1662-4 (1932) & CA **27**, 241 (1933) 7)N.H.Coy & H.Spooner, PhysRev **53**, 495(1938) & CA **32**, 4075(1938) 8)N.H. Coy & H.Spooner, PhysRev **58**, 709-13(1940) & CA **35**, 29(1941) 9)W.Jevons, ProcRoySoc **56**, 211-12(1944) & CA **39**, 2030(1945) 10) Gmelin System No **18**, Teil B2 (1949), 392

Arsenic Triazide, $As(N_3)_3$, mw 327.05, N 62.73%; prepn attempted by Vournazos (Ref 1) by reacting Na azide with As tribromide: $3NaN_3 + AsBr_3 = 3\ NaBr + As(N_3)_3$, but he obtained instead wh needles of the complex sodium arsenic bromoazide, $Na_8[AsBr_3(N_3)_8]$, in methyl alc soln (Ref 2)

There are no expl props given in the literature

Refs: 1)A.C.Vournazos, ZAnorgChem, **164**, 264(1927) & CA **21**, 3841(1927) 2)Mellor **8** (1928), 337

Barium Diazide (formerly called Barium Trinitride), $Ba(N_3)_2$, mw 243.43 N 51.79%; wh monoclinic prisms, mp expl at 150° (Ref 16), ignites 190-200° (Ref 11), d 2.936, Q_{soln} at 19.8° −7.8 kcal/mol (Ref 2), Q_f 5.32 kcal/mol (Ref 27), enthalpy of formation, free energy and entropy (Ref 27); very sol in w (12.5% at 0°, 16.2% at 10.5°, 16.7% at 15° and 17.3% at 17°), v sl sol in alc (0.017% at 16°), insol in eth (Refs 3 & 10). The toxicity is discussed by Sax (Ref 28) and is considered very sl (Ref 19). First prepd in 1890 by Curtuis (Ref 1) by neutralizing pure hydrazoic acid with Ba hydroxide soln. This method of prepn is also described by Audrieth (Ref 12) and in Refs 4 & 18. Can also be prepd by the action of hydrazoic acid on Ba oxide or carbonate (Ref 10). When evapd over sulfuric acid, crysts of monohydrate barium azide, $Ba(N_3)_2 \cdot H_2O$, are formed which have a mean idex of refraction of 1.7 (Ref 4). A safe, semi-industrial method has been developed for the production of $Ba(N_3)_2$ using ethyl nitrite, $Ba(OH)_2$ and hydrazine hydrate (yield 44-55%) (Ref 19). An alternative method consists of treating $Ba(ClO_4)_2$ with an equimolar quant of KN_3, yielding 80% Ba azide (Refs 10a & 19)

Ba azide is not as powerful an expl as Ca azide, but it is nearly as powerful as Sr azide. According to Curtius and Rissom (Ref 3), Ba azide does not expl by percussion and behaves on a hot plate like Ca azide. In a capillary it expl at 217-221°, and at about 180° metallic Ba is present. Tiede (Ref 6) observed that in vacuo, Ba azide begins to decomp at 120° and evolves N_2 at 160°. Hitch (Ref 8) noted that Ba azide undergoes no change until 180° when N_2 is evolved; at 225° the salt explodes. Wöhler and Martin gave 152° as the temp at which the salt expl (Ref 7).

The thermal decompn of solid Ba azide was studied by Gyunter et al (Ref 14) and by Yoffe (Ref 18a), the former investigators reporting a heat of decompn of 13.7 kcal. They supposed that expln occurs as a result of another primary reaction with the formation of nitride or of a strong exothermal reaction between the primary products of decompn (See also Ref 25). According to Ryabinin (Ref 17) to achieve decompn of a thin tablet of Ba $(N_3)_2$ within 3 min at atm press, a temp of 170° is required. This temp rises with the press on the tablet: at 2000 kg/cm² it is ca 210°, at 10,000 kg/cm² ca 225° and at 45,000 kg/cm² ca 235°. Garner & Reeves (Ref 26) found that the thermal decompn of $Ba(N_3)_2$ obeys a 6th power law, whereas, $Ca(N_3)_2$ and $Sr(N_3)_2$ obey a 3-rd power law. The mechanism of the thermal decompn of unirradiated and of briefly preirradiated Ba azide was postulated by Mott (Ref 13) and studied by Thomas, & Tompkins (Ref 20). However, on detailed examination of the photo and ionic conductivity of this salt, the latter authors found that their results did not agree with the mechanism postulated previously (Ref 21). Jacobs and Tompkins (Ref 23) in their study of the ionic conductance of solid metallic azides found that all salts obeyed the equation: $\log k = \log A - (E/2.303RT)$ in which, for Ba azide, $\log A = -5.99$ and $E = 11.6$ kcal/mol in the temp range 295 to 380°K

According to Ebler (Ref 5) Ba azide is not decompd by exposure to radium. Gyunter et al (Ref 15) also found that, unlike other azides, Ba azide is not decompd by X-rays of radium. X-rays of less than 0.7 A° also have no effect while soft X-rays produce a weak blue fluorescence. By using a Hadding tube, Gyunter et al, decompd approx 6% of $Ba(N_3)_2$ with more than half of the decompn product appearing as nitride. This fact was connected with the impact sensitivity of Ba azide. Groocock & Tompkins (Ref 24) described a technique for studying the effects of pre-irradiation and of prolonged bombardment with 100 and 200 V electrons on Ba azide at RT

The expl props of Ba azide were studied by Ficheroule & Kovache (Ref 19) who found that this salt detond 14% of the time with a 2 kg wt at a height of 100 cm. It is extremely sensitive to friction. When laid in a train it

does not behave like a primary expl, but large quantities deflagrate violently and lead to explosion. According to Haid et al (Ref 11) dry Ba azide may be safely transported in cardboard boxes in quantities up to 500 g, but with a water content of 10%, larger quants of it can be transported without danger.

The spectra of Ba azide were photographed by Petrikaln (Ref 9) who observed not only triplet lines but also that those of the singlet system were emitted. In addition the oxide bands of the molecule were present in all spectra of the azides of Ca, Sr and Zn. For Raman Effect of the cryst Ba azide see Ref 17a

Ba azide has not been used as an expl, but it has been used in the manuf of fluorescent lamps and radio tubes (Ref 12a & 19). It has also found use as a blowing agent during the vulcanization of cellular rubber (Ref 22). The expl reaction of Ba azide is prevented by adding gelatin, machine oil or Neugen (polyethylene glycol laurate)

Refs: 1)T. Curtius, Ber **23**, 3032 (1890) 2) M.Berthelot et al, BullFr [3] **11** 747(1894); AnnChimPhys [7] **2**, 144 (1894) & JCS **66** II, 352(1894) 3)T.Curtius & J.Rissom, JPrakt Chem **58**, 261-309(1898) & JCS **76** II, 91(1899) 4)L.M.Dennis & C.H.Benedict, JACS **20**, 229 (1898); JCS **74** II, 426(1898) & ZAnorgChem **17**, 18-25(1898) 5)E.Ebler, Ber **43**, 2613 (1910) 6)E.Tiede, Ber **49**, 1742(1916) 7) L.Wöhler & F.Martin, ZAngChem **30**, 33(1917) & JSCI **36**, 570 & CA **11**, 3432(1917) 8)A.R. Hitch, JACS **40**, 1195(1918) 9)A.Petrikaln, ZPhysChem **37**, 610-8(1926) & CA **20**, 2791 (1926) 10)Mellor **8** (1928), 350 10a)W.Hoth & G.Pyl, ZAngChem **42**, 888-91(1929) & CA **23**, 5547(1929) 11)A.Haid et al, Jahresber-CTR **8**, 102-8(1931) & CA **26**, 3669 (1932) 11a)Gmelin, System No **30**(1932), 134-8 12) L.F.Audrieth, ChemRevs **15** 169(1934); CA **29**, 700(1935) & InorgSynth **1**, (1939), 79-81 12a)Thorpe **1** (1937), 563 13)N.F.Mott, Proc RoySoc **172A**, 325(1939) 14)P.L.Gyunter et al, KhimReferatZhur **1940**, No 10-11, 120 & CA **37**, 1270(1943) 15)P.L.Gyunter et al, KhimReferatZhur **1940**, No 10-11, 80-3 & CA **37**, 1271(1943) 16)Davis(1943), 411 17)Y.N.Ryabinin, ZhurFizKhim **20**, 1363-6 (1946) & CA **41**, 2970(1947) 17a)L.Kahovec & K.W.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666-7(1948) 18)Matérial Téléphonique SA, FrP 938,720(1948) & CA **44**, 291(1950) 18a)A.D.Yoffe, ProcRoySoc **208A**, 196(1951) & CA **46**, 5845(1952) 19) H.Ficheroule & A.Kovache, MP **33**, 7-19 (1951) & CA **47**, 6617(1953) 20)J.Thomas & F.Tompkins, ProcRoySoc **210A**, 111-25 (1951) & CA **46**, 11000 (1952) 21)J.Thomas & F.Tompkins, JChemPhys **20**, 662-6 (1952) & CA **46**, 11000 (1952) 22)J.Tanaka & K. Yasuda, Repts Osaka Perfect Ind Res Inst **4**, No 1, 32-6(1952) & CA 46, 11743(1952) 23)P.W.Jacobs & F.C.Tompkins, JChemPhys **23**, 1445-7(1953) & CA **49**, 15336(1955) 24) J.M.Groocock & F.C.Tomkins, ProcRoySoc **223A**, 267-82(1954) & CA **48**, 8059(1954) 25)P.I.Byal'kevich, KhimReferatZhur **1954**, No 23239 & CA **49**, 15530(1955) 26)W.E. Garner & E.L.Reeves, TransFaradSoc **51**, 694-704(1955) & CA **49**, 15398(1955) 27) P.Gray & T.Waddington, ProcRoySoc **235A**, 106-19 (1956); **235A**, 489(1956) & CA **50**, 12627(1956) 28)Sax (1957), 329 29)H. Rosenwasser, USArmyEngrRes&Develop LabsRpt 1551-TR, 26(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Beryllium Diazide (formerly called Beryllium Trinitride), $Be(N_3)_2$ mw 93.07, N 90.31%; wh solid, sol in tetrahydrofuran, insol in eth and easily hydrolyzed by w(Ref 1). Obtd by Wiberg & Michaud (Ref 4) when Me_2Be was sublimed and reacted with a dry eth soln of excess HN_3 at −116°. Removal of eth and excess HN_3 by vac distn yielded $Be(N_3)_2$. In a reactn betw a beryllium salt and an azide, Curtius & Rissom (Refs 1 & 2) obtd an impure beryllium azide; it detonated only sl in a flame and is insensitive to initiation by percussion (Ref3)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 277(1898) & JCS **76** II, 92(1899) 2) Mellor **8** (1928), 350 3)Gmelin, System No **26**

(1930), 102 4)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 502(1954) & CA **49**, 768 (1955) 5)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1551-TR, 48(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Bis (Hydroxylamino) Azide (called Dihydroxyl-Ammonium Trinitride by Dennis & Isham), $(NH_2OH)_2 \cdot HN_3$, mw 109.10, N64.20%; col, trans, leaf-like crysts, mp 66°; v sol in w, sol in alc and insol in eth. This compd was prepd in 1906 by Dennis & Isham (Ref 1) upon evapg a mixt of hydroxylamine and hydrazoic acid, both in methyl alc solns. The recovered crysts were purified by dissolving in a mixt of 1 g methyl alc and 20 p ether, filtering and rapidly evapg the solvent in a vacuum desiccator (See also Ref 2). No expl props were determined (Also see Hydrazoic Acid)

Refs: 1)L.M.Dennis & H.Isham, JACS **29**, 22-4(1907) & CA **1**, 528(1907) 2)L.F.Audrieth, ChemRevs **15**, 200-202(1934) & CA **29**, 700 (1935)

Bismuth Triazide, $Bi(N_3)_3$, mw 335.07, N 37.63%. The prepn of this compd was attempted by Vournazos (Ref 1) who treated Bi iodide with an equimolar part of Na azide. The following reaction occurred: $2\,Bi\,I_3 + 2\,NaN_3 + H_2O = BiOI + BiI_3 + 2HN_3 + 2NaI$ and with twice this amt of Na azide: $BiI_3 + 2NaN_3 + H_2O = BiOI + 2NaI + 2HN_3$. It was thought by Vournazos that an unstable bismuth iodo-diazide, $Bi\,I(N_3)_2$, was formed but immediately hydrolyzed (Ref 2)

Refs: 1)A.C.Vournazos, ZAnorgChem **164**, 263(1927) 2)Mellor **8** (1928), 337

Boron Triazide, $B(N_3)_3$, mw 136.89, N 92.10%; wh hex crysts, sol in tetrahydrofuran insol in eth; prepd by Wiberg and Michaud (Ref 1) by the addn of diborane to a frozen eth soln of excess HN_3 at –20° and thawing at RT. The residue was isolated by distn at –65° to –45° for 4½ hrs. $B(N_3)_3$ is extremely explosive and deton with water or eth vapor, but it can be stabilized as $NaB(N_3)_4$ by reaction of an eth soln of excess $B(N_3)_3$ and NaN_3. Upon addn of an eth soln of Me_3N to $B(N_3)_3$, the partial azides, $BH(N_3)_2$ and BH_2N_3, form stable addn compds

An eth sol of **Lithium Boroazide,** $LiB(N_3)_4$ is formed in 90% yield upon evapn to dryness of a reaction mixt consistg of excess HN_3 in eth soln and frozen $LiBH_4$ in eth soln. The wh solid residue is an expl very sensitive to pressure and to percussion. It is sol in eth and easily hydrolyzed. $B(N_3)_3$ and LiN_3 are assumed to be intermediate products (Ref 1)

Refs: 1)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 497-9(1954) & CA **49**, 767(1955) 2)H. Rosenwasser, USArmy EngrRes & Develop LabsRpt 1551-TR, 48(1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Bromine Azide (Bromoazide), BrN_3, mw 121.94, N 34.45%; orange-red liq, fr p-45°; bp expl, misc in all proportions with eth, less sol in benz or ligroin (Refs 2 & 3). Prepd by Spencer (Ref 1) by passing a stream of bromine, diluted with N_2, over Na or Ag azide and condensing the resulting liq bromoazide. It may be prepd also by heating NaN_3 or AgN_3 with a soln of bromine in eth, benz or ligroin (Refs 1&3)

Bromoazide is a very powerful expl compd and extremely sensitive to heat and to mechanical shock. Eth, benzene or ligroin solns of BrN_3 are stable in the dark, but when concd, they are likely to expl on shaking, and gradually decompose on standing. In general, BrN_3 resembles IN_3 but is more volatile and is immediately decompd by water. The only evidence for the existence of BrN_3 in aq soln was the formation of a little HN_3 with consequent dimunition of free N_2 (Ref 1). The liq BrN_3 expl in contact with P, As, Na and Ag foil, but the vapor, when diluted with N_2 and passed over Ag or Na leaf, gives a film of the corresponding azide and bromine (Ref 2)

Bromoazide gives a pungent vapor which

irritates the eyes. It has toxicological props similar to hydroazoic acid, causing giddiness, headache, and slakening of the muscles when inhaled (Ref 6). It is dangerous, when heated, emitting highly toxic fumes of bromine and explodes. Reacts with water or steam to produce toxic or corrosive fumes, and it can react on contact with reducing materials (Ref 6)

Refs: 1)D.A.Spencer, JCS **127**, 216-24(1925) & CA **19**, 1106 (1925) 2)Mellor **8**(1928), 336 3)Gmelin, System No **7**(1931), 334 4)Thorpe **1** (1947), 581 5)Kirk & Othmer **7** (1951), 594 6)Sax(1957), 383

Cadmium Diazide $Cd(N_3)_2$, mw 196.46, N 42.78%; wh biaxial crysts, mp expl 291° (Ref 3); d 3.24 at 20° (Ref 12), Q_e 558-625 cal/g (Refs 2 & 4); sol in water and is hygroscopic. First prepd by Curtius & Rissom (Ref 1) by dissolving $CdCO_3$ in 16-17% hydrazoic acid, and by Brown et al (Ref 5) by electrolyzing solns of Amm azide in liq NH_3 using a cadmium anode. Birckenbach(Ref 10) prepd the pure compd by the method of Curtius & Rissom and Bassière from a mixt of solns of $Cd(NO_3)_2$ and NaN_3 by evapn in cold over H_2SO_4, after removal by filtration of the first ppt formed (Ref 12). The usual method of prepn is by the action of hydrazoic acid on CdO or $CdCO_3$ (Refs 1,7,8,9&11)

$Cd(N_3)_2$ is an extremely sensitive and dangerous expl which detonates on heating or on rubbing with a horn spatula (Ref 10). Thermal decompn in high vac between 100 and 120° leads to the reaction $3Cd(N_3)_2 = Cd_3N_2 + 8N_2$. Wöhler and Martin reported the following expl props: *Impact Sensitivity* by 0.964 kg falling wt on 0.01 to 0.02 g compressed sample reqd energy of 18.54 kg m/cm² vs 4.76 kg m/cm² for LA under 0.600 kg impact on same sample wt (Ref 3); *Loading Density* at 1100 kg/cm² – 3.200 g/cc (Ref 4); *Minimum Initiating Charge:* required for tetryl– 0.01g,for PA – 0.02g, for TNT – 0.04 g and for TNA 0.10g (Ref 4); *Temperature on Explosion* 3829° (Ref 4) and *Work Density* (an approx measure of deton value) 116.8 kg/cm² (Ref 4)

According to Wöhler (Ref 6), Cd azide is much more powerful than LA but is more difficult to prepare because of its high soly in water (Ref 13)

The cryst structure of $Cd(N_3)_2$ was studied by Bassière (Ref 12). Bowden and Singh (Refs 14 & 15) studied the effects of irradiation and nuclear bombardment on $Cd(N_3)_2$ crysts. They observed no ignition or detonation due to bombardment with slow neutrons, fission products, α-particles or γ-particles. Irradiation with intense electron beams by X-rays, Hydrogen, Argon or Mercury-ions led to explns which proved thermal in origin. The critical thickness for thermal initiation and growth to expln is 24μ at 320°, 20μ at 325° and 17μ at 330° (Ref 15). According to Bowden and Singth, neutron bombardment of $Cd(N_3)_2$ does not affect its rate of deton, given as 4200 m/-sec (Ref 15)

$Cd(N_3)_2$ in aq soln, forms with pyridine a col, cryst compd, $Cd(N_3)_2 \cdot 2C_5NH_5$ (Ref 1). Other complex salts described in Ref 13a (See also Table A under Ammines)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 261-309(1898) & JCS **76** II, 91-2(1899) 2)L.Wöhler & F.Martin, Ber **50**, 586-96 (1917); JCS**112** I, 383-4(1917) & CA **11**, 2900-1(1917) 3)L. Wöhler & F.Martin, ZAngChem **30**, 33-9 (1917); JSCI **36**, 570(1917) & CA **11**, 3432 (1917) 4)L.Wöhler & F.Martin, SS **12**, 1-13, 18-21, 39-42, 54-7, 74-6(1917) & CA **12**, 629 (1918) 5)A.W.Browne et al, JACS **41**, 1769-76 (1919) & CA **14**, 28(1920) 6)L.Wöhler, ZAngChem **35**, 545(1922) & CA **17**, 1144(1923) 7)Gmelin, System No **33**(1925), 75 8)Mellor **8** (1928), 351 9)Marshall **3** (1932), 158 10) L.Birckenbach,ZAnorgChem **214**, 94-6(1933) & CA **27**, 5267(1933) 11)L.F.Audrieth, Chem Revs **15**, 208, 214(1934) & CA **29**, 700(1935) 12)M.Bassière, CR **204**, 1573-4(1937) & CA **31**, 5238(1937) 13)Davis(1943), 183,411&412 13a)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314(1939) 14)F. Bowden & K.Singh, Nature **172**, 378-80(1953) & CA **48**, 1003(1954) 15)F.Bowden & K.Singh, ProcRoySoc **227A**, 22-37(1954) & CA **49**, 4991 (1955)

Calcium Diazide (formerly called Calcium Trinitride), $Ca(N_3)_2$, mw 124.13, N 67.71; col, rhmb ndls (Ref 2); mp dec 100° (in vacuo); N_2 evolved at 110° (Refs 2,3 & 15) expl 158° (Refs 5 & 11) Q_e 625 cal/g (Ref 4), Q_f −11.0 k cal/mol (Ref 20) sol in W (38.1% at 0° & 45% at 15.2°), v sl sol in alc (0.211% at 16°) and insol in eth (Ref 1). Its toxicity is discussed by Sax (Ref 21) under azides. $Ca(N_3)_2$ was first prepd in 1898 by Dennis & Benedict (Ref 2) and by Curtius & Rissom (Ref 1) by dissolving calcium oxide or carbonate in dil aq HN_3 and concg the filtrate by evapg over sulfuric acid (Refs 7&9)

The expl props, as detd by Haid et al (Ref 8), indicate that Ca azide is the most powerful of the alkaline earth azides. Although Curtius found that this salt did not expl by percussion, Wöhler & Martin (Ref 5) and Haid et al (Ref 8) obtained deton by impact (Refs 7&12). When heated rapidly $Ca(N_3)_2$ expl between 144-156°. Heated in a capillary tube, metallic Ca appears at 120-130° (Ref 11) and in vacuo expl between 160-170° (Ref 14). The kinetics of the thermal decompn has been studied by Andreev (Ref 10), Garner & Reeves (Ref 19) and others; ionic conductance of the solid by Jacobs & Tompkins (Ref 18) in the temp range 290-370°K, and initiation and propogation of expln by Bowden & Williams (Ref 16) who measured the rate of deton as 770 m/sec. Haid et al (Ref 8) ignited $Ca(N_3)_2$ by rubbing a small sample in a mortar and in the Lead Block Expansion test obtained a value of 120 ml

The spectra of calcium azide explns were photographed by Petrikaln (Ref 6) and the Raman Effect studied by Kahovec & Kohlrausch (Ref 13)

$Ca(N_3)_2$, as well as $Ba(N_3)_2$ or NaN_3, has been recommended as a cellulating agent in the prepn of sponge rubber (Ref 17)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 261-309(1898) & JCS **76** II, 91(1899) 2) L.M.Dennis & C.H.Benedict, JACS **20**, 228 & 231(1898); JCS **74** II, 426(1898) & ZAnorgChem **17**, 18-25(1898) 3)E.Tiede, Ber **49**, 1742(1916) 4)L. Wöhler & F.Martin, Ber **50**, 586-96(1917); JCS **112** I, 383-4 & CA **11**, 2901(1917) 5)L.Wöhler & F.Martin, ZAng Chem **30**, 33-9(1917); JCSI **36**, 570(1917) & CA **11**, 3432(1917) 6)A.Petrikaln, ZPhys Chem **37**, 610-8(1926) & CA **20**, 2791(1926) 7)Mellor **8** (1928), 349 8)Haid et al, Jahresber CTR **8**, 102-8 (1931) & CA **26**, 3669(1932) 9)L.F.Audrieth, ChemRevs **15**, 169(1934) & CA **29**, 700(1935); Inorg Synth **1** (1939) 80-1 10)K.K.Andreev, KhimReferat Zhur **1940**, No 10-11, 120-1 & CA **37**, 1271 (1943) 11)Davis (1934), 411 12)Thorpe **1** (1947) 562 13)L.Kahovec & K.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666-7 (1948) 14)A.D.Yoffe, ProcRoySoc **A208**, 188-99(1951) & CA **46**, 5845(1952) 15)Kirk & Othmer **7** (1951), 594 16)F.P.Bowden & H.T.Williams, ProcRoySoc **208A**, 176-88 (1951) & CA **46**, 5844-5(1952) 17)J.Tanaka & K. Yasuda, ReptsOsakaPrefectIndResInst **4**, No 1, 32-6(1952) & CA **46**, 11743(1952) 18)P.W.Jacobs & F.C.Tompkins, JChem Phys **23**, 1445-7(1953) & CA **49**, 15336(1955) 19)W.E.Garner & L.E.Reeves, TransFarad Soc **51**, 694-704(1955) & CA **49**, 15398(1955) 20)P.Gray & T.Waddington, ProcRoySoc **235A**, 106-19(1956) & CA **50**, 12627(1956) 21)Sax(1957), 424 22)Gmelin, System No **28**, Teil B, Lieferung 2(1957), 329-31 23)H. Rosenwasser, USArmyEngrRes&Develop LabsRpt 1551-TR, 27(1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Calcium Diazide Monohydrazinate (formerly called Calcium Trinitride Hydrazinate), $Ca(N_3)_2 \cdot N_2H_4$, mw 154.16, N 72.69; wh, fluffy pwd, mp dec at 120° (losing hydrazine); bp expl violently at 308°; v sol in w. Prepd by the gradual dehydrazination of Ca diazide dihydrazinate in N_2 at 100° and its chem identity established by means of press-concn and press-temp curves (Refs 1&2). No references to expl props found.

Refs: 1)A.L.Dressler & A.W.Browne, JACS **53**, 4238-42(1931) & CA **26**, 666(1932) 2) Gmelin, System No **28**, Teil B, Lieferung 2 (1957), 331

Calcium Diazide Dihydrazinate (formerly called Calcium Trinitride Dihydrazinate) $Ca(N_3)_2 \cdot 2N_2H_4$, mw 188.22, N 74.37; wh, rect, ortho rhombic crysts, mp dec slowly at RT, bp exp violently at 335°; v sol in w, sol in anhyd hydrazine (34.7% at 23°), mod sol in methanol (7% at 23°) v sl sol in alc (0.4% at 23°); pract insol in CCl_4, chlf, benz, acet, diethyl eth, et acetate or CS_2. Prepd from a nearly satd soln of $Ca(N_3)_2$ in anhyd hydrazine by either evapn at RT over sulfuric acid or treatment with absol alc. Chemical identity established by means of press-concn and press-temp curves in addn to chemical analysis (Refs 1&2)

Refs: 1)A.L.Dressler & A.W.Browne, JACS **53**, 4236-8(1931) & CA **26**, 666(1932) 2) Gmelin, System No **28**, Teil B, Lieferung 2 (1957), 331

Carbonyl Diazide (formerly called Carbonyl Nitride & Carbazoimide) (Called Kohlensäure-diazid, Carbazid or Stickstoffkohlenoxyd in Ger), $CO(N_3)_2$, mw 112.06, N 75.00%; extremely volat, long ndls very sol in w, alc and eth but insol in petr eth; undergoes hydrolysis to yield CO_2 and HN_3; it has a penetrating odor and like other carbonyl compds is highly toxic and dangerous (Ref 7)

$CO(N_3)_2$ was first prepd in 1894 by Curtius & Heindenreich (Refs 1 & 2) by the action of sodium nitrite on the hydrochloride of carbohydrazide, $CO(NH \cdot NH_2HCl)_2$. Kesting (Ref 3) found that this reaction did not always proceed homogenously and that hydrazidicarboxyazide, $(NHCON_3)_2$, was always formed as a by product in ca 20% yield. The two compds could be separated by carrying out the diazotization under benz. Kesting (Ref 3) obtd $CO(N_3)_2$ in about 70% yield from $CO(OEt)_2$ refluxed for 2 days on a w bath with 99% $N_2H_4 \cdot H_2O$

Carbonyl diazide is an extremely dangerous expl as it may explode violently, even under H_2O, on sl friction or when exposed to light (Refs 1,2,3,5 & 6)

Like sulfuryl azide, $CO(N_3)_2$ decomposes in such solvents as benz and aniline, and converts aromatic hydrocarbons into pyridine bases and also into primary amines (Ref 1 & 4). Kesting (Ref 3) found that $CO(N_3)_2$ in H_2O and $NaNO_2$ under benz, when slowly treated with HCl yielded $(NHCON_3)_2$ and when $CO(N_3)_2$ in alc was heated with piperidine, it gave hydrazidocarboxy piperidide, mp 179°

No addnl info on $CO(N_3)_2$ was found in the literature.

Refs: 1)Beil **3**, 130 & [102] 2)T.Curtius & K.Heindenreich, Ber **27**, 2684(1894); JPrakt Chem **52**, 454(1895) & JCS **68** I, 12(1895) 3) W.Kesting, Ber **57B**, 1321-4(1924) & CA **19**, 245(1925) 4)T.Curtius & A.Bertho, Ber **59**, 565(1926) & CA **20**, 2500-1(1926) 5)L.F. Audrieth, ChemRevs **15**, 216-7(1934) 6) Thorpe **2** (1938), 278 & 323 7)Sax (1957), 442

Cerium Hydroxydiazide, $Ce(OH)(N_3)_2$, mw 241.19, N34.85%; yel expl residue obtained by Curtius & Darapsky from freshly pptd Ce hydroxide dissolved in hydrazoic acid and evapn of the soln formed (Refs 1&2). No references to expl props found

Refs: 1)T.Curtius & A.Darapsky, JPrakt Chem **61**, 408(1900) & JCS **78** II, 474(1900) 2)Mellor **8**(1928), 354

Cerium Triazide, $Ce(N_3)_3$, mw 266.20, N47.36%; expl ppt obtained by Curtius & Darapsky (Ref 1) by boiling a mixt of Ce nitrate and Na azide (Ref 2) No references to expl props found

Refs: 1)T.Curtius & A.Darapsky, JPrakt Chem **61**, 408(1900) & JCS **78** II, 474(1900) 2)Mellor **8**(1929), 354

Cesium Azide (formerly called Cesium Trinitride or Cesium Azoimide), CsN_3, mw 174.93, N24.02%; col, clear, tetrag ndles (Ref 1); mp 310-18° (Ref 2), 320° (Ref 3) or 326° in vacuo (Refs 4 & 12); bp dec at 350° evolving N_2 (Ref 3), Q_f 2.37 k cal/mol (Ref 13); lattice energy 146 k cal/mol (Ref 14); v sol in w (307% at 16°), sl sol in alc

(1.04% at 16°), insol in eth (Ref 2). Its toxicity is not known. First prepd in 1898 by Dennis & Benedict (Ref 1) and also by Curtius & Rissom (Ref 2) by dissolving freshly pptd CsOH in aq HN_3 and evapg the soln in air or over sulfuric acid. Moldenhaur & Möttig (Ref 6) prepd the compd by reacting Cs metal with N_2 activated by an electrical discharge. It can also be prepd by neutralizing 3% hydrazoic acid with an aq soln of pure CsOH, concg the soln on steam bath, acifying with HN_3 and adding 2 vols alc to the soln cooled in an ice bath. The ppt was collected by filtration and washed with alc and ether (Refs 4,8,9 & 10)

CsN_3 does not expl on impact; it behaves like KN_3 on a hot plate and like RbN_3 at temps above its mp (Ref 5). The thermal decompn of CsN_3 has been studied by Clusius (Ref 7), Klaus & Mollet (Ref 15) and by Tiede (Ref 3). The Raman Effect was studied by Kahovec & Kohlrausch (Ref 11).

Refs: 1)L.M.Dennis & C.H.Benedict, JACS **20**, 227(1898); ZAnorgChem **17**, 20(1898) & JCS **74** II, 426(1898) 2)T.Curtius & J.Rissom, JPraktChem **58**, 261-309(1898) & JCS **76** II, 92(1899) 3)E.Tiede, Ber **49**, 1742(1916) & CA **11**, 2176(1917) 4)R.Suhrmann & K.Clusius, ZAnorgChem **152**, 52(1926) & CA **20**, 1962 (1926) 5)Mellor **8** (1928), 348 6)W.Moldenhaur & H.Möttig, Ber **62**, 1955(1929) & CA **24**, 1300(1930) 7)K.Clusius, ZAnorgChem **194**, 47-50(1930) & CA **25**, 889(1931) 8)L.F. Audrieth, ChemRevs **15**, 202-3(1934) & CA **29**, 700(1935) 9)Gmelin, System No **25**(1938), 115-6 10)A.W.Browne, InorgSynth V1(1939), 79 & CA **36**, 2488(1942) 11)L.Kahovec & K.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666-7(1948) 12)Kirk & Othmer, **7**(1951), 593-4 13)P.Gray & T.C.Waddington, Proc RoySoc **235A**, 106-19(1956) & CA **50**, 12627 (1956) 14)P.Gray & T.C.Waddington, Proc RoySoc **235A**, 481-95(1956) & CA **50**, 15203 (1956) 15)P.Klaus & H.Mollet, Helv **39**, 363-75(1956) & CA **50**, 15413(1956)

Chlorine Azide or Chloroazide, (Chlorazid in German), ClN_3, mw 77.48, N 54.24%; col gas at RT; yel-orange liq, bp −15°; yel, v expl solid at below −100° (Ref 8); sl sol in w, readily sol in butane, pentane, benz, MeOH, EtOH, diethyl ether, acet, chlf, CCl_4 & CS_2 (Ref 8). According to Sax (Ref 15) the toxicity of ClN_3 azide is severe and acute on single exposure or inhalation, causing injury to skin or mucous membranes of sufficient severity to threaten life or cause permanent physical damage. The effects of continuous or repeated exposure are unknown.

Chlorine azide was first prepd in 1908 by Raschig, (Ref 1) upon acidification with acetic or boric acid of a mixt of Na hypochlorite and Na azide in aq soln:

$$NaOCl + 2HAc + NaN_3 \rightarrow 2NaAc + H_2O + ClN_3$$

Although Raschigs' method was satisfactory, Frierson et al (Ref 8) preferred, for the lab prepn, passing chlorine gas into an ethereal suspn of silver azide at RT:

$$AgN_3 + Cl_2 \rightarrow AgCl + ClN_3$$

Chlorine azide gas, with a sweetish odor similar to that of HClO, is an extremely dangerous expl. It expl violently in contact with a flame, on exposure to sunlight and sometimes even spontaneously (Refs 1,5,6,7, 10,11 & 12). Gleu (Ref 4) found ClN_3 decompd at 400° and 2 mm press without expln into the elements, N_2 and Cl_2. Decompn was accompanied by red radiation and intense short wave radiation in the blue and ultraviolet regions. Pannetier (Ref 13) observed that the deton of pure ClN_3 by a simple electric spark, resulted in a continous spectrum from ultraviolet to red with max intensity at 5000-5500A°. The kinetics of expln corresponded to complete rupture of the mol, recombination of the individual atoms giving rise to the spectra. Expln of ClN_3 occurred at all press above 0.1 mm (Ref 14).

The chemical reactions of ClN_3 have been studied by Raschig (Ref 2), Gutmann (Ref 3), and by Frierson et al (Refs 8 & 9). Reaction with lq ammonia resulted in the formation of an expl liq (Refs 8 & 9); reaction with pentane gave hydrazoic acid (Ref 8); and reaction

with Na and P resulted in violent explns, with spontaneous deton occurring within a few minutes in the case of P (Ref 8). Gutmann (Ref 3) found that tertiary sodium arsenite, Na_3AsO_3, does not react with the inorg salts of HN_3, but with chloro- and iodoazides it gives the alkali azide and halide and is ozidized to arsenate. Chlorine azide with silver azide forms *Azino-Silver Chloride,* N_3AgCl, which is a deep blue solid, stable only below −30° and expl violently in the dry state (Ref 9), decompg into AgCl and N_3. When moistened with non aq liqs, the compd, N_3AgCl, decomp rapidly but without expln as the temp is raised. It is not sensitive to mech shock but extremely sensitive to temps above −30° (Ref 9)

Refs: 1)F.Raschig, Ber **41**, 4194-5(1908) & CA **3**, 622(1909) 2)F.Raschig, "Schwefel-Stickstoff-Studien," Leipzig (1924), 204 & CA **18**, 2584(1924) 3)A.Gutmann, Ber **57B**, 1956-8(1925) & CA **19**, 1253(1925) 4)K.Gleu, ZPhysik **38**, 176(1926) & CA **21**, 1229(1927) 5)Gmelin, System No **6**(1927) 417 6)Mellor **8** (1928) 336 7)L.F.Audrieth, ChemRevs **15**, 215(1934) & CA **29**, 700(1935) 8)W.J. Frierson et al, JACS **65**, 1696-8(1943) & CA **37**, 6576(1943) 9)W.J.Frierson & A.W. Browne, JACS **65**, 1698-1700(1943) & CA **37**, 6576(1943) 10)Thorpe **1** (1947), 581 11) Ephraim (1949), 675-6 12)Kirk & Othmer **7** (1951), 594 13)G.Pannetier, CR **233**, 168-70 (1951) & CA **46**, 1871(1952) 14)G.Pannetier, BullFr **1954**, 1068-70 & CA **49**, 7247-8(1955) 15)Sax(1957), 464

Chromium Triazide (Chromium Azoimide or Chromium Azide) $Cr(N_3)_3$, mw 178.08, N 70.79%; dk green crysts, sol in w. Reported in 1898 by Curtius & Rissom (Ref 2) to be formed in soln by dissolving chromiun hydroxide in aq hydrazoic acid but the product was not isolated because it decompd on evaporg the soln. Curtius & Darapsky (Ref 3) found that chrome alum, $Cr_2(SO_4)_3 \cdot K_2SO_4 \cdot 24H_2O$, and NaN_3, gave a green soln of $Cr(N_3)_3$ which however, was completely hydrolyzed on boiling; the resulting basic Cr azide pptd as a green salt when alc and ether were added to the soln. They observed that the chrome-alum solns reacted with NaN_3 in a manner similar to that described by Dennis (Ref 1) with the elements separating as the hydroxides. Oliveri-Mandalà (Ref 5) treated a soln of chromic nitrate with 3 mols of Na azide and noted that the soln became violet and then green, but nothing separated even when concd solns were used. The normal Cr azide was obtained by Oliveri-Mandalà & Comella (Ref 6) by evapg $Cr(NO_3)_3$ in abs alc with excess NaN_3 in vacuo over KOH. If the alc soln contains some w, basic azides of Cr: *chromium hydroxydiazide,* $Cr(N_3)_2OH$ and *chromium dihydroxyazide,* $CrN_3(OH)_2$ are formed (Ref 6). The expl props of the basic Cr azides were not investigated.

Other methods of prepn Cr azide are given In Refs 7 & 9

Aq solns of Cr azide hydrolyze slowly and have the green color of Cr complexes. They do not ppt $Cr(OH)_3$ when NH_4OH is added or ppt AgCl when $AgNO_3$ is added (Refs 5 & 6). When boiled, its concd aq solns deposit mixts of basic salts, the compns of which depend upon the duration of heating and concn of the solns. One basic Cr azide, $Cr(N_3)_2OH \cdot 2H_2O$, was analyzed and found to be far less expl than the original $Cr(N_3)_3$ (Refs 6 & 7). Other Cr azide complexes are described below

Chromium Azide Complexes In attempting to prepare Cr azide from a soln of $Cr(NO_3)_3$ and NaN_3 in pyridine, Oliveri-Mandalà (Ref 5) found that when more than 3 mols of C_5H_5N were added a greenish-violet ppt slowly separated. This subst was washed with cold w, alc and then acet to give a green crust of *chromium triazide pyridine complex,* $Cr(N_3)_3 \cdot 3C_5H_5N$, which was insol in most org solvents but sl sol in C_5H_5N or glycerol. The dried (in vacuo) subst exploded violently on heating (Refs 5 & 7). Another complex *sodium chromium azide,* $CrN_9 \cdot 3NaN_3$, green crysts, was prepared by Oliveri-Mandalà & Comella (Ref 6) by adding 3 mols of NaN_3 in alc to a soln of

of freshly prepd $Cr(OH)_3$ in concd aq HN_3. Aq solns of sodium chromium azide did not react with Cr or N_3 *but with* $AgNO_3$ *gave an expl complex salt.* The sodium chromium azide is considered to be the Na salt of chromihydrazoic acid, $H_3Cr(N_3)_6$, but this acid was not isolated because it decomposed too readily. Attempts to obtain it by the methods of Wöhler & Martin (Ref 4) failed

Strecker & Schwinn (Ref 8) prepd the following chromium azide complex salts: $[Cr(NH_3)_6](N_3)_3$ and $[Cr(NH_3)_5Cl](N_3)_2$ {See Table A under Ammines and also see Ref 10 for prepn and props of *Cis-diazidobisethylenediamine chromium azide,* cis-$[Cr(en)_2(N_3)_2]N_3$.

Refs: 1)L.M.Dennis, ZAnorgChem **6**, 35 (1894) & JACS **18**, 947(1896) 2)T.Curtius & J.Rissom, JPraktChem **58**, 266(1898) & JCS **76** II, 92(1899) 3)T.Curtius & A.Darapsky, JPraktChem **61**, 408-22(1900) & JCS **78** II, 474-5(1900) 4)L.Wöhler & F.Martin, Ber **50**, 595(1917); JCS **112** I, 383-4(1917) & CA **11**, 2900(1917) 5)E.Oliveri-Mandalà, Gazz **49** II, 43-6(1919) & CA **14**, 701(1920) 6)E.Oliveri-Mandalà & G.Comella, Gazz **52** I, 112-5(1922) & CA **16**, 2089(1922) 7)Mellor **8** (1928), 354 8)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314(1939) 9)Thorpe **3** (1946), 109 10)M.Linhard & M.Weigel, ZAnorgChem **271**, 131-7(1952) & CA **47**, 7360 (1953) 11)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1551-TR, 46(1958) "Hydrazoic Acid and the Metal Azides" a literature survey)

Cobalt Triazide (formerly call Cobalt Trinitride or Cobalt Azoimide) $Co(N_3)_2$, mw 142.99, N58.78%; red-brn crysts (anhyd), mp det 148° (0.02 g in 5 sec) (Ref 5), hygroscopic and easily hydrolyzed (Ref 4). The basic cobalt azide, $Co(OH)N_3$ was first prepd in 1898 by Curtius & Rissom (Ref 1). An aq soln of cobalt azide was studied by Dennis & Isham (Ref 2). The anhyd salt, prepd by the action of cobaltous carbonate on hydrazoic acid, was prepd by Wöhler (Ref 3) and by Wöhler & Martin (Ref 4). Methods of prepn are also described in Refs 6,7,8 & 10)

According to Wöhler (Ref 3), Co azide is extremely easily detond by friction,and a 0.01-0.05g compressed sample is detond by impact (Ref 5). Wöhler & Martin (Ref 4) consider Co azide even more expl and more dangerous than either Pb or Ag azide. A thin layer of Cr azide crysts exploded by a hot wire gave a measured vel of deton of 3400 m/s (Ref 10). After being subjected to neutron bombardment no measurable difference in vel of deton was observed.

Cobalt Azide Complexes – Curtius & Rissom (Ref 1) found that *potassium cobaltoazoimide,* $[KN_3 \cdot Co(N_3)_2]$, pptd when strong solns of the two azides were mixed. This compd appeared as bright-blue crysts (pink in soln) which expl at 225°, The *ammonium anologue,* $[(NH_4)N_3(CO(N_3)_2]$, was similar in appearance and props (Ref 1).

Dennis & Isham (Ref 2) observed that on adding pyridine to an aq soln of Co azide, a pink ppt formed which partially dissolved in excess pyridine and on evapn in air yielded a green cryst ppt, insol in water. Another portion of the pink ppt was filtered, washed with water, then with a small amt of pyridine and finally redissolved in excess pyridine. A dark-red soln was obtained which on evapn in a desiccator produced small red transp crysts, $[CoN_6 \cdot 6C_5H_5N]$, insol in w, and which became opaque in w or on exposure to air (Ref 2).

Strecker & Oxenius (Ref 9) were unable to prep by the usual methods Co complexes contg the azido group, because of the tendency of $Co(N_3)_2$ to hydrolyze. They succeeded, however, in prepg by using other methods the following complexes: a) *Hexamminecobaltic azide,* $[Co(NH_3)_6](N_3)_3$, yel solid– by interacting hexamminecobaltic sulfate with Ba azide in aq soln b)*Chloropentammine-cobaltic azide* $[Co(NH_3)_5Cl](N_3)_2$, dk red solid by treating chloropentamminecobaltic sulfate with Ba azide in aq soln (See Table C under Ammines) c)*Tetramminediazidocobaltic azide,* $[Co(NH_3)_4(N_3)_2] \cdot N_3$, red-brn– by treating

tetramminediazidocobaltic chloride with hydrazoic and (See also Table B under Ammines) d)*Diethylenediamminediazidocobaltic azide,* $[Co(C_2H_4NH_2)_2(N_3)_2]N_3$, grn – by treating diethylenediamminediazidocobaltic chloride with hydrazoic acid. All these complexes are sol in w and are expl when dry

Several other complexes of this type are described in Refs 9 and 6

Refs: 1)T.Curtius & J.Rissom, JPrakt Chem **58**, 261-309(1898) & JCS (2) **76**, 92 (1899) 2)L.M.Dennis & H. Isham, JACS **29**, 22(1907) & CA **1**, 528(1907) 3)L.Wöhler, ZAngChem **27**, 335-6(1914) & CA **9**, 1115 (1915) 4)L.Wöhler & F.Martin, Ber **50**, 592 (1917); JCS **112** I, 383-4(1917) & CA **11**, 2900(1917) 5)L.Wöhler & F.Martin, ZAng Chem **30**, 33-9(1917), JSCI **36**, 570(1917) & CA **11**, 3433(1917) 6)Mellor **8**, (1928),355 7)Gmelin, System No **58**, Teil A (1932), 251 8)L.F.Audrieth, ChemRevs **15**, 199 & 201 (1934) & CA **29**, 700(1935) 9)W.Strecker & H.Oxenius, ZAnorgChem **218**, 151(1934) & CA **28**, 5002(1934) 10)F.P.Bowden & K. Singh, ProcRoySoc **227A**, 24(1955) & CA **49**, 4991(1955) 11)H.Rosenwasser, USArmy EngrRes&DevelopLabsRpt 1551-TR 45(1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Cupric Azide (formerly called Cupric Azoimide or Copper Trinitride) $Cu(N_3)_2$, mw 147.59, N 56.93%; dk brn with red tinge, rhmb crysts, mp-begins to dec slowly ca 120° and rapidly ca 150° (Ref 13), deton ca 174° (Ref 4); d at 25° 2.20 to 2.25 (Ref 17), Q_f 139.4 kcal/mol (Ref 20); sol in all acids and in most org bases, sl sol in w, hydrolyzed by boiling w to CuO, insol in neutral solvents (Ref 10)

Cupric azide, with ½ or 1 mol H_2O, was first prepd in 1898 by Curtius & Rissom (Ref 1) by mixing dil aq solns of Cu sulfate and Na azide, washing the ppt with ice w and drying it in a desiccator (Ref 18) They also obtd it by the action of 3.87% hydrazoic acid on Cu pptd by Zn. Browne et al (Ref 6) found that, in addn to $Cu(N_3)_2$, some CuN_3 was formed on electrolysis of a soln of Amm azide in liq NH_3 at –67°, using a copper anode. Pure, anhyd $Cu(N_3)_2$ was prepd by Straumanis & Cirulis (Refs 11 & 13) by the following methods: a)from $Cu(NO_3)_2 \cdot 3H_2O$ and aq $NaNO_3$ b)from $Cu(NO_3)_2 \cdot 3H_2O$ and $LiN_3 \cdot H_2O$ in abs alc c)from $Cu(N_3)_2 \cdot 2NH_3$ by decompn at 100-5° d)from Cu pdr and aq HN_3, as dk coarse crysts and e) from CuO and concd HN_3, as dk grn crysts of intermediate size and particularly sensitive to expln (Ref 13). Although these azides differed slightly in appearance, they all had the same crystn structure as shown by X-ray photographs (Ref 11). For addnl info on prepn of cupric azide, see Refs 7,9,19 & 21

According to Curtius and Rissom (Ref 1), the(ahyd)$Cu(N_3)_2$ was considered to be very sensitive to shock or heat, even when water wet. Based on more recent data, Cirulis (Ref 11 & 13) states that the product is sensitive only when dry or wet with ether; the moist product wet with alc is rather insensitive to friction or shock. The sensitivity of the dry azide to friction is so great that it explodes while being removed from filter paper (Refs 7,18 & 21)

Explosive Properties – *Brisance* – sl greater than $Pb(N_3)_2$ (Ref 13)
Detonation Rate – 5000 to 5500 m/sec (Ref 11)
Explosion Temperature – 174° for 0.02g sample/5 sec (Ref 4) to 202-5° (Refs 9 & 11); not decompd thermally without expln (Ref 5)
Friction Sensitivity – extremely sensitive (Refs 7,11,13,18 & 21)
Gas Volume on Explosion – 607 l/kg as compared to 308 l/kg for LA (Ref 13)
Impact Sensitivity (1 kg wt) – det under impact (Ref 4); 1 cm for crystn product and 2 cm for amor product against 4 cm for LA (Refs 11,13, & 21)
Initiating Efficiency – very small quantities are needed to initiate other expls, for example PETN is initiated by 0.0004g $Cu(N_3)_2$ compared with 0.0025g by LA and 0.18 g by MF (Ref 13)

Power by Trauzl Test – 115 cc/10g sample (Refs 9&11)

Stability in Storage – No loss in wt on storage at RT for one year (Ref 13).

The use of polyvinyl alc or gelatin for the desensitization of cupric azide, its decompn in moist air or high temp and its use in detonators were described recently (Ref 20a)

Infrared absortion spectra were obtained by Delay et al (Ref 17) in the range 3-19μ.

Cirulis (Ref 13) found that LA loaded into copper caps can form copper azide if moisture is present. Hydrazine and hydroxylamine reduce $Cu(N_3)_2$ to white, cuprous azide, CuN_3 (Ref 11). Other reactions involving cupric azide are described below:

Cupric Amminoazide (Ammoniate of Copper Azide), *Diammine copper azide*, $[Cu(NH_3)_2](N_3)_2$, mw 181.65, N 61.69%; green crysts, expl when heated or struck. Obtained by Dennis & Isham (Ref 2) by shaking freshly pptd black cupric hydroxide, while still moist, with an excess of hydrazoic acid, and washing and dissolving the ppt in aq ammonia. This compd was also prepd by Browne et al (Ref 6) and studied by Strecker & Schwinn (Ref 8) and by Straumanis & Cirulis (Ref 11) (See also Ref 21, p 149 and Table D under Ammines in this dictionary). **Tetrammine copper azide**, $[Cu(NH_3)_4](N_3)_2$, mw 215.72, N 64.90%, blue crysts, expl at 202° and on impact. Prepd by Strecker & Schwinn (Ref 8) and by Straumanis & Cirulis (Ref 11) from cupric azide and NH_3, (either liq or the dry gas). Only the di- and tetrammino- compds were prepd, (See also Ref 21, p 149 and Table D under Ammines)

Cupric Azide, Basic (Anhydrous Cupric Oxyazide), $CuO{\cdot}Cu(N_3)_2$, mw 227.13, N 37.00%; yel solid, expl 203-5° (Ref 11), ignites 245° (Ref 3). Cirulis & Straumanis (Ref 11) assigned to it the formula $Cu(OH)N_3$. Basic cupric azide was first prepd by Wöhler & Krupko (Ref 3) on heating cupric azide in w at 70-80°, followed by drying in air free from CO_2, until hydrazoic acid is evolved. This compd expl at 7 to 8 cm under 1 kg impact (Ref 11) and is about one third as sensitive as normal cupric azide (Refs 7&21, p 154). The basic azide, $Cu(N_3)_2{\cdot}Cu(OH)_2$, is formed on retention of water by the oxyazide or prepared by reacting an alc soln of $Cu(NO_3)_2$ with an aq soln of dimethyl or diethylamine and NaN_3 (Ref 11, p 332-4). Another basic azide, $Cu(N_3)_2{\cdot}2Cu(OH)_2$, was prepd by treating $Cu(NH_3)_2(N_3)_2$ with water at 80° until the water becomes col. This product is a yel grn powder, insol in water, hydrolyzed by water above 80°, and is sol in acids and bases. It expl at 199-200° and under 1kg impact at 8 cm (Ref 11, p 332-4) (See also Refs 7,9&21, p 155)

Cupric Azide Complexes. Cupric azide forms numerous complex compds, such as $[Cu(C_5H_5N)_2](N_3)_2$ and $[Cu(C_2H_4(NH_2)_2)_2](N_3)_2$, wherein the azide group is analogous to the corresponding halides (Ref 8). The *cupric pyridine azide*, $Cu(N_3)_2{\cdot}2C_5H_5N$, mw 305.78, N 36.65%; brn ndls insol in water but readily sol in dil acids. Was first prepd by Dennis & Isham (Ref 2) by the action of pyridine on cupric azide. It was studied by Strecker & Schwinn (Ref 8) and by Cirulis & Straumanis (Ref 11, p 341). This compd expl at 205° and under a 1kg impact at 20 cm. It is an expl weaker than $Cu(N_3)_2{\cdot}2NH_3$ (Refs 7 & 21) The *copper azide chloride*, $Cu(N_3)_2{\cdot}3CuCl_2 6H_2O$ or $Cu(N_3)_2{\cdot}3CuCl_2$ prepd by Straumanis & Cirulis (Ref 16) expl at 207-8°. The hydrate cannot be dehydrated. Therefor the anhyd compd should be prepd from abs alc

The general types of copper azide addn compds: a)$[Cu(N_3)_6]^{----}$ b)$[Cu(N_3)_4]^{--}$ c) $[Cu(N_3)_3]^{-}$ and d)$[(N_3)_2CuN_3Cu(N_3)_2]^{-}$ have been prepd and studied by Straumanis & Cirulis (Ref 16). These brn to grn azido cuprates were prepd by dissolving $Cu(N_3)_2$ in aq or alc solns of sol azides. Compds a)&b) are stable in concd aq solns, c) is stable only in alc soln, and d) only in the presence of an excess of RNH_3N_3 or HN_3. The same authors have prepd and studied nonelectrolyte complexes (Ref 11, p 335 & Ref 14) and other azido cuprates with org cations in Refs 12,

15 & 16. Many of these compds are expl and deton when heated or struck (See also Ref 21, p 150-4 and Azido Complexes under Ammines)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 295(1898) & JCS (2) **76**, 92(1899) 2)L.M. Dennis & H.Isham, JACS **29**, 19(1907) & CA **1**, 528(1907) 3)L.Wöhler & W.Krupko, Ber **46**, 2055(1913); JCS **104** II, 703(1913) & CA **7**, 3088(1913) 4)L.Wöhler & F.Martin, Ang Chem [1] **30**, 33-9(1917); JSCI **36**, 570(1917) & CA **11**, 3432(1917) 5)A.R.Hitch, JACS **40**, 1195-1204(1918) & CA **12**, 1951(1918) 6)A.W.Browne et al, JACS **41**, 1770-2(1919) & CA **14**, 28(1920) 7)Mellor **8** (1928), 348 8)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314(1939) 9)A. Cirulis, Naturwissenschaften **27**, 583(1939) & CA **33**, 9175 (1939) 10)M.Straumanis & A.Cirulis, ZAnorgChem **251**, 315-31(1943) & CA **37**, 6573(1943) 11)A.Cirulis & M. Straumanis, ZAnorgChem **251**, 332-54(1943) & CA **37**, 6573-4(1943) 12)M.Straumanis & A.Cirulis, ZAnorgChem **252**, 121-5(1943) & CA **38**, 1701(1944) 13)A.Cirulis, SS **38**, 42-5(1943) & CA **38**, 1879(1944) 14)A.Cirulis & M.Straumanis, JPraktChem **162**, 307-28 (1943) & CA **38**, 1969-70(1944) 15)A.Cirulis & M.Straumanis, Ber **76B**, 825-30(1943) & CA **38**, 1970-2(1944) 16)M.Straumanis & A. Cirulis, ZAnorgChem **252**, 9-23(1943) & CA **38**, 3564(1944) 17)A.Delay et al, CR **219**, 329-33(1944) & CA **40**, 4273(1946); BullFr **12**, 581-7(1945) & CA **40**, 2386(1946) 18) Thorpe **1** (1947), 562 19)Kirk & Othmer **7** (1951), 594 20)T.Waddington & P.Gray, ComptRend 27e CongInterChimInd, Brussels 1954, **3**; IndustrieChimBelge 20, Spec No 327-30 (1955) & CA **50**, 16328(1956) 20a) S.Okubo et al, TokyoKôgyoShikenshoHôkoku **52**, 311,315(1957) & CA **52**, 8559(1958) 21) Gmelin, System No **60**, Teil B, Liefurung 1 (1958), 142-4 & 149-55 22)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1551-TR, 43(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Cuprous Azide (formerly called Cuprous Trinitride or Cuprous Azoimide), CuN_3, mw 105.56, N39.81%; wh or sl yel-grn crysts changing under sunlight to deep red with a violet tinge; mp – deflg ca 174° (Ref 5) to 220° (Ref 4) expl 217° in 5 sec(Ref 19); d 3.26 (Ref 15); Q_e 58.7 k cal/mol (Ref 7); Q_f −67.2 k cal/mol(Ref 17); theor temp on expln 3152° (Ref 7); practically insol in water (0.08g/l) and in 2% HN_3 (0.29g/l) at RT (Ref 14)

Curtius in 1890 (Ref 1) described the existence of cuprous azide, (deep red in color), obtained by treating cuprous oxide with hydrazoic acid. Wöhler & Krupko (Ref 4) reported a new subst, CuN_3, prepd by gradually adding a soln of NaN_3 to an excess of a concd soln of copper sulfite to which K sulfite had been previously added, followed by AcOH in sufficient quant to dissolve the ppt. Browne et al (Ref 8) found that some CuN_3 was formed by electrolysis of a soln of ammonium azide in liq NH_3 at −67° using Cu electrodes, although earlier investigations by Turrentine & Moore (Ref 3) produced electrochemically a compd corresponding to the formula $CuN_3 \cdot 2H_2O$. Straumanis & Cirulis (Ref 14) found that in the reaction of Cu with HN_3, CuN_3 was formed as an intermediate which was subsequently oxydized to $Cu(N_3)_2$. Cirulis reported (Ref 11) that CuO or $Cu(OH)_2$ with an aq soln of HN_3 yielded CuN_3 as fine moss-grn crysts which expld when dry by whisking with a brush. Denigès (Refs 12 & 13) in a study of the analogy between the azide ion and the halogen ion described the prepn of CuN_3 which was obtained as white hexahedral crysts whose props paralled those of the cuprous halides

In a study of the cryst structure of CuN_3, Wilsdorf found (Ref 15) that the azide obtained either by reduction of a $CuSO_4$ soln by $KHSO_3$ and addn to NaN_3 or by treating Cu powdr with NH_4OH show the same X-ray patterns. Suzuki (Ref 16) made thermodynamic studies of CuN_3 and from the reaction Cu + $\frac{3}{2}$ N_2 = CuN_3 obtained the following results: ΔF° = 71.219 cal & ΔH° = 60,230 cal

Cuprous azide is highly sensitive to heat,

impact and friction. The brisance is close to that of Ag azide. Its expl props are affected by cryst size: small crysts (0.06 to 0.09 mm) expl on impact and, when 3 mm in size and dry, may expl by the touch of a feather. Spontaneous deton can occur even under water (Ref 2). It will expl in contact with a red-hot wire and deton either in open air or in vacuo by mechanical effect (Ref 10). Deton by impact under 0.60 kg falling wt occurs from 9.5 cm for 0.01 g sample to 24 cm for 0.05 g sample of small crysts (Refs 4 & 5)

A recent study of the sensitivity of cuprous azide to heat and impact by Singh (Ref 20) confirms earlier investigations showing that sensitivity increases with an increase in cryst size. The activation energy involved in its thermal decompn has been established as 26.5 k cal (Ref 20). In addn to heat of deton, temp develpd on expln, work density and loading density, Wöhler & Martin (Refs 6 & 7) also report the following info with respect to the initiating efficiency of CuN_3:

	Minimum Priming Charge Reqd to Initiate HE's		
HE	CuN_3, g	LA, g	MF, g
Tetryl	0.025	0.025	0.29
PA	0.045	0.025	0.30
TNT	0.095	0.09	0.36
TNA	0.375	0.28	0.37
TNX	0.40	–	0.40

Cuprous azide is thus indicated to be an efficient initiator. For addnl info on prepn of cuprous azide see Refs 9 & 18

In a large variety of detonators, in which LA has been loaded into brass containers, cuprous azide is formed on the surface of containers stored under hot and humid conditions. Extreme care should be exercised in handling cuprous azide or any components of ammunition in which it may be formed

Refs: 1)T.Curtius, Ber **23**, 3023(1890) 2) L.Wöhler, ZAngChem **24**, 2096(1911) & Chem Ztg **35**, 1096(1911) 3)J.W.Turrentine & R.L. Moore, JACS **34**, 375-82(1912) & CA **6**, 1410 (1912) 4)L.Wöhler & W.Krupko, Ber **46**, 2053(1913); JCS **104** II, 703(1913) & CA **7**, 3088(1913) 5)L.Wöhler & F.Martin, ZAng Chem **30**, 33-9(1917) & CA **11**, 3432(1917) 6)L.Wöhler & F.Martin, Ber **50**, 595(1917); JCS **112** I, 384(1917) & CA **11**, 2901(1917) 7)L.Wöhler & F.Martin, SS **12**, 2, 18, 41 & 57(1917) & CA **12**, 629(1918) 8)A.W.Browne et al, JACS **41**, 1772(1919) & CA **14**, 28 (1920) 9)Mellor **8** (1928), 348 10)H.Muraour et al, TransFaradSoc **34**, 991(1938) & CA **32**, 9502(1938) 11)A.Cirulis, Naturwissenschaften **27**, 583(1939) & CA **33**, 9175(1939) 12)G.Denigès, BullTransSocPharmBordeaux **79**, 7 & 12(1941); Bull Fr[5] **10**, 177-80(1943); CR **214**, 651-4(1942) & CA **38**, 6224(1944) 13)G.Denigès, BullTransSocPharmBordeaux **80**, 97-104(1942); ChemZtr **1943** I, 2674-5 & CA **38**, 4876(1944) 14)M.Straumanis & A. Cirulis, ZAnorgChem **251**, 315(1943) & CA **37**, 6573(1943) 15)H.Wilsdorf, ActaCryst **1**, 115-8(1948) & CA **42**, 7594(1948) 16)S. Suzuki, JChemSocJapan, PureChemSec **74**, 269(1953) & CA **47**, 11934(1953) 17)T. Waddington & P.Gray, ProcRoySoc **235A**, 489(1956) 18)Gmelin, System No **60**, Teil B, Lieferung 1 (1958) 142-4 19)H.Rosenwasser, USArmyEngrRes&DevelopLabsRpt 1551-TR, 43(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey) 20)K. Singh, TransFaradSoc **55**, 124(1959)

Cyanazide, See under Cyanocompounds and Derivatives

Cyanuric Triazide, See under Cyanocompounds and Derivatives

Dicyandiazide, See under Cyanocompounds and Derivatives

Fluorine Azide, FN_3, mw 61.02, N68.86%, grn-yel solid at −154°, mp explodes on evapg and at RT decomps to N_2 and N_2F_2. It was prepd by A.W. Browne & J.F. Haller in 1942 by treating HN_3 with fluorine in a stream of N_2 [J.F.Haller, Dissertation, Cornell U (1942) cited by N.V.Sidgwick, "The Chemical Elements and their Compounds", Vol 1 (1950) p 718 Oxford University Press, London]. No further work is known to have been reported since the original prepn of fluorine azide

Gallium Triazide, $Ga(N_3)_3$, mw 195.79, N 64.39%; col crysts, sol in tetrahydrofuran, sens to moisture. Prepd by Wiberg & Michaud (Ref 1) by adding an eth soln of excess HN_3 to a frozen eth soln of GaH_3 and thawing the mixt at RT. Excess HN_3 and eth were removed by distn at −25° and heating to RT in vacuo. The eqn for the reaction is $GaH_3 + 3HN_3 \rightarrow Ga(N_3)_3 + 3H_2$. Analysis of the azide gave $GaH_{0.2}(N_3)_{2.8}$. No expl props were given

Refs: 1)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 502-3(1954) & CA **49**, 768(1955) 2)H. Rosenwasser, USArmyEngrRes&DevelopLabs Rpt 1551-TR, 48(1958) "Hydrazoic Acid and the Metal Azides (a literature survey)

Gold Azide (Aurous Azide), AuN_3, mw 239.22, N17.5%; orange ndls extremely expl were obtained by Curtius & Rissom (Ref 1) on evapg a soln of a mixt of gold chloride and Na azide. They also obtd a *Sodium Gold Azide,* as an orange, crystn, extremely expl residue, on evapg a soln of a mixt of aurochloric acid and Na azide. The structure of these compds was not supported with evidence. While investigating methods of prepg Au azide, Rogers (Ref 3) obtd sodium gold azide and detd its props and structure: $NaAu_{1.43}N_{9.05}$, or-red ndls; mp begins to dec at 90°, rapid decompn at 117° and expl at ca 130°; extremely sol in w giving unstable aq solns which dec on standing in light, sol in alc or acet, sl sol in dry eth and insol in chlf or CCl_4. Its method of prepn was the treating a soln of aurochloric in eth with dry Na azide. The color of the soln changed rapidly from bright yel to deep rd-brn indicating immediate reaction. After standing overnight with intermittent shaking the soln was filtered and evapd to dryness at RT. Purification was made by washing with eth and crystg from abs alc (Ref 3)

Microscopic examination and qualitative analysis proved the existence of a pure compd which was extremely expl and unstable even in aq solns (Ref 3)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 304(1898) & JCS **76** II, 92(1899) 2)Mellor **8** (1928), 349 3)G.T.Rogers, JInorgNuclear Chem **5**, 339(1958) & CA **52**, 8815(1958) 4) H.Rosenwasser, USArmyEngrRes&Develop LabsRpt 1551-TR, 43(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Hydrazine Azide (formerly called Hydrazine Azoimide, Hydrazine Trinitride or Hydrazonium Azide) (called by Curtius Diammonium Azide, N_5H_5), $N_2H_4 \cdot HN_3$, mw 75.08, N93.29%; rhmb, hygr crysts; mp 75.4(Ref 6); v sol in w, sol in hydrazine(190% at 23°), methanol (6.1% at 23°) and in alc (1.2% at 23°C), not appreciably sol in chlf, carbon tetrachloride benz, carbon disulfide, ethyl acetate or diethyl ether (Ref 6). First prepd by Curtius in 1891 (Ref 1) by neutralizing hydrazoic acid with hydrazine

hydrate or by pouring hydrazine hydrate over ammonium azide and evapg the mixt in a flat dish placed in a desiccator. This latter method of prepn was patented by Müller in 1936 (Ref 10). The cryst product obtained by Curtius was in the form of long lustrous plates or prisms (mp ca 50°) which detonated violently on rapid heating but the azide also burned quitely with a smoky yel flame when heated slowly (Ref 1). The moist salt is also expl (Ref 11). Curtius & Rissom (Ref 2) reported that hydrazine azide begins to melt at 65° and decomp energetically at 108°. Dresser & Browne (Ref 6) prepd a very pure material (mp 75.4°) which was relatively stable since it showed only very sl decompn when heated to 110° in vacuo for several days. Thrown upon a hot plate it burned with a puff, but without deton. According to Dresser & Browne, hydrazine azide is entirely insensitive to ordinary mech shock or impact (Ref 6). This compd reacts readily with benzaldehyde and with acet and is regarded by Ephraim (Ref 11) as a polymer of imide, $(NH)_x$ (See also Refs 4,7 & 9)

Hydrazine Azide Monohydrazinate, $N_2H_5N_3 \cdot N_2H_4$, mw 107.13, N 91.53%; wh delq crysts, mp 66.4°; v sol in w or in anhyd hydrazine. Prepd by treatment of a nearly satd soln of hydrazine azide in anhyd hydrazine with an equal vol of abs alc (Ref 6). This solvate was first obtained by Riegger in the lab of Cornell Univ (Ref 3) and has the same empirical formula as normal hydrazonitrous acid (3-hydrazinopentazane) or as heptazane (Refs 5 & 6). In order to establish the identity of this monohydrazinate and establish whether higher solvates exist, the temp-concn diagram was detd for the system hydrazine azide-hydrazine. This investigation showed only one solvate formed, with eutectics located at 51° and −17°C (Ref 6) (Also see Refs 7 & 9). No expl props were mentioned

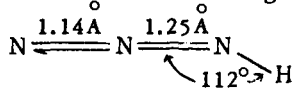
Hydrazine Azide Hemiammonate, $(N_2H_5N_3)_2NH_3$, mw 167.21, N 92.16%; wh delq crysts which exhibit extreme hygr on exposure to air but stable in the absence of moisture. It was isolated and identified by Howard & Browne (Ref 8) in a study of equilibria in the system hydrogen azide-ammonia. It is easily prepd by condensing liq ammonia upon solid hydrogen azide and permitting the soln to evap to dryness upon warming to RT (Ref 8, p 2352). In liq ammonia, the hemiammonate undergoes ammonolysis to an extent that varies directly with temp and with the concn of ammonia. (See also Ref 9). No expl props were determined.

Refs: 1).T.Curtius, Ber (2) **24**, 3342,3348-9 (1891); JCS **62** I, 113(1892) & Ber **29**, 772 (1896) 2)T.Curtius & J.Rissom, JPraktChem **58**, 261(1898) & JCS **76** II, 92(1899) 3)H.E. Riegger, PhD Thesis, "Hydronitric Acid and Hydrazine Trinitride," Cornell University, Ithaca, NY(1910) 4)Mellor **8** (1928), 344 5) A.W.Browne & F.Wilcoxon, JACS **48**, 683 (footnote 13) (1926) 6)A.L.Dresser & A.W. Browne, JACS **55**, 1963(1933) & CA **27**, 3417 (1933) 7)L.F.Audrieth, ChemRevs **15**, 202 (1934) & CA **29**, 700(1935) 8)D.H.Howard, Jr & A.W.Browne, JACS **56**, 2348(1934) & CA **29**, 700(1935) 9)Gmelin, System No 23 (1936), 549-50 10)E.Müller, GerP 634688 (1936) & CA **31**, 511(1937) 11)Ephraim (1943), 662

HYDROGEN AZIDE AND HYDRAZOIC ACID

(formerly called *Azoimide, Ammonitric Acid, Hydrazonitrous Acid or Hydronitric Acid)* (Stickstoffwasserstoffsäure in Ger). It exists as anhydrous and as aqueous hydrogen azide. The latter is called hydrazoic acid. The structure of hydrogen azide and of hydrazoic acid has been the subject of a number of investigators, such as Mendeléeff (Ref 2), Thiele (Ref 27 & 28), Turrentine (Ref 35), Franklin (Ref 62), Hendricks & Pauling (Ref 63), Herschberg et al (Ref 98), Pauling & Brockway (Ref 103), Davis (Ref 104), Buswell et al (Ref 105), Eyster (Ref 111), Shomaker & Spurr (Ref 118), and Lieber et al (Ref 140). Various other investigators studied the behavior of azides and proposed structures

(See Refs 58,69,71,79,90,96,101,117,136,137, 142,147,148,150 & 154). It is now generally agreed that the hydrogen azide molecule has a hydrogen atom linked by a bond at an angle 112° to the linear azide group:



Hydrogen Azide, Anhydrous, HN_3, mw 43.03, N 97.66% Col liq, fr p −80° (Ref 18), bp 35.7° (Ref 99) & 37° (Ref 18), $d_t = 1.126/(1+0.0013t)$, where t = 0 to 20° (Ref 99); $Q_{decompn(liq)}$ 67 ± 5% kcal/mol(Ref 77), $Q_{f(gas)}$ −70.9 ± 0.5 kcal/mol (Ref 99); Rossini (Ref 146) gives for heat of formn at 25° −70.3 kcal/mol; $Q_{soln\ in\ H_2O}$ 9.7 ± 0.1 kcal/mol (Ref 99), Q_{vapzn} at 12.4° 7.3±0.01 kcal/mol(Ref 99); vapor press can be calcd from the formula $\log_{10} P = 8.198 - 1643/T$ mm Hg (Ref 130). Values for the dipole moment were measured by Sidgwick (Ref 91) and by Amble & Dailey (Ref 137)

Browne & Lundell(Ref 23) found pure anhyd HN_3 to have low electrical conductivity but the addn of potassium azide very greatly increased its conductivity. Hydrogen azide is sol in w, alc or eth and is itself a solvent for many substs as found by qual investigations of McKinney(Ref 59). Vapors of HN_3 are considered dangerous (Refs 94,123,124 & 160); low concns produce eye irritation and headache, high concns affect the central nervous system and continued exposure may cause death (Refs 3,67,84,102,131 & 133). The gas, aq solns and its salts act as protoplasmic poisons (Refs 67,84,145 & 151). Two cases of accidental poisoning have been reported (Refs 66,86)

Anhydrous hydrogen azide was first prepd in 1891 by Curtius & Radenhausen by fractionally distilling the aq soln with fused calcium chloride(Ref 4). In 1907 Dennis & Isham isolated the pure compd in larger quanty and detd some physical and chem props (Ref 18). The pure compd is extremely expl but it can be kept for days at RT in sealed tubes without change (Refs 18 & 130). After months of storage its tendency to expl spontaneously becomes much greater (Ref 93). It expl readily when subjected to the slightest shock or when heated. Thermal decompn takes place at 290° and at 4 cm press, 9-11% of HN_3 decompd in 25 min (Ref 80). Traces of impurities catalyze the reactn to expln (Ref 93). Gaseous HN_3 also decomp explosively under the influence of an elec spark, with emission of yel light, at all press of HN_3 above 5 mm (Ref 156). Introduction of H_2, N_2 or A into the system markedly inhibits expl reaction, H_2 exerting the largest inhibitive effect (Ref 156). When frozen at 4°K, the products from the decompn of gaseous HN_3 by elec discharge formed a blue solid identified as NH_4N_3 (Ref 152). Deposition of HN_3 at 77°K, in the absence of elec discharge, gave a clear col glass that became polycrystn at 148°K. No other prods were observed above 148°K (Refs 46,141 & 152). Ultra-violet light of 2200 Å decomposes gaseous HN_3 into N_2, H_2 and NH_4N_3 (Ref 72). Beckman & Dickinson (Ref 85) have proposed a mechanism for this decompn and they detd the no of mols decompd by measuring the press of the N_2 and H_2 produced

The Hg photosensitized (at 2537 Å) decompn of gaseous HN_3 at press from 0.3 to 20 mm is reported to parallel closely its photochem decompn (Ref 97). The variation of absorption as a function of press and the influence of other gases are given by Verdier (Ref 121), mol coefficients of absorption by Bonnemay & Verdier(Ref 129a), and fluorescence of the HN_3 mol by Gaviola & Wood (Ref 73)

The spectra of the expln of HN_3 at 18° to 130° and of HN_3–Hg vapor mixts have been studied by Tolmachev (Ref 115) who found a similarity with the spectra of slow thermal decompn flames. Pannetier (Ref 138), in a study of the expln of HN_3 vapor, observed two new bands of the NH radical at 3240 and 3253 Å. A review by Bonnemay(Ref 127) on the photochem, thermal and elec decompn of HN_3 supports the hypothesis (Ref 89) that in gaseous, liq or solid phases a chain

reaction is propagated by atoms of nitrogen. The free radical of N*, or possibly N_3, has been detected by Thrush (Ref 158). Infrared spectroscopic studies of the decompn of HN_3 have been reported by Dowes et al (Ref 157) and by Becker & Pimentel (Ref 159), and an exptl value of the dissocn energy has been detd by Pannetier & Gaydon(Ref 139)

The mechanism of the expl decompn of pure hydrogen azide and of its mixts with H_2 has been studied in some detail by Avanesov & Rukin(Ref 108). The propagation of deton is periodic and consists of the transfer of the elastic impulse in the still unchanged gas and of the decompn of the particles which have been subjected to the impulse(Ref 108)

Use. Liq HN_3 has been found by McKinney (Ref 59) to act as a solvent for many substs, especially inorg compds

Hydrogen Azide, Aqueous or Hydrazoic Acid (HA), $HN_3 + nH_2O$ (aq distillate has contd up to 27% HN_3, Ref 94, p 183) was first obtained in 1890 by Curtius(Ref 1) on treatment of benzoyl azide with NaOH, followed by distn with H_2SO_4. Subsequently Curtius used instead of NaOH, Na ethylate (Ref 5) and also alc ammonia(Ref 8). Methods of prepn employed by other investigators may be divided into the following general classes: a)*direct syntheses* (Refs 78,88 & 131a) b) *interaction of hydrazine and nitrous acid*(Refs 7,9,11,35,55,62 & 64) c)*oxidation of hydrazine* (Refs 15,19,20,21,22,36,42,48,49,52,68,74 & 95) d)*ammonolysis of nitrous oxide and nitrates*(Refs 6,12,14,64,81 & 87) and e) *oxidation of triazines and decompn of higher hydronitrogens*(Refs 7,16,43,83,110 & 153). Addnl info on methods of prepn may be found in the books of Mellor(Ref 75), Gmelin(Ref 100), Thorpe(Ref 132) & Kirk & Othmer(Ref 144) and in a review by Audrieth(Ref 94)
Note: See also "A Safe Method for Preparation of Uncontaminated Hydrazoic Acid" by M.D.Kemp, JChemEduc **37**, 142 (March 1960)

Ultra violet decompn of HA has been studied by many investigators (Refs 70,119, 122,126,129,135 & 143). The threshold of photodecompn is at 2550 Å (Ref 119) and decompn occurs after a short induction period (Refs 122 & 126) at a rate indicated by the gases evolved (Ref 126). The energy of activation of the decompn of HA was calcd as 695 cal/mol (Ref 129b). In the electrolysis of aq solns of HA, low intensity UV emission has been observed in the gaseous phase (Refs 107,112 & 120)

West(Ref 13) made conductivity measurements of HA and calcd the limiting value to be 1.86×10^{-5}. Oliveri-Mandalà also made conductivity measurements and calcd a dissociation constant and an ionization constant. Also see values reported by Quintin (Ref 113) and by Yui(Ref 116)

Roth & Müller(Ref 77) calcd the Q_f (dil soln) as -53.3 kcal/mole and Q_{neutn} by $Ba(OH)_2$ as 10.0 kcal/mol and Q_{neutn} by NH_3 as 8.2 kcal/mol. Rossini et al(Ref 146) reported Q_f values at 25°C for HA solns of various concns. Other thermodynamic props have been calcd by Eyster & Gillette(Ref 114) and by Waddington & Gray(Ref 155). Racz(Ref 125) has reviewed the literature on the stability, thermodynamics and photochemistry of HA

The chemical reactions of HA have been the subject of numerous investigations. It reacts with acids (Refs 17,21,26,33,37,45, 56,60,64,82,92 & 109), oxidizing agents (Refs 14,32 & 76), reducing agents (Ref 34), and it forms expl derivs such as azides (Refs 38, 39 & 44), tetrazoles(Refs 24,25,30,31,40 & 60) and others (Refs 46,47 & 61). Platinum black and Raney nickel decomp solns of hydrazoic acid to form ammonia and nitrogen (Refs 51 & 106). Other reactions are also described (Refs 29,41,43,53,57,59,128,132, 147a & 149)

Explns involing HN_3 or HA may be avoided by carrying the gas as it is formed into a stream of nitrogen or air and absorbing the gas in Ba hydroxide soln(Ref 134). During the investigation of the vel of deton of hydrazoic acid, the bottle contg it exploded on agitation, killing a man (Ref 65). The vel of deton of pure HN_3 has been measured by photographic techniques as 2650 ± 100m/sec(Ref 108)

Uses - The acid character of hydrazoic acid

has made it the basis for use in several analytical methods (Refs 10,13 & 56). It is a unique compd of hydrogen and nitrogen, many derivatives of which are expl and, being solids, are used in comml and military expls.

Refs: 1)T.Curtius, Ber **23**, 3025(1890) & JCS **60**I, 57(1891) 2)D.Mendeléeff, Ber **23**, 3464(1890) 3)O.Loew, Ber **24**, 2948(1891) 4)T.Curtius & R.Radenhausen, JPraktChem **43**, 207(1891) & JCS **60** I, 524(1891) 5)T. Curtius, Ber **24**, 3341(1891) 6)W.Wislicenus, Ber **25**, 2084(1892); JCS **62** II, 1151(1892) 7) T.Curtius, Ber **26**, 1263(1893) 8)T.Curtius, Ber **29**, 781(1896) 9)M.Dennstedt & W. Göhlich, ChemZtg **21**, 876(1897) & JCS **74** II, 425-6(1898) 10)T.Curtius & J.Rissom, JPraktChem **58**, 261(1898) & JCS **76** II, 92 (1899) 11)S.Tanatar, Ber **32**, 1399(1899) 12)E.C.Szarvasy, JChemSoc **77**, 606(1900) 13)C.A.West, JCS **77**, 705(1900) 14)L.M. Dennis & A.W.Browne, JACS **26**, 577(1904) & ZAnorgChem **40**, 68(1904) 15)A.W.Browne, JACS **27**, 551(1905) 16)A.Darapsky, Ber **40**, 3033(1907) & CA 1, 2479(1907) 17)L.M. Dennis & H.Isham, JACS **29**, 18(1907) & CA **1**, 528(1907) 18)L.M.Dennis & H.Isham, JACS **29**, 216(1907); Ber **40**, 458(1907) & CA **1**, 1102(1907) 19)A.W.Browne & F.F. Shetterly, JACS **29**, 1305(1907); Ber **40**, 3953(1907) & CA **2**, 234(1908) 20)R.Stollé, GerP 205683(1908) & CA **3**, 1805(1909) 21) J.Thiele, Ber **41**, 2681(1908) & CA **2**, 3315 (1908) 22)R.Stollé, FrP 400445(1909) & CA **5**, 977(1911) 23)A.W.Browne & G.E.Lundell, JACS **31**, 435(1909) & CA **3**, 1373(1909) 24) F.C.Palazzo, AttiAccadLinceiMem[5], **191**, 218(1910) & CA **4**, 2455(1910) 25)E.Oliveri-Mandalà, AttiAccadLinceiMem[5], **191**, 228 & CA **4**, 2455(1910) 26)E.Oliveri-Mandalà & A.Coppola, AttiAccadLinceiMem[5], **19** I, 563(1910) & CA **4**, 2455(1910) 27)J.Thiele, Ber **44**, 2522(1911) & CA **5**, 3820(1911) 28) J.Thiele, Ber **44**, 3336(1911) & CA **6**, 630 (1912) 29)E.Oliveri-Mandalà & B.Alagna,Gazz **40** II, 441(1910); ChemZtr **1911** I, 662 & CA **5**, 3571(1911) 30)E.Oliveri-Mandalà, Gazz **41** I, 59(1911) & CA **5**, 2636(1911) 31)E.Oliveri-Mandalà & T.Passalacqua, Gazz **41** II, 430 (1911) & CA **6**, 625(1912) 32)H.E.Riegger, JACS **33**, 1569(1911) & CA **6**, 46(1912) 33) E.A.Werner, ProcChemSoc **28**, 257(1911) & CA **7**, 2931(1913) 34)J.W.Turrentine & R.L. Moore, JACS **34**, 375 & 382(1912) & CA **6**, 1410(1912) 35)J.W.Turrentine, JACS **34**, 385(1912) & CA **6**, 1410(1912) 36)H.Staudinger, GerP 273667(1913) & CA **8**, 2787 (1914) 37)E.Oliveri-Mandalà & F.Noti, Gazz **43** I, 304(1913) & CA **7**, 2934(1913) 38)E. Oliveri-Mandala & F.Noti, Gazz **43** I, 514 (1913) & CA **7**, 3495(1913) 39)E.Oliveri-Mandalà & E.Calderaro, Gazz **43** I, 538(1913) & CA **7**, 3755(1913) 40)E.Oliveri-Mandalà & T.Passalacqua, Gazz **43** II, 465(1913) & CA **8**, 1272(1914) 41)P.J.Kirkby & J.E. Marsh, ProcRoySoc **88A**, 90(1913) & CA **7**, 3875 & 3932(1913) 42)F.Sommer, ZAnorg Chem **86**, 71(1914) & CA **8**, 1932(1914) 43) E.Oliveri-Mandalà, Gazz **44** I, 662 & 670(1914) & CA **9**, 70-1(1915) 44)L.Wöhler, ZAng Chem **27** I, 335(1914) & CA **9**, 1115(1915) 45)F.Sommer, Ber **48**, 1833(1915) & CA **10**, 342(1916) 46)E.Oliveri-Mandalà & E.Calderaro, Gazz **45** I, 307(1915) & CA **10**, 596-7(1916) 47)E.Oliveri-Mandalà, Gazz **45** II, 120(1915) & CA **10**, 1514(1916) 48)A.W.Browne & O.R.Overman, JACS **38**, 285(1916); CA **10**, 727(1916) & ZAnorgChem **94**, 217(1916) 49) F.Sommer, ZAnorgChem **96**, 75(1916); JCS **112** II, 30(1917) & CA **11**, 1374(1917) 50) E.Oliveri-Mandalà, Gazz **46** I, 298(1916) & CA **11**, 1142(1917) 51)E.Oliveri-Mandalà, Gazz **46** II, 137(1916) & CA **11**, 1608-9(1917) 52)F.Sommer & H.Pincas, Ber **49**, 259(1916) & CA **10**, 1138-9(1916) 53)E.Oliveri-Mandalà, Gazz **48** II, 35(1918) & CA **13**, 844 (1919) 54)A.Angeli, AttiAccadLinceiMem [5], **27** II, 389(1918); JCS **116** II, 149(1919) & CA **13**, 3100(1919) 55)W.R.Hodgkinson, BritP 128014(1918) & CA **13**, 2425(1919) 56)E.Oliveri-Mandalà, Gazz **51** I, 138(1921) & CA **15**, 3039(1921) 57)E.Oliveri-Mandalà, Gazz **52** II, 139(1922) & CA **17**, 1642(1923) 58)A.W.Browne & A.B.Hoel, JACS **44**, 2116 (1922) 59)A.E.McKinney, "The Behavior of

Anhydrous Hydronitric Acid Toward Various Inorganic Salts," PhDThesis, Cornell U, NY (1923) 60)K.F.Schmidt, Ber **57B**, 704(1924) & CA **18**, 2868(1924) 61)A.Korczyński & S.Namyslowski, BullFr **35**, 1186(1924) & CA **19**, 644(1925) 62)E.C.Franklin, JACS **46**, 2137(1924) 63)S.B.Hendricks & L.Pauling, JACS **47**, 2917(1925) 64)A.W.Browne & F.Wilcoxon, JACS **48**, 682(1926) & CA **20**, 1185(1926) 65)A.Cooper-Key, "50th Annual Rpt of HMInspExpls," Pamphlet(1926) 45 pp & CA **20**, 3085(1926) 66)R.Stern, Klin Wochschr **6**, 304(1927) & CA **21**, 1847(1927) 67)W.Biehler, ArchExptlPathPharmakol **126**, 1-9(1927) & CA **22**, 823(1928) 68)J.Martin, JACS **49**, 2133(1927) 69)A.Angeli, Atti AccadLinceiMem [6],**5**, 732(1927) & CA **21**, 3603(1927) 70)K.Gleu, Ber **61B**, 702(1928) & CA **22**, 2324(1928) 71)H.Lindemann & H.Thiele, Ber **61B**, 1529(1928) & CA **22**, 3598(1928) 72)A.O.Beckman & R.G.Dinkinson, JACS **50**, 1870(1928) & CA **22**, 3840(1928) 73)E.Gaviola & R.W.Wood, PhilMag[7] **6**, 1191(1928) & CA **23**, 771(1929) 74)R.E. Kirk & A.W.Browne, JACS **50**, 337(1928) 75) Mellor **8** (1928), 328-35 & 341-2 76)K.Gleu & E.Roell, ZAnorgChem **179**, 233(1929) & CA **23**, 3179(1929) 77)W.A.Roth & F. Müller, Ber **62B**, 1188(1929) & CA **23**, 4400 (1929) 78)W.Moldenhaur & H.Möttig, Ber **62B**, 1954(1929) 79)V.K.Pershke, Ber **62B**, 3054(1929) & CA **24**, 1624(1930) 80)H.C. Ramsperger, JACS **51**, 2134(1929) & CA **23**, 4617(1929) 81)W.Hoth & G.Pyl, ZAngChem **42**, 888(1929) & CA **23**, 5547(1929) 82)A. Hantzsch, Ber **63B**, 1784(1930) 83)L.F. Audrieth, JChemEd **7**, 2055 (1930) 84)W. Biehler, KnollsMitt **1927**, 21-6; BergesPhysiol ExptlPharmakol **49**, 829(1930) CA **24** 659(1930) 85)A.O.Beckman & R.G.Dickinson, JACS **52**, 124(1930) & CA **24**, 1034(1930) 86)Z.Kocher, KlimWochschr **9**, 2160(1930) & CA **25**, 1907 (1931) 87)J.Meissner, BritP 369,529(1930); CA **27**, 2255(1933) & ChemZtr **1920** II, 715 88)H.Wattenberg, Ber **63**, 1667(1930) 89) D.Alekséev, ZhFizKhim **2**, 535(1931) & CA **27**, 5979(1933) 90)A.Hantzsch, Ber **66B**, 1349(1933) & CA **28**, 985-6(1934) 91)N.V. Sidgwick, TransFaradSoc **30**, 801(1934) 92) E.C.Franklin, JACS **56**, 568(1934) & CA **28**, 2289(1934) 93)R.Meyer & H.J.Schumacker, ZPhysChem **170A**, 33(1934) & CA **28**, 7133 (1934) 94)L.F.Audrieth, ChemRevs **15**, 169 (1934) & CA **29**, 700(1935) 95)M.Möller, KglDanskeVidenskabSelskabMath-fysMedd **12**, No 16, 61 pp (1934) & CA **29**, 3930(1935) 96)A.Hantzsch, Ber **67B**, 1674(1934) 97) A.E.Myers & A.O. Beckman, JACS **57**, 89 (1935) & CA **29**, 2089(1935) 98)G.Hershberg et al, ZElectrochem **41**, 522(1935) & CA **29**, 7806(1935) 99)P.Günther et al, ZPhysChem **175A**, 154(1935) & CA **30**, 2094(1936) 100) Gmelin, System No 4, Lieferung 3(1936), 285-303 101)W.Engler & K.W.Kohlrausch, ZPhysChem **34B**, 214(1936) & CA **31**, 1296 (1937) 102)F.Hildebrandt & D.F.Schmidt, ArchExptlPathPharmakol **187**, 155(1937) & CA **32**, 8557(1938) 103)L.Pauling & L.O. Brockway, JACS **59**, 13(1937) 104)M.M. Davis, TransFaradSoc **35**, 1184(1939) & CA **33**, 9141(1939) 105)A.M.Buswell et al, JACS **61**, 2809(1939) & CA **33**, 9141(1939) 106)L.Irrera, AttiX°CongrInternChim **3**, 206 (1939) & CA **33**, 9175(1939) 107)R.Audubert & E.T.Verdier, CR **208**, 1984(1939) & CA **33**, 6162(1939) 108)D.S.Avanesov & L.G.Rukin, KhimReferatZh **1939**, No 6,4 & CA **34**, 3496 (1940) 109)G.Caronna & B.Sansone, Gazz **69**, 739(1939) & CA **34**, 5363-4 (1940) 110) L.F.Audrieth & C.F.Gibbs, InorgSynth **1** (1939), 77-9 & CA **36**, 2488(1942) 111)E.H. Eyster, JChemPhys **8**, 141(1940) & CA **34**, 1562(1940) 112)R.Audubert & C.Racz CR **210**, 217(1940) & CA **34**, 2709(1940); BullFr **7**, 907(1940) & CA **36**, 2209(1942) 113)M. Quintin, CR **210**, 625(1940) & CA **34**, 4647 (1940) 114)E.H.Eyster & R.H.Gillette, JChemPhys **8**, 369(1940) & CA **34**, 4663-4 (1940) 115)Y.M.Tolmachev, ZhFizKhim **14**, 10 & 615(1940) & CA **35**, 975 & 3904(1941) 116)N.Yui, BullInstPhysChemRes(Tokyo) **20**, 390(1941); EngAbstract in SciPapers InstPhysChemRes(Tokyo) **38**, Nos 1034-5 & CA **36**, 1230-1(1942) 117)E.Oliveri-Mandalà & G.Caronna, Gazz **71**, 182(1941) & CA **36**, 2804(1942) 118)V.Schomaker &

R.Spurr, JACS **64**, 1184(1942) & CA **36**, 3996 (1942) 119)M.Bonnemay & E.T.Verdier, CR **214**, 228(1942) & CA **37**, 3341(1943) 120)E. T.Verdier, CR **214**, 617(1942) & CA **37**, 6194 (1943) 121)E.T.Verdier, CR **214**, 953(1942) & CA **38**, 4511(1944) 122)M.Bonnemay, CR **215**, 65(1942) & CA **38**, 5457(1944) 123)L.T. Fairhall et al, USPubHealthRpt **58**, 607 (1943) & CA **37**, 3833(1943) 124)Anon,Natl-SafetyCouncil, Inc,WartimeSafetyDigest (1943) & CA **37**, 6461(1943) 125)C.Racz, JChimPhys **40**, 109(1943) & CA **39**, 1799 (1945) 126)M.Bonnemay, CR **216**, 230 & 882 (1943) & CA **39**, 1594 & 2255(1945) 127)M. Bonnemay, JChimPhys **40**, 231(1943) & CA **39**, 2939(1945) 128)Davis(1943), 426-30 129a)M.Bonnemay & E.T.Verdier, JChimPhys **41**, 113(1944) & CA **40**, 2384(1946) 129b) M.Bonnemay, JChimPhys **41**, 18(1944) & CA **39**, 3205-6(1945); JChimPhys **41**, 56(1944) & CA **40**, 1736(1946) 130)K.Stewart, Trans FaradSoc **41**, 663(1945) & CA **40**, 3312(1946) 131)N.S.Pravdin & S.B.Shakhnovskaya, Farmakol i Toksikol **8**, No 5,50(1945) & CA **40**, 7410(1946) 131a)K.Stewart, Nature **157**, 191(1946) & CA **40**, 3057(1946) 132)Thorpe, **1**(1947), 580-1 & **2**(1946), 572 133)J.D.Graham et al, JIndHygToxicol **30**, 98(1948) & CA **42**, 3571-2(1948) 134)MatérielTeléphonique, FrP 938720 (1948) & CA **44**, 291(1950) 135) E.Oliveri-Mandalà & G.Werber, Gazz **80**, 177 (1950) & CA **44**, 9262(1950) 136)A.Bonnemay & R.Daudel, CR **230**, 2300(1950) & CA **44**, 8711(1950) 137)E.Amble & B.P.Dailey, J ChemPhys **18**, 1422(1950) & CA **44**, 10430 (1950) 138)G.Pannetier, CR **232**, 817(1951) & CA **45**, 5514(1951) 139)G.Pannetier & A.G.Gaydon, JChimPhys **48**, 221(1951) & CA **46**, 1354(1952) 140)E.Lieber et al, Annal Chem **23**, 1594(1951) & CA **46**, 3857-8(1952) 141)F.O.Rice & M.Freamo, JACS **73**, 5529 (1951) & CA **46**, 6206(1952); JACS **75**, 548 (1953) & CA **47**, 5291(1953) 142)J.D.Rogers & D.Williams, PhysRevs **82**, 131(1951) & CA **46**, 6925(1952) 143)E.Oliveri-Mandalà & G. Valenti, RicercaSci **21**, 232(1951) & CA **47**, 10352(1953) 144)Kirk & Othmer **7**(1951), 591-5 145)B.Chance, JBiolChem **194**, 483(1952) & CA **46**, 5107(1952) 146)F.D.Rossini et al, "Selected Values of Chemical and Thermodynamic Properties," USGovtPrintgOffice (1952), 55 147)W.J.Thomas, JCS **1952**, 2383 & CA **46**, 10714(1952) 147a)C.L. Arcus et al, JCS **1953**, 178 & 3698; JCS **1954**, 4319 148)W.J.Thomas, TransFaradSoc **49**, 855(1953) & CA **48**, 5570(1954) 149)J. Bandoz-Lambling, AnnChim(Paris) **8**, 586 (1953) & CA **48**, 10474(1954) 150)W.V. Bhagwat & R.P.Shukla, AgraUnivJRes(Sci) **2**, 187(1953) & CA **49**, 2293(1955) 151)W. Scheler et al, ZBiochem **325**, 258 & 401 (1954); CA **48**, 8286(1954) & **49**, 5555(1955) 152)I.L.Mador & M.C.Williams, JChemPhys **22**, 1627(1954) & CA **49**, 42-3 (1955) 153) R.T.Merrow & R.W.VanDolah, JACS **76**, 4522(1954) & CA **49**, 13069(1955) 154)K. Singh, ProcRoySoc **225A**, 533(1954) & CA **49**, 15585-6(1955) 155)T.C.Waddington & P. Gray, CR, 27^{e} CongrInternChimInd(Brussels) **3**(1954); IndChimBelge **20**, Spec No 327-30 (1955) (in English); ProcRoySoc **235A**, 106 (1955) & CA **50**, 12627 & 16328(1956) 156) G.Pannetier & M.Lecamp, BullFr **1954**, 1068 & CA **49**, 7247-8(1955) 157)D.A.Dowes et al, JChemPhys **23**, 1258 & 1606(1955); CA **49**, 14484-5(1955) & CA **50**, 676(1956) 158) B.A.Thrush, ProcRoySoc **235A**, 143(1956) & CA **50**, 12660(1956) 159)E.G.Becker & G.C.Pimentel, JChemPhys **25**, 228(1956) & CA **50**, 15244(1956) 160)Sax(1957), 761 161)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1151-TR, 1-9 (1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Iodine Azide or **Iodoazide** (called Jodazid or Azoimidjodid in Ger), IN_3 mw 168.93 N 24.88%; yel volat solid, extremely dangerous expl when exposed to heat or shock; sol in w, giving a neutral soln which on standing hydrolyzes into hydrazoic and hypoiodous acid; also sol in many org solvs in which it slowly decomps into iodine and nitrogen. Its toxicity is severe, as a single exposure can cause injury to the skin or mucous membranes of sufficient severity to threaten life or cause permanent

physical impairment (Ref 10). Iodine azide was first prepd in 1900 by Hantzsch (Ref 1) on adding an ethereal soln of iodine to an aq suspn of silver azide cooled to 0° followed by extracting with eth at below 0° (Refs 4,5,7,8&9)

When in a dry state, iodine azide may decomp spontaneously with great violence into its elements. According to Gutmann (Ref 2) tertiary sodium arsenite, Na_3AsO_3, does not react with inorg salts of hydrazoic acids (metal azides) but with chloro- or iodoazides it gives sodium azide, alkali halide and sodium arsenate. By means of a photon counter, the radiation emitted on deton of iodine azide (prepd by a modn of Hantzsch's method) clearly showed UV radiation which was not found on deton of nitrogen triiodide, NI_3, an extremely sensitive and dangerous expl (Ref 6)

According to Mellor (Ref 3), A.C.Vournazos prepd some complex azides of iodine by reacting zinc nitrate with sodium iodoazide forming *Sodium Zinc Iodoazide,* $Na[ZnI_2N_3]$, which reacted with silver iodide to form *Sodium Silver Zinc Iodoazide,* $Na(ZnI_2N_3)$-AgI-$Na(ZnI_2N_3)$, and with lead iodide to form *Sodium Lead Zinc Iodoazide,* $PbI_2[(ZnI_2N_3)Na]_4$. No expl props of these complex iodine azides were described

Refs: 1)A.Hantzsch, Ber **33**, 523(1900) & JCS **78** II, 274(1900) 2)A.Gutmann, Ber **57B**, 1956(1924); CA **19**, 1253(1925) & ZAnalChem **65**, 252(1924) 3)Mellor **8** (1928), 337 4)L.C.Ramon, Afinidad **10**, 211(1930) 5)L.F.Audrieth, ChemRevs **15**, 215(1934) 6)R.Audubert & R.Ralea, CR **208**, 983(1939) & CA **33**, 4131(1939) 7)Thorpe **1**(1947), 581 8)Ephraim(1949), 675 9)Gmelin, System No 8, Lieferung 2(1955), 600 10)Sax(1957), 785-6

Iron Azide or Ferric Triazide, $Fe(N_3)_3$ or $[Fe(N_3)](N_3)_2$, mw 181.92, N 69.30%; dk brn hygro lfts easily hydrolyzed and very unstable, mp-expl 200°(Ref 10). It was first prepd and isolated, in 1917 by Wöhler & Martin (Ref 5), by treating dry ferric sulfate with sodium azide in abs methanol, removing the pptd sodium sulfate by filtration, and concg the soln of ferric azide in a vac desiccator. Earlier, Turrentine (Ref 4) obtained ferric azide in soln by electrolysis of a dil sodium azide soln using iron electrodes and later by Browne et al (Ref 6) on using iron electrodes in liq amm solns of ammonium azide, but the product was ammonolyzed to an *Ammono-Basic Ferric Azide.* Curtius et al (Refs 1&2), with ferric alum and NaN_3, obtained ferric azide in soln and they also reported that iron dissolved in dil aq hydrazoic acid to form the azide, but the soln decompd on evapn giving either the basic azide or hydroxide. In 1934 Franklin (Ref 9) noted that aq hydrazoic acid reacts on iron to form ferric azide, nitrogen and ammonia together with a small amt of hydrazine. According to Franklin *Ferrous Azide,* $Fe(N_3)_2$, is formed first and oxidizes to ferric azide when the soln is warmed with excess hydrazoic acid present. Ricca (Ref 11) studied the reactions of the ferric ion with hydrazoic acid on electrolysis of their solns and obtained results which would indicate that ferric azide has the structure $[Fe(N_3)](N_3)_2$

Aq solns of ferric azide have a deep red coloration similar to that produced by $Fe(CNS)_3$. This characteristic coloration is also produced when ferric salts are added to aq solns of hydrazoic acid, thus serving as a colorimetric test for HN_3(Ref 3)

Racz (Ref 10) studied the thermolysis and UV radiation emitted by ferric azide. He reported that, when enclosed, ferric azide expl at ca 200° in air and at 230° in nitrogen. Decompn of this azide was accompanied by strong UV emission which began at 270°, at activation energies of 47, 33 and 64 kcal. By comparison with the results of other azides, it is inferred that the processes occurring at 47 and 64 kcal energies are independent of the metal, while that occurring at an activation energy of 33 kcal indicates a different mechanism of thermolysis (Ref 10). Santappa (Ref 12) studied the ferric azide-vinyl monomer system when irradiated with UV light of wave

length 300-400 mμ and found the quantum yield varied linearly with the monomer conc, and the rate of Fe^{2+} production was dependent on the org impurities present. Vinyl monomers studied were acrylonitrile, methyl methacrylate and methacrylic acid

The toxicity or hazardous nature of ferric azide is not given in Sax nor were there found any other expl props reported in the literature (Refs 7 & 8)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 291(1898) & JCS **76** II, 91(1900) 2)T. Curtius & A.Darapsky, JPraktChem **61**, 408 (1900) & JCS **78** II, 474(1900) 3)L.M. Dennis & A.W.Browne, JACS **26**, 603(1904); ZAnorgChem **40**, 100(1904) 4)J.W.Turrentine, JACS **33**, 820(1911) 5)L.Wöhler & F.Martin, Ber **50**, 594(1917) & JCS **112** I, 384(1917) 6)A.W.Browne et al, JACS **41**, 1775(1919) & CA **14**, 28(1920) 7)Mellor **8** (1928), 354 8)Gmelin, System No 59, Teil B (1932), 156 9)E.C.Franklin, JACS **56**, 568(1934) & CA **28**, 2289(1934) 10)C.Racz, CR **209**, 534 (1939) & CA **34**, 30(1940) 11)B.Ricca, Gazz **75**, 71(1945) & CA **41**, 4397-8(1947) 12)M.Santappa, CurrentSci(India) **23**, 145 (1954) & CA **48**, 12561(1954) 13)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1551-TR, 14(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Lanthanum Triazide, $La(N_3)_3$, mw 222.97, N37.25%, Curtius and Darapsky prepd the basic salt, *Lanthanum Hydroxyazide,* $La(OH)(N_3)_2 \cdot 1\frac{1}{2}H_2O$, by boiling a soln of lanthanum nitrate and sodium azide. The white slimy mass of basic lanthanum azide was obtained either on evapg the mixed soln in vacuo or on treating it with a mixt of alc & eth. No props of the product were reported nor were there any addnl refs found in which any attempts were made to prepare and isolate the lanthanum azide

Refs: 1)T.Curtius & A.Darapsky, JPrakt Chem **61**, 408(1900) & JCS **78** II, 474(1900) 2)Mellor **8** (1928), 352

LEAD AZIDE OR LEAD DIAZIDE (LA)

(Formerly called *Lead Trinitride, Plumbic Nitride, Plumbamide, or Lead Hydronitride)* (called Bleiazid in Ger, Azoture or Nitrure de plomb in Fr, Acido di piombo or Azotidruro di piombo in Ital, Azida de plomo, Plumbazido or Nitruro de plomo in Span, Azid svintsa in Russian and Chikkaen in Japan), $Pb(N_3)_2$, mw 291.26, N 28.86%, Col crysts which exist in two modns: orthorhombic (α) and monoclinic (β) forms (Refs 44,45,113,137,142 & 144); mp - decomp into Pb and N_2 (Ref 143) at 245 to 250° (Ref 17), expln temp LA 315° to 360° (Refs 16,17,28,44,73,106,124 & 149) and 275° for dextrinated (Ref 106); cryst d 4.71 (α) (Refs 92 & 144), 4.93 (β) (Ref 144), 4.38 (dextrinated) (Ref 141), apparent d cryst 0.8 (Ref 103) & dextrinated 1.5 (Ref 141); Q_{expln} 260 cal/g (Ref 73) to 367 cal/g (Refs 16,91,111 & 167) $Q_{activation}$ 10-11 kcal/mol (Ref 70) to 55 kcal/mol (Refs 47,58,84, 106,111 & 133) Q_f^P −114.5 kcal/mol (Refs 137 & 149) to −126.3 kcal/mol (Ref 123), −115.5(α) and −115.8(β) kcal/mol (Ref 137)

LA is very sol in AcOH, sol in w to the extent of 0.02% at 18° and 0.09% at 70° (Ref 141); Ref 128a gives 0.05/100 g H_2O at 100°; almost insol in eth, acet, alc, ammonia or org solvents. The sol of α-LA in w is 8.5×10^{-4} mol/l which gives a concn solubility product of 2.6×10^{-9} and a thermodynamic solubility product of 1.8×10^{-9} (Ref 134). LA may be dissolved in monoethanolammine or in a 50/50 mixt of monoethanolammine/ammonia, from which it may pptd by addg dil AcOH. The resulting prod, according to Majrich (Ref 66) is impure

LA is not considered particularly toxic but inhalation of its dust should be avoided as this causes headaches and distention of blood vessels. It has been recommended that the LA content of air should be less than 0.2 mg/cubic meter in order to avoid toxicity by inhalation (Ref 141). An investigation of LA as an industrial hazard (Ref 96) has indicated that the storage and distribution of Pb from LA in tissues following ingestion are similar, in general, to other Pb salts. The acute toxicity of LA is associated with the azoimide radical (See Hydrazoic Acid in this section) rather than with the lead. Also, see Sax (Ref 150), Eddy (Ref 95), Schwartz (Ref 90), Ref 97 and Siefert (Ref 102) for further discussions of the toxic effects of LA

LA was first prepd in 1891 by Curtius (Ref 1) by adding Pb acetate to a soln of Na or Amm azide. Curtius & Rissom (Ref 2) prepd it by the action of hydrazoic acid on a lead salt. Turrentine (Ref 6) obtd LA during electrolysis of a 3% soln of Na azide on lead anodes. Browne et al (Ref 20) found LA was formed when a soln of NH_4N_3, in liq NH_3 at −67°, was electrolyzed with a Pb anode. Some details of prepg LA have been described by Hyronimus (Ref 3), Stettbacher (Refs 13,85 & 114), Hodgkinson (Ref 18), Hale (Ref 29), Matter (Ref 32), Wallbaum-Wittenberg (Ref 79), Meissner (Ref 78) and by many others (See Refs 15,19,35,41,59, 64,68,72,86,94,122a,126 & 151). Plant methods of manufg LA have been given by Meissner (Ref 60) von Herz (Refs 53 & 61), Gómez (Ref 141a) and Stettbacher (Ref 52) and procedures for continuous manufg by Matter (Ref 39a), Meissner (Ref 40), Greceanu (Ref 93) and others (Ref 104), Moskovich (Ref 75) prepd stable gelatine emulsions of LA. Darier & Goudst (Ref 22) described a procedure for preventing expln of LA by effecting the reaction within the interstices of a porous inert absorbent material

The hazards involved in the manuf of the pure crystalline material delayed its practical use for many years. Although manufd and used in foreign countries since 1920, its military and coml uses in the USA, since 1931, have been restricted to an impure colloidal form or "dextrinated" LA (Ref 122a). The prepn of colloidal LA has been described by Rintoul & Weir (Ref 21), Snelling (Ref 23), Lowndes (Ref 24), Rinkenbach (Ref 51), Fleischer & Burtle (Ref 108), Moskovich (Ref 74), Böstrom et al (Ref 145) and Fonda & Fonda (Ref 125). In these procedures the objective was to obtain rounded aggregates of

uniform size and to prevent the formation of large crysts (See Ref 72). The existence of LA in (α) and (β) forms was first reported in 1931 by Garner & Gomm (Ref 44) and by Miles (Ref 45). According to Moskovitch & Aleksandrovich (Ref 65) and Hattori & McCrone (Ref 144) the stable form (α) is prepd by mixing equal vols of 1M $Pb(NO_3)_2$ and 2M NaN_3 solns and recrystg the pptd LA from sodium acetate soln. The less stable (β) form is prepd by slow diffusion of Pb^{++} and N_3^- ions into water. β-LA transforms readily in soln to α-LA (See also Manufacture of LA, which follows Laboratory Methods)

Laboratory Methods of Preparation of Dextrinated LA

(Caution: All operations should be conducted behind a barricade of safety glass or transparent plastic)

I) Method Used in Some American Laboratories: a) Dissolve 2.33 g of Na azide and 0.058 g NaOH in 70 ml H_2O (distilled or permutite treated) by shaking in a 125 ml separatory funnel. This is soln A b)Dissolve 6.9 g Pb nitrate and 0.35 g dextrin in 90 ml H_2O (distilled or permutite treated) in a 250 ml tall form beaker, and add 1 or 2 drops of 10% NaOH to bring the pH to ca 5. This is soln B c)Heat soln B to 60-65° on a water bath and agitate it with a formaldehyde plastic or hardwood stirrer. The stirring should be as efficient as possible to prevent the formation of large crysts. Stirring while vigorous should not produce any spattering of the mixt and the stirrer should not rub against the walls of the beaker d)Add soln A(which is in the 125 ml separatory funnel) dropwise to soln B, with continuous agitation. The addition should require about 10 mins e)Remove the beaker from the water bath and continue stirring the mixt in beaker while cooling to room temp (about 1 hr) f)Remove the stirrer and rinse it into a beaker with a stream of distilled w g)Allow the ppt of LA to settle and filter by suction the contents of the beaker through a filter paper placed in a 100 ml plastic Büchner funnel h)Stop the suction, add 50 ml of distilled H_2O to the Büchner and stir the ppt with a plastic rod or spatula, taking care not to tear the filter paper i)Remove the H_2O by suction and repeat the operation of washing two more times j)Set aside the mother liquor and the wash waters and (under a hood) destroy the azides in soln by adding the required amt of Na nitrite, followed by a slow addn of 92% sulfuric acid:

$$NaN_3 + NaNO_2 + H_2SO_4 \rightarrow Na_2SO_4 + N_2O + N_2 + H_2O.$$

When the azide is destroyed, which is indicated by the sample turning litmus paper red and starch-iodide paper blue, pour the soln into a sink (See also under Destruction or Killing of LA) k)By means of a plastic or wooden spatula transfer the wet ppt to an open Al dish l)Dry the sample for 8-15 hrs (but no more than 24 hrs) at 65° and examine it under the microscope. The LA crystals should be approximately spherical in shape, opaque in appearance, averaging not over 0.07 mm in diam. They should be free flowing and contain an average of 92.5% $Pb(N_3)_2$. There should be no needle-shaped crysts. The yield will be ca 5 g m)If it is desirable to save the sample, wet it with a small amt of H_2O and transfer to a rubber-stoppered plastic or hard rubber bottle

Note: It is not advisable to use a sintered glass funnel or crucible for filtering because friction between the glass and LA might result in an expln, even under water, especially if a glass rod or spatula is used for stirring or transferring the sample

II) Argentine Naval Powder Factory Method (Azul, ProvBuenosAires): a)Dissolve 8-9 g of Pb nitrate in filtered water free of Cl_2, chlorides and gritty materials b)Dissolve 3g of Na azide in 100 ml w and det the approx alkalinity by titrating a 5 ml of soln with N/10 H_2SO_4, using phpht as an indicator. If the amt of acid required to discolor the phpht is 8 to 10 ml, the soln is satisfactory; if less than that add a few drops of NaOH soln c) Dissolve 0.3 g of potato dextrin in a small

amt of hot w d)Provide a reaction vessel, consisting of a round bottom stainless steel beaker of about 0.5 l capacity, highly polished inside, equipped with a water jacket and a stainless steel agitator with two double blades placed one above the other and not touching the walls or the bottom of the vessel e)Transfer the Pb nitrate soln in the vessel, start the agitator and circulate water preheated to ca 50° through the jacket. Test the neutrality of soln by placing a few drops on a glazed white porcelain plate and adding one drop of methyl orange indicator. If the soln is acidic, add a few drops of aq NaOH and retest the soln f)Add the dextrin soln and while stirring as rapidly as possible (in order to obtain small uniform crysts) run in the Na azide soln slowly while maintaining the temp at 50+5° g)Decant the mother liquor using a rubber tube syphon. h)Refill the reaction vessel with w, agitate for a few mins, allow to settle and decant i)Tilt the vessel and transfer its contents (using a jet of water) into a filter cloth bag and wash the bag and contents in three changes of w j)Store the bag in a plastic container k) Dry it when required at 60-65°. The yield will be ca 6.5g

Note: This laboratory method is essentially the same as the Argentinian plant procedure; the only difference is in quantity of materials used. When using 440-450 g of Pb nitrate, 150 g Na azide and 15 g dextrin about 330 g LA are obtained (Some European plants use as much as 10 times these quantities, producing up to 3300 g in one batch) (Ref 141a)

III) German Method is essentially the same as the plant procedures described in Ref 157a, under Bleiazid, except that the quantities are much smaller

IV) Italian Method is essentially the same as the plant procedure described in Ref 126, pp 230-33, except that the quantities are much smaller

V) Spanish Method. The procedure used at the Pirotecnia de Sevilla on an industrial scale, is described by Vivas, Feigenspan & Ladreda (Ref 104a, pp 316-22). The same method can be used in the laboratory provided the quantities are much smaller

Plant Manufacture of Dextrinated LA by the duPont Method. The procedure briefly described below is essentially the same as was observed by B.T.Fedoroff at the Kankakee Ordnance Works, Joliet, Illinois, which was operated by the United States Rubber Company. This method is based on the duPont method developed before WW II [See Bleiazid, pp Ger 12-13 of PATR 2510 (1958)]

In the manuf of LA, the size and shape of the crystal is most important. The crysts produced by the duPont method are free-flowing, lt buff, spherical in shape, opaque in appearance under the microscope and averaging not over 0.07 mm in diam. Their $Pb(N_3)_2$ content is ca 92.5%

Dextrin is used as a colloiding agent, which prevents the formation of large sensitive crysts of LA and regulates, to some extent, the shape, though not so much as NaOH and the agitation. It has been found that an unsatisfactory dextrin could be much improved by its pptn from an aq soln of alcohol. Furthermore, the addn of various substances such as K or Na ferrocyanide, Na oxalate, Na tartrate and Rochelle salt improves the colloiding action of dextrine. The use of amts of dextrine in excess of the prescribed duPont method tends to lower the purity and make the final LA product more hygroscopic. In the duPont process, yellow potato dextrin with not more than 0.2% insol material is used

As the concn of solns employed in the manuf of LA affects cryst size and shape to some extent it is advisable not to deviate from the following concns: 7.325±0.075% $Pb(NO_3)_2$, 3.175 ± 0.025% NaN_3 and 4.1 g dextrine per liter of Pb nitrate soln

Unless the Pb nitrate content is sufficient to give 10% excess over theory, fine white crysts appear in the mother liquor which will not settle out properly and which mix with

the LA to give it a white appearance. This condition is not particularly serious, but the yield and purity are lowered and the resulting product tends to cake on the drying pans and to be dusty

Procedure a)Prepare the stock soln A by dissolving 169 lb (ca 73.9 kg) lead nitrate in about 750 l of water treated by the permutit demineralizing process. Care must be taken to remove all grit and insol matter. As this soln is usually acidic (pH 4.2-4.6), add slowly 25 to 30 g NaOH in dil aq soln. NaOH serves to neutralize all acid either occluded or that due to hydrolysis of lead nitrate. An excess of NaOH must be avoided because it tends to produce elongated crysts of LA which are very sensitive. Add to the stock soln 9 lb(ca 4.08 kg) potato dextrin previously dissolved in about 100 l of water and then bring the soln to 7.325 ± 0.07% Pb $(NO_3)_2$ content. b)Prepare the stock soln B by diluting with treated w the *refined solution* (contg ca 27% NaN_3),delivered from the Sodium Azide (qv) plant,to 3.175± 0.02% NaN_3 content. Add 794 g NaOH in pellets. This quantity of caustic is supposed to be sufficient to keep neutralized most of the free acid formed during the interaction of lead nitrate soln for pptg LA. This quantity of NaOH is also just sufficient to control the purity of the finished LA, because it ppts a small amt of lead as $Pb(OH)_2$ or $Pb(OH)N_3$. Adjust the concn of stock soln B to 3.175 ± 0.025% NaN_3 content. c)Drop 60 l of stock soln B(10% in excess) from a 1000 l storage tank to the stainless steel measuring tank of ca 100 l capacity, where the soln is heated by means of hot water coils. Drop the preheated soln into the precipitator (reactor). This is a stainless steel round bottom spouted kettle of 120 l capacity with a steel jacket and four-bladed stainless steel agitator which rotates at 95 RPM. d)Raise and maintain the temp of soln A at 135-140°F (57.5-60°) by circulating 170°F (ca 76.5°) water through the jacket of the reactor. e)Drop 50 l of stock soln B(NaN_3),from the 1000 l storage tank to a second stainless steel measuring tank of ca 100 l capacity. f)While agitating the soln A at 95 RPM and maintaining the temp at 135-140°F add the soln B at a rate of 2 l per min. When the soln is all in, start to circulate cold w through the jacket, while continuing the agitation until the temp drops to 90° or lower. g)Stop the agitation and when LA settles, decant the mother liquor to the "killing tank," where the azide content is destroyed with $NaNO_2$ and HNO_3, as described under Destruction (Killing) of Azides. h)Wash the ppt of LA into a rubber bucket and transfer on filter cloth spread over a vacuum Nutsch. Rinse the ppt with four changes of w and send it to the killing tank. i)Leave on the last wash just enough w to cover the LA and pack the wet LA (24-26% moisture) in drums for shipment

The overall pptn time cycle is 60 mins,and 7.6 lbsLA(92.3% purity) is produced per pptn

Analytical procedures for plant control and for finished products are described under Lead Azide, Analytical Procedures and under Sodium Azide Analytical Procedures

Note: A plant method used in Argentina is similar to the laboratory method described above, except that much larger quantities of lead nitrate, sodium azide and dextrin were used. A plant method used in Germany before and during WWII is described in Ref 157a, under Bleiazid. A plant method used in Italy is described in Ref 126, pp 230-33 and the method used in Spain is described in Ref 104a, pp 316-22

Explosive Properties of LA. Many compds have been proposed as substitutes for MF but none has been found more suitable than LA. The others are either too sensitive, too expensive or less effective than LA in initiating efficiency. As early as 1893 the Prussian Government investigated the azides of Pb, Ag and Hg for possible uses as detonants, but a fatal accident caused these experiments to be discontinued. No further work was done with LA until Wöhler in 1907 called attention to it again as a possible substitute for MF (Refs 15 & 59). Since that time

considerable interest in and study of the expl props have resulted:

Brisance by *Sand Test.* Dextrinated LA 95% as brisant as pure, crystalline LA; 75% as MF and 37% as diazodinitrophenol (Ref 141); calcd by *Kast formula* LA 107×10^6 and MF 128×10^6 (Ref 73) by *Lead Plate Test,* LA is much less effective than MF (Ref 13). Stewart (Ref 11) claimed LA had same brisance as MF

Detonation Rate. 4500 m/sec at d 3.8 to 5300 m/sec at d 4.6 (Refs 28 & 73), 5400 m/sec at max d (Ref 91). For addnl values and discussions see Roth (Ref 169), Bowden & MacLaren (Ref 138), Cook (Ref 107) and others (Refs 13,43,48,50,57,59,67,86,118, 141 & 151). Deton effect on plastic material of various shapes is described by Kolsky & Shearman (Ref 115). The deton characteristics of LA, MF and other expls, using an assumed equation of state, have been calcd (Ref 146)

Explosion Temperature. 315° for pure LA and 275° for dextrinated LA (min temps for ignition in 5 sec) (Ref 106) (See also Refs 33,79,83,110,111,122a & 132)

Friction Sensitivity. LA is more sensitive than MF (Refs 73,79 & 110). Bowden & Gurton (Ref 112) detd the effect of grit particles for frictional initiation of LA

Gas Evolution on Explosion. 308 l/kg for LA vs 315 l/kg for MF (Ref 91)

Impact Sensitivity. Dextrinated LA is less sensitive to impact than MF, Pb styphnate, diazodinitrophenol, tetracene or crystalline LA. When wet with water or alc LA is still sensitive to deton by impact (See also Refs 9,11,16,29,33,35,73,79,81,91,110,122a,131, 136,140,159 & 166); with 2 kg wt LA 12 cm vs MF 5 cm (Ref 82), with 500 g wt LA 30-40 cm vs 10.5 cm for MF (Ref 35a)

Initiating Efficiency. More efficient initiator than MF and slightly less efficient than diazodinitrophenol(See Table under Mercurous Azide and Refs 16,29,38,46,48,49,59,71,73, 79,81,94,101 & 116)

Power by Trauzl Test. Dextrinated LA is 89% as powerful as pure LA and 80% as powerful as MF (Ref 141); 181 cc expansion for 10g LA vs 128 cc for MF calcd by Kast formula (Ref 73) (See also Refs 11 & 153)

Pressure Developed on Explosion (own vol). 94,930 kg/cm² vs 90,260 kg/cm² for Ag azide, both at loading d 3.0 g/cc under 1100 kg/cm² press (Ref 16). Noddack & Grosch (Refs 132 & 147) calcd the surface press on explosion as 11,900 kg/cm² for LA vs MF 14,3000 kg/cm².

Stability to Heat. % loss in wt in 75°C *International Test:* 0.17 for LA vs 0.18 for MF; *100°C Heat Test:* 0.5% loss 1st 48 hrs and 0.1% 2nd 48 hrs vs expln for MF under same test conditions; *100°C Vac Stab Test:* 0.4cc gas evolved in 40 hrs (Ref 141). The thermal stability of both dextrinated and pure LA is exceptional (Ref 116)

Specific Energy 4380 kg/l (Ref 28), 361.2 joules/gm (Ref 169)

Stability in Storage. LA has been found unchanged with respect to purity or brisance after storage for 25 months at 50° or after storage under a w-alc mixt at RT; storage at 80° for 15 months caused no decrease in brisance, and after similar storage, a priming compn contg LA showed no decrease in sensitivity to stab action (Ref 141) (See also Refs 11,28, & 79). On the otherhand, MF stored at 80° for 1 day was reduced to 92% purity and its initiating efficiency was practically destroyed (Ref 141)

Temperature Developed on Explosion. 3420° (Ref 16) to 3450° (Refs 73 & 91); 3484° (Ref 128a, p 1)

Thermal Decomposition of LA has been the subject of study by a large number of investigators: Hitch (Ref 17) found LA extremely hard to decomp without explg. Garner & Gomm (Ref 44) in studying the rate of decompn of α and β-forms found that β-LA decompd much more rapidly than α-LA; the critical increments were α-form 47,600 cals and β-form 38,800 cals. The kineties of

thermal decompn of α-LA were studied by Evans (Ref 165), Hill (Ref 156) and Griffiths & Goocock (Refs 152 & 155) who found initial rapid evolution of gas, followed by the formation of surface nuclei which grow three-dimensionally. The decay stage followed the contracting sphere mechanism. Garner (Refs 88 & 161) also investigated the reaction kinetics of LA decompn while Hawkes & Winkler (Ref 106) indicated that thermal expln of LA may be spontaneous. The min energy requirements for ignition of LA and other expls have been reported (Ref 164). According to Suzuki (Ref 133), Ryabinin (Ref 105), Tsukerman (Ref 103), Apin (Ref 84), Yoffe (Ref 121), Ubbelohde et al (Ref 111) expl decompn occurred after an induction period. Moskovich & Aleksandrovitch (Ref 65) found that during the induction period, Pb atoms formed at the cryst surface were autocatalytic in accelerating decompn. Experiments by Apin (Ref 84) showed that the decompn velocity increased slightly during the induction period, then rapidly, and finally expln occurred. Muraour (Ref 77), Andréev (Ref 168) and Bowden et al (Refs 120 & 148) have studied the effects of pressure and confinement on the decompn rate of azides, fulminates and other expls. Also studies of LA decompn in vacuo have been reported by Schumacher (Ref 55) and Burlot (Ref 56) Muraour & Schumacher (Ref 56a) and of the influence of heat have been reported by Belyaev (Ref 83), Weyl (Ref 122) and the effect of shock from electrons by Muraour (Ref 54). The critical amt of LA for ignition in H_2-O_2 mixt and CH_4-air mixt has also been detd (Ref 117)

Andréev (Ref 154) published a book on the thermal decompn and expln of substs which includes a discussion on azides. Recent exptl work on azide research has been reviewed in a symposium on the initiation and growth of explosions in solids [Proc Roy Soc 246A, pp 145-297 (1958)]. Wyatt (Ref 160) discussed ignition by elec discharge, Bowden (Ref 162) ignition by neutrons, α-particles and fission products, Kaufman (Ref 163) discussed the effect of nuclear radiation and Groocock (Ref 155) Todd and Parry (Ref 172) the effect of high energy x-rays on the thermal decompn of LA. Low x-ray dosage caused LA crysts to decrepitate with heat and to increase in hardness. Higher x-ray dosage produced severe damage, 98% destruction of a Service LA sample was observed after an x-ray dose of 3.5×10^8 röntgen. In air the solid decompn prod was basic lead carbonate, $2PBCO_3 \cdot Pb(OH)_2$. In the absence of CO_2 but in the presence of w, the prod was Basic Lead Azide of unknown formula. According to Renaud (Ref 119) LA when treated for 20 min with a supersonic intensity of 100 w/sq cm and 1 megacycle/sec showed no explosibility. Audubert (Ref 76) found that slow thermal decompn of LA gave rise to UV radiation (See Refs 67a & 69)

Effects of Radiation. LA exposed to gamma radiation by Warren et al and by Rosenwasser, as reported in Ref 139, exhibited post-irradiation gas evolution as measured by vac stab test appar. Bowden & Singh (Ref 135) irradiated Pb, Ag and Cd azides with electrons, neutrons, fission prods and x-rays. All azides were exploded by an intense 75-kv electron stream. Thermal neutron irradiation did affect the subsequent decompn of Li and Pb azides. Muraour & Ertaud (Ref 129) also subjected LA to a neutron flux. Raney (Ref 158) reported that a total flux of 7.5×10^{16} n/sq cm converts LA to Pb carbonate. According to Berchtold & Eggert (Ref 128) ignition of LA by exposure to energy from a photographic "electron" flash bulb at a distance of 6 cm, reqd 240 W-sec energy. The dissociation of LA by absorption of light energy was described by Eggert (Refs 130 & 167)

Other Properties. Delay et al (Ref 100) and Mohler (Ref 98) obtd infrared absorption spectra of LA and other expls in the range 3 to 19 μ Kahovec & Hohlrausch (Ref 109) detd the Raman Effect of crystalline LA. Wöhler (Ref 7) observed that LA decompd in

direct sunlight quicker than other azides. In sunlight or under w, LA becomes yel-brn and then lt yel; NH_3 is evolved as a result of the reduction, by Pb, of the HNO_3 formed on hydrolysis; the Pb is oxidized to form a Basic Lead Azide $PbO{\cdot}Pb(N_3)_2$ (qv) (Ref 9). Belyaev & Matyushko (Ref 87) measured the heat conductivity of LA and obtd a *Specific Heat* value of 0.09 cal/gm/°C vs a value of 0.1 for MF. Roth's (Ref 169) value of the ratio of mean specific heats is 1.337. Hattorie & McCrone (Ref 144) measured the *Refractive Index* and the *Molecular Refraction* of form I (α) and form II (β) LA

According to Stewart (Ref 11) moist LA is not affected by contact with steel or Fe whereas MF changes under storage in contact with these metals. Also Cu, brass and Al had considerably less effect on LA than on MF (Ref 28). LA does corrode Cu with the formation of the extremely sensitive Cupric Azide (qv) (Ref 99). Eschback & Löbbecke (Ref 39) avoided the reaction of LA with Cu or brass parts by coating them with Cd. Warren (Ref 89) has also studied the action of LA on copper

According to Seavey & Kerone (Ref 63) LA can be made safe for handling by wetting it with a non-flammable liq, such as dichloroethyl ether, which is a non solvent and is less volatile than w but is capable of complete removal by drying. Moskovich (Ref 75) prepd stable gelatin emulsion of LA and detd their props. Strecker & Claus (Ref 26a) found that selenium monobromide reacted with LA suspended in benz forming Pb chloride and selenium. Klatt (Ref 80) noted that LA in HF produced an insol ppt of PbF_2 with evolution of gaseous HN_3. The characteristics of LA have been modified by Birkenbach & Rorig (Ref 30) by the formation of mixed crysts or double salts, such as $Pb(N_3)_2{\cdot}PbCl_2$ and $Pb(N_3)_2{\cdot}PbBr_2$. The double salt with Pb bromide was not exploded by a 10-kg hammer falling through 100 cm, whereas, the Pb chloride double salt exploded when the same hammer fell 65 cm. Pure LA exploded when a 2-kg hammer fell 35-40 cm. Friederich (Ref 25) by simultaneous or successive pptn obtd LA in mixed or double crysts with other substs, such as basic lead azide (qv), heavy metal hydroxides, carbonates, basic chlorides and sulfates, and neutral and basic salts of nitro compds. Such mixed LA crysts are claimed to be suitable for use in expl compns

Destruction or Killing of LA. Explosives of the initiating type, such as LA, cannot be burned, hence relatively large quants are destroyed by detonating them; small quants are decompd chemically. LA can be chemically destroyed by any one of the following methods (Ref 141): (a) mix LA with at least 5 times its weight of a 10% NaOH soln and allow the mixt to stand for 16 hrs with occassional stirring. The resulting supernatant soln of Na azide is decanted and disposed of by drainage into the ground. (b) dissolve LA in a 10% ammonium acetate soln and add a 10% Na or K bichromate soln until no more yell lead chromate is pptd. (c) Wet LA with 500 times its weight of w, slowly add 12 times its weight of a 25% sodium nitrite soln, agitate, and then slowly add 14 times its weight of a 36% nitric acid or glacial acetic acid soln. A red color produced on adding ferric chloride soln indicates LA is still present. Toxic fumes of nitrogen oxides (See Sax, Ref 150, p 950-1) are liberated in this process: $Pb(N_3)_2 + 2NaNO_2 + 4HNO_3 \rightarrow Pb(NO_3)_2 + 2NaNO_3 + N_2O_2 + 2N_2 + 2H_2O$. (d) dissolve LA in 50 times its weight of a 15% cerric ammonium nitrate. The LA is decompd with the evolution of nitrogen

Uses. Hyronimus of France (Ref 3) should be credited with the first success, in 1907, in the attempt to use LA in the expl industry. He proposed the use of LA in detonators to replace either wholly or in part the MF which had been used theretofore. In 1908 and later Wöhler (Ref 4) also secured patents for the use of LA as a substitute for MF in filling detonators and primers. Soon afterwards LA was manufd in Germany and in France and compd detonators were used in Europe during WWI. Some years later the manuf of LA

detonators was begun in the USA but, since 1930, its military and coml uses have been restricted to "dextrinated" LA

A number of investigators have conducted tests or reviewed the literature relative to the use of LA as a detong agent. These include reports by Stettbacher (Refs 13,31,85 & 114) Taylor & Cope (Ref 14), Hale (Ref 29), Taylor & Rinkenbach (Ref 33), Audrieth (Ref 59), Ubbelohde et al (Ref 111), Rosenwasser (Ref 170), Evans et al (Ref 171) and others (Refs 5,8,10,12,26,27,34,36,37,42,62, 127,141, & 157). The large and extensive patent literature is evidence of the importance of LA as a detonating expl

LA is used as an initiating agent in military ammo and in priming compns which are physical mixts of materials that are very sensitive to impact or percussion, and when exploded undergo very rapid auto combustion. LA has many advantages over MF: a) it is safer to handle. b) its nitrogen content is higher. c) it possesses a lower sensitivity to impact and percussion when pressed and is more easily detond by flame than by shock or friction. d) while MF and some other initiating compds become "dead" at high press, LA acquires a higher brisance and penetrating power when compressed to high density. e) it is less expensive than MF to prepare and f) its raw materials are readily available (See also Lead Azide Explosive, Primer and Detonator Compositions)

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Lead Azide, Basic, $PbO{\cdot}Pb(N_3)_2$, mw 514.47, N 16.34%, ndls, mp-explodes at 390°. Basic LA was first prepd by Wöhler & Krupko (Ref 1) in three ways: a)heating aq suspensions of $Pb(N_3)_2$ and $Pb(OH)_2$ in a sealed tube at 140° for 12-15 hrs, b)leading CO_2 free air thru a boiling aq suspension of LA until the calcd amt of HN_3 was evolved,and c)heating the requisite quants of LA and $Pb(OH)_2$ on a water bath for 24 hrs. The latter two methods

yielded uniform products. Feitknecht & Sahli (Ref 4) prepd basic LA by hydrolysis of LA with H_2O, by reaction of LA with NaOH, and by pptn from Pb salt solns by a mixt of NaN_3 and NaOH

X-ray examination (Ref 4) showed three forms of $Pb(N_3)_2 \cdot PbO$: Iα, the unstable reaction product of $Pb(N_3)_2$ with 1 equiv NaOH; Iβ, the stable reaction product of $Pb(N_3)_2$ with 1 equiv NaOH; and Iγ, the reaction product of $Pb(N_3)_2$ with $PbO \cdot \frac{1}{2}H_2O$ and the hydrolysis product of $Pb(N_3)_2$ with H_2O. Three forms of $3Pb(N_3)_2 \cdot 5H_2O$ were identified: IIα, the reaction product of $Pb(N_3)_2$ with 1.2 equiv NaOH; IIβ, the ppt from $Pb(NO_3)_2$ with 1 NaOH and 1 NaN_3; and IIγ, the ppt from $Pb(NO_3)_2$ with 1.2 NaOH and 0.8 NaN_3. Basic LA, form III, $Pb(N_3)_2 \cdot 2PbO - Pb(N_3)_2 \cdot 3PbO$; form IV, $2Pb(N_3)_2 \cdot 7Pb(OH)_2$; and form V, $Pb(N_3)_2 \cdot 4PbO$ to $Pb(N_3)_2 \cdot 9PbO$ were also reported by Sahli (Ref 4).

Basic LA is less sensitive to impact or temp than normal LA. According to Mellor, however, (Ref 3) an intimate mixt of LA and oxide in proportions necessary to form the sub-oxide showed the same sensitivity as pure LA

Friederich (Ref 2) proposed the use of basic LA in mixed or double crysts with other substs, such as LA, heavy metal carbonates etc. The double crysts were obtd by simultaneous or successive pptn.

Refs: 1)L.Wöhler & W.Krupko, Ber **46**, 2052 & 2054(1913) & CA **7**, 3088(1913) 2)W. Friederich, Brit P 180605(1921) & CA **16**, 3399(1922) 3)Mellor **8** (1928), 353-4 4)W. Feitknecht & M.Sahli, Helv **37**, 1423-39(1954) & CA **48**, 13505-6(1954)

Lead (IV) Azide. Treatment of Pb_3O_4 with aq HN_3 soln yielded a yel-red soln with a considerable Pb(IV) azide content. Normal HN_3 soln and Pb_3O_4 gave compds of PbN_9 to PbN_{10}, while concd HN_3 solns yielded a compd slightly lower than PbN_{12}. These aq Pb(IV) azide solns decompd spontaneously with evolution of N_2 and pptn of $Pb(N_3)_2$. Attempts to prepare solid Pb(IV) azide by mixing ethyl acetate or acetone solns of $(NH_4)_2 PbCl_6$ and NaN_3 gave a dk-red soln. This soln on treatment with petr eth gave an unstable expl azide thought to be NH_4Pb (IV) azide

Ref: H.Möller, ZAnorgChem **260**, 249-54 (1949) & CA **44**, 5750-1 (1950)

Lead Azide, Various Military Types. Although LA has been known since 1891 and patented in 1907 for use in detonators (see general discussion and Uses under Lead Azide), its adoption for military purposes was slow due to hazards involved in its manuf. Notwithstanding its many advantages over MF, some countries (such as Russia) still did not replace MF with LA in all primers and detonators. It is known that Germany, GtBritan and US started to use LA for military purposes in the early thirties. Germany and US adopted dextrinated LA, while GtBrit preferred cryst LA built around a nucleus of Pb carbonate (called "service" LA). Commercial manuf of LA in the US began ca 1932 by the duPont Co and this material (dextrinated LA) was adopted some time later by the US Ordnance Corps. Another type of LA, the so-called colloidal, was known since about 1918 (see Refs 21, 23, 24 etc under general discussion on Lead Azide), but it was not until 1932 that it was investigated at PicArsn with a view of its use for military purposes (Ref 1). The material investigated at PicArsn was prepd by the method patented by Rinkenbach (Ref 2). This investigation showed that CLA was much less hygroscopic than the DLA supplied by the du Pont Co and that it was about 3 times as efficient when used in detonators. Another type of non-dextrinated LA, the so-called PVA-LA (polyvinyl alcohol LA) was developed and patented in 1947 by Fleischer & Burtle (Ref 6) and assigned to Olin-Mathieson Chem Corp. This substance proved to be superior not only to DLA but also to CLA, as was later shown by Wagner et al at PicArsn (Refs 23 & 24)

According to Wagner (Ref 24), the DLA was considered satisfactory for military purposes until it was required to produce a small detonator for use in 20 mm ammo. The development of this detonator, which began at PicArsn ca 1947 is described in detail by Seeger (Ref 14). This detonator, designated as M47 or T32E1, was a short, stab-type contg as a primary chge ca 15 mg NOL No 130 [basic LSt 40, LA 20, tetracene 5, Sb_2S_3 15 & $Ba(NO_3)_2$ 20%], as a base charge not less than 34 mg RDX, and as an intermediate charge LA. As the dimensions required for this detonator were very, small (0.290" long and 0.145" diam, outside), there was not enough room to contain the amt of DLA needed to achieve what was required from this detonator. This meant that a more efficient expl than DLA was required. As no other initiating expls seem to be superior to LA, it was decided to investigate the non-dextrinated LA's and to compare their properties with dextrinated LA's. A total of six LA's (including the dextrinated) were investigated at PicArsn (see table, p A559), while experimental LA's RD-1343 and RD-1352 were investigated in GtBritain. Since Brit reports ERDE 7/R/58 and ERDE 10/R/57, describing prepn and props of RD-1343 and RD-1352 are conf, they were not used.

Following LA's are listed by Wagner (Ref 24):

A) Dextrinated Lead Azide: (abbrd as DLA), known also as *Type I LA (US).* Its prepn and props are described under Lead Azide

B) Service Lead Azide: (abbrd as SLA) (Brit). It is the std United Kingdom LA consisting of LA crysts each contg a nucleus of Pb carbonate. Its method of prepn is probably conf. No repts describing its prepn were available at PicArsn. Its props were detd at PicArsn on a sample procured from the Western Cartridge Co, which used to manuf this material for Canada during WW II (Ref 24). Most of these props are listed in the table, p A559

Brit service LA is practically non-hygroscopic and is superior in functioning characteristics to dextrinated LA, but it offers no substantial advantage over RD-1333 LA. It has been considered at Gt Brit that storage of SLA under w is hazardous due to the possibility of growth of crysts and formation of agglomerates which deton spontaneously. At least one expln was attributed to this cause. Long-term storage tests conducted at PicArns did not show any growth of crysts (Refs 22 & 24)

C) *Colloidal Lead Azide:* (abbrd as CLA) or *Type II (US)* is nondextrinated LA of very small (3-4 micron) particle size, patented in 1933 by Rinkenbach (Ref 2). For its prepn, to an aq soln contg 4% of Na azide was rapidly added, while mechanically agitating and maintaining the temp at 25°, a soln of Pb nitrate or Pb acetate in slight excess of the amt necessary for the equation:

$$2NaN_3 + Pb(NO_3)_2 \rightarrow Pb(N_3)_2 + 2NaNO_3$$

The resulting slurry was filtered and the ppt washed with several portions of w and dried. This product could be handled and pressed without danger of expln (Ref 2). This statement is not consistent with the impact test values given in table, p A559. This table also gives some other props of CLA, as was reported in Ref 24. Its loading d was not reported at 15000 psi, but in Ref 1 it is given as 2.77 at psi 3000 vs 2.93 for crystalline LA. Stability in storage is given in Ref 18

According to Ref 24, the CLA is not suited for uses requiring good flow characteristics, but, because of its very fine particle size it is ideal as a spot charge and a priming chge in low energy electric initiators. It successfully replaced the milled dextrinated LA formerly used for this purpose, thus eliminating the milling operation which was always considered dangerous, even under carefully controlled conditions. For prepg a spot charge for a low energy elec detonator, a dry colloidal LA is mixed with a concd soln of NC in eth-alc (or other solvent) and a small quantity of resulting paste is placed on the bridge wire to form a droplet (spot). For a type of detonator in which the bridge wire is located inside a cavity, the charge of LA can be made in the form of a pellet by pressing wet LA into the cavity.

US Military requirements and tests for colloidal LA (called also Type II) are given in Ref 19. The tests are essentially the same as for DLA (Type I) (See item VII under Lead Azide, Plant Analytical Procedures), except the particle size detn

D) *Polyvinylalcohol Lead Azide:* (abbrd as PVA-LA), patented by Fleischer & Burtle (Ref 6) can be prepd by adding to the soln of NaN_3 a soln of Pb nitrate contg ca 3% of PVA in soln. The resulting product consists of LA crysts coated with PVA. This method of prepn is very similar to prepn of dextrinated LA described in detail under Lead Azide. PVA-LA possesses practically the same sensitivity to impact as DLA, but is much more efficient in detonators and is practically non-hygroscopic. Its assay is usually 93-96% LA. Its ignitability is practically the same as for straight LA and better than for DLA. Some props of PVA-LA contg 96.07% LA are given in the table. For more info on PVA-LA see Refs 23 & 24

Note: Fleischer & Burtle (Ref 6) also patented LA's prepd by pptg in presence of one of the following substances: polyethylene glycols (such as "Carbowax" and hexaethylene-glycol) and ureaformaldehyde polymer (such as "Uformite). Their props were claimed to be similar to those of PVA-LA

E) *RD-1333 LA (Brit).* It is an experimental expl developed as a possible substitute for the SLA, which has been considered to be too sensitive for some operations. Method of prepn of RD-1333 is described in conf Brit rept (Ref 12). Its props were examined at Pic Ars and are given in conf rept (Ref 21). The props of RD-1333 listed in the Table, p A559 were taken from an unclassified rept (Ref 24)

F) *Dextrinated Colloidal Lead Azide* (abbrd as DCLA), was prepd on an experimental basis by the Olin-Mathieson Chem Corp, in essentially the same way as the DLA except that process controls were requlated to give an end product of a very small (1-2 micron) particle size. Its props are described in Ref 24. See also the table on p A559

G) *RD-1343 LA* (Brit). It is an experimental LA, considered to be an improved version of RD-1333-LA. Its prepn and props are described in conf Brit rept (Ref 20)

Various Types of Lead Azide (Comparison of Some Properties)

Properties	Dextrinated (Type I, US)	British Service	Colloidal (Type II, US)	PVA (US)	RD-1333 (Brit Exptl)	Dextrinated Colloidal	RD-1343 (Brit Exptl)	RD-1352 (Brit Dextr)
Color	Buff	Wh	Wh	Wh to buff	–	–	–	–
Lead azide, %	92.7	98.1	99.9	96.0	98.7	95.3		
Total lead, %	69.3	71.5	71.67	71.6	71.06	69.99		
Particle size, mean, microns	24.5	55.0	3.4	19.0	34.5	1.74		
Apparent density, g/cc (Ref 1)	1.83	–	0.85	–	–	–		
Density (pressed at 15000 psi), g/cc	3.14	3.31	–	3.81	–	–		
Sand test values	13.8**	–	15.0**	–	–	–		
Expln temperature (5 secs), °C	340	350	344**	340	345	–		
Impact sensitivity:								
PicArsn App, 2 kg wt, inches	4-6	2	2-3	4-5	5	3-6(?)		
charge wt, mg	28	37	25	30	23	18		
BurMinesApp, 2 kg wt, cm	13-28	30(?)	–	13-16	15	–		
PicArsn App, 500 g wt, in	12(?)	–	6**	18	15	–		
Charge wt, mg	28	–	–	31	21	–		
BurMines App, 500 g wt, cm	100+	–	35**	100+	100+	–		
Vacuum stability (avg):								
100°, gas evolved, ml/g/40 hrs	0.32	–	–	0.20	–	–		
120°, gas evolved, ml/g/40 hrs	0.46	–	–	0.44	0.43	–		
100°Heat Test:								
Loss in sample wt in 8 hrs, %	–	0.11	–	0.12	0.30	–		
Loss in sample wt in 48 hrs, %	0.34	0.08	–	0.13	0.30	–		
Loss in sample wt in 96 hrs, %	0.39	0.16	–	–	0.30	–		
Explosion in 100 hrs	None	None	–	None	None	–		
Minimum chge in mg required to initiate 60 mg RDX base chge of M47 Detonator *	90	25	–	30	25	–		
Hygroscopicity at 90% RH and RT (% gain in 56 hrs)	1.18	0.07	0.02**	0.03	–	–		
Solubility, g/100 g H_2O								
At ca 10°	0.056	0.019	–	0.017	–	–		
At ca 35°	–	0.032	–	0.041	–	–		
Soly in 50% alc at ca 35°	–	0.009	–	0.022	–	–		

Prepn and some props are described in Brit Rept ERDE No 7/R/58, which is conf

Prepn and some props are described in Brit Rept ERDE No 10/R/57, which is conf

* LA was used as an intermediate charge; as a primary chge was used 15 mg of NOL No 130 mixt (basic LSt 40, LA 20, tetracene 5, Ba nitrate 20 & Sb sulfide 15%). All chges of M47 were consolidated at ca 15000 psi

** Values given in PATR 255 (1932)

H)*RD-1352-LA* (Brit). It is an experimental dextrinated LA prepd with the idea of improvg the existing Brit LA's. Its prepn and props are given in Brit conf rept (Ref 17)

Remarks on Table: a)LA content was detd by measuring the nitrogen content of the sample and calcg % LA [See US Ordnance Corps Gasometric Method, item III C a, under Lead Azide Plant, Analytical Procedures] b)The % lead in a sample on a LA basis is always less than that given by a total lead analysis (See item III D, under Lead Azide Plant, Analytical Procedures). A sample calcn showing this is outlined below: mw of $Pb(N_3)_2$ = 291.26; at wt of Pb 207.21 and % Pb in $Pb(N_3)_2$ = 207.21/291.26 = 71.08. If % $Pb(N_3)_2$ in a DLA is 92.8, then % Pb in sample based on LA is 71.08 × 92.8 = 65.8, as against 69.3 found by analysis. The % excess of Pb: 69.3 - 65.8 = 3.5 is due to Pb contg impurities, believed to be organic complexes of lead hydroxide. This is also true for other LA's contg organic matter (such as PVA LA, DC LA, RD-1333, RD-1343 and RD-1352). The Pb contg impurity in case of Brit SLA is Pb carbonate c)Particle size was detd either according to procedures given in Ref 19 or by the improved microscopic method of Lavitt (Ref 10)

Note: According to Wagner (Ref 24), it has always been generally accepted that a few of the accidental explns which have occurred with LA during its history were caused by the fracture of crysts which were too large or too easily fractured. Some of the recent investigators, such as Garner & Gomm (Ref 3) and Miles (Ref 4) are inclined to think that accidental explns might be caused by the beta variety of crysts which are formed during manuf together with alpha-crysts and remain in stored LA. This view seems inconsistent with the recent work conducted at the Armour Research Foundation (Ref 15), in which it was shown that if any beta-variety were made, it would be rapidly transformed to the alpha-form when the material is stored under water

According to Wagner (Ref 24) it is believed that the sensitivity of LA is due at least in part, to internal stresses formed in the crystal which are an inherent result of the manufg process. Consequently both large and small crysts could be stressed, making them extremely sensitive to impact. More evidence that cryst size is not a controlling influence on impact sensitivity may be found in Ref 11 d)Apparent density given in the table for duPont DLA and for CLA was detd by Burton & Hopper (Ref 1) by filling with a slight tapping a tared 5 ml graduate to the mark and weighing the ensemble

e)Density of pressed sample was detd by the mercury displacement method. For this a tared No 6 cap was filled with Hg and weighed. After emptying the cap, it was filled with a sample, compressed at 15000 psi and accurately weighed. After adding some Hg to fill the empty space in the cap created by compressing, the cap was reweighed

$$d = A \Big/ \frac{B-C}{13.546}, \text{ where}$$

A = wt of sample, B = wt of Hg necessary to fill the cap C = wt of Hg to fill the empty space and 13.546 the d of Hg

f)Sand test values were taken from Ref 1. They were detd by the proced described in Ref 7

g)Explosion temperature values were detd as described in Ref 7

h)Impact sensitivities were detd as described in Ref 7

Note: Most of the impact sensitivity values recorded in the table, p A559 are averages of those reported in various CLR and GLR reports of PicArsn. The values which appeared to be doubtful are marked with (?). It seems that the values obtained for LA with PicArsn app are more reliable then those obtained with BurMines app. This unrealibility is particularly noticeable when a 2 kg wt is used with BM app. As readings for LA's are very small when using the 2 kg wt, it is difficult to differentiate between samples because their sensitivities are so similar. It is easier to

evaluate the sensitivities if larger readings are obtained, such as when using a 500 g wt
i)Vacuum stability test and 100° Heat test were detd according to Ref 7. Judging from the data as recorded in the table, p A559, all types of LA's investigated at PicArsn were of satisfactory stability
j)Minimum chge of LA required to initiate 60 mg of RDX, was detd by loading each of many caps used for M47 detonator with 60 mg RDX (base chge) and different amts of LA (initiating chge). The smallest amt of LA required to cause high order detonation (as judged by the diam of hole punched in a lead disk on firing a cap against it) in 100% of firings was considered the minim chge
k)Hygroscopicity was detd according to the proced described in Refs 9 & 13. The same info is given in Ref 14

In this procedure a 2-3 g dry sample was transferred by means of a wooden spatula to a tared Petri dish (ca 90 mm diam and 13 mm high). After redrying the sample at 95° for ca 30 mins, the ensemble was cooled for 20 mins in a desiccator and reweighed. All weighings were made as quickly as possible. The dried sample and dish were placed in a humidor maintained at 90% RH and 30° and left there for a specified period, such as 24, 48, or 56 hours

Note: Results in the table, p A559 indicate that of all the examined types of LA's only DLA is very hygroscopic. This means that it can pick up moisture in excess of permitted max 0.5% from the time of its removal from the dry house until it is used for loading detonators (Ref 24). From this it is logical to assume that some of the picked up moist would have to be volatilized before DLA could be raised to a temp high enough to be initiated by flame. This means that the higher the moist content, the harder it will be to initiate DLA especially by heat transfer and the lower will be the output. This decrease in output might be the result of a substantial decrease in the stable detonation rate or the inability of DLA to attain a high rate of deton in a limited column length. If moist content of DLA is 0.8% or higher, the detonators used by the US Ordnance Corps usually function low order. It has been tentatively established that in order to obtain a high order-detonation a column length of at least 0.1" is required if dry DLA is used and an appreciably longer column must be used if DLA is moist. Most US detonators are long enough to permit the use of DLA with a moist content 0.5% or sl higher, but this does not apply to the M47 detonator, which has a LA column only ca 0.11" long and ca 0.12" in diam. As it is practically impossible to have DLA completely dry, its use in short detonators, such as M47 cannot assure 100% functioning high order. For these reasons, it has been proposed to use in short detonators non-hygroscopic materials, such as PVA-LA, RD-1333-LA etc, which are besides more effective than DLA

Aside from impairing the functioning of LA, moisture in a detonator has other detrimental effects, such as hydrolysis with formation of small quantities of hydrazoic acid. This acid can react with Cu or Cu-bearing alloys such as gilding metal cup forming extremely sensitive $Cu(N_3)_2$ (See under Copper Azide). In order to prevent this danger the Brit use in their detonators, tin-coated copper cups, while the US practice includes coating of gilding metal cups with lacquers (such as Red No 1105) or paints (such as acid-proof black paint Type I or II). With these methods there is always the possibility of scraping off part of the coating thus leaving Cu exposed to the action of LA. For this reason, the US Navy prefers to use Al cups, but the Ordnance Corps considers Al not very suitable because it does not provide sufficient confinement and also because some difficulties are encountered with shrinkage of Al cups during loading. The ideal material seem to be stainless steel because it is not attacked by LA and because it provides sufficient confinement (Refs 22 & 24)

Another approach to prevent the formation

$Cu(N_3)_2$ has been suggested by the Linden Laboratories, Inc in their Final Rept Contract DAI-28-017-501-ORD-(P)-1405, April 1955. The method consists of treating the crysts of LA with a limited amt of H_2CO_3, H_2S or HI, so as to form lead carbonate, sulfide or iodide only on the surface without penetration into the crystal (Ref 22). This treatment will unquestionably reduce the efficiency of LA because it will be contaminated by inert materials

l)Solubility of LA in water or in 50% alcohol was detd as described in item VII F under Lead Azide Plant Analytical Procedures

In addn to above listed tests, the various LA's were loaded in M47 caps as intermediate chges together with NOL No 130 as a primary chge and RDX as a base chge and subjected to the following tests given in the Purchase Description PA-PD-202, with Rev 1 dated 30 Sept 1952 and Amend 1 dated 27 Jan 1953:

A) Detonator acceptance test, conducted by firing each detonator against a lead disc gave satisfactory results because all samples punched holes not smaller than 0.156" in diam

B)Detonator periodic functioning test, conducted by firing detonators, previously subjected to long-term storage at 71°, against a lead disc as in previous test, also gave satisfactory results

C)Detonator safety test, conducted by assembling M47 detonators into M505 fuzes and firing statically in M97A1 HEI 20 mm shells, gave satisfactory results

Other tests included: ballistic firing test, booster initiation test and waterproofners test. They all gave satisfactory results

In conclusion, it may be said that although all six types of LA's investigated at Pic Arsn are satisfactory, the PVA-LA and the RD-1333 LA stand out as being generally superior to DLA. The other types investigated: Brit SLA, CLA and DCLA, while superior to DLA in some respects such as output and hygroscopicity, have certain disadvantages, as can be concluded from the table, p A559 (See also Ref 24)

The latest Brit exptl LA's, RD-1343 and RD-1352 cannot be discussed because they are classified materials

Refs: 1)O.E.Burton & J.D.Hopper, PATR 255 (1932), "Study of the Explosive Characteristics of Lead Azide Prepared Commercially" 2)Wm.H.Rinkenbach, USP 1,914, 530(1933), "Method of Producing Noncrystalline Explosive Azide" 3)W.E.Garner & A.S.Gomm, JCS **1931**, 2123-34 (Thermal decompn and deton of LA crysts) 4)F.D. Miles, JCS **1931**, 2532-42 (Formation and characterization of crysts of LA and some other initiating expls) 5)K.S.Warren, PATR 1152 (1942), "Study of the Action of Lead Azide on Copper" 6)J.Fleischer & J.B. Burtle, USP 2,421,778 (1947) "Initiating Explosives" 7)Wm.H.Rinkenbach & A.J. Clear, PATR Rev 1(1950), "Standard Laboratory Procedures for Sensitivity, Brisance and Stability of Explosives" 8)U.S.Military Specification MIL-L-3055, Amend 1(1952) (Requirements and tests for dextrinated lead azide) 9)J.Bernstein, GLR 51-HI-2332, Pic Arsn (1952) "Hygroscopicity of Dextrinated Lead Azide" 10)J.W.Lavitt, PATR 1957 (1953), "An Improved Microscopic Method for the Determination of the Crystal Size Distribution of '2-Micron' RDX" 11)F.P. Bowden & K.Singh, Nature **172**, 378(1953) (Size effects in the initiation and growth of explosives) 12)J.W.C.Taylor, A.T.Thomas & K.J.Holloway, ERDE (Explosives Research & Development Establishment) Rept No 17/R/53(1953) (Manuf of RD-1333 LA) (Conf) 13) M.Falcione, GLR 54-HI-1354, PicArsn (1954) "Hygroscopicity of Dextrinated Lead Azide" 14)D.E.Seeger, PATR 2198(1955), "Development of the M47 (T32E1) Detonator" 15) Armour Research Foundation, Contract No DAI-11-022-ORD-(P)-18, Rept No 6, 25 Apr 1956(Crystallographic props of primary expls) 16)B.Furini, Jr, EPR (Eastern Process Rept)-220, No 10, Serial No 30, E.I. du Pont de Nemours & Co (1956), "Special RD-1333 Lead

Azide for Picatinny Arsenal" (Conf) 17) K.J.Holloway, G.W.C.Taylor & A.T.Thomas, ERDE Rept No 10/R/57(1957) (Prepn and props of exptl dextrinated LA RD-1352) (Conf) 18)T.W.Stevens, PicArsn Expls DevelSection Rept No 8 (1957) (Storage stability tests of colloidal LA for use in low energy electric detonators) 19)Purchase Description X-PA-PD-1217(1957) (Tentative spec for Type I and Type II LA's used in the US for military purposes) 20)K.J.Holloway, G.W.C.Taylor & A.T.Thomas, ERDE Rept No 7/R/58(1958) (Prepn and props of RD-1343 lead azide) 21)R.L.Wagner, PicArsn EDS Rept No 18(1958), "Investigation of RD-1333 Lead Azide for Use in Detonators" (Conf) 22)R.L.Wagner, K.G.Sheffield & D.E.Seeger,PicArsn EDS Rept No 57(1959), "Investigation of British Service Lead Azide." 23)R.L.Wagner, K.G.Sheffield & D.E.Seeger, PicArsn EDS Rept No 60 (1959) "Investigation of Polyvinyl Alcohol Lead Azide for Use in Detonators" 24)R.L. Wagner, PATR 2662(1960), "Lead Azide, Its Properties and Use in Detonators"

LEAD AZIDE PLANT, ANALYTICAL PROCEDURES. The lead azide plant of the Kankakee Ordnance Works (KOW), Joliet, Illinois, operated by the US Rubber Co manufd dextrinated crystalline LA from Na azide and Pb nitrate in presence of dextrin. Na azide also was manufd at KOW; the analytical procedures are described under Sodium Azide, Analytical Procedures

Most of the procedures (unless otherwise stated) described below were taken from the "Lead Azide Laboratory Manual" (Ref 9) with grateful acknowledgment to the US Rubber Co

The procedures given below include not only the analysis of LA,but also analyses of primary materials used in the manuf of LA, as well as various solns, wastes, etc.

I) Lead Nitrate, intended for use in the manuf of LA is a commercial, specially washed product, which has to comply with the requirements of the US Military Spec MIL-L-20549, which are: a)Color-white, b)Moisture - 1.25% (max), c)Purity 98.0% min (calcd on the basis of the material as received), d) Water-insoluble matter – 0.20%(max), e) Acidity 0.50% max (calcd as HNO_3), f)Copper– none

Procedures:

a)Color – by visual examination

b)Moisture. Accurately weigh approximately 10g in a tared glass-stoppered dish, remove the stopper and heat the dish for 5 hrs at 125°. Cool in a desiccator, stopper and reweigh

$$\% \text{ Moisture} = \frac{\text{Loss of wt}}{\text{Wt of sample}} \times 100$$

c)Purity. Transfer an accurately weighed sample (ca 1g) to a 250 ml beaker and dissolve in 100 ml distd w. Add dropwise 10 ml of satd Na sulfate soln contg 5% of sulfuric acid. Catch the ppt on a tared Gooch or Selas No 2001 crucible, wash with a 1% sulfuric acid soln and then with 50% alc. Heat the crucible and contents in a muffle furnace to const wt, cool in a desiccator and weigh

$$\% Pb(NO_3)_2 = \frac{\text{Wt of } PbSO_4 \times 1.0922}{\text{Wt of sample}} \times 100$$

Note: When using a Selas crucible, it is important to have it properly cleaned after each test. For this remove as much ppt as possible by inverting the crucible and tapping lightly, but do not use a glass or metal rod to pry the ppt. Convert the remaining sulfate to chloride by immersing the crucible in hot 1:1 HCl; wash it with hot distd w, employing both straight and reverse washings; dry thoroughly in oven and weigh to const wt. Repeat the operations until two successive washings agree within at least 0.005 g

d)Water-insoluble matter. Dissolve a 25 g sample (weighed on a trip balance) by heating it in a 500 ml beaker with 250 ml distd w. Filter the hot soln through a tared Gooch or Selas No 2001 crucible and wash the residue

thoroughly with hot distd w. Dry the crucible at 100° for 2 hrs, cool in a desiccator and weigh

$$\% \text{ WIM} = \frac{\text{Gain in Wt}}{\text{Wt of sample}} \times 100$$

Note: For cleaning the Selas crucible after this test use reverse washings with a hot acid (except hydrofluoric), followed by hot distd w until two successive weighings of dried crucible agree within at least 0.0005 g

e)Acidity. Dissolve a 25 g sample (weighed on a trip balance) in 500 ml distd w, add a few drops of methyl orange indicator and titrate with 0.2N NaOH soln to reddish end-point

$$\% \text{ Acidity as } HNO_3 = \frac{0.063 \times N \times \text{ml of NaOH}}{\text{Wt of sample}} \times 100$$

f)Copper. Dissolve a 100 g sample (weighed on a trip balance) in distd w and ppt all lead as sulfate by slowly adding a satd aq soln of Na sulfate contg 5% sulfuric acid. Filter through a filter paper, catching the filtrate in a Nessler tube. Add NH_4OH until alkaline and note if any Cu is present as indicated by the appearance of a blue color

Note: A more sensitive test for Cu is the addn of a few drops of a dil soln of $K_4Fe(CN)_6$ to the slightly acidic filtrate. A reddish-brown color due to $CuFe(CN)_6$ indicates the presence of Cu

II) Dextrin. There seems to be no US military spec for potato dextrin used in manuf of LA, but there is Spec MIL-D-3994 covering the requirements of corn dextrin for use in pyrotechnic mixts

The following tentative specs were proposed by the Eastern Laboratories of the E.I. du Pont Co (Ref 9, p 24) for yel potato dextrin suitable in manuf of "dextrinated LA": a)Soly in w at 2-4° –10 g/l(min) b)Insolubility in w at 90° –0.3%(max) c)Starch – present or absent (see Note below) d)Acceptance- after plant trial prepn of LA

Procedures:

a)Solubility in Water. Agitate mechanically for 1 hr at 2-4° a 20 g sample in 1 l distd w and, after allowing the slurry to settle for 30 mins, decant and filter about 150 ml of supernatant liquid. Pipette 100 ml of clear liq to a tared 150 ml beaker and evaporate to dryness over steam. Dry for 2 hrs in an oven at 100°, cool in a desiccator and weigh

$$\text{Soly g/l} = \text{Gain in wt} \times 10$$

b)Insolubility in Water. Dissolve in hot w a 5 g sample, digest it on a steam bath for 1 hr and filter while hot through a tared Gooch crucible. Wash the crucible with hot w, dry for 2 hrs at 100°, cool and weigh

$$\% \text{ Insol} = \frac{(\text{Gain in wt}) \times 100}{5}$$

c)Starch. Dissolve a 5 g sample in 50 ml hot w and add an excess of N/10 iodine soln [previously prepd by dissolving 12.692 g cryst iodine in 25 ml of soln contg 15 g KI (free from iodate) in w and diluting to 1 l with distd w]. Blue color is produced if starch is present

Note: The color obtained should be recorded, as it is a definite clue to the compn of the dextrin sample. The iodine test can indicate the extent of hydrolysis dextrin has gone through, and the soly characteristics of the dextrin

d)Acceptance. Prepare in the lab a sample of LA as described under Laboratory Preparation of Lead Azide, using the dextrin under test. See if the resulting LA meets the requirements of military specs

Note: Requirements of MIL-D-3994 for corn starch used in pyrotechnic mixts are more numerous and include: a)Granulation – not less than 99.5% must pass a No 80 US Std sieve, b)Water, uncombined – not more than 5.0%, c)Ash – not more than 0.15%, d)Water insoluble material – not more than 2.5%, e) Acidity as AcOH – not more than 0.18% and

f)Reducing sugars, as dextrose – not more than 4.0%

III)Lead Nitrate Dilution Tank. According to Ref 9, p 43, a basic unit at KOW consisted of a 1000 liter batch composed of 163 lb (ca 73.9 kg) Pb nitrate, 9 lb dextrin (ca 4.08 kg) and sufficient amt of NaOH to raise pH to 4.6-4.8 (each 10g NaOH raises pH about 0.1). Sodium hydroxide was USP or CP grade pellets or flakes and it was not required to analyze it. The $Pb(NO_3)_2$ content in the tank was 73.25±0.75 g/l

A 6 oz sample was taken by the operators after each batch was made and analyzed in the lab as follows

a)Determination Lead Nitrate Content: Method I. Pipette 5 ml sample into a 400 ml beaker contg 150 ml distd w and 20 ml acetate buffer soln (previously prepd by mixing 200 ml 50% $CH_3COO{\cdot}NH_4$ soln with 800 ml 1:4–CH_3COOH/H_2O). Add dropwise 5 ml of 10% $K_2Cr_2O_7$ soln and boil on hot plate until the color of soln becomes orange. Remove from heat and let settle. Filter through No 40 paper and wash the ppt with hot w until wash w becomes colorless

The following reactions took place:

$$Pb(NO_3)_2 + 2CH_3COO{\cdot}NH_4 \rightarrow (CH_3COO)_2Pb + 2NH_4NO_3$$

$$2(CH_3COO)_2Pb + K_2Cr_2O_7 + H_2O \rightarrow 2PbCrO_4 + 2CH_3COOK + 2CH_3COOH$$

Discard the filtrate and transfer the ppt quantitatively to an iodine reaction flask by successive washings with 3N HCl and hot w. Cool, add 10 ml of 10% KI soln and titrate with N/10 $Na_2S_2O_3$ soln until near discoloration. Add 5 ml of 0.5% starch soln and continue titration to greenish coloration

Following reactions take place:

$$2PbCrO_4 + 6KI + 16HCl \rightarrow 2PbCl_2 + 2CrCl_3 + 6KCl + 8H_2O + 3I_2$$

$$3I_2 + 6Na_2S_2O_3 \rightarrow 6NaI + 3Na_2S_4O_6$$

Calculation:

$Pb(NO_3)_2$ g/l = (ml $Na_2S_2O_3$) × N × F, where F is the factor calcd from the formula:

$$\frac{Pb(NO_3)_2}{3000} \times \frac{1000}{5} = 22.082$$

b)Determination of Lead Nitrate Content: Method II. Pipette 25 ml sample in 400 ml beaker. Add 50 ml distd w and 4-5 drops of methyl red indicator. If the soln is alkaline, neutralize it with N/10 HNO_3. Titrate with std Na sulfite soln (prepd by dissolving 18g of CP anhyd Na_2SO_3 in a mixt of 900 ml distd w and 100 ml methanol or ethanol and stored in the dark) to faint, but definite yel end-point. The wh ppt forming during the titration is an aid in observing the end-point

$$Pb(NO_3)_2 \text{ g/l} = \frac{\text{ml } Na_2SO_3 \times 1000 \times F}{\text{ml sample}}$$

where F represents grams of $Pb(NO_3)_2$ corresponding to ml of std Na_2SO_3 soln. The stdzn of Na_2SO_3 soln is conducted in the same manner, using 1.8 g of CP lead nitrate weighed to the nearest mg

c)Determination of pH by Bromcresol Green. Place 10.0 ml sample in a test tube and add 1.0 ml of 0.04% brom cresol green indicator (prepd by dissolving 100 mg powdered indicator in 2.9 ml N/20 NaOH and diluting to 250 ml with distd w). After shaking the mixt compare its color with standards in the LaMotte comparator. With brom cresol green, the pH should be 5.2 to 5.4

Note: More exact control of titre can be achieved by using a pH meter, such as The Coleman Industrial Tester No 15

IV)Refined Sodium Azide Liquor was made at the Sodium Azide Plant (qv) and transfered to the Lead Azide Plant

According to Ref 9, p 37, the capacity of the basic unit at KOW was 900-950 lbs of soln with the following average analysis: NaN_3 320 g/l, NaOH 1-7 g/l, Na_2CO_3 0.01-0.05% and IM (insol matter) <0.008%

An 8 oz sample was taken by the operator while the soln was in the "refined scale tank" and brought to the lab. After detg the sp gr of the soln by means of a 1.1-1.2 hydrometer, the following determinations were made:

a)Determination of NaN_3 Content. Assemble the apparatus consisting of "gas evolution flask" [Pyrex Erlen flask ca 130 mm high and ca 68 mm diam, std taper 29/42 with hollow stopper, having inside permanently attached in the center of bottom, a cylindrical vial, 25 mm diam and 30 mm high; a "gas measuring burette" 100 ml capacity provided with a bulb and a levelling bulb (reservoir)]

Prepare an aliquot by pipetting 20 ml of liquor into 1000 ml volumetric flask, diluting to the mark with distd w and shaking the flask. Pipette exactly 20 ml of this soln into the inside of the Erlen flask surrounding the vial ("outer space") and exactly 5 ml of 43% ceric ammonium nitrate soln into the vial ("inner space"). Connect the Erlen flask to the gas measuring burette filled with w (satd with N_2 & CO_2) and let stand for 10 mins. Remove burette stopcock for 10 secs, in order to equalize the pressure inside and outside the reaction chamber. Raise levelling bulb until just above level of stopcock hole. When burette is full of w and it begins to seep into stopcock chamber, replace the stopcock and the levelling bulb. Open stopcock and if there are any bubbles in top of gas burette, repeat the previous operation. Loosen the evoln flask from the clamp and shake, gently at first. Replace flask in clamp and after allowing to stand for 10 mins level the water in the bulb with that in the gas burette and take reading. Note temp of burette jacket and of const temp bath and correct barometric press to 0°

$$A = NaN_3\ g/l = 0.63639 \times V \times \frac{100\,(P - W)}{100 + 0.366\,t^\circ}$$

V - is observed vol of N_2, P - corrected press in mm, W - vap press of w at observed temp t° in °C. The value 0.63639 is obtained from the formula

$$\left(\frac{2NaN_3}{3N_2} \times \frac{1000}{4} \times 1.25057\right) \Big/ 760.0,$$

where 1.25057 g/liter is density of N_2 at std conditions

Note: If Na_2CO_3 is present as an impurity, the value V includes CO_2 because ceric ammonium nitrate, being sl acidic reacts with carbonates liberating CO_2

b & c)Determination of NaOH and Na_2CO_3 Contents by a modification of the method described in vol 2, p 514 of "A Manual for Explosives Laboratory," Lefax, Philadelphia 1943)

Procedure: Pipette a 10 ml sample into a 250 ml Erlen, dil with ca 100 ml CO_2-free distd w, add 1 drop of phpht indicator and titrate with N/10 std sulfuric acid just to the disappearance of pink color. This occurs when all NaOH is neutralized and the carbonate has been converted to bicarbonate. Take the burette reading as R_1. Add 5 drops of methyl-yellow indicator and titrate to the first slight indication of change of color from yel to reddish. This occurs when all bicarbonate is converted to sulfate. Take the burette reading as R_2

Calculations:

% Total alkalinity as NaOH =

$$\frac{R_2 \times N \text{ of acid} \times 0.0401 \times 100}{\text{Wt of sample in aliquot}}$$

As the reading $(R_2 - R_1)$ corresponds to bicarbonate obtained by conversion of carbonate, the amt corresponding to Na carbonate must be $2(R_2 - R_1)$ ml

$$C = \%\ Na_2CO_3 = \frac{2(R_2 - R_1) \times N \text{ of and} \times 0.053 \times 100}{\text{Wt of sample in aliquot}}$$

As the reading R_3 corresponds to NaOH plus bicarbonate, the amt corresponding to NaOH must be; $R_1 - (R_2 - R_1) = (2R_1 - R_2)$ ml and

$$B = \frac{(2R_1 - R_2) \times N \text{ of acid} \times 0.0401 \times 100}{\text{Wt of sample in aliquot}}$$

Note: It was mentioned in the previous proced that the value V includes CO_2. For calcn of actual % NaN_3, the formula

$$\frac{A}{10 \times (\text{sp gr of soln})} - C \times 0.6134 \text{ is given}$$

in Ref 9, p 37. As the amt of Na_2CO_3 in refined NaN_3 liquor is only ca 0.05%, it is sufficient to calc % NaN_3 from the formula

$$\frac{A}{10 \times (\text{sp gr of soln})}$$

d)Insoluble Matter (IM) in Composite. Pipette a 25 ml sample into a bottle contg several other samples of refined liquor. Shake the composite, det its sp gr by a 1.1 to 1.2

hydrometer. Filter 100 ml of composite through a tared No 2001 Selas porcelain crucible. Wash the crucible with several portions distd w, heat at 100° for 2 hrs. cool in a desiccator for 30 mins and weigh

$$\% \text{ IM} = \frac{\text{Wt of IM} \times 100}{100 \times \text{sp gr}}$$

V)Sodium Azide Feed Tank. Soln in this tank was pumped from the "refined storage" tank (see IV). The basic unit consisted at KOW (Ref 9, p 39) of 825 l of soln. The concns of NaN_3, NaOH and Na_2CO_3 were the same as in the "refined storage" tank. Analytical procedures were the same as in IV, except that no Na_2CO_3 and IM detns were made

VI)Sodium Azide Dilution Tank. The basic unit of KOW(Ref 9, p 40) consisted of 1000 l soln of 31.75 kg NaN_3 and 794 g NaOH.Specifications: NaN_3 31.75 ± 0.25 g/l, NaOH 0.794 g/l (approx) and NaN_3/NaOH 40.0 ± 0.5. An 8 oz sample is taken by sampler for the following determinations:

a)Determination of NaN_3 Content – same as proc (a) under IV

b)Determination of NaOH Content. Fill a 100 ml vol flask with sample and transfer it quantitatively to a 500 ml Erln flask. Titrate with N/10 sulfuric acid using phpht as indicator

$$\text{NaOH g/l} = \frac{(\text{ml } H_2SO_4) \times N \times 40.01}{100}$$

where N is normality of H_2SO_4 and 40.01 is equiv wt of NaOH

Note: Calculations used in making sodium azide dilutions are discussed in Ref 9, pp 41-2

VII)Lead Azide by Military Specification Methods. US Govt Spec MIL-L-3055 issued in 1949 was revised in 1952 and then replaced in 1957 by the Purchase Description X-PA-PD-1217, which was issued only for use pending final revision of MIL-L-3055, which has not yet been made at this time (1960)

The original MIL-L-3055 deals only with one type of LA-crystalline and lists the following requirements: a)Color – wh to buff, b)Form – aggregates free from needle-shaped crysts having a max dimension of 0.1 mm, c)LA content – 91.5% (minim) d) Total lead 68.50 to 71.15%, e)Acidity – none, f)Solubility in water – 1.0% (max), g) Sand test – when 0.15 g LA is used to initiate 0.40 g of tetryl in the test, not less than 45 g of sand shall be crushed

The X-PA-PD-1217 deals with two types of LA: Type I (crystalline) and Type II (colloidal). Requirements for crystalline are the same as in MIL-L-3055 whereas requirements for colloidal LA are: a)Color – wh to buff b)Form – average particle size shall be not greater than 5 microns and the max size of any particle shall be 10 microns c) LA Content – 99.0% (minim)

Following tests are taken mostly from MIL-L-3055 and X-PA-PD-1217:

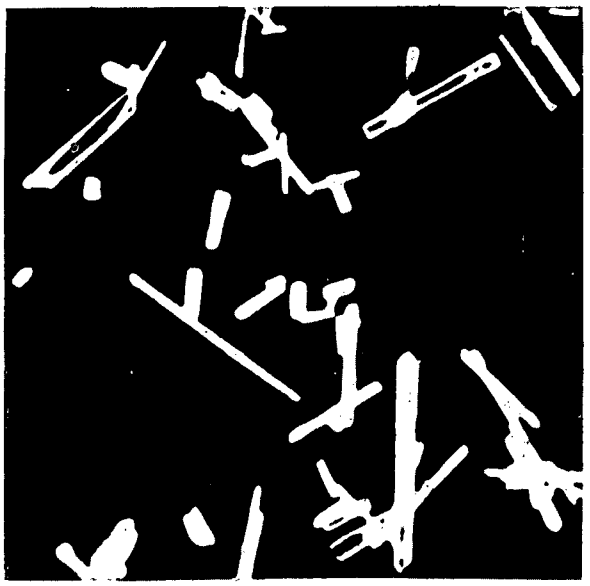
A)Color. Det by visual examination

B)Form. For Type I LA: spread a thin layer of crysts on a glass slide, allow to dry in the air at RT and examine under a microscope using a magnification 150 × (approx). If

LA NORMAL CRYSTALS



LA NEEDLE SHAPED CRYSTALS



needle shaped crysts are present, measure their max dimensions. Type II LA: use a microscope equipped with a Filar type micrometer eye-piece and such an objective (approx 43×) that the total magnification of the optical system is approx 550×. Detailed description of procedure is given on pp 7-10 of X-PA-PD-1217

Note: For the tests described below use samples previously dried in a vacuum oven at 65° to const wt. This requires ca 3 hrs (Never go beyond 25 hrs)

C)LA Content. Several methods are known of which the following direct titration method described by J.D.Hopper & O.E. Burton in PATR 255 (1932) seems to be the simplest, although it is not as accurate as the *gasometric method* used by the US Ordnance Corps

Procedure. Dissolve 0.25 g sample in 10 ml of 2N NaOH soln and add 200 ml of freshly boiled and cooled distd w. Neutralize the soln with dil sulfuric acid using litmus as indicator. Add (from a pipette) exactly 25 ml of 0.1N ceric sulfate soln and after allowing the mixt to stand 5 mins, add from a burette an excess of 0.1N ferrous ammonium sulfate soln, recently standardized by 0.1N K permanganate soln, as evidenced by the disappearance of yel coloration. Titrate with 0.1N K permanganate soln until the appearance of a permanent faint pink coloration. The reactions involved in the analysis are:

$$Pb(N_3)_2 + 2NaOH \rightarrow 2NaN_3 + Pb(OH)_2$$

$$2NaOH(excess) + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

$$2NaN_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2HN_3$$

$$2HN_3 + 2Ce(SO_4)_2 \rightarrow 3N_2 + Ce_2(SO_4)_3 + H_2SO_4$$

$$2Ce(SO_4)_2\ (excess) + 2(NH_4)_2\ Fe(SO_4)_2 \rightarrow Ce_2(SO_4)_3 + Fe_2(SO_4)_3 + 2(NH_4)_2\ SO_4$$

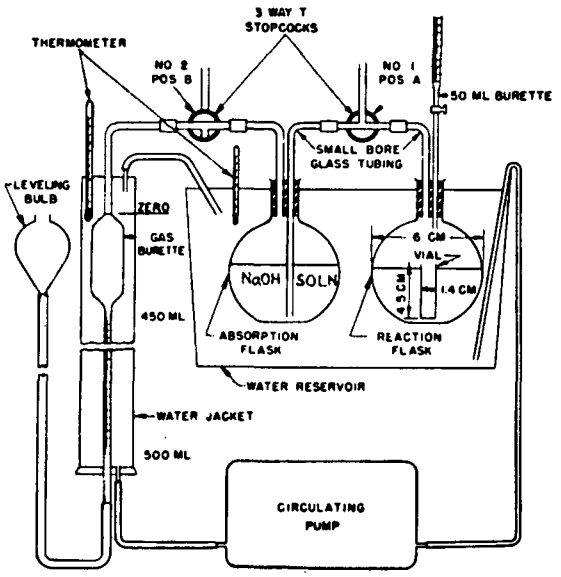
$$10(NH_4)_2\ Fe(SO_4)_2(excess) + 2KMnO_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 10(NH_4)_2\ SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$$

Due to the fact that doubts have been expressed concerning the validity of the values obtained from the assay of the *gasometric method* of analysis, Croom & Pristera of Pic Arsn (Ref 15) investigated this method. They also studied the US Navy *distillation–titration method* and the *British direct titration method* not only from the point of view of their precision but also of their applicability to LA's contg additives, inorganic (Pb carbonate in Brit "Service" LA) or organic (dextrin, PVA, etc)

a)USOrdnance Corps Gasometric Method, known also as the *Modified Nitrogen Evolution Method.* It was developed in 1947 at PicArsn by F.Pristera et al and originally described in ChemLabRept 120347. It was later incorporated in the US Military Spec MIL-L-3055(1950) and then in the Purchase Description X-PA-PD-1217 (1957). The previously used method described in the US Spec 50-13-12 was not applicable to LA's contg carbonates (such as the Brit "Service" LA). Therefore the modified method was developed. Following is its description

Apparatus: Assemble the app as shown on fig

and insert the burette contg distd w satd with nitrogen into one hole in the rubber stopper of the reaction flask. Add 90 ml of 10% NaOH soln satd with nitrogen to the 125 ml carbon dioxide absorption flask. Fill the gas burette and levelling bulb with a 0.1% soln of "Nacconol" (or other approved org detergent) satd with nitrogen. Control the temp of the system by circulating water by means of a pump betw the water-reservoir which serves as a jacket for the reaction flask and the glass jacket of the gas burette



Procedure. Place a part of a wet sample in a tared Büchner funnel. Remove the bulk of liquid by suction and the rest by air-drying followed by heating in a vacuum oven at 65° to const wt (requires 3 or more hrs, but not more than 25 hrs). Transfer from the funnel an accurately weighed portion of dried LA (ca 1.7g) to a glass vial shown on fig. Add 3 ml of distd w to the vial and place it erect in the reaction flask contg 75 ml of 15% ceric ammonium nitrate soln satd with nitrogen and containing ca 2 ml of 50% aq soln of Dow Corning Antifoam AFEmulsion. Connect the reaction flask to the apparatus, taking care not to upset the vial contg the sample. In order to insure that the reaction and absorption flasks are connected to the apparatus without air leaks, apply a coat of molten paraffin wax to all rubber-to-glass joints. Open stopcocks 1 and 2 to the atmosphere by adjusting them as shown by position A on fig. Adjust the water level in the gas burette to zero with the aid of the levelling bulb and after waiting 10 mins to allow the system to come to temp equilibrium, read the thermometer in the water jacket to 0.1°. Turn stopcocks 1 and 2 to position B and shake the reaction flask so that the vial inside of it is upset and assumes a horizontal position, thus allowing the sample to react with ceric ammonium nitrate, also called ammonium hexanitrocerate:

$$Pb(N_3)_2 + 2(NH_4)_2\,Ce(NO_3)_6 \rightarrow 3N_2 + 2Ce(NO_3)_3 + 4NH_4NO_3 + Pb(NO_3)_2$$

Note: If a carbonate is present, it would react with Amm hexanitrocerate (in acid medium) with formation of CO_2. If this gas was allowed to go to the measuring burette the results for LA content will be too high. In order to remove CO_2 from the gas mixt, a flask contg NaOH soln ("caustic trap") is inserted between the reaction flask and the burette (see fig). As the gas is evolved from the reaction flask, lower the levelling bulb so that the liquid level in the bulb is sl below that in the gas burette. Occasionally gently agitate the reaction flask to aid in completing the decompn of LA. As soon as the evoln of gas ceases, add to the flask (if necessary) a measured amt of w from the 50 ml burette until the water level in the burette will be between 450 and 500 ml divisions of the burette. Allow the temp of the system to adjust itself to within 0.1° of its temp at the beginning of the detn and then measure the vol of gas at the existing atm press, detd to the nearest 0.1 mm Hg with the aid of a mercurial barometer having a brass scale. Correct the observed reading to 0°

$$\%\ LA = \frac{0.1558 \times (A-B) \times (C-D)}{(273 + t) \times W}, \text{ where}$$

A = ml of gas measured in gas burette, B = ml of w added to reaction flask from burette, C = atm press in mm Hg, D= vapor press of w in mm Hg at temp t, t = temp of w in the jacket surrounding gas burette, W = wt of dry sample in grams

Note: For routine plant analysis at KOW, detn of LA content was done by the *gasometric method* similar to detn of NaN_3 content described under IV Refined Sodium Azide Liquor, Ref 9, pp 40 & 53a. This method was simpler and more rapid although probably not as accurate as the Ordnance Corps method

b)US Navy Distillation – Titration Method, known also as NOL Method was developed at the NavalOrdnLab and described in Ref 10. It consisted of treating LA with dil sulfuric to produce volatile hydrazoic acid. This was distilled into a measured excess of Amm hexanitrocerate and the excess cerate was then detd by titration with std Na oxalate

As this method proved to be inaccurate for some types of LA (see Discussion, which follows the description of methods), the following modified version was developed at PicArsn and described in Ref 14, pp 5-6 and Ref 15, pp 5-6

Apparatus consists of a heating mantle, a 125 ml round-bottom reaction flask a three-way side-arm adapter, a 50 ml burette with a No 5 rubber stopper, a water-cooled condenser, a support with a clamp, a curved adapter and a 125 ml Erlen flask, serving as a receiver. Std tapered ground glass joints are used to connect the distilling flask side-arm adapter, condenser and curved adapter. The three-way side-arm adapter has a female joint, at its upper end, to which the burette is fitted with the No 5 rubber stopper. A thin film of silicone grease may be applied to ground-glass joints

Procedure. Air-dry a portion of the sample in a Büchner funnel and then heat in a vacuum oven at 55 to 60° for 3 hrs or until const wt is obtained. Transfer an accurately weighed portion of from 2 to 3 milli-equivalents (0.2991 to 0.4366 g) of the dried sample to a small porcelain (or glass) crucible and cover the sample with w. Add to the reaction flask a small amt of 50% Dow Corning Antifoam AF Emulsion and place the crucible in the flask, using rubber-tipped tweezers. Connect the ground glass joints of the apparatus and insert in the upper end of the three-way adapter a 50 ml burette, using a No 5 rubber stopper. Fill this burette with 3N perchloric acid and place 40-50 ml (accurately measured) of 0.1N Amm hexanitrocerate in the Erlen flask. Arrange this flask in such a position that the adapter from the condenser extends below the surface of liq. After checking all the joints for tightness, run the perchloric acid from the burette to the reaction flask and close the stopcock of burette. Heat the flask for about 12 mins to distil off hydrazoic acid formed as result of reaction: $Pb(N_3)_2 + 2HClO_4 \rightarrow Pb(ClO_4)_2 + 2HN_3$. The acid reacts with Amm hexanitrocerate in the Erlen flask as follows: $2HN_3 + 2(NH_4)_2Ce(NO_3)_6 \rightarrow 3N_2 + 2Ce(NO_3)_3 + 2HNO_3 + 4NH_4NO_3$. Disconnect the adapter and rinse it by a stream of distd w into Erlen flask, add 1 drop of 0.25M 5-nitro-1,10-phenanthroline (nitroferroin) and titrate the excess of cerate with std 0.1N Na oxalate until the color changes from red to pale greenish blue. Make a blank detn on the reagents and apply correction if necessary. Standardize the cerate by titrating 40.0 ml of soln with std Na oxalate obtained from the NBS (National Bureau of Standards)

$$\%\ LA = \frac{(A \times B) - [(C-E) \times D] \times 4.2}{0.2886 \times W}, \text{ where}$$

A = ml of Amm hexanitrocerate soln in Erlen flask, B = normality, C = ml of Na oxalate, D = its normality, E = ml Na oxalate soln used in blank detn, W = wt of sample in grams

c)British Direct Titration Method, known also as *ERDE* (Explosives Research and Development Establishment) *Method,* was described in ERDE Report 17/R/53. Essentially it consists of prepg a soln of the sample and quantitatively pptg the azide with a known excess of std Ag nitrate soln. The excess Ag nitrate

is then titrated with std thiocyanate (See also under Discussion, which follows)

This method is also described in the Brit Spec IG 237, a copy of which is included as Appendix in PicArsn, ExplDevSectn Rept No 57(1959), Appendix. Other Brit tests for LA, described in the same Spec include: matter insoluble in nitric acid, acidity, moisture and bulk density

Discussion. In the opinion of Croom & Pristera (Ref 15, pp 4-5), although the Brit ERDE method is applicable to all LA's and is the simplest (because it does not require any special equipment), it is undesirable because it is very tedious and because Ag azide pptd in the course of analysis is a very sensitive expl. The NOL method (Ref 10 & Ref 15, pp 2 & 5-6) is undesirable because it is rather tedious, is inapplicable to LA's contg certain organic additives (such as PVA) and often gives low results with other types of LA. This inaccuracy is probably due to partial coating of unreacted LA with insol Pb sulfate (formed on treating LA with dil sulfuric acid), thus preventing this coated portion from completely reacting with sulfuric acid. Replacement of sulfuric acid by perchloric acid as described in the distillation-titration procedure given below gave satisfactory results

The US Ordnance Corps method (Ref 15, pp 3-5 and Ref 14, pp 4-5) requires a complicated apparatus difficult to assemble, but if the method is used for control work, these disadvantages are encountered only on initial installation, because the equipment can be used countless times without any readjustments or repairs. Once the equipment is assembled, the method enjoys the advantage of being applicable to all types of LA. Also, it has a shorter working time, eliminates the tedious prepn and standardization of reagents, it uses a larger sample (which tends to reduce any error in weighing and also lessens the possibility of obtaining a non-homogeneous sample), and it is relatively safe because the sample is placed under water immediately after weighing

D)Total Pb Content in LA. This test, not required now for the Army material, is simple and can be used for the detn of purity of LA samples, if desired

Procedure: Dissolve with const stirring a ca 1g accurately weighed sample (previously dried at 65° for 3 hrs) in 50 ml of satd Amm acetate soln heated in a 400 ml beaker. The principal reaction is probably:

$$Pb(N_3)_2 + 2\,CH_3COONH_4 \rightarrow 2NH_4N_3 + (CH_3COO)_2Pb$$

Add ca 200 ml distd w, heat to boiling and while stirring rapidly, add gradually 10 ml of a 10% potassium chromate soln:

$$(CH_3COO)_2Pb + K_2CrO_4 \rightarrow PbCrO_4 + 2\,CH_3COOK$$

Digest on a steam bath for 1 hr with frequent stirring and filter through a tared Gooch or Selas No 2010 crucible. Wash the ppt of $PbCrO_4$ in crucible with hot distd w, dry for 2 hrs at 100°, cool in a desiccator and weigh

$$\%\ Pb = \frac{0.64109 \times \text{Wt of ppt} \times 100}{\text{Wt of sample}}$$

Note: For cleaning the Selas crucible, remove as much of ppt as possible by inverting the crucible and lightly tapping the base with the fingers. Dissolve the rest of ppt in warm 1:1 HCl and wash the crucible with hot distd w, employing straight and reverse washings. Dry in an oven until two successive weighings agree within at least 0.0005 g

E)Acidity in LA by the Standard Method (Army). Mix the original wet sample and transfer ca 10 g portion to a tared Gooch or Selas No 2001 crucible. Wash with five 20 ml portions of cold (0 to 15°) distd w, which has been boiled and cooled prior to the test. Allow each portion of w to remain in contact with LA for 3 mins. Add to the filtrate 5 drops of 0.1% methyl orange indicator and note if the sample is free from acidity as shown by the absence of a red tinge

Note: For the routine analysis of LA intended for the *Navy* a dried sample (ca 1g) is

accurately weighed in a tared Grooch or Selas crucible and, after subjecting it to the same treatment as above, the filtrate is tested for acidity. The crucible, with washed LA, is dried at 65° to const wt and the loss of wt gives the solubility of LA in water (see Solubility in Water). The Navy test has the advantage of determining the acidity and soly on the same sample. Its disadvantage seems to be a too small sample (1 g) to permit accurate detn. It probably would be better to use a larger sample

F)Solubility of LA in Water. Transfer ca 5 g of air-dry sample to a tared filtering crucible, dry in a vacuum oven at 65° to const wt (ca 3 hrs), cool in a desiccator and weigh. Wash the dry LA with five 10 ml portions of distd w at 0 to 15°, allowing each portion to remain in contact with the sample for 3 mins. Remove each wash by suction and aspirate for 5 mins after the last wash. Dry the crucible at 65° to const wt, cool in a desiccator and reweigh

$$\% \text{ Soly} = \frac{\text{Loss of wt} \times 100}{\text{Wt of sample}}$$

Note: The same method can be used for soly in 50% alcohol, etc

G)Sand Test, used to det initiating efficiency of LA, is conducted according to description given in Ref 6, p 5 and Ref 7, pp 7-9

VIII)Moisture in Dried LA. LA's dried at the Plant usually contain small amounts of moisture (0.3-0.4%). The following two methods for the detn of this moisture were studied at PicArsn by Bernstein (Ref 8):

a)Closed Vessed Technique. Transfer carefully by a wooden spatula ca 2 g sample to a tared, flat top Pyrex, weighing vessel approx 55 mm in diam and 30 mm high, provided with a ground joint cap. Close the dish and accurately weigh the ensemble. Remove the cap and heat the vessel in an oven at 65° for 1 to 1½ hrs. Cool for 20 mins in a desiccator over freshly prepd $CaCl_2$, cap the vessel and reweigh

$$\% \text{ Moisture} = \frac{\text{Loss in wt} \times 100}{\text{Wt of sample}}$$

b)Open Vessel Technique. The test is essentially the same as above except that an open dish, approx 55 mm in diam and 30 mm high is used. The weighing operation must be conducted as quickly as possible to avoid absorbtion of atm moisture

Note: Investigation of the above two methods conducted at PicArsn (Ref 8) have shown that values obtained by the "closed vessel technique" are on the average 0.06% higher (when tests are conducted at 43-47% RH) then obtained by the "open vessel technique"

IX)Moisture in Wet LA. According to Ref 9, p 54, LA manufd at the Kankakee OW was packed wet (contg 22-27% of water or 50% alc) in cloth bags, each contg the equivalent of 25 lbs of dry azide. Five such bags were placed in a drum and surrounded by sawdust wetted with 50% alc. As the azide was packed, a sample of ca 25 g was taken from each drum, placed in a tared Al dish and reweighed. After drying the sample at 65° for 16 hrs, the ensemble was cooled and reweighed

$$\% \text{ } H_2O = \frac{(\text{Loss in wt}) \times 100}{\text{Wt of wet sample}}$$

Note: Since the azide had been packed usually a day or more before the moisture detn was completed, it was necessary to calculate in advance the wt of wet azide to pack in order that each bag should contain 25 lbs of dry LA. For this purpose it was assumed that the moisutre content of the lot (four drums) being packed would be approx the same as the ave of the ten preceding analyzed lots. From this ave value, the wt of wet material equiv to 25 lbs of dry LA was calcd. This result, plus a tare of containers constitutes the **pack weight** (Ref 9, p 54)

Eg: If average moist content of 10 preceding lots was 25.26% and tare of containers 10 lbs 6 oz, the ave wt of wet LA equiv to 25 lbs of dry LA $= \frac{25}{100-25.26} = 33.449$ lbs = 33 lbs 7oz.

This gives for pack weight = 33 lbs 7 oz + 10 lbs 6 oz = 43 lbs 13 oz

X)Moisture in Wet LA by Density Measurements. For rapid estimation of moisture in Nutsch blends of LA going into a drum, the following procedure was used at the Kankakee OW

A wet sample was packed to about ⅔ capacity of a tared calibrated 125 Erlen flask and the ensemble weighed on a trip balance. Water was added to the mark and the ensemble reweighed. The gain of wt in grams was approx equal to the vol in ml of w added and vol of wet LA was equal to capacity of flask (ca 125 ml) minus vol of w add. By dividing the wt of wet LA by its vol, the density was detd (Ref 9, p 55)

Calculation. Percentage of moisture in wet LA may be calcd from the formula:

$$\%H_2O = \left[\left(\frac{D}{d} - 1\right) \Big/ \left(D-1\right)\right] \times 100, \text{ where}$$

D = density of dry sample, d = density of wet sample and 1 = density of water

Eg: If D = 4.38 and d = 2.18, then

$$\%H_2O = \left[\left(\frac{4.38}{2.18} - 1\right) \Big/ \left(4.38-1\right)\right] \times 100$$

$$= \frac{1.01 \times 100}{3.38} = 29.9\%$$

XI) Ball Drop Test. In order to det the sensitivity of LA to impact, a composite sample was taken from each lot manufd at the Kankakee OW, dried as usual and subjected to the following test described in Ref 9, p 56:

A steel ball, ½" diam and weighing 8.33 g, was dropped from a height 25" on a 0.08" layer of LA spread on 1" thick SAE 3260, nickel-chrome, hardened steel block which rested on a 1¼" thick by 12" diam cast iron base. Ten consecutive drops should produce no detonations in a properly manufd LA, but when the ball was dropped from the height of 45", it should deton in all trials. Care was taken not to drop the ball on the same spot twice, as a pellet made by the first drop would usually fire if hit a second time. If detonations occurred at 25" drop, lower heights were tried until no detons were produced

XII)Ethyl Alcohol Solution. Since LA is very sensitive, it was shipped and stored at KOW wet with not less than 20% (usually 25-27%) of a 50 ± 0.5% (by wt) denatured alcohol (Ref 9, p 57). Alcohol served as an antifreezing agent. The compn of an alc soln can be detd from the table, density vs % alc after detg the density by means of a Leach pycnometer standardized at 25°, using the following formula:

$$d = \frac{\text{Wt of pycn with alc soln} - \text{Wt of pycn empty}}{\text{Vol of pycn at } 25^\circ}$$

Note: Alcohol used at the Kankakee plant was Grade SD No 1, delivered in No 50 drums. It contained 5 parts of methanol per 100 parts of ethanol. No analysis is necessary because alc is produced and shipped under Govt supervision

XIII)Killing Tank at KOW contained, according to Ref 9, p 59, various waste liquids and slurries in which azides are likely to be present. When a sufficient quantity of such material was accumulated (not less than 1000 liters), the operator stirred the contents of the tank and took a 6 oz sample. If a small portion of the sample gave a positive test for azides (a red ppt of iron azide obtained on adding few drops of 1% $FeCl_3$ soln), a quantitative test was conducted as follows:

Determination of Azide as NaN_3. Pipette a 25 ml portion of the sample into a 500 ml Erln flask and dil with w to 150-200 ml. Titrate with 40% sulfuric acid to just to the phpht end-point, not overrunning it. Add N/10 ceric sulfate soln in 5 ml portions until the soln becomes deep yel, indicating an excess of ceric sulfate. Record the exact amt used. Add 10 ml of 40% sulfuric acid and 2 drops of o-phenanthroline indicator (prepd by dissolving 14.85 g of o-phenanthroline monohydrate, $C_{12}H_8N_2 \cdot H_2O$ in 1 l of 0.025 M freshly prepd ferrous sulfate

soln). Add N/10 ferrous sulfate soln to an orange (or pink) end-point in order to reduce the residual ceric sulfate to cerous salt

The following reactions take place:

$$2\,Ce(SO_4)_2 + 2NaN_3 \rightarrow 2N_3 + Na_2SO_4 + Ce_2(SO_4)_3$$

$$2\,Ce(SO_4)_2 + 2\,FeSO_4 \rightarrow Fe_2(SO_4)_3 + Ce_2(SO_4)_3$$

Calculation:

$$NaN_3\ g/l = \left[\left(\begin{matrix}FeSO_4\text{ equivalent of}\\CeSO_4\text{ used, ml}\end{matrix}\right) - (FeSO_4\text{ used, ml})\right] \times \frac{NaN_3 \times N\text{ of }FeSO_4}{25}$$

Note: Killing of contents of the tank was usually done by adding a mixt of 1 part 30% $NaNO_2$ and 2 parts of 30% HNO_3

$$NaN_3 + NaNO_2 + 2HNO_3 \rightarrow 2NaNO_3 + N_2O + N_2 + H_2O$$

$$Pb(N_3)_2 + 2NaNO_2 + 4HNO_3 \rightarrow Pb(NO_3)_2 + 2NaNO_3 + 2N_2O + 2N_2 + 2H_2O$$

XIV)Nitric Acid for Killing was used (Ref 9, p 61) in mixt with 30% $NaNO_2$ soln and should be ca 30% and not below 22%

Procedure: Cool to 20° the sample collected in an 8 oz glass-stoppered bottle and fill a hydrometer jar to about ⅔. Insert hydrometer of range 1.0-1.2 and take reading at 20°. Det approx concn from tables, density vs concentration, such as

d at 20°	% HNO_3
1.115	20.0
1.120	20.8
1.125	21.6
1.130	22.4
1.134	23.0
1.140	24.0
1.152	25.8
1.156	26.4
1.160	27.0
1.164	27.6
1.168	28.2
1.170	25.0
1.172	28.8
1.176	29.4
1.180	30.0

XV)Disposal of Laboratory Samples Containing Azides at KOW. According to Ref 9, pp 69-71 the following were some of the rules observed in the lab of Kankakee OW:

a)No more than 25 g of dry LA was allowed in the lab at a time

b)No azide-contg sample was allowed to be mixed with an acid since this would form the extremely toxic hydrozoic acid (gas)

c)No azide-contg sample was allowed to be mixed with salts of heavy metals, especially of Cu, Cd, Ag, and Pb since these would form sensitive azides

d)All samples contg a high percentage of NaN_3, such as crude, refined, mother liquor, clear liquor, lime treatment, feed tank and wringer cake (see under Sodium Azide), were saved and returned to operations, as an economy measure

e)All LA moisture samples were, after final weighing, saved and returned to the plant for mixing with finished product

f)All Government composite samples were, after removal of the portion for analysis, wetted with 95% alc and returned to the Govt Inspector

g)All LA samples, suspected to be impure (such as contaminated, exposed to direct sunlight, exposed to temps in excess of 65°, etc) were returned to the plant to be destroyed as described under Killing Tank. Small amts of azide were killed in the lab with ceric ammonium nitrate soln:

$$2NaN_3 + 2Ce(NH_4)_2(NO_3)_6 \rightarrow 4NH_4NO_3 + 2NaNO_3 + 2Ce(NO_3)_3 + 3N_2$$

XVI)Laboratory Disposal of LA by the Method of Wm. H. Rinkenbach. Disperse with stirring ca 1 oz of waste LA in 1.5 gal 10% aq Amm acetate soln, add 2.5 oz of Na nitrate dissolved in 1 pint of w, and then 7 oz of glacial AcOH or its equivalent of weaker acid. After allowing the soln to stand in a warm place for at least 1 hour, dispose of it

Notes: A)The above proced results in a clear soln, which contains no toxic materials and can be easily disposed of by pouring it into

a sink or a stream B)This proced does not possess the disadvantages of other methods, such as: a) Solution in Amm acetate and decompn with Na bichromate. This method produces a sludge which cannot be disposed of by dumping into a sink b)Decompn with ceric ammonium nitrate soln. This reagent is expensive and somewhat corrosive c)Decompn by Na nitrite and nitric acid. This mixture is very corrosive and the procedure requires stirring during decompn to keep the heavy LA in suspension C)The procedure of WHR can be used to remove azides from bags used for storing wet LA or for catch-bags placed underneath sinks, without injuring the fabric. For this, the bags are impregnated with Amm acetate soln and then subjected to treatment with Na nitrite soln and AcOH. For removing LA or other azides from machinery, tables, floors, etc, it is usually sufficient to wipe the object with a rag impregnated with Amm acetate soln, followed by wiping with Na nitrite soln and AcOH

XVII)Laboratory Disposal of LA by the British Method. Disperse with stirring ca 1 oz of LA in 250 ml of 15% aq Na nitrite soln, and add slowly 250 ml of 25% aq AcOH soln. Carry out this operation in the hood behind a protecting screen

XVIII)Laboratory Disposal of LA by the Method Used at the du Pont's Plant at Pompton Lakes, NJ. Disperse slowly with stirring ca 1 g sample in ca 16 ml of 25% aq ceric ammonium nitrate soln and allow to stand. Fumes from the decompn are not toxic

XIX)Laboratory and Plant Disposal of LA by the Method Used at the du Pont's Plant at Pompton Lakes, NJ. Disperse with stirring ca 1 g sample in 500 ml w and mix it with 9 ml of a 30% aq Na nitrite (commercial grade) soln. Finally add ca 18 ml of a 24% aq nitric acid and test with ferric chloride for the completion of destruction

XX)Laboratory Disposal of LA by the Method Used at Picatinny Arsenal. Disperse with stirring ca 1 oz sample in 1.5 gal of 10% Amm acetate soln and add sufficient Na bichromate soln to ppt all lead as chromate. Test for the completion of destruction by transferring a portion of slurry to a filter paper and treating the filtrate with a few drops of Na bichromate soln. Confirm the absence of LA by washing,with distd w,the residue on filter,free of sol azide salts, drying it,and subjecting a 20 mg portion to the impact test, using 2 kg wt. The material should not detonate

XXI)Laboratory Test for the Presence of LA. The following proced was used at PicArsn and described in ChemLabRept 94772(1943) for detection of azides in loaded housings of friction primers:

Procedure: Place three of the loaded housings in a 50 ml beaker, add 20 ml of 25% aq Amm acetate soln and heat to boiling. Allow to boil gently for 5 mins. Remove the housings, washing them with a stream of distd w. Boil the soln for another 5 mins and filter, catching the filtrate in a small beaker. Acidify the filtrate with 3 drops of concd nitric acid and add 5 ml of 10% aq Ag nitrate soln. After allowing to stand for 30 mins, filter and wash the ppt on filter paper with w. Remove the filter paper, spread it on a watch glass and pour ca 10 ml of 10% ceric ammonium nitrate soln on top of ppt. The evolution of odorless gas (nitrogen) indicates the presence of an azide (in this case, it is silver azide)

Refs: 1)M.Marqueyrol & P.Loriette, MP **18**, 93-5(1921) & CA **16**, 1667(1922) (Detn of LA in primer compns as conducted at the Laboratoire Centrale des Poudres, Paris) 2)S.M. Moskovich, Visti Institutu Fizichnoi Khemii-Akademiya Nauk Ukraina (Russia), **6**, 179-87 (1936) & CA **31**, 6579(1937) (Potentiometric detn of LA was conducted by dissolving 0.02 g LA in 50 ml of 2% Ba nitrate, making the soln acid with 1 ml of 2N nitric acid, plus 2 ml of 2N AcONa and titrating with 0.1N Ag nitrate) 3)J.W.Arnold, IEC, AnalEd **17**, 215-17(1945) & CA **39**, 2267(1945) (Assay of LA & Na azide by cerate oxidimetry) 4) R.Haul & E.Scholz, Naturwissenschaften **32**,

294-5(1944) & CA **40**, 2764(1946) (Polarographic detn of azide ion in general and in LA in particular) 5)D.F.Vasil'ev, TrudyKomissiiAnalKhim, OtdelKhimNauk, **2**, [5], 90-5(1949) & CA **44**, 9300-1(1950) (Polarographic detn of Pb content of LA) 6)US Military Specification MIL-L-3055(1949) and Amendment 1 (1952) (Requirements for crystalline LA intended for use in manuf of detonators, fuzes and priming compns) 7)W.H. Rinkenbach & A.J.Clear, "Standard Laboratory Procedures for Sensitivity, Brisance and Stability of Explosives", PicArsnTech Rept 1401 (1944), Revised in 1950 8)J. Bernstein, "Determination of Moisture Content of Lead Azide", PicArsnGenLab Rept 51-H1-2331 (1951) (Comparison of closed and open vessel techniques) 9)US Rubber Co, Kankakee Ordnance Works, Joliet, Illinois, "Lead Azide Laboratory Manual", Revised in 1953 by B.C.Carlson (Lab procedures used in the manuf of Na and Pb azides and a brief description of manufg process developed by the Du Pont Co) 10)S.G. Landsman & J.M.Rosen, "Improved Method for the Assay of Lead Azide", Navord Rept 4191(1955) 11)J.Vřeštál et al, Chem Průmysl **6**, 50-2(1956) & CA **50**, 14229(1956) (Chelatometric detn of Pb in LA) 12)Y. Mizushima & S.Nagayama, JIndExplosives Soc Japan **17**, 113-15(1956) & CA **50**, 16557 (1956) (Microdetn of Pb, Cu & Na in azides) 13)Ibid, Rept Govt Chem Ind Res Inst **51**, 320-2(1956) & CA **51**, 4216 (1957) (Gasometric detn of Pb, Ca & Na azides with ceric ammonium nitrate) 14)US Military Purchase Description X-PA-PD-1217(1957) issued for use pending revision of MIL-L-3055 (Requirements for crystalline and colloidal lead azides) 15)R.Croom & F.Pristera, "Investigation of Methods for the Analysis of Lead Azide", SFAL, Tech Rept 2486(1958), PicArsn, Dover, NJ 16)D.G.Young, formerly of Kankakee OW, Joliet, Ill; private communication, 1960 (info on manuf and analysis of LA)

Lead Azide Explosive, Primer and Detonator Compositions. LA has replaced MF for many purposes. Its chief applications are as an initiating agent for less sensitive HE charges and as an ingredient of priming compns which are very sensitive to impact or percussion. Priming compns are used for the ignition of initial detonating agents, BkPdr, small arms propellants, etc. Most military priming compns consist of a mixt of one or more initial detong agents, oxidizing agents, fuels, sensitizers and binding agents (Ref 98)

Some typical primer and detonator compns used in USA are as follows (Refs 88,95,97 &98): *Percussion Type* – LA/$KClO_3$/Sb_2S_3/ ground glass 33.6/14.3/21.5/30.6 and LA/ $KClO_3$/Sb_2S_3/$Pb(SCN)_2$ 5/53/17/25 (Ref 87); *Friction Type* – LA/$KClO_3$/Sb_2S_3/carborundum 28.3/33.4/33.3/5.0; *Relay Type* – LA Pressed at 5000 psi and covered with an onion skin; and *Detonator Type* – LA generally sensitized by the addn of lead styphnate (LSt), $PbO_2C_6H(NO_2)_3 \cdot H_2O$, to lower the ignition temp

Grant and Tiffany (Ref 84) detd that the order of initiating efficiency of priming compositions increased as follows:

Priming Composition	%	Order
Lead styphnate (LSt)	100	1
LA/LSt	20/80	2
Mercury fulminate/$KClO_3$	80/20	2
LA	100	3
LA/LSt/Aluminum	80/17/3	3
LA/LSt	40/60	3
Diazodinitrophenol/$KClO_3$	75/25	3
LA/LSt/Al/$KClO_3$	80/18/0.5/1.5	4
LA/LSt	60/40	4
LA/LSt	80/20	5

The sand crushed per unit wt of expl in the detonator was taken as a measure of the

initiating efficiency of the expl chge. This test is called the *miniature - cartridge test* (Ref 85). The testing of detonators was described in previous reports (Refs 9,32,43,73 &80)

Modern detonators are compd detonators loaded with a base chge, a priming chge and sometimes an ignition chge. The common base charges in use are tetryl, PETN and TNT (Refs 5,11,15,18,30,31,36,38,39,42,44, 48,49,53,59,60,65,81,82,86,94 & 98). RDX was proposed in 1922 (Ref 23) as a base detonator chge but it has not found practical use. Grant and Tiffany (Ref 84) found the order of increasing efficiency of detonator base charges to be: MF/$KClO_3$ 80/20, tetryl, PETN and RDX. The influence of other factors such as priming chge, reinforcing capsule and outside diameter of shell was also investigated

Although Blechta (Ref 33) concluded that LA was too sensitive and unsuitable for practical use, the large and extensive patent and technical literature is evidence of the interest and application of LA in detonators. Hyronimus (Ref 1) was the first to propose the use of LA in priming chges. The use of layers of a HE nitro compd and a covering layer of LA suitable for blasting caps, primers or detonators was patented by Wöhler (Ref 2) Rheinisch-Westfälische AG (Ref 3), Burkard (Ref 7), Will (Ref 5), Matter (Refs 11 & 31), Eschbach (Ref 15), Friederich (Ref 24), Nobels Expl Co & Morris (Ref 30), Oerlikon (Ref 36), Symmes (Ref 38), Hercules Powder Co (Ref 39), Eschbach & Wippenhohn (Ref 42), Meissner (Ref 44), Lewis (Refs 48 & 53), Biazzi (Ref 49), Johnson (Ref 60), Rubenstein & Imperial Chem Inds (Ref 65), Burrows (Ref 81), Lyte (Ref 82) and Bain & Carl (Ref 89). General discussions of the use of LA in detonator compositions may be found in Refs 5, 18,29,51,52,79,84,88,97 & 98). According to Kast (Ref 29) LA loaded into detonators at 600 to 5000 kg/cm² showed increased initiating efficiency

Various multi-component LA compns or admixtures with LA to alter its sensitivity characteristics have also been developed, for example: LA/$KClO_3$ by Wöhler (Ref 8); PETN/LA/$KClO_3$ or tetryl/LA/$KClO_3$ by Claessen (Ref 4); NC/LA 3/1p or NC/LA/NaN_3 11/6/3p by Buell (Ref 6); LA/TNT 80/20 or LA/TNT/gum arabic 70-95/5-30/ 0.2-0.4p by Runge (Ref 10); NC/PA/LA by Stine (Ref 12); LA/LSt/resin by Eschbach (Ref 16); NC/LA 10-20/90-80 or NG/NC 97-70/7-30% with LA by Hudson (Ref 13); paraffin/cork flour/LA by Sprengluft-Ges (Ref 14); LA/tetryl 40-95/60-5% by Cook & Grotta (Ref 21); Pb salt of trinitrophloroglucinol/LA or Pb derivs of tetrazole/LA by Rathsburg (Ref 19); LA/LSt by vonHerz (Ref 22); LA/soot or powd cork by Kowatsch (Ref 25); LA/basic LA or basic salts of nitro compds by Friederich (Ref 20); LA/fatty subst 0.05-20% (paraffin) by Harlé (Ref 27); LA/TNT, tetryl or PETN by Matter (Ref 28); LSt/LA/Al or graphite (3%) by Ashcroft & Imperial Chem Inds (Ref 37); LA/diazodinitrophenol by Symmes (Ref 38) and 80-20/20-80% mixt by Hercules Powder Co (Ref 39); LA/powd glass 98/2% or LA/powd quartz 99/1% and LA/tetracene/$CaSi_2$ 80/10/10% by Eshbach & Wippenhohn (Ref 42); LA/$CaSi_2$/$Ba(NO_3)_2$/tetracene by Imperial Chem Inds & Weale (Ref 40); LA/2,4,6-trinitro-1,3,5-triazidobenzene by Turek (Ref 45); LSt or dibasic picrate/LA (10-20%)/resin by Eshbach & Friederich (Ref 50); LA/binder/ester gum, cellulose acetate or Canada balsam by Olsen et al (Ref 55); LA/tetracene with or without other substs such as PbO_2, Sb sulfide, $Ba(NO_3)_2$ or $CaSi_2$ by McNutt (Ref 56); LA with $KClO_3$/S/Pb thiocyanate -40/ 10/50% by duPont (Ref 58); LA/natural, synth or rubber substitute by Snelling (Ref 63); LA/ $Ba(NO_3)_2$/tetracene by Weale (Ref 62); LA/ diazoguanidine picrate with or without tetryl by Imperial Chem Inds (Ref 67); LA/nitrosoguanidine by Olsen & Seavey (Ref 61); LA/ nitratohypophosphite by Brun & Burns (Ref 68); LA/ground glass by Hatch (Ref 70); LA/ various expl additives which lower flash point, by Dynamit-AG (Ref 72); LA/Pb nitrosoresorcinate by Kerone & Carroll (Ref 71); LA/

PETN or tetryl or NC(30-60%) by Lawrence (Refs 74&78); LA/tetracene 85-90/10-15% by vonHerz (Ref 75); LA/BkPdr or smokeless powd 60-95/5-40% by Hanley (Ref 83) and LA/normal Pb salt of 2,4-dintroresorcinol by Rubenstein (Ref 92)

LA and its priming or detong compns have also been desensitized by special treatment or devices, for example: addn of 0.05 to 20% by wt of oil, grease or paraffins (Ref 26); removal of insol carbonates of Pb in LA (Ref 34); for an elec blasting cap the use above the LA chge of Pb thiocyanate/$KClO_3$/ground pyro pdr loose mixt free from admixture with LA (Ref 47); LA wetted with non flammable, non solvent liq of low volatility, such as dichloroethyl ether, but capable of complete removal by water (Ref 54); a stratified primer reqd a relatively small quantity of LA compn (Ref 64); a detonator particularly suitable for coal mining consists of an outer polyvinyl resin shell and an inner iron walled capsule charged with LA (Ref 76); by blending with LA a small quant (0.5 to 3%) of finely divided Ca stearate, blasting caps with an approp base chge were made less sensitive to shock or frictional impact (Ref 86); the addn of 4-20% of NC having a fiber length 30 to 250μ to LA reduced its sensitivity and improves loading characteristics (Ref 91); by using an elec ign device the priming compn LA/LSt 80/20% was replaced by LA alone (Ref 93); detonators for use in presence of firedamp were made safe by mixing from 3 to 20% of an inert material, such as KCl, Na_2CO_3, $NaHCO_3$, KBr or wax, with both the primary (LA or LA/LSt-65/35%) and secondary (PETN or tetryl) charges (Ref 94) and by the use of dextrinated LA for initiating compns (Ref 96)

Since LA reacts with Cu or brass detonator capsules to form extremely dangerous Cupric Azide(qv), this difficulty has been overcome by the use of Al, Al alloy, iron or paper containers; for example: Al detonator shells were patented by Eschbach (Ref 15), Biazzi (Ref 49), Lewis (Ref 53), duPont (Ref 58), Noddin (Ref 59), Rubenstein & Imperial Chem Inds (Ref 65) and others; according to Galewsky (Ref 18), German military detonators of WWI substituted for the Cu shells first Zn, then Fe and finally Al; Eschbach (Ref 35) also proposed protecting LA, in Cu containers, from moisture and CO_2 by applying a cast seal to container mouth equipped with an elec igniter; Noddin (Ref 59) coated the Al shell surface with Al_2O_3 to make it corrosion resistant; detonator casings for LA expls were also made of an alloy contg Cu or Zn (Ref 17) contg Cu 90-95% and Al 5-10% or replacing Al in part by Mg (Ref 41), nitrited alloy steel (Ref 47), alloy of Zn 95.0-98.5%, Cu 4.0-1.5% and Ag 1.0-0.1% (Ref 57) or high Al alloy subjected to anodic oxidation (Ref 58); vonHertz (Ref 46) proposed the use of vulcanized fiber casings, Lignoza-Spolka Akcyjna (Ref 66) casings made wholly of Pb; and Dhôme & Deffrenne (Ref 69) the use of a steel shell protected by an exterior layer of Pb; Salzberg (Ref 77) treated the gilding metal shell with lauryl mercaptan and Martin (Ref 90) separated the priming chge from the main chge by a foil of D'Arets alloy or Woods metal to obtn more uniform ignition

Refs: 1)F.Hyronimus, FrP 384792(1907); JSCI **27**, 524-5(1907) & CA **3**, 1690(1909); USP 908674(1909) & CA **3**, 1088(1909); Brit P 1819(1908); GerP 224669(1910) & Chem Ztr **1910** II, 771 2)L.Wöhler, BrtiP 4468 (1908); FrP 387640(1908); GerP 196824 & ChemZtr **1908** I, 1439 and USP 904289(1909) & CA **3** 717(1908) 3)Rheinisch-Westfälische Sprengstoffe AG, GerP 238942(1910) & CA **6**, 2170(1912) 4)C.Claessen, FrP 459979 (1913) & CA **8**, 3238(1914); GerP 284400 (1916) & CA **10**, 970(1916) and SwedP 40749 & 40955(1916) & CA **10**, 2525 & 2800(1916) 5)W.Will, SS **9**, 52-3(1914) & CA **8**, 1508(1914) 6)W.H.Buell, BritP 21082 (1914) & CA **10**, 970(1916); USP 1174669(1916) & CA **10**, 1435 (1916) 7)E.Burkard, BritP 16405(1914) & CA **11**, 889(1917) 8)L.Wöhler, USP 1128394(1915) & CA **9**, 1118(1915) 9)C.G.Storm & W.C.Cope, BurMinesTechPaper **125** (1916) 10)W.Runge, USP 1168746 & 1185830 (1916) & CA **10**, 822

& 2045(1916); CanP 181129(1917) & CA **12**, 631(1918) 11)O.Matter, USP 1239613(1917) & CA **11**, 3438(1917); CanP 176610(1918), USP 1254147(1918) & CA **12**, 226 & 766(1918) 12)C.M.Stine, USP 1313650 (1919) & CA **13**, 2763 (1919) 13)W.G.Hudson, USP 1329525 (1920) & CA **14**, 1045(1920) 14)Sprengluft-Ges, BritP 152335(1920) & CA **15**, 756(1921) 15)W.Eschbach, BritP 151572 (1920) & CA **15**, 599-600(1921); USP 1438431(1923) & CA **17**, 882-3(1923) 16)W.Eschbach BritP 156429(1920) & CA **15**, 1815(1921); ChemZtr **1921** II, 765 17)W.Eschbach, BritP 204277 (1923) & CA **18**, 905(1924) 18)P.Galewsky, SS **15**, 153,163,190,197,203 & 212(1920) & CA **15**, 2986 (1921) 19)H.Rathsburg, BritP 177744(1921); CA **16**, 3399(1922) & ChemZtr **1922** IV, 810; BritP 190215(1921) & CA **17**, 3101(1923) 20)W.Friederich, BritP 180605 (1921) & CA **16**, 3399(1922) 21)R.M.Cook & B.Grotta, USP 1385245(1921) & CA **15**, 3751(1921) 22)E.vonHerz, BritP 187012 (1921); CA **17**, 1147 (1923) & ChemZtr **1923** II, 781; USP 1498001(1924) & CA **18**, 2605 (1924) 23)E.vonHerz, USP 1402693(1922) & CA **16**, 1014(1922) 24)W.Friederich, USP 1424462(1922) & CA **16**, 3399(1922) 25)A. Kowatsch, USP 1424487(1922) & CA **16**, 3400(1922); CanP 222375(1922) & CA **16**, 4067(1922) 26)Etablissements Davey, Bickford, Smith et Cie, BritP 196593(1923); CA **17**, 3791(1923) & ChemZtr **1923** IV, 306 27)E.C.Harlé, USP 1488787(1924) & CA **18**, 1911(1924) 28)O.Matter, BritP 280249(1926); CA **22**, 3048(1928) & ChemZtr **1928** I, 2599 29)H.Kast, SS **21**, 188-92(1926) & CA **21**, 1184(1927) 30)Nobel'sExplCo, Ltd & G.Morris, BritP 297853(1927) & CA **23**, 2827(1929) 31)O.Matter, BritP 303975(1927); CA **23**, 4822 (1929) & ChemZtr **1931** I, 2712 32)M. Sukharevskii, SS **22**, 17(1927) 33)F.Blechta, ChemObzor **3**, 330-6(1928) & CA **23**, 1271 (1929) 34)O.Matter, FrP 663841(1928) & CA **24**, 696(1930) 35)W.Eschbach, BritP 304144(1928) & CA **23**, 4822(1929) 36)Werkzeugmaschinenfabrik Oerlikon, BritP 309114 (1928); CA **24**, 502(1930) & ChemZtr **1931** I, 1053 37)G.A.Ashcroft & ImperialChemInds, Ltd, BritP 317023(1928) & CA **24**, 1983(1930) 38)E.M.Symmes, BritP 333534(1938) & CA **25**, 595(1931) 39)Hercules Powder Co, FrP 675638(1929) & CA **24**, 2886(1930) 40) ImperialChemInds, Ltd & A.Weale, BritP 362048(1930) & CA **27**, 1177(1933) 41)W. Eschbach & W.Friederich, FrP 696663(1930) & CA **25**, 2853(1931) 42)W.Eschbach & Wippenhohn, BritP 374060(1930) & CA **27**, 3336(1933) 43)A.Haid & H.Koenen, SS **25**, 393,433 & 463(1930) 44)J.Meissner, BritP 373516(1931) & CA **27**, 3336(1933) 45)O. Turek, USP 1824848(1932) & CA **26**, 309 (1932) 46)E.vonHerz, BritP 382247(1932) & CA **27**, 5982(1933) 47)H.A.Lewis, USP 1877772(1933) & CA **27**, 420(1933) 48)G.A. Noddin, USP 1906869(1933) & CA **27**, 3612 (1933) 48)H.A.Lewis, USP 1918920(1933) & CA **27**, 4931(1933) 49)M.F.Biazzi, BritP 387545(1933) & CA **28**, 328-9(1934);USP 1950019(1934) & CA **28**, 3235(1934) 50)W. Eschbach & W.Friederich, BritP 417763(1934) & CA **29**, 2744(1935) 51)L.F.Audrieth, Chem Revs **15**, 223-4(1934) 52)I.E.Moisak, Trudy KazanKhimTekhnolInst No **2**, 81-5(1935) & CA **29**, 5271(1935) 53)H.A.Lewis, USP 1991857(1935) & CA **29**, 2360(1935) 54)F.R. Seavey & E.B.Kerone, USP 2000995 (1935) & CA **29**, 4586(1935) 55)F.Olsen et al, USP 2001212(1935) & CA **29**, 4586(1935) 56)J.D. McNutt, USP 2004505(1935) & CA **29**, 5274 (1935) 57)E.T.Lednum, CanP 348649(1935) & CA **29**, 3518(1935) 58)E.I.duPont, BritP 451668(1936) & CA **31**, 542(1937) 59)G.A. Noddin, CanP 361815 (1936) & CA **31**, 1616 (1937) 60)C.R.Johnson, CanP 361816(1936) & CA **31**, 1616(1937) 61)F.Olsen & F.R. Seavey, USP 2060522(1937) & CA **31**, 542 (1937) 62)A.Weale, USP 2065929(1937) & CA **31**, 1212(1937) 63)W.O.Snelling, USP 2067213(1937) & CA **31**, 1616(1937) 64)F.R. Seavey, USP 2068516(1937) & CA **31**, 2010 (1937) 65)L.Rubenstein & ImperialChemical Inds, Ltd, BritP 470418(1937) & CA **32**, 1456 (1938) 66)LignozaSpolka-Akcyjna, BritP 474495(1937) & CA **32**, 3156(1938) 67)Imperial ChemicalInds, Ltd, AustralianP 102202(1937) & CA **32**, 2753(1938) 68)W.Brün & J.E.Burns,

USP 2116878(1938) & CA **32**, 5214(1938) 69)A.Dhôme & P.D.Deffrenne, FrP 826286 (1938) & CA **32**, 7728(1938) 70)G.B.Hatch, USP 2156942(1939) & CA **33**, 6600(1939) 71)E.B.Kerone & C.C.Carroll, USP 2177657 (1940) & CA **34**, 1176(1940) 72)Dynamit-AG vorm AlfredNobel & Co, BritP 528299 (1940) & CA **35**, 7716(1941); FrP 852495 (1940) & CA **36**, 2414(1942) 73)R.L.Grant & A.B.Coates, BurMines RI 3696(1943) 74)R.W.Lawrence, CanP 398139(1941) & CA **35**, 6796(1941) 75)E.vonHerz et al, GerP 702269 (1941) & CA **35**, 8299(1941) 76)W. Eschbach, GerP 715101 (1941) & CA **38**, 2212(1944) 77)P.L.Salzberg, USP 2255600 (1942) & CA **36**, 274(1942) 78)R.W.Lawrence, BritP 546276 (1942) & CA **37**, 3274(1943) 79)Davis(1943), 424-30 80)R.L.Grant, Bur Mines RI 3696(1943) 81)L.A.Burrows, CanP 411756(1943) & CA **37**, 3943(1943); USP 2427899(1947) & CA **42**, 764(1948) 82)G.A. Lyte, USP 2360698(1944) & CA **39**, 1294 (1945) 83)E.J.Hanley, USP 2363863(1944) & CA **39**, 3672(1945) 84)R.L.Grant & J.E. Tiffany, BurMinesTechPaper **677**, 34pp (1945); IEC **37**, 661-6(1945) & CA **39**, 3671(1945) 85)R.L.Grant & J.E.Tiffany, IEC, AnalEd **17**, 13-19(1945) & CA **39**, 1053-5(1945) 86) L.A.Burrows & W.E.Lawson, USP 2402235 (1946) & CA **40**, 5568 (1946) 87)L.F. Audrieth, USP 2410801(1946) & CA **41**, 866 (1947) 88)All & EnExpl(1946), 66 89)C.J. Bain & L.R.Carl, USP 2415806(1947) & CA **41**, 2901(1947) 90)C.A.Martin, USP 2423837 (1947) 91)L.Rubenstein & B.Campbell, USP 2464777(1949) & CA **43**, 6828(1949) 92)L. Rubenstein, USP 2493551(1950) & CA **44**, 3022(1950) 93)Dynamit-AG, vorm Alfred Nobel & Co, GerP 803644(1951) & CA **45**, 5930· (1951) 94)H.Elsner, GerP 803645(1951) & CA **46**, 1260(1952) 95)Kirk & Othmer **6** (1951), 8 96)L.Rubenstein, USP 2653863 (1953) & CA **48**, 2376(1954) 97)Armament-Engrg(1954), 47-9 98)TechMan TM9-1910 & Tech Ord TO 11A-1-34(1955), 113-6

Lead Azide Explosive, Primer and Detonator Compositions, Analytical Procedures. Following are some typical methods of analysis:

I)Analysis of an Unknown Sample by the Method Used in the Laboratoire Centrale des Poudres as described by M. Marqueyrol & P.Loriette in MP **18**, 93–5(1921):

a)Extract a weighed portion of sample with ether in a tared filtering crucible and evaporate the extract at RT. Dry at 60°, weigh and test for nitro compds, such as PA, TNT, tetryl, etc

b)Extract the residue on filter with a small amt of w, evaporate the extract at 60° in a tared crystallizer and weigh it. Test the contents of crystallizer for chlorates, nitrates, etc

c)Add to the residue left after extraction with eth and with w, 10 ml of cold 5% aq soln of KCN and leave for 2 hrs while periodically agitating. Filter and wash the ppt with a few ml of KCN soln and then with w. This treatment dissolves MF if it is present. Test for its presence by adding a drop of nitric acid to few drops of soln. If this causes some pptn, det the MF content(by electrolysis of soln and weighing the Hg deposited on cathode

d)Place the residue insol in cyanide in a small distillation flask, add few ml w contg ca 1 ml AcOH, cool the mixt and collect the distillate in a Ag nitrate soln of ca 5% strength. Formation of ppt of AgN_3 indicates the presence of LA in the original sample. Remove AgN_3 by filtration, wash it with w, then alc and finally with ether. Dry in air and weigh

II)Analysis of Mixtures Containing Lead Azide, Antimony Sulfide, Lead Sulfocyanate and Potassium Chlorate. As an example of such mixts may be cited the T–4 Primer Composition used in M15A2 Detonator. Its ave analysis is: LA 5.0, Sb sulfide 17.0,

Pb sulfocyanate 25.0 & K chlorate 53.0%. It can be analyzed as described under Method 1 and Method 2

Method 1. US Military Spec MIL–D–2493 (1950) superseding the US Army Spec 50–78–7(1946), describes the following procedures

a)Moisture content. Dry to const wt at 55–65°, or in a desiccator contg Ca chloride, an accurately wt sample 0.40 to 0.70 g and calculate the loss in wt as the percentage of moisture (M)

b)Antimony sulfide content. Transfer ca 1g of air-dried sample, accurately weighed, to a beaker contg 100 ml of 1% nitric acid and allow to digest for 10 mins at RT, with occasional stirring. Filter through a Selas or Gooch crucible and wash the residue with distd w. Retain the filtrate and washings for the next procedure. Rinse the residue in crucible with alc & eth, dry at ca 70° for 1 hr, cool in a desiccator and weigh. Calc % Sb_2S_3 on moisture-free basis

$$\% \ Sb_2S_3 = \frac{\text{Wt of residue in crucible} \times 101}{W(1 - 0.01M)}$$

where W = wt of sample and M = % of moisture

c)Lead sulfocyanate content. Dilute the combined filtrate and aq washings of the previous proced to ca 300 ml with distd w. Add, while vigorously agitating, 15 ml of 10% Ag nitrate soln and continue agitation until the ppt coagulates. Allow to stand for ca 10 mins and filter through a Gooch crucible having a thick asbestos mat. Wash the residue, which consists of a mixt of Ag sulfocyanate and Ag azide, with distd w. Assemble the crucible to another suction flask. Add to the crucible, while applying a very light suction, 25 ml of 3% ceric ammonium sulfate soln and cover the crucible with a watch glass to retain spatterings due to gas evolution. Adjust the rate of suction so that the 25 ml of soln passes through the crucible in 5 mins. Repeat this treatment and wash the material accumulated on the watch glass into the crucible with a stream of distd w. Using a rubber policeman on a glass rod, tamp the ppt in the crucible into compact pad which will not crack when air is drawn through it. Continue treating the residue with ceric ammonium sulfate until no further evolution of gas is observed and then repeat the treatment three more times. Be sure to return to the crucible any asbestos which might pass into the suction flask. Wash the residue(Ag sulfocyanate) with distd w, followed by alc and eth. Aspirate until free of ether, dry at ca 135° for 1 hr, cool in a desiccator and weigh. Calc % $Pb(SCN)_2$ on a moisture-free basis

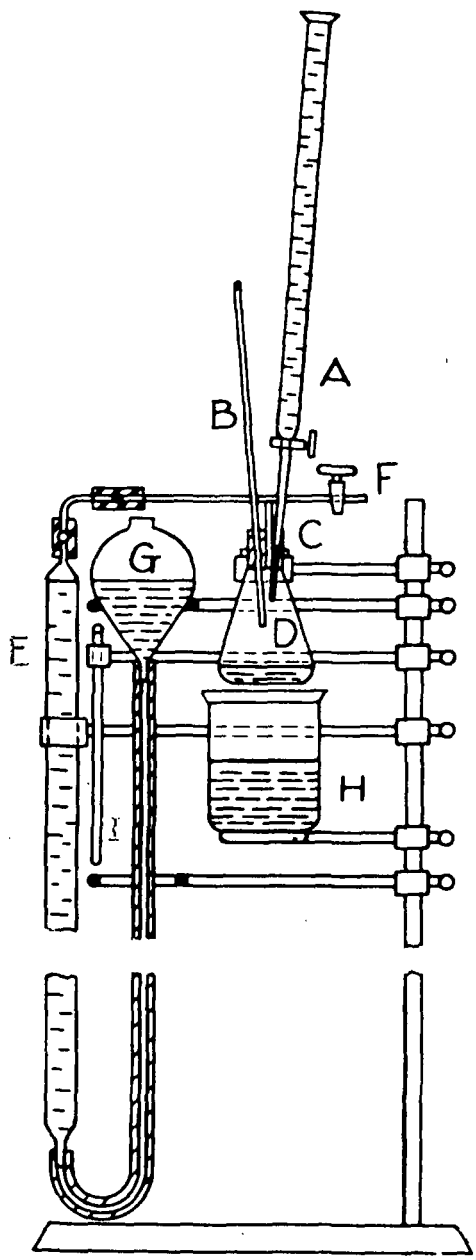
$$\% \ Pb(SCN)_2 = \frac{A \times 97.4 \times 0.985}{W(100 - 0.01M)}, \text{ where}$$

A = wt of residual sulfocyanate, M = % moisture and W = wt of sample

Note: This method is tedious, requiring up to 10 hrs, and it is not sufficiently accurate

d)Lead azide content. Assemble the apparatus shown in the fig, p A582, and protect it by screens from effects of air currents in order to maintain the temp around the app as constant as possible. B and I are thermometers

Procedure: Transfer ca 2 g, accurately weighed, to a 125 ml Erlen flask D, add 5 ml of 10% Ag nitrate soln and 10 ml distd w. Shake the flask and wash down its sides with a stream of distd w. Fill a 5 ml delivery burette A with 60% ceric ammonium nitrate soln and connect the flask D, by means of a tightly fitting rubber stopper C, to the gas measuring burette E (such as a 100 ml burette calibrated in 0.1 ml). Clamp this assembly in position, open stopcock F and, using the leveling bulb G, adjust the water level in the burette E to the zero point. Close the stopcock F and test the system for gas-tightness by raising and lowering the bulb G and releveling the water in G with that in E. Consider the system to be gas-tight if the w level in E returns to zero point



Add to the flask D, exactly 5 ml of ceric ammonium sulfate from the burette A, lower the bulb G to the vicinity of the 30 ml mark of the gas burette E. Shake the flask D(together with the clamp and stand) vigorously until no further increase in gas volume is observed. Add to the 400 ml beaker H ca 200 ml of w of the same temp as shown by the thermometer I, and raise the beaker until the flask D is covered with w to about 2" above the bottom. Clamp the beaker in position and allow the app to stand with occasional shaking until the temp as indicated by B is the same as that indicated by I. Adjust the height of G so that the level of w in it is the same as in the burette E. Record the observed level in E, the temp of the gas and the atm pressure. Calc % $Pb(N_3)_2$ on a moisture-free basis

$$\%Pb(N_3)_2 = \frac{(E-5.0)\times(P-V)\times 0.0570\times 1.08}{W(1-0.01M)\times(1+0.00367t)},$$

where E = ml of gas collected in burette E, P = atm press, mm Hg(see Note), V = vapor press of w at t°, M = % moisture in the sample, t = temp shown by thermometers B and I, W = wt of sample, grams

Note: If the atm press is observed on a mercurial barometer having a brass scale, calc the corrected atm press(P) as follows:

$P = P_1 - 0.000163P_1t_1$, where P_1 = reading in mm Hg of mercurial barometer and t_1 = temp of barometer

e)Potassium chlorate content is calcd on a moisture-free basis by subtracting from 100 the sum of the percentages of Sb_2S_3, $Pb(SCN)_2$ and $Pb(N_3)_2$

Method 2 for Analysis of Primary Mixtures Used in Detonators T–4, T–32, etc. It has been observed that the analysis of this primer by the methods of US Military Spec MIL–D–2493(1950) usually gives too high results for Pb sulfocyanate and Sb sulfide contents and too low results for K chlorate content. It has also been observed that the detn of Pb sulfocyanate by washing with ceric ammonium sulfate(as described in proced c of Method 1) is an extremely slow operation(usually requires ca 10 hrs) and different analysts do not check with each other. It is believed that one of the reasons for the inaccuracy of

results is due to the fact that no satisfactory test is provided for the detn of completeness of washing with ceric ammonium sulfate. A similar condition exists in the detn of Sb sulfide(proced b of Method 1), where no test is provided for the completion of washing of the sample with 1% nitric acid. The only detn of Method 1 which gives accurate results is the proced d for detn of the LA content

In view of the above mentioned difficulties in analysis by Method 1, Method 2 was developed at PicArsn by B.T.Fedoroff, M.L.Mauger & O.J.Hearn and described in ChemLabRept 130535(1951). The method was incorporated in the Purchase Descriptions PA–PD–202(1952) and PA–PD–124 (1953)

Method 2

a)Moisture content – same as proced a) in Method 1

b)Potassium chlorate content. Weigh accurately a (ca 1g) moisture-free sample directly in a tared 30 ml sintered glass crucible of medium porosity. Add from a burette, 3 ml of Solution No.1[prepd by shaking in a 1 l amber or blue glass bottle ca 800 ml dist w with 40 g KSCN, followed by 1 g $Pb(SCN)_2$, 1 g Sb_2S_3 and 1 g $Pb(N_3)_2$. After allowing to stand overnight, filter a portion of the soln reqd for analysis into another smaller bottle]. Swirl the crucible for exactly 1 min, taking care not to spill any liq; aspirate by suction and wipe the bottom and the side of crucible on the outside with tissue or filter paper. Continue the washing using one more 3.0 ml portions, two 2.0 ml and two 1.0 ml, making a total of 12.0 ml. Remove the crucible from the adapter and wash it on the outside with a stream of w. Empty the suction flask and wash it,as well as the adapter,with a stream of distd w. Place a clean test tube in the flask and insert the stem of the adapter. Insert the crucible and wash its contents with 1 ml of Soln No 1 as above; aspirate with suction catching the filtrate in the test tube. Remove the test tube, add 1 ml of distd w and test the soln for the presence of chlorate ion as follows:

Incline the tube at an angle of ca 45° and run down the side of the tube from a dropper, ca 0.5 ml of a soln contg 1.0 g DPhA in 100 g of concd sulfuric acid, so that there will be two distinct layers. If an appreciable amt of chlorate ion is present, a distinct blue ring will be visible at the border of the two layers. On shaking the tube, the ring disappears, but the contents assume a blue coloration, which lasts several seconds depending on the amt of chlorate ion present. If only a trace of chlorate is present the blue ring may not form, but upon shaking the test tube a slight bluish coloration will appear momentarily, lasting only a fraction of a second. If a definite blue ring forms in this test, repeat the washing of residue in the crucible using one 1 ml and one or two 0.5 ml portions of Soln No. 1 depending on the outcome of the test for the chlorate ion

Note: If a total of more than 14.0–14.5 ml of Soln No. 1 is required to remove the chlorate, it is advisable to repeat the whole proced b starting with a new sample and performing all operations exactly as described

After complete removal of the chlorate ion, wash the inside of the crucible with 1.0 ml of Solution No. 2[prepd by shaking vigorously in a 500 ml amber or blue glass bottle ca 400 ml distd w with 1 g of $Pb(SCN)_2$, 1 g Sb_2S_3 and 1 g $Pb(N_3)_2$, allowing to stand overnight and filtering a portion of liq required for analysis into another smaller bottle] and aspirate immediately. Wipe dry the bottom and the sides of crucible on the outside with tissue or filter paper and dry the crucible in an oven at 80 ± 1° for 30 mins; cool in a desiccator and weigh. Calc % $KClO_3$ on a moisture-free basis

$$\% KClO_3 = \frac{B \times 100}{W}$$, where B = loss in wt

of the crucible with sample and W = wt of moisture-free sample

c)Antimony sulfide content. Weigh accurately ca 1 g of air-dried sample directly in a tared 30 ml sintered glass crucible of medium porosity. Add 5 ml of 1% nitric acid and swirl the crucible constantly for 1 min taking care not to spill its contents in order to dissolve the bulk of the ingredients of the compn except Sb sulfide. Filter the mixt into a vacuum flask by applying suction, remove the crucible from adapter and wipe the bottom and sides dry on the outside with tissue or filter paper. Repeat the operation of washing seven more times making a total of 40 ml of 1% nitric acid used. Remove the crucible from the adapter and wash it on the outside with a stream of distd w. Empty the suction flask and wash it, as well as the adapter with a stream of distd w. Place in the flask ca 1 ml of a satd soln of ferric ammonium sulfate[prepd by shaking vigorously in a 500 ml bottle 125 ml of $(NH_4)_2Fe_2(SO_4)_4 \cdot 24H_2O$ with ca 120 ml distd w, allowing to stand overnight and filtering a portion into a smaller bottle], insert the adapter with crucible. Add 5 ml of 1% nitric acid and aspirate. If the liq in the flask turns red(due to the formation of ferric sulfocyanate), repeat washing of residue with one or two 5 ml portions of 1% nitric acid and test again for the presence of the sulfocyanate ion

Note: If more than 50 ml of 1% nitric acid is required to complete the removal of the sulfocyanate ion, repeat the entire proced c, starting with a new sample

Rinse the crucible and contents with factory alc and then with eth. Aspirate until the disappearance of the eth odor and dry the ensemble in an oven at 80 ± 1° for 30 mins. Cool in a desiccator and weigh. Calc % Sb_2S_3 on a moisture-free basis

$$\% Sb_2S_3 = \frac{C \times 100}{W(1 - 0.01M)}$$, where C = wt of residue(Sb_2S_3) in the crucible, M = % moisture in air-dried sample and W = wt of air-dried sample before washing it with 1% nitric acid

d)Lead azide content – same as proced d) in Method 1

e)Lead sulfocyanate content. Calculate % $Pb(SCN)_2$ content in the sample on a moisture-free basis by subtracting from 100 the sum of the percentages of $KClO_3$, Sb_3 and $Pb(N_3)_2$

III)Analysis of Mixtures Containing Lead Azide, Potassium Chlorate, Antimony Sulfide and Carborundum(or Glass). As examples of such mixts may be cited: *Primer Composition I:* $KClO_3$ 33.4, LA 28.3, Sb_2S_3 33.2 & carborundum 5.1%; and *Primer Composition II:* $KClO_3$ 15.4, LA 33.4, Sb_2S_3 21.1 & glass 30.1%.

Methods of analysis of such mixts were developed at PicArsn by T.D.Dudderar & E.F.Reese and described in Chem Lab Rept 42863(1935). These methods were incorporated in the US Army Spec 50–78–7 (1946) which was superseded by the US Military Spec MIL–D–2493(1950) and the Purchase Description PA–PD–124(1953)

Following are the procedures:

a)Moisture content – same as proced a in Item II

b)Potassium chlorate content. Weigh accurately in a small (ca 15 ml), previously ignited, cooled and tared Grooch or Selas crucible ca 1 g moisture-free sample. Add 3 ml of distd w, previously satd with LA, at temp 25 ± 2° and agitate for exactly 1 min, breaking up(very cautiously) any lumps with a rubber policeman attached to a glass rod. Apply suction and repeat the operation 5 times, making a total of six 3 ml extractions. Rinse the sample in the crucible with a few ml of alc and then with eth; dry for ca 15 mins at 95°, cool in a desiccator and weigh. Save the residue for the next proced

$$\% KClO_3 = \frac{\text{Loss in wt} \times 100}{W}$$, where W = wt of sample

Note: For a mixt contg glass instead of carborundum, four 3 ml extractions are sufficient

c)Lead azide content. Extract the residue of proced b with 5 ml portions of satd amm acetate soln at ca 25°(hot soln must not be used as it dissolves Sb sulfide to some extent), agitating each portion for ca 30 secs. Continue the washings until they no longer give a yel ppt with a few drops of K bichromate soln. The use of more than 60 ml of amm acetate soln should be avoided. Wash the residue with distd w, followed by a few ml of alc and then eth. Dry for ca 15 mins at 100 ± 10°, cool in a desiccator and weigh. Save the residue for the next proced. Calcd on a moisture-free basis

$$\% LA = \frac{\text{Loss in wt} \times 100}{W}$$, when W = wt of sample used in proced b)

d)Antimony sulfide content. Treat the residue from proced c directly in the filtering crucible with cold concd HCl until nearly all the sulfide is removed. This can be approx judged by the disappearance of hydrogen sulfide odor. In order to remove the last traces of sulfide, rinse the residue with hot concd HCl. Finally wash it with w, alc and eth and ignite to remove separated sulfur and organic matter; cool in a desiccator and weigh

$$\% Sb_2S_3 = \frac{\text{Loss in wt} \times 100}{W}$$, where W = wt of sample used in proced b)

e)Carborundum or glass content. Subtract the tare of ignited empty crucible(see proced b) from the wt of crucible with residue after the Sb sulfide detn and calculate the difference as the percentage of carborundum in the sample

IV)Analysis of Mixtures Containing Lead Azide, Potassium Chlorate, Antimony Sulfide, Glass and Shellac. As an example of such mixts may be cited the primer composition contg: $KClO_3$ 14.0, LA 33.0, Sb_2S_3 21.0, glass 30.0 & shellac 2.0%. Its method of analysis was developed at PicArsn by T.D.Dudderar and described in ChemLab Rept 49334(1937). It does not seem to be incorporated in any specs

Following are the procedures:

a)Moisture content – same as proced a under Item II

b)Shellac content. Weigh accurately ca 2g moisture-free sample (dried at 55° for 30 mins) in a tared 50 ml beaker and add 5 ml of absolute alcohol(previously satd at RT with $KClO_3$, which is appreciably sol in alc). Warm cautiously on a steam bath over a thin sheet of asbestos for 15 mins, breaking up any lumps by very cautious use of a rubber policeman attached to a glass rod. Cool to RT, let settle and decant the alc shellac soln through a small tared, previously ignited and cooled, 30 ml Gooch or Selas crucible. Repeat the extraction with new portions of abs alc until the shellac is completely removed(about 3 times). Transfer the residue in the beaker by means of a rubber policeman to the above crucible, rinse the beaker with several portions of chlf into the crucible and after aspirating, dry the crucible with the residue at 55° for 15 mins, cool in a desiccator and weigh. Save the residue for use in the next proced. Calc % shellac on a moisture-free basis

$$\% \text{ Shellac} = \frac{\text{Loss of wt in the crucible} \times 100}{W}$$,

where W = wt of sample

c)Potassium chlorate content. Extract the residue of previous proced with distd w satd with LA, etc as described in proced b under Item III

d)Lead azide content. Extract the residue of previous proced with amm acetate soln, etc, as described in proced c)under Item III

e)Antimony sulfide content. Extract the residue of previous proced with HCl, etc, as described in proced d)under Item III

f)Glass content – same as proced e)under Item III

V)Analysis of Mixtures Containing Lead Azide, Barium Nitrate, Basic Lead Styphnate and Antimony Sulfide. As an example of such mixts may be cited the *NOL No 130 Primer Mixture* used in T–32E1 and M47 Detonators: LA(dextrinated) 20.0, $Ba(NO_3)_2$ 20.0, LSt(basic) 40.0, tetracene 5.0, & Sb_2S_3 15.0%. Its max moisture content is 0.3%. The method of analysis of such mixts was developed at PicArsn by J.Campisi, ChemLabRept 52–H1–2114(1952) and was incorporated in the Purchase Descriptions PA–PD–202(Rev 1)(1952) and PA–PD–124 (1953)

Following are the procedures:-

a)Moisture content. Place ca 0.5 g of air-dried sample into a tared weighing bottle with outside ground cap and reweigh the ensemble accurately. Remove the cap and heat the bottle in an oven maintained at 60 ± 5° for 2 hrs, cool in a desiccator and reweigh

$$\% \text{ Moisture} = \frac{\text{Loss in wt} \times 100}{\text{Wt of sample}} = M$$

b)Barium nitrate content. Place ca 0.5 g moisture-free sample in a tared 20 ml, medium porosity, sintered glass crucible and reweigh the ensemble accurately. Add 3 ml of LA-satd distd w at temp 5 ± 2°, agitate by swirling for exactly 1 min and, if necessary, break up gently any lumps with a rubber policeman attached to a glass rod. Remove the liq by suction and repeat these procedures 5 times, making a total of six 3 ml extractions. Rinse the sample in the crucible 3 times with factory alcohol (90–95% by vol), dry in an oven at 60 ± 5° for 30 mins, cool in a desiccator and weigh. Save the residue for the next proced. Calc % $Ba(NO_3)_2$

$$\% \text{ } Ba(NO_3)_2 = \frac{\text{Loss in wt} \times 100}{W}, \text{ where}$$

W = wt of sample

c)Basic lead styphnate content. Extract the residue of previous proced with six 5 ml portions of satd amm acetate soln at temp not higher than 25°. Agitate by swirling each portion for ca 30 secs, allowing the liq to remain in the crucible for ca 2 mins and then remove it by suction. Finally wash the residue with w until the filtrate is colorless. This treatment dissolves not only basic LSb but also LA. Transfer quantitatively the extract and washings to a 250 ml volumetric flask. Dilute to the mark with distd w and mix thoroughly. Pipette accurately 2 ml of this soln to a 50 ml volum flask and dilute to mark with distd w. Fill a "Corex" glass spectrophotometric cell, having a width of ca 1 cm with this soln, and det the optical density of combined styphnate and acetate ions at a wavelength of 410 millimicrons (azide ions do not interfere at this wavelength) by using a "Quartz Ultraviolet Spectrophotometer" as described in the JourAmerOpticalSoc **31**, 683 (1941) or a "Beckman Spectrophotometer Model DU". Fill a 2nd cell(which is identical in optical characteristics as the 1st cell) with straight satd amm acetate soln and det its optical density. Save the crucible with residue for detn of tetracene. Calc % basic LSt on a moisture-free basis

$$\% \text{ Basic LSt} = \frac{28(A - B)}{W \times D}, \text{ where } A = \text{optical}$$

density of basic LSt soln, B = optical density of amm acetate soln, W = wt of sample of proced b), and D = width of "Corex" cell in cm

Note: If the cells are not identical, it is necessary to correct the optical density for the difference in the amt of light which the two cells

scatter and absorb. To do this, fill both cells with amm acetate soln and measure the optical density of the cell, which originally contained the basic LSt soln, at a wavelength of 410 millimicrons.

d)Tetracene content. Wash the wet residue of previous proced 3 times with factory alc(90 –95% by vol), remove alc by suction, dry the crucible in an oven at 60 ± 5° for 30 mins, cool in a desiccator and weigh(C). Transfer the dried residue in the crucible to a 125 ml beaker, with a stream of distd w. Add 25 ml distd w and boil the slurry on a hot plate for 5 mins. Filter through the above crucible, wash the residue 3 times with boiling w and then with factory alc. This treatment removes the tetracene. Dry the crucible & the residue in an oven at 60 ± 5° for 30 mins, cool in a desiccator and weigh(D). Calc % Tetracene on a moisture-free basis

$$\% \text{ Tetracene} = \frac{(C - D) \times 100}{W}$$, where C = wt of crucible with residue after completion of proced c, D = same after boiling with w to remove tetracene, and W = wt of sample of proced b

e)Antimony sulfide content. The residue in crucible after removing all other ingredients of sample is Sb_2S_3. Calcd on a moisture-free basis

$$\% Sb_2S_3 = \frac{(D - E) \times 100}{W}$$, where D = wt of crucible with sample(see proced d), E = tare of crucible, W = wt of sample of proced b)

f)Dextrinated lead azide content. Calculate by subtracting from 100 the combined percentages of Ba nitrate, basic LSt, tetracene and Sb sulfide

Note: LA was removed from mixt together with basic LSt on treatment with satd amm acetate(see procedure d)

VI)Analysis of Mixtures Containing Lead Azide and Aluminum. As an example of such mixts may be cited the *M41 Primer Mixture:* LA 90 & Al 10%. Its method of analysis was developed at PicArsn by F.Pristera & L.May, ChemLabRept 113523(1945)

Following are the procedures:

a)Moisture content – same as in proced a in Item II

b)Aluminum content. Transfer an accurately weighed ca 1 g moisture-free sample to a 150 ml beaker and add with stirring 2 ml of 50% alc, followed by 50 ml distd w, 3 ml glac AcOH and 5 ml of 25% Na nitrite soln. Cover the beaker with a watch glass and lift it as soon as the evolution of gas subsides. Stir the mixt and allow it to stand for ca 3 mins with occasional stirring. Total reaction of destruction of LA shall not last more than 5 mins. Decant the supernatant liquid immediately and transfer Al residue quantitatively to a tared sintered glass crucible of fine porosity, in order to be able to retain the superfine Al. Wash the Al in the crucible with cold w, followed by acetone, dry at 100° for 30 mins, cool in a desiccator and weigh. Calc % Al on a moisture-free basis

$$\% \text{ Al} = \frac{(A - B) \times 100}{W}$$, where A = wt of crucible with Al, B = wt of empty crucible, and W = wt of sample

c)Lead azide content. Subtract from 100 the percentages of Al and of moisture

Refs for Analytical Procedures are listed before each method of analysis

Lithium Azide (formerly called *Lithium Azoimide or Lithium Trinitride),* LiN_3, mw 48.96, N85.83%; anisotropic, col crysts, mp-expl 115° to 298° (Ref 1); sol in w (36.1% at 10° and 66.4% at 16°), sol in alc (20.3% at 16°) and insol in eth (Ref 1); Q_f^v −2.58 kcal/mol at 298°K, lattice energy 194 kcal/mol at 298°K (Ref 21). Prepd in 1898 by Curtius & Rissom (Ref 1) by the action of a soln of lithium sulfate on barium azide and evapn of the clear liq. In the same year, Dennis & Benedict (Ref 2) prepd lithium azide by dissolving lithium hydroxide in hydrazoic acid and allowing the soln to evap in air. They obtd the hydrated salt with 1 mol of water of crystn, $LiN_3 \cdot H_2O$ (Ref 3). Hoth & Pyl (Ref 8) made lithium azide by interaction of sodium azide and lithium chloride in aq alc soln. Frankenburger & Zimmermann (Ref 9) produced LiN_3 by passing O_2-free N_2 over Li heated to 500-600°. Nitrogen, contg very small amts of O_2 produced a glow in the gas in immed contact with the solid azide. More recently Hofman-Bang (Ref 22) prepd 99.5% pure Li azide by dissolving NaN_3 and $LiSO_4 \cdot H_2O$ in water and adding 96% alc. The filtrate from this mixt was evapd to near dryness on a w bath and finally at 80° in an oven. According to Browne & Houlehan (Ref 4), anhyd Li azide is best prepd by reacting metallic Li with a liq NH_3 soln of NH_4N_3

Dennis & Benedict (Ref 2) claimed that Li azide when crystd from aq soln always contd 1 mol of w of crystn while Curtius & Rissom (Ref 1) stated that Li azide and Ba azide were both obtd without water of crystn. Dennis & Browne (Ref 3) prepd both azides contg 1 mol of w, but by long continued drying over concd H_2SO_4 they dehyd these salts completely to the anhyd azides. In a study of the system LiN_3-water, Rollet & Wohlgemuth (Ref 11) found that LiN_3 was deposited above 68.2°, $LiN_3 \cdot H_2O$ from 68.2 to −31° and $LiN_3 \cdot 4H_2O$ from −31° to the eutectic point of −47.5°. They observed a considerable tendency to supersatn in spite of inoculation of the system

Explosive Properties. Li azide, although detond with difficulty, propagates at a velocity of 990 m/sec (Ref 15). Wohler & Martin (Ref 5) obtd an expln temp of 245° for 0.02 g of the subst which detond violently after 5 sec, but this compd could not be detond by impact. The photochemical decompn of Na, K & Li azides in solns irradiated by UV light of 2537Å wave length was studied by Bonnemay (Refs 13). For low concns the reactn was homogenous and decompn proceeded at a vel proportional to the concn, but independent of the cation. At high concns the vel of decompn was not explained by a simple law (for example Beer's Law) but showed, after an induction period, that reaction proceeded by chains which formed at the start of photolysis. Crystalline Li azide can be initiated to expln by intense electron streams but not by slow neutron bombardment (Ref 16)

Other Properties. The mol refractions of Li, Na and K azides were detd in solns of varying concns by Petrikalns & Ogrins (Ref 12). They also detd the density and refractive index for crystn Na and K azides. The ionic conductance of solid Li azide, as detd by Jacobs & Tomkins (Ref 18), obeyed the general equation: $\log k = \log A - (E/2.303RT)$ where k is the specific conductivity in $ohm^{-1}\ cm^{-1}$; A is a constant and E is activation energy in kcal/mol. For Li azide log A = 0.840, E is 19.1 and T, the temp range 300-370°K. The Raman Effect of crystn Li azide was detd by Kahovec & Kohlrausch (Ref 14); the observed frequency, 1368.7 cm^{-1}, corresponded to the oscillation in a linear triatomic molecule.

The chem reaction between LiN_3 and benzene diazonium chloride has been described by Huisgen (Ref 20). The formation of an expl *Lithium Boroazide,* $LiB(N_3)_4$, a wh solid sol in ether, easily hydrolyzed and very sensitive to impact and pressure was reported by Wiberg & Michaud (Ref 19). This compd was obtd on evapg to dryness a mixt of ether solns of excess HN_3 and $LiBH_4$; Li azide and B azide were assumed as intermediate products in the stepwise reaction

The reaction between Ba and Li azides

and N_2 at 400–500° and 280–320 atms was studied by Ariya & Prokof'eva (Ref 17) but no expl props were det'd. Their enthalpies of formation were recalcd as 0.1 and 3.1 kcal/mol for the respective azides. (See also refs 6,7,10 & 23)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 277(1898) & JCS **76** II, 92(1899) 2)L.M. Dennis & C.H.Benedict, JACS **20**, 226(1898); ZAnorgChem **17**, 18(1898) & JCS **74** II, 426 (1898) 3)L.M.Dennis & A.W.Browne JACS **26**, 601(1904); JCS **86** II, 558(1904) & ZAnorg-Chem **40**, 97(1904) 4)A.W.Browne & A.E. Houlehan, JACS **33**, 1747(1911) 5)L.Wöhler & F.Martin, ZAngChem **30**, 33(1917); JSCI **36**, 570(1917) & CA **11**, 3432(1917) 6)Gmelin, System No 20(1927), 86 7)Mellor **8** (1928), 345 8)W.Hoth & G.Pyl, ZAngChem **42**, 888 (1929) & CA **23**, 5547(1929) 9)W.Franken-burger & W.Zimmermann, ZPhysChem **10**, Abt B, 238(1930) & CA **25**, 642(1931) 10)L.F. Audrieth, ChemRevs **15**; 202(1934) 11)A.P. Rollet & J.Wohlgemuth, CR **198**, 1772(1934) & CA **28**, 3999(1934) 12)A.Petrikalns & B.Ogrins, Radiologica **3**, 201(1938); ChemZtr **1939** II, 327 & CA **35**, 3145-6(1941) 13)M. Bonnemay, CR **215**, 65(1942) & CA **38**, 5457 (1944); JChemPhys **41**, 18(1944) & CA **39**, 3205(1945) and JChemPhys **41**, 113(1944) & CA **40**, 2384(1946) 14)L.Kahovec & K.W. Kohlrausch, Monatsch **77**, 180(1947) & CA **42**, 6666(1948) 14a)Thorpe **7** (1948), 368 15)F.P.Bowden & H.T.Williams, ProcRoy Soc **208A**, 185(1951) & CA **46**, 5884-5(1952) 16)F.P.Bowden & K.Singh, Nature **172**, 378 (1953) & CA **48**, 1003(1954); ProcRoySoc **227A**, 28(1955) & CA **49**, 4991(1955) 17)S. M.Ariya & E.A.Prokof'eva, SbornikStatei-ObschKhim **1**, 9(1953) & CA **48**, 12522(1954) 18)P.W.Jacobs & F.C.Tompkins, JChem Phys **23**, 1445(1953) & CA **49**, 15336(1955) 19)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 499(1954) & CA **49**, 767(1955) 20)R.Huisgen, Chimica(Switz) **10**, 266(1956) & CA **51**, 16326 (1957) 21)P.Gray & T.C.Waddington, Proc RoySoc **235A**, 106 & 488(1956) & CA **50**, 12627 & 15203(1956) 22)N.Hofman-Bang, ActaChem(Scand) **11**, 581(1957) & CA **52**, 6996(1958) 23)H.Rosenwasser, USArmy EngrRes & DevelopLabsRpt 1551-TR, 10(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Magnesium Diazide (formerly called *Magnesium Azoimide or Magnesium Trinitride,* $Mg(N_3)_2$, mw 108.37, N 77.56%; wh ppt sol in w, insol in eth or tetrahydrofuran. The prepn of Mg diazide was attempted in 1898 by dissolving the metal in dil HN_3, but the product decompd on evapg the soln and was not isolated (Ref 1). Turrentine (Ref 2) studied the electrochem corrosion of Mg in Na azide soln and obtd a wh flocculent ppt, probably basic magnesium azide, $Mg(OH)N_3$, but he did not identify the product. Browne & Houlehan (Ref 3) reported that metallic Mg reacted vigorously with a liq NH_3 soln of Amm azide to form Mg azide; however, this compd probably united with NH_3 to form an ammonate. Wiberg & Michaud (Ref 6) obtd $Mg(N_3)_2$ in almost quant yield from an eth soln of excess HN_3 and a frozen ether-dioxane soln of Et_2Mg. The reaction began below 0° and ended in about 30 min at RT. Distn removed the excess solvent and HN_3. Mg azide was found to deton only slightly in flame and to be sensitive to moisture. According to Wiberg and Michaud (Ref 6) it can be isolated from w only as $Mg(OH)N_3$ and it can not be volatilized in high vac at RT

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 291(1898) & JCS **76** II, 91(1899) 2)J.W. Turrentine, JACS **33**, 811(1911) 3)A.W.Browne & A.E.Houlehan, JACS **33**, 1750(1911) 4) Mellor **8** (1928), 350 5)Gmelin, System No 27, Teil B, Lieferung 1 (1939),73 6)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 501(1954) & CA **49**, 768(1955) 7)H.Rosenwasser, USArmy EngrRes & DevelopLabsRpt 1551-TR, 48(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Manganese Diazide (formerly called *Manganese Azoimide or Manganese Trinitride),* $Mn(N_3)_2$, mw 138.99, N 60.47%; wh hygro powd easily hydrolyzed, mp-expl at 203 in 5 sec, (Ref 5),

Q_e^v 676 cal/g (Ref 4), Q_f^p −92.2 kcal/mol (Ref 4). By dissolving Mn in dil HN_3, Curtius & Rissom (Ref 1) obtd a basic manganese azide, $Mn(OH)N_3$, but the soln decompd on evapn. On continuing this work, Curtius & Darapsky (Ref 2) found that an aq soln of Mn alum and Na azide on pptn with alc and eth gave the basic Mn azide previously obtd. Wöhler (Ref 3) studied the reaction of Mn carbonate on HN_3 in acet and obtd $Mn(N_3)_2$ which was not as easily detond as Co azide but exploded more violently than Zn azide. It was prepd by shaking together finely divided dry basic Mn azide with HN_3 in acetone until the solid became entirely sol in w (Ref 4). The expl temp for a 0.02 g sample to det in 5 sec was 203° and a compressed sample detond under impact of a 2 kg falling wt (Ref 5). Franklin (Ref 6) reported that Mn azide was prepd by reactg the metal with aq hydrazoic acid, HN_3

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 261(1898) & JCS **76** II, 90(1899) 2)T. Curtius & A.Darapsky, JPraktChem **61**, 408 (1900) & JCS **78** II, 474(1900) 3)L.Wöhler, ZAngChem **27** I, 335(1914) & CA **9**, 1115(1915) 4)L.Wöhler & F.Martin, Ber **50**, 594(1917); JCS **112** I, 383(1917) & CA **11**, 2901(1917) 5)L.Wöhler & F.Martin, ZAngChem **30** I, 33 (1917); JSCI **36**, 570(1917) & CA **11**, 3432 (1917) 6)E.C.Franklin, JACS **56**, 569(1934) & CA **28**, 2289(1934)

Mercuric Azide (formerly called *Mercuric Trinitride* (called Quecksilberazide in Ger), $Hg(N_3)_2$, mw 284.66, N 29.52%; clear to lemon yel crysts, existing as ortho prisms (stable α-form) or aggregates terminating in prisms or ndls (abnormally sensitive, unstable β-form) (Refs 8 & 12); mp – begins to dec with gas evoln ca 212°, bp ca 215°, expl at 220° (Ref 3) to 300° (Ref 4); sl sol in cold w (0.26 g in 100 g sol at 20°), sol in hot w; its toxicity is not known (see Mercurous Azide)

It was first prepd in 1894 by Berthelot & Vieille (Ref 1), by Wöhler (Refs 2 & 3) and later by Stettbacher (Ref 6) all essentially from a soln of NaN_3 decompd by concd H_2SO_4 and/or passing the HN_3 formed into mercuric oxide in boiling w. The HgO was quantitatively converted to $Hg(N_3)_2$ which crystd from the slowly cooled soln. An alternative method of prepn consisted in mixing a concd soln of NaN_3 and mercuric nitrate; $Hg(N_3)_2$ pptd as wh powdery mass was less sensitive than LA, but according to Stettbacher (Ref 6) it could be converted into the highly sensitive β-form by soln and crystn. Alpha-$Hg(N_3)_2$ was prepd by Miles (Ref 8) by mixing a satd soln of mercuric chloride with an equiv soln of NaN_3 made sl acid with hydrazoic acid. According to Miles, a mixt of α and β crysts was always obtd when the α azide was recrystd from w or acetone. Stettbacher (Ref 6) considers the prepn of mercuric azide as one of the most dangerous and treacherous of chem operations as this subst presents one of the few examples of "Crystal Tension" (qv) (Ref 7)

According to Stettbacher, mercuric azide develops the same vol of gas on deton as MF but it is 20 times more brisant. Wöhler & Krupko (Ref 3) observed that its sensitivity depended on the cryst size of the azide. Mercuric azide is considered to be more sensitive to impact and friction than MF and is so unstable that it frequently undergoes spontaneous deton at the slightest touch even under w (Refs 5 & 7). Hitch (Ref 4) noted this sensitivity expecially when the azide was prepd from mercuric nitrate and Na or K azide solns but by careful thermal studies he decompd it quanty into its elements without expln below 300°. Miles (Ref 8) reported that in every case when β-crysts of $Hg(N_3)_2$ were present the material was likely to expl, and in w or more rapidly in mercuric nitrate soln, the β-type was unstable being transformed to the α-type, as in the parallel case of LA.

Klatt (Ref 10) studied the bp rise of solns of $Hg(N_3)_2$ in HF (hydrogen fluoride) and found indications that 3 ions per mol were formed: $HgN_6H_2^{++}$ and $2F^-$

The ignition of $Hg(N_3)_2$ by exposure to the intense light produced from a photographic

"electron" flash bulb at 6 cm dist was detd by Eggert (Ref 11) as requiring 300 w-sec elec energy. Wöhler (Ref 2) observed that $Hg(N_3)_2$ remained unchanged in the dark under water but on exposure to sunlight or heat the yel color changed successively to orange, brn, black and finally grey, yielding the metal

A compd called in Ger *Ammon-basisches Mercuriazide*, Hg_2NN_3, was obtd by Strecker & Schwinn (Ref 9) as yel crysts, insol in w or alc, which detond violently on heating or on impact. It was prepd by several methods, one of which was the dropwise addn of concd NH_3 to a soln of mercuric azide in hot w, as long as any yel ppt formed, followed by filtration and drying. The second crop of crysts were obtd by heating the mother liquor on a w bath and filtering the ppt

Refs: 1)M.Berthelot & P.Vieille, AnnChim Phys [7] **2**, 339(1894) & BullFr[3] **11**, 747 (1894) 2)L.Wöhler, ChemZtg **35**, 1096(1911) & CA **6**, 2895(1912) 3)L.Wöhler & W.Krupko, Ber **46**, 2056-7 (1913) & JCS **104** II, 703(1913) 4)A.R.Hitch, JACS **40**, 1202 (1918) & CA **12**, 1951(1918) 5)B.Oddo, AnnalChimAppl **11**, 165-98(1919) (A monograph on prepn, props & applications) & CA **13**, 3011(1919) 6)A. Stettbacher, SchweizChemZtg **27**, 273-4(1920); CA **14**, 3531(1920) & SS **15**, 211-2(1920) 7) Mellor **8** (1928), 351 8)F.D.Miles, JCS **1931**, 2536 & CA **26**, 848(1932) 9)W.Strecker & E.Schwinn, JPraktChem **152**, 214(1939) & CA **33**, 5314(1939) 10)W.Klatt, ZPhysChem **185A**, 306(1939) & CA **34**, 1899(1940) 11)J. Eggert, Naturwissenschaften **40**, 55(1953) & CA **47**, 11735(1953) 12)H.Rosenwasser, US ArmyEngrRes & DevelopLabsRpt 1507-RR, 18 (1957)

Mercurous Azide (formerly called *Mercurous Trinitride)* (called Stickstoffquecksilberoxydul or Stickstoffcalomel in Ger), HgN_3, mw 242.63, N 17.32%; wh anisotropic ndls; mp – started to dec with evoln of gas at 215°, expl at 270° (Ref 8); Q_{expln} 266 cal/g or 64.4 kcal/mol (Ref 7); Q_f 70.2(Ref 21) to 77.3 kcal/mol (Ref 19); v sl sol in w (0.025 g) in 100 g (Ref 6). According to Sax (Ref 22), mercurous azide is highly toxic. When heated it emits fumes of Hg and may expl on exposure to light or heat

In 1890-1 Curtius (Refs 1 & 2) prepd HgN_3 by reactg solns of HN_3 or Na azide with $HgNO_3$ to ppt the azide (Ref 2a). Berthelot & Vieille, prepd it by adding a dil aq soln of NH_4N_3 to $HgNO_3$ followed by washing the product (Ref 3) and later also Wöhler & Krupko (Ref 6) who detd some of its expl characteristics and its decompn by light

According to Curtius (Ref 2), HgN_3 is more stable than either Ag or Pb azide but it does become yel on exposure to light and yields a blk compd with aq NH_3. Wöhler (Ref 5) observed that HgN_3 turned yel because of the formation of colloidal Hg; the yel color passed to orange, brn, blck and finally to grey when exposed to light. In darkness, Wöhler & Krupko kept the salt under water for several months without change; the dry salt, in vacuo and darkness, did not change in 24 hrs at 120-140°(Ref 6). Wöhler & Martin (Ref 7) reported an expl temp of 281° in 5 sec for a 0.02 g sample and deton of a compressed sample under impact. Taylor & Rinkenbach (Ref 11) obtd the following values for sensitivity to impact and friction:

SENSITIVITIES OF DETONATING COMPOUNDS

	HgN_3	$Pb(N_3)_2$	AgN_3
Impact Test, US BM App:			
500 kg wt, 0.02 g sample, cm	6	43	41
Pendulum Friction Test: (10% point for min wt and height to cause expln)			
Added wt, kg	1	0.45	4.35
Fall, cm	50	37.5	33
Swings, No	16-17	12	39

(See also Refs 13,14,16 & 18 for addl info on prep & props)

Noddack & Grosch (Ref 20) calcd the expln temp, measured the gas press produced and obtd the energy output from HgN_3 in primers set off in a closed bomb. For a 1 g compressed charge, they obtd an expln press value of

10,900 kg/sq cm (Ref 20)

Infrared absorption spectra of HgN_3 in the range 3 to 19 microns were obtd by Delay et al (Ref 17). The formation of a complex salt involving Hg^+ azide, $[Hg(C_2H_5N)](N_3)_2$, is described by Strecker & Schwinn (Ref 15)

Uses. The great sensitivity of certain metallic azides to heat, impact and friction suggested their possible use as detonants. As early as 1893 (Refs 5 & 14), the Prussion Govnt investigated mercurous and other azides for their possible application in detonators and Wöhler & Martin (Ref 7) detd the min amt of the various azides necessary to initiate deton in different HE's, as follows:

INITIATION EFFICIENCY OF AZIDES

	Min Amt of Initiator Reqd to Detonate HE				
Initiator, g	*Tetryl*	*PA*	*TNT*	*TNA*	*TNX*
Cd azide	0.01	0.02	0.04	0.1	–
Ag azide	0.02	0.035	0.07	0.26	0.25
Pb azide	0.025	0.025	0.09	0.28	–
Cu^+ azide	0.025	0.045	0.095	0.375	0.40
Hg^+ azide	0.045	0.075	0.145	0.55	0.50
Tl azide	0.07	0.115	0.335	–	–

Mercurous azide, although ranking 5th in the above comparative efficiency rating, was suggested by Wöhler & Martin (Ref 7) and proposed by others (Refs 4,9,10 & 12) as a constituent of priming mixts for use in detonators. Grotta (Refs 9 & 10) patented a mixt of HgN_3/MF/$KClO_3$–20/60/20%. He claimed that it had great brisance, was not readily "dead pressed" and, unlike other mixts contg MF, it was not hygroscopic nor rendered ineffective by moisture. It was claimed further that this mixt does not attack copper to form the dangerous Cu azide, thus providing an advantage over Pb azide which does react with copper [Also see patents by Blechta (Ref 12)]

Refs: 1)T.Curtius, Ber **23**, 3032(1890) & JCS **60** I, 56(1891) 2)T.Curtius, Ber **24**, 3345(1891) & JCS **62** I, 112(1892) 2a)T. Curtius, & J.Rissom, JPraktChem **58** II, 261(1898) & JCS **76** II, 91(1899) 3)M. Berthelot & P.Vieille, AnnChemPhys [7] **2**, 339(1894) 4)L.Wöhler, GerP 196824(1907) & CA **2**, 2302(1908) 5)L.Wöhler, ZAngChem **24**, 1111 & 2089(1911); SS **6**, 253(1911) & CA **5**, 3730(1911) 6)L.Wöhler & W.Krupko, Ber **46**, 2050(1913) & JCS **104** II, 702(1913) 7) L.Wöhler & F.Martin, a)Ber **50**, 595(1917); JCS **112** I, 383(1917) & CA **11**, 2900(1917); b)ZAngChem **30** I, 33(1917); JCS **112** II, 466 (1917); JCSI **36**, 570(1917) & CA **11**, 3432 (1917) and c)SS **12**, 1,18,39,54 & 74(1917) & CA **12**, 629(1918) 8)A.R.Hitch, JACS **40**, 1201(1918) & CA **12**, 1951(1918) 9)B.Grotta, a)USP 1439099(1922) & CA **17**, 883(1923); b)USP 1453976(1923); JSCI **42**, 804A(1923) & CA **17**, 2506(1923); c)CanP 246338(1925); ChemZtr **1926** I, 553 & CA **19**, 1349(1925) 10) B.Grotta, IEC **17**, 134(1925) 11)C.A.Taylor & W.H.Rinkenbach, JFrankInst **204**, 374(1927) 12)F.Blechta, AustrP 126,150(1931) & CA **26**, 2320(1932); FrP 704,994(1931) & Chem-Ztr **1932** I, 1325 13)Mellor **8** (1928),351 14) L.F.Audrieth,ChemRevs **15**, 204-14(1934) 15)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314(1939) 16)Davis (1943), 183,411,412 & 420 17)A.Delay et al, CR **219**, 329(1944) & CA **40**, 4273-4(1946); BullFr **12**, 581(1945) & CA **40**, 2386(1946) 18)Kirk & Othmer **6** (1951), 19 & **7** (1951), 594 19)S.Suzuki, JChemSocJapan, PureChemSect **73**, 278(1952) & CA **40**, 6907(1952) 20)W. Noddack & E. Grosch, ZElectrochem **57**, 632 (1953) & CA **49**, 8602(1955); Explosivst **4**, 69(1956) & CA **51**, 9162(1957) 21)P.Gray & T.C.Waddington,"ComptesRendues, 27e CongrInternChimInd,Brussells 1954,"3 IndustrieChimBelge **20**, Spec No, 327-30(1955) & CA **50**, 16328(1956); ProcRoySoc **235A**, 106(1956) & CA **50**, 12627(1956) 22)Sax (1957), 865

Nickel Diazide (formerly called *Nickel Trinitride or Nickel Azoimide),* $Ni(N_3)_2$, mw 142.76, N 58.88%; sandy, hygr grn pdr, mp–expl ca 200°, Q^P_e 656 cal/g(Ref 4), Q^P_f –91.9 kcal/mol (Ref 4); very sol in w which it holds tenaciously (ca 13%) but soon undergoes

hydrolysis (Ref 4). In 1898 Curtius & Rissom (Ref 1) obtd *Basic Nickel Azide,* $Ni(OH)N_3$, with some $Ni(N_3)_2$ (?) by reacting nickel carbonate with aq HN_3. It was a grn cryst compd exploding at 247-71°. Curtius & Darapsky (Ref 2) continuing this work found that solns of Ni alum and Na azide, pptd with alc and eth, produced the neutral $Ni(N_3)_2$ + H_2O. Wöhler & Martin (Ref 4) obtd Ni azide by shaking together finely divided Ni carbonate or the basic Ni azide with an ethereal soln of HN_3 until the solid became partly sol in w. Franklin reported (Ref 10) that Ni azide was formed by reacting the metal with aq hydrazoic acid, HN_3. Ni azide is a very sensitive expl, detong violently even at the slightest touch (Ref 5)

Dennis & Isham (Ref 3) prepd the addn compds, $Ni(N_3)_2(C_5H_5N)_4$ and $Ni(C_6H_5N)_6$, as grn ppts by treating Ni azide with pyridine. Both compds were unstable in air and non expl. Browne et al (Ref 6) obtd an *Ammonobasic Nickel Azide* by electrolyzing solns of NH_4 azide in liq NH_3, using Ni electrodes. The pink deposit which formed on the electrodes exploded on heating. It turned grn on treatment with w, gradually dissolved and settled out as an expl ppt. Ricca & Pirrone (Ref 8) prepd an addn compd from 1 vol of 15% $NiSO_4$ and 3 vols of a 5% aq soln of a compd obtd by mixing equal vols of satd aq $Hg(CN)_2$ and N NaN_3. The grn compd, $Hg(CN_2)_2 \cdot Ni(N_3)_2$, did not explode when heated. A lt blue solid, sol in w and exploding violently was described by Strecker & Schwinn (Ref 11). This complex compd called *Hexamminenickel Azide,* $[Ni(NH_3)_6](N_3)_2$, is listed in Table E under Ammines (See also Refs 7 & 9). Double salts, such as $Ni(N_3)_2 \cdot NH_4N_3$ and $Ni(N_3)_2 \cdot KN_3$, have been reported (Refs 1 & 4)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 299(1898) & JCS **76** II, 92(1899) 2)T. Curtius & A. Darapsky, JPrakt Chem **61**, 418 (1900) & JCS **78** II, 474(1900) 3)L.M.Dennis & H.Isham, JACS **29**, 21(1907) & CA **1**, 528 (1907) 4)L.Wöhler & F.Martin, Ber **50**, 593 (1917); JCS **112** I, 383(1917) & CA **11**, 2900 (1917) 5)L.Wöhler & F.Martin, AngChem **30** I, 35-9(1917); JSCI **36**, 570(1917) & CA **11**, 3432(1917) 6)A.W.Browne et al, JACS **41**, 1775(1919) & CA **14**, 28(1920) 7)Mellor **8** (1928), 355 8)B.Ricca & F.Pirrone, Gazz **59**, 564(1929) & CA **24**, 309(1930) 9)L.F. Audrieth, Chem Revs **15**, 199-200(1934) 10) E.C.Franklin, JACS **56**, 569(1934) & CA **28**, 2289(1934) 11)W.Strecker & E.Schwinn, JPrakt Chem **152**, 217(1939) & CA **33**, 5314 (1939)

Nitrosyl Azide, NON_3, mw 72.04, N77.79%, yel unstable compd (above −50°), mp −66 to −57 depending on method of prepn; bp 1.5° (extrapolated value); Qvapzn 5.6 kcal/mol; vapor pressure 30 mm at −66°, 60 mm at −58° and 200 mm at −32° represented by log p = 7.306 − 1215.6/T where p = mm press and T = degrees Kelvin; Trouton constant 20.2

Nitrosyl azide was first prepd in 1957 by H.W.Lucien [Ref JACS **80**, 4458-60(1958)] from the reactions between: a)NaN_3 and nitrosyl chloride b)NaN_3 and nitric acid, c) NaN_3 and nitrosyl hydrogen sulfate and d) hydrazoic acid and nitrosyl hydrogen sulfate at temps below −30°. It was necessary to exercise due precaution against explns in all reactions. Successful prepns were obtd only when the reagents were slowly mixed at the lowest practical temp and gradually warmed to the desired reaction temp. According to Lucien, explns occurred at least once in each reaction type except in those experiments in which either ether was used as a solv or anhyd NaN_3 was used. Of seven attempts to treat NaN_3 with nitrosyl hydrogen sulfate, only two were successful. Almost the same record of success was reported for the reaction between NaN_3 and wh fuming nitric acid. Low yields, not exceeding 6%, were attributed to the instability of NON_3 and to the slow and incomplete reactions by which it was prepd. The yields of the various reactions decreased in the order:

$NOHSO_4 + NaN_3$
$NOCl + NaN_3$ (moist)
H_2SO_4/HNO_3(1:1) + NaN_3
HNO_3 (70%) + NaN_3
$NOHSO_4 + HN_3$
$NOCl$ (anhyd) + NaN_3

Although the extremly low yields (1%) in the last reaction were increased (to 5%) by adding water, excessive water resulted in reactions difficult to control

Nitrosyl azide was characterized by conventional analytical data and a study of its decompn into equimolar quants of nitrous oxide and nitrogen. The infrared spectra of samples from each of the procedures of prepn were compared and all showed similar absorption patterns. No other properties of NON_3 were reported

Phosphorus-Nitrogen Azide, $[PN(N_3)_2]_3$, mw 387.09, N 75.99%, col oil, insol in w, sol in org solvs, stable to alkali, decompd by concd HNO_3. It was prepd by reacting $(PNCl_2)_3$ with Na azide in acetone under N_2 to form the trimeric phosphonitrile azide which was readily detond by friction

Refs: 1)C.Grundmann & R.Ratz, ZNaturforsch **10b,** 116-7(1955) & CA **49**, 13007(1955) 2)H. Rosenwasser, USArmyEngrRes&DevelopLabs Rpt 1551-TR, 49(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Potassium Azide (formerly called *Potassium Trinitride or Potassium Azoimide),* KN_3, mw 81.12, N 51.80%, wh tetrag crysts (Refs 2,12 & 50); mp 320°(Ref 7), 343°(Ref 13), 350°(Refs 10 & 48) and decompg 355°−360° (Refs 2,7 & 13); d 2.038 g/cc (Ref 12), 2.045 g/cc (calcd in Ref 12) and 2.056 g/cc (Ref 14); Q_f 0.33 kcal/mol(Ref 45) $Q_{hydration}$ 157 kcal/mol (Ref 45), ionic conductance of crysts, E = 30.1 kcal/mol in temp range 390–500°K for log A = 4.59 in equation log k = log A − (E/2.303RT) (Ref 42). KN_3 is sol in w (49.2 g in 100 g solv at 17°), sl sol in alc (0.14 g in 100 g solv at 16°), insol in eth (Ref 2), and its soly in NH_3 is considerable and approxs that of KBr; in liq SO_2 potassium azide the salt becomes yel and explodes (Ref 5). The refractive index and conductivity of aq solns and soly of KN_3 in alc, w and benz were detd by Cranston & Livingstone (Ref 14). The density, refractive index and mol refraction of crystn K azide also have been reported (Ref 27). According to Sax (Ref 49) its toxicity is similar to that

of other azides; its expln hazard is moderate but it must be considered a dangerous material.

Potassium azide was claimed to be first prepd in 1898 by Dennis & Benedict (Ref 1b) and in the same year by Curtius & Rissom (Ref 2), both by methods involving evapn of a soln of KOH neutralized with a slight excess of hydrazoic acid, HN_3. This same method of prepn was described in 1894 by Dennis (Ref 1a). Browne & Houlehan (Ref 3) obtd KN_3 by reactg metallic K with NH_4N_3 in liq NH_3. Other methods of prepn are described by Hoth & Pyl (Ref 16), Moldenhauer & Möttig (Ref 17), Wattenburg(Ref 17a), Franklin (Ref 19), Audrieth et al (Refs 21, 30 & 37) and others (Refs 15, 22, 29, 39 & 51)

According to Curtius & Rissom (Ref 2), K azide was neither volatile nor hygro. When heated the salt melted, boiled and gave off nitrogen; the residue inflamed with a feeble deton. It did not explode under impact of a hammer (Ref 8). Hitch (Ref 9) studied the slow thermal decompn at high temps and found that K azide behaved similarly to Ba azide, depositing metallic K, but not decompg violently enough to break the apparatus. Audubert (Ref 26) reported an energy of activation of 20-22 kcal/mol for its thermal decompn; Garner & Marke (Ref 24) 36.1 kcal/mol with decompn appreciable at 220° in the presence of K vapor, and in vacuo decompn occurred in two stages at 330–350° (see Ref 32). Jacobs & Tompkins (Ref 41), who also observed that decompn was catalyzed by a constant vapor pressure of K, proposed a mechanism for its decompn and detd a value of 41.5 kcal/mol as the energy reqd

The photo-chemical decompn of aq solns of K azide was accompanied by intense UV (emission) (Refs 25, 28, 33, 34, & 35). Tompkins & Young (Ref 46) noted that color centers developed and the salt decompd into its elemetns when freshly pptd K azide was irradiated with UV light. In studying the electrolysis of aq K azide solns, Audubert & Racz (Ref 31) observed that low intensity UV radiation appeared. The Raman Effect of both cryst and K azide in soln has been reported by Kahovec & Kohlrausch (Ref 38). Wohlgemuth (Ref 20) studied the $KN_3 - H_2O$ system and found the eutectic at −12.9° contained 26.2% KN_3. The satd soln contained 29.3% KN_3 at 0°, 51.4% at 100° and formed no hydrate. The optical props of K azide solns were reported by Ångstrom (Ref 6)

The structure of the K azide mol has been studied by Frevel (Ref 23) and others who have obtd diffraction data (Ref 36), neutron diffraction measurements (Ref 40) and its mol refraction (Ref 43)

According to Browne & Hoel (Ref 11) K azide reacts with iodine in the presence of carbon disulfide to form K iodide and liberate nitrogen. When manganese dioxide is gently heated with K azide, the reaction proceeds with considerable violence forming K manganate (Ref 4). These authors also used K azide impregnated paper to detonate Ag azide in a lecture demonstration. According to Mellor (Ref 18), R.Stan studied the reduction of K azide by chromous salts

The mechanism of K azide formation with labeled N^{15} and its reactions are discussed by Clusius et al (Refs 44 & 47).

Potassium azide can be used for the qualitative detection of thorium and for its quantitative detmn either alone or in the presence of other rare earths (Ref 1a)

Refs: 1a)L.M.Dennis, AmChemJ **16**, 79-83 (18) & JCS **66** II, 256(1894); JACS **18**, 947-52(1896) & JCS **72** II, 232(1897) 1b)L.M. Dennis & C.H. Benedict, JACS **20**, 227(1898); ZAnorgChem **17**, 18-25(1898) & JCS **74** II, 426(1898) 2)T.Curtius & J.Rissom, JPrakt Chem **58**, 279(1898) & JCS **76** II, 91(1899) 3)A.W.Browne & A.E.Houlehan, JACS **33**, 1749(1911) 4)H.Goldberg, JACS **34**, 886-90 (1912) & CA **6**, 3236(1912) 5)F.Friedrichs, ZAnorgChem **84**, 390(1914) 6)A.K.Ångström, ZPhysChem **86**, 525-8(1914); JCS **106** II, 229 (1914) & CA **8**, 1538(1914) 7)E.Tiede, Ber **49**, 1745(1916) 8)L.Wöhler & F.Martin, ZAng Chem **30** I, 33-9 (1917); JSCI **36**, 570(1917) & CA **11**, 3432(1917) 9)A.R.Hitch, JACS **40**, 1195(1918) & CA **12**, 1951(1918) 10)W.R.

Hodgkinson, BritP 129152(1918) & CA **14**, 805(1920) 11)A.W.Browne & A.B.Hoel, JACS **44**, 2106 & 2117(1922) & CA **16**, 4154 (1922) 12)S.B.Hendricks & L.Pauling, JACS **47**, 2908(1925) & CA **20**, 318(1926) 13)R. Suhrmann & K.Clusius, ZAnorgChem **152**, 56 (1926) & CA **20**, 1962(1926) 14)A.Cranston & A.Y.Livingstone, JCS **1926**, 502 15)Mellor **8** (1928), 347 16)W.Hoth & G.Pyl, ZAngChem **42**, 888(1929) & CA **23**, 5547(1929) 17)W. Moldenhauer & H.Möttig, Ber **62B**, 1954-9 (1929) & CA **24**, 1300(1930) 17a)H.Wattenberg, Ber **63B**, 1667-72(1930) & CA **24**, 4998 (1930) 18Mellor **11** (1931), 368 19)E.C. Franklin, JACS **56**, 568-71(1934) & CA **28**, 2289(1934) 20)J.Wohlgemuth, CR **199**, 601-3(1934) & CA **28**, 7129(1934) 21)L.F.Audrieth, ChemRevs **15**, 199&202(1934) 22)Gmelin, System No 22, Teil 2 (1936), 247-50 23)L. K.Frevel, JACS **58**, 779-82(1936) & CA **30**, 4756(1936) 24)W.E.Garner & D.J.Marke, JCS **1936**, 657-64 & CA **30**, 6270(1936) 25) R.Audubert & H.Muraour, CR **204**, 431(1937) & CA **31**, 2518(1937) 26)R.Audubert, CR **204**, 1192(1937) & CA **31**, 4210(1937) 27) A.Petrikaln & B.Ogrins, Radiologica **3**, 201(1938); ChemZntr **1939** II, 327 & CA **35**, 3145-6(1941) 28)R.Audubert, TransFarad Soc **35**, 197-204(1939) & CA **33**, 2806-7(1939) 29)A.Chrétien & O.Hoffer, BullFr **6**, 1587 (1939) & CA **34**, 3197(1940) 30)InorgSynth **1**(1939), 79-81 & CA **36**, 2488(1942) 31)R. Audubert & C.Racz, CR **210**, 217(1940) & CA **34**, 2709(1940); BullFr **7**, 907(1940) & CA **36**, 2209(1942) 32)W.E.Garner, Chim & Ind (Paris) **45**, Suppl to No 3, 111-8(1941); Chem Zentr **1942** II, 365-6 & CA **37**, 4571-2(1943) 33)M.Bonnemay, CR **214**, 826-8(1942) & CA **38**, 3540(1944); CR **215**, 65-7(1942) & CA **38**, 5457(1944); CR **216**, 52&154(1943) & CA **38**, 4868(1944); CR **216**, 230(1943) & CA **39**, 1594(1945) 34)M.Bonnemay, JChimPhys **41**, 18-41(1944) & CA **39**, 3205(1945) 35)M. Bonnemay & E.T.Verdier, JChimPhys **41**, 113-24(1944) & CA **40**, 2384(1946) 36)L.K. Frevel et al, IEC, AnalEd **18**, 83-93(1946) & CA **40**, 2051 (1946) 37)M.W.Miller & L.F. Audrieth, InorgSynth **2** (1946), 139-41 & CA **40**, 6356(1946) 38)L.Kahovec & K.W. Kohlrausch, Monatsh **77**, 180(1947) & CA **42**, 6666(1948) 39)Kirk & Othmer **7** (1951), 593-4 40)S.W.Peterson & H.A.Levy, PhysRevs **87**, 462-3(1952) & CA **46**, 9436(1952) 41)P.W. Jacobs & F.C.Tompkins, ProcRoySoc **215A**, 254-77(1952) & CA **47**, 4206(1953) 42)P.W. Jacobs & F.C.Tompkins, JChemPhys **23**, 1445-7(1953) & CA **49**, 15336(1955) 43)S.S. Batsanov, Vestnick Moskov Univ **9**, No 9, Ser Fiz Mat i Estestven Nauk No **6**, 95-108 (1954) & CA **49**, 8652(1955) 44)K.Clusius & E.Effenberger, Helv **38**, 1843-7(1955) & CA **50**, 11871-2(1956) 45)P.Gray & T.C. Waddington, ProcRoySoc **235A**, 106&481 (1956) & CA **50**, 12627&15203(1956) 46) F.C.Tompkins & D.A.Young, ProcRoySoc **236A**, 10-23(1956) & CA **50**, 15241(1956) 47)K.Clusius & M.Vecchi, Helv **39**, 1469-83 (1956) & CA **51**, 3483(1957) 48)B.L.Evans & A.D.Yoffe, ProcRoySoc **238A**, 568-74 (1957) & CA **51**, 15129(1957) 49)Sax(1957), 1063 50)C.N.Rao & C.W.Hoffman, JScInd-Res(India) **16B**, 267-8(1957) & CA **51**, 17317 (1957) 51)R.W.Dreyfus & P.W.Levy, Proc-RoySoc **246A**, 233-40(1958) 52)H.Rosenwasser, USArmyEngrRes&DevelopLabs Report 1551-TR, 12,13,17&50(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Rubidium Azide (formerly called *Rubidium Trinitride or Rubidium Azoimide),* RbN_3, mw 127.50, N32.96%; col tetrag, sl hygro crysts (Ref 1); mp 260°, N_2 evolved in vacuo (Ref 3), 300° (Ref 3), 321° (Ref 5), 330° (Ref 2), 395° in vacuo (Ref 5) and regular evolution of N_2 at 310° once decompn has begun (Ref 3); d 2.937 g/cc (Ref 8); Q_f^v 0.07 kcal/mol (Ref 18); lattice energy 152 kcal/mol (Ref 18); sol in w (114g/100g w at 17°), sl sol in alc (0.182g/100g alc at 16°) insol in eth (Ref 2)

Rubidium azide was first prepd in 1898 by Dennis & Benedict (Ref 1) & in the same year by Curtius & Rissom (Ref 2) both by neutralizing RbOH with HN_3 and allowing the soln to evap in air. It is also formed

when N_2 activated electrically reacts with Rb metal (Refs 7 & 9). The toxicity of Rb azide is not discussed in Sax (Ref 19). Also see general Refs 6, 13, 14 & 20

This compd was found to be sensitive to impact by drop hammer (Ref 8), but stable to heat and light at RT (Ref 3). During electrolysis of its solns N_2 is liberated in an active form (Ref 4). In the thermal decompn of Rb azide, the residue contains the nitride, Rb_3N, which is a grn grey pdr extremely sensitive to moisture (Ref 11). The structure of Rb azide was detd by Pauling (Ref 10) and by Büssen et al (Ref 12); its diffraction data tabulated by Frevel et al (Ref 16) and the Raman Effect of both crystn pdr and in soln was reported by Kahovec & Kohlrausch (Ref 17)

Rubidium pentazido cuprate, $Rb[(N_3)_2CuN_3Cu(N_3)_2]$, crystallizes from a concd soln of RbN_3 and $Cu(N_3)_2$; the corresponding Cs salt is very expl (Ref 15). The expln temp of the Rb salt is 230-3° and it deton in a flame

Refs: 1)L.M.Dennis & C.H.Benedict, JACS **20**, 227 & 231 (1898); ZAnorgChem **17**, 20 (1898) & JCS **74** II, 426(1898) 2)T.Curtius & J.Rissom, JPraktChem **58**, 280-2(1898) & JCS **76** II, 91-2(1899) 3)E.Tiede, Ber **49**, 1742-5 (1916) & CA **11**, 2176(1917) 4)E.Briner & P.Winkler, Helv **6**, 429-35(1923); JChimPhys **20**, 201-16(1923) & CA **17**, 2841(1923) 5)R. Suhrmann & K.Clusius, ZAnorgChem **152**, 52 (1926) & CA **20**, 1962(1926) 6)Mellor **8** (1928), 347-8 7)W.Moldenhauer & H.Möttig, Ber **62**, 1954-9(1929) & CA **24**, 1300(1930) 8)P.Günther et al, ZPhysChem, Abt B **6**, 461 (1930) & CA **24**, 2930(1930) 9)H.Wattenberg, Ber **63**, 1667-72(1930) & CA **24**, 4998(1930) 10)L.Pauling, ZPhysChem, Abt B **8**, 326-8 (1930) & CA **24**, 5561(1930) 11)K.Clusius, ZAnorgChem **194**, 47-50(1930) & CA **25**, 889 (1931) 12)W.Büssen et al, ZPhysChem **15B**, 58(1931) & CA **26**, 1170(1932) 13)L.F. Audrieth, ChemRevs **15**, 202(1934) & CA **29**, 700(1935) 14)Gmelin, System No 24 (1937), 113-4 15)M.Straumanis & A.Cirulis, ZAnorgChem **252**, 121(1943) & CA **38**, 1701-2(1944) 16)L.K.Frevel et al, IEC, AnalEd **18**, 83-93 (1946) & CA **40**, 2051(1946) 17)L.Kahovec & K.W.Kohlrausch, Monatsh **77**, 180(1947) & CA **42**, 6666(1948) 18)P.Gray & T.C.Waddington, ProcRoySoc **235A**, 106 & 481(1956) & CA **50**, 12627 & 15203(1956) 19)Sax(1957), not listed 20)H.Rosenwasser, USArmy Engr-Res & DevelopLabsRpt 1551-TR, 11,45 & 50 (1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Silicon Tetrazide, $Si(N_3)_4$, mw 196.19, N85.68%; wh crysts, sol in bz and eth. It was prepd by the reaction of $SiCl_4$ and NaN_3 in dry benz in the presence of $LiAlH_4$ in ether. The soln was heated under reflux on a w bath for 20-30 hrs, decanted and the benz sublimed at high vac and 0°. Attempts to prep Si azide by the reaction of SiH_4 and HN_3 were unsuccessful

$Si(N_3)_4$ is highly expl and sensitive to moisture.

Refs: 1)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 500(1954) & CA **49**, 768(1955) 2)H. Rosenwasser, USArmyEngrRes & DevelopLabs Rpt 1551-TR, 48(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

SILVER AZIDE (formerly called *Silver Azoimide or Silver Trinitride)* (called Silberazid in Ger; Nitrure d'argent in Fr; Acido d'argento o Azotidruro d'argento in It and Ácido de plata in Span), AgN_3, mw 149.90; N 28.03%, col ortho ndls from NH_3 (Ref 88) mp 250° (Ref 1) in vacuo 185°(Ref 21); N_2 evolved above 254°(Ref 21) and expl 297-300°(Refs 10,21 & 60); d 5.1g/cc (Ref 50) to 4.8 g/cc (Ref 37a). Q_f^v -74.2 kcal/mol (Ref 80), Q_{react} 68kcal/mol (Ref 60), lattice energy 204.7 kcal/mol(Ref 80), ΔF°_{298} 78.69kcal/mol from elec chem cell (Ref 63); $Q_{activation}$ 20–21 kcal/mol (Ref 41), 35 kcal/mol (Ref 79) to 41 kcal/mol (Refs 65 & 71); Q_{deton} 65.5 kcal/mol (Refs 14 & 16) and sp heat 0.12 cal/g (Ref 66). The soly of Ag azide in w has been reported as 3.9×10^{-5} mol/l at 17° (Ref 68), 5.1×10^{-5} mol/l at 25° (Ref 43), and 8.4×10^{-3} g/l

(Ref 37).(Also see Refs 7,8,38 & 74). It is non-hygro and only very sl sol in alc, eth or acet(Ref 27). Ag azide, like other Ag compds, can be absorbed into the body circulation and subsequently deposited in various body tissues causing a generalized greyish pigmentation of the skin–a condition known as "argyria" (Ref 89). According to Sax there is no known method by which silver deposited in a body can be eliminated. The expln hazard of AgN_3 is severe when it is exposed to shock or heat

Silver azide was first prepd in 1890-1 by Curtius (Ref 1) by passing hydrazoic acid, HN_3, into neutral silver nitrate soln. This and other methods of prepn were later described by Thiele (Ref 2), Angeli (Ref 3), Dennis (Ref 4), Curtius & Rissom (Ref 5), Dennis & Isham (Ref 8), Wöhler & Matter (Ref 9), Hodgkinson (Ref 20) & Hitch (Ref 21). Turrentine (Ref 9a) electrolyzed 3% Na azide soln using Ag anode; Brown et al by electrolysis at the Ag anode in Amm azide soln (Ref 24). See also Darier & Goudet (Ref 25), Meissner (Ref 30), Taylor & Rinkenbach (Ref 27), Majrich (Ref 38), Wallbaum-Wittenburgh (Ref 46), Stettbacher (Ref 73), Bertho & Aures (Ref 76) and others (Refs 29, 57, & 58). Darier & Goudet (Ref 25) describe the prepn with a min risk of expln by effecting the reaction within the interstices of a porous absorbent material which is inert and maintains the expl crysts separate from each other. Taylor & Rinkenbach (Ref 27) prepd Ag azide in the pure state, as white colloidal aggregates, by mixing fairly concd solns of $AgNO_3$ and NaN_3. The colloidal prod was more stable and less sensitive than the crysts. Meissner (Ref 30) described an app for the prepn of Ag azide by a continuous process and Stettbacher (Ref 73) detailed a recent lab procedure for its prepn

Explosive Properties:

Brisance by Sand Test, 41.1 g sand crushed vs 37.2 g by MF (Ref 27)

Detonation Rate, 1500 m/sec (unconfined and initiated by hot wire), 1700 m/sec (unconfined and initiated by impact with grit particle), and 1900 m/sec (unconfined in vacuo at 0.1 mm Hg) (Ref 59)

Explosion Temperature, °C. 297° in 5 sec for a 0.02g sample (Ref 15) to 308° in 1 sec for a 0.02 g sample (Ref 58)

Friction Sensitivity, extremely sens, but more stable to friction than either Cu, Ni or Co azides (Ref 15) (also see Ref 28)

Impact Sensitivity, 3 in with 2 kg wt and 6 cm with 1 kg wt or 41 cm with 500 g wt vs 43 cm for LA both in BM App (Refs 28 & 58)

Initiating Efficiency, see table under Mercurous Azide (or Ref 16)

Lead Block Expansion, 22.6 cc for 2g sample vs 25.6 cc for MF(Ref 9, p 247)

Stability, color remains white when kept in the dark but on exposure to sunlight cyrsts darken. It is stable at 75° (Refs 28 & 46)

Temperature Developed on Explosion 3545° vs 3420° for LA(Ref 16)

Work Density, 96.5 kg/cc vs 98.9 kg/cc for LA (Ref 16). Other expl props have been described in Refs 12,13,16,18,32,35,48,51, 72,75,81,83 & 94

General Properties: The x-ray cryst structure of Ag azide was detd by Bassière (Ref 36) and his results were confirmed by West (Ref 39). Pfeiffer(Ref 55) also studied the x-ray struct of Ag azide and detd the Ag-N bond dist as 2.56 A° and the Ag-Ag bond dist as 3.00 A°. The optical and elec props, dielectric const, UV absorption spectra and photo conductivity were detd by McLaren & Rogers (Ref 84). Suzuki(Ref 64) calcd the std free energy, $\Delta F°$; entropy, $\Delta S°$; and the heat content, $\Delta H°$ for the reactn $AgN_3 + Hg = HgN_3 + Ag$. The normal AgN_3–Ag electrode potential, referred to hydrogen, was measured by Brouty (Ref 49) as 0.2945V. Bowden & McLaren (Ref 90) studied the expln of Ag azide in an elec field and found that with 45V across the cryst, expln occurred when the current rose to ca 150 μA within minutes. Berchtold & Eggert (Ref 67) observed that Ag azide exposed to the energy from a photographic "electron" flash bulb at a dist of

6 cm, reqd 300 w-sec for ignition to deton. Bowden & Singh (Ref 69) found that Ag and other azides were all exploded by an intense electron stream but not by slow neutron bombardment

Decomposition. The thermal decompn of Ag azide has been the subject of considerable investigation. Freshly prepd, pure, col crystn Ag azide in sunlight or Hg light becomes violet, gray, and finally black with the evolution of N_2. The compd remains unchanged when kept in the dark at RT but evolution of N_2 continues, even in the dark, when Ag azide is heated (Ref 10). Bowden & McAuslan (Ref 82) studied the slow thermal decompn by means of a scanning electron microscope and observed between 120° and 250° a crystallographic phase change at 180°. Small crysts of Ag azide irradiated in vacuo with UV light (2000-3600Å) reqd a critical amt, corresponding to 8×10^{-4} cal/sq mm of cryst surface,to initiate expln (Ref 77). Light emitted by the explosn of one cryst of azide did not initiate expln of another cryst, but tiny flying fragments did. The effect of light on Ag azide and other expls was recently reported by Eggert (Ref 92) and by McAuslan (Ref 93). The UV absorption and UV irradiation on thermal decompn of Ag azide has been studied by many investigators (Refs 31,40, 42, 45 & 85), also IR absorption (Refs 53). Audubert & Calmar (Ref 86) found that surface dissocn of Ag azide showed nitrogen active with 2 half-lives characterized by different emission spectra. Thermal decompn has also been studied by Evans & Yoffe (Ref 87), Bowden (Ref 70), Sawkill(Ref 78), Gray & Waddington (Ref 80), Bartett et al (Ref 91) and others. Bartett et al (Ref 91) investigated the thermal decompn of both allotropic forms of Ag azide and obtd activation energies of 44 to 46 kcal/mole for the low-temp form and 31 to 32 kcal/mol for the high-temp form

Chemical Reactions. Hantzsch (Ref 6) reacted iodine with Ag azide and obtd Iodine Azide (qv) and Spencer (Ref 26) reacted bromine with Ag azide to form the highly unstable Bromine Azide (qv); Frieson & Browne (Ref 52) formed *Azino Silver Chloride,* N_3AgCl, stable only below −30°,by reacting chlorine azide (qv) with Ag azide. Friedlander (Ref 22) reacted tetramethylammonium iodide with Ag azide and obtd *tetramethylammonium azide* (qv). *Guanidine Azide* (qv), $HNC(NH_2)_2 \cdot HN_3$, was prepd by reacting quanidine chloride with Ag azide (Ref 34). Silver azide in anhyd acet reacts with α-acetobromo sugars to form *β-acetoazido sugars* (Ref 56). The formation of complexes between Ag and azide ions has been described by Leden & Schöön (Ref 74). Klatt(Ref 47) noted that the molal bp rise of a soln of Ag azide in HF indicated formation of 4 ions per mole: $AgFH^+$, HN_3F^+ and $2F^-$. Tingle (Ref 23) warned against the expl nature of ammoniacal silver oxide solns due to the unexpected ready formn of Ag azide. A method of analyzing components contg small quants of Ag azide and its identification by behavior under impact or flame are given by Loriette & Loriette (Ref 19). Cyanimide ions have been introduced into the Ag azide cryst lattice by coprecipitation (Ref 79)

Uses. As early as 1893 the Prussian Govt investigated Ag azide for its possible use in detonators (Refs 10,32 & 35). Originally the cost and extreme sensitiveness acted as serious deterrents to its extended use. Blechta (Ref 33) proposed mixing Na azide with granular substs, such as tetryl, PETN, MF, etc and adding $AgNO_3$ soln to ppt Ag azide as a film over them. The efficiency of such initiators was about the same as that of pure Ag azide. In Italy Ag azide was manufd, by the analogous method used for Pb azide, in the form of an amor powder for use in some priming compns (Refs 54 & 62). Sprenger (Ref 17) described a method for opening and examining blasting caps contg Ag azide, but C.G.Storm considered the procedure dangerous. Ag azide is photosensitive and gelatin emulsions of it, prepd by methods analogous to those for AgBr emulsions, were relatively insensitive to shock or temp rise (Ref 11) (Also see Refs 44 & 61)

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SODIUM AZIDE (Formerly called Sodium Azoimide or Sodium Trinitride) (called Natriumazid or Stickstoffnatrium in Ger; Azothydrure or Nitrure de sodium in Fr; Acido di sodio or Azotidruro di sodio in Ital; Azida de sodio or Nitruro de sodio in Span and Azid natriya in Russian), NaN_3, mw 65.02, N 64.63%. Wh hex crysts, mp decomp (in vacuo) with evolution of N_2 from 275° (Refs 17 & 29) to 330° (Refs 17, 20, 48, 50 & 69) at atm press for complete decompn; $d_4^{25°}$ 1.8473 (Refs 25, 27 & 30), $Q_{activation}$ 10 to 34 kcal/mol below 365° and 20 to 50 kcal/mol above 365° (Refs 69, 72, 77 & 115); Q_f -5.08 kcal/mol at 298°K (Ref 188); lattice energy 175 kcal/mol at 298°((Refs 78 & 188); ionic conductance of crysts obeyed equation log k = log A - (E/2.303RT) where log A = 0.490 and E = 25.0 kcal/mol in temp range 375 to 490°K (Ref 156); sp heat from 0° to 100° is 0.2934 cal/gm/°C (Ref 86)

The soly of NaN_3 in acet, $CHCl_3$, hexane, cyclohexane, CCl_4, trichloroethylene or ethyl acetate at 25° is less than 0.005 g/100 ml of soln (Ref 130); the soly in methanol at 25° is 2.48 g/100 ml soln (Ref 130); in ethanol at 0° 0.22 g/100 ml solvent, 0.46 at the bp of soln; and in benz at bp soly is 0.10 g/100 ml solv (Ref 30). Curtius (Ref 1) found NaN_3 soly in H_2O to be 40.2 g/100 g at 10° and 41.7 g/100 g H_2O at 17°; it is insol in ether. According to Friedrichs (Ref 12) NaN_3 was

insol in sulfur dioxide but readily sol in liq NH_3. Wohlgemuth (Ref 60) studied the system H_2O-NaN_3 and found a eutectic at −15.1° (21.6% NaN_3), a point of transition at 2.1° (27.8% NaN_3), and a metastable eutectic at −20° (26.8% NaN_3). The satd aq soln at 0° contd 28% NaN_3 and at 100° 35.6% NaN_3. Crysts of $NaN_3 \cdot 3H_2O$ were isolated by Wohlgemuth (Ref 60). Cranston & Livingstone (Ref 30) and Günther & Perschke (Ref 46) detd densities, refractive indices and elec conductivity of aq NaN_3 solns; the electrolysis also was studied by Turrentine (Ref 11), Briner & Winkler (Ref 24), Schmidt (Ref 140) and by Semenchenko & Serpinskii (Ref 58), Audubert et al (Refs 82 & 87), Verdier (Ref 93), and Jolibois & Clérin (Ref 95); mol refraction by Petrikalns & Ogrins (Ref 79), optical props by Ångstrom (Ref 13). From his studies of the props of aq NaN_3 solns, Yui (Ref 88) detd the true dissociation constant of HN_3. Nuclear spin resonance of aq NaN_3 and other Na compds has been reported in Ref 187

Structure. NaN_3 was assumed by Curtius & Rissom (Ref 4) to be hexagonal crysts. This form was established by A.C.Gill as reported by Dennis & Benedict (Ref 3). According to West (Ref 59) the hexagonal units of NaN_3 contain 3 mols. He detd their cryst dimensions and showed by powd photographs their anisotropic nature on thermal expansion. By means of X-ray and Laue photographs Hendricks & Pauling (Ref 27) detd the rhombohedral unit cell dimensions and the interatomic distances of NaN_3. The configuration of NaN_3 was detd also by Frevel (Ref 68) who found the N–N distance of 1.150 ± 0.016 Å in good agreement with the value 1.17 Å reported by Hendricks & Pauling. However, Bassière (Refs 81 & 102) claimed that the azide ion in NaN_3 was asymetric with N–N distances of 1.10 and 1.26 Å (see also Ref 75). Dreyfus & Levy (Ref 200) observed that NaN_3 and KN_3 subjected to thermal or mech shock showed distortion along slip planes. By X-ray diffraction techniques Krasner & Keating (Ref 199) investigated stacking faults introduced into NaN_3 by grinding or exposure to radiation. Joebstl & Rosenwasser (Ref 202) in a study of the optical and electron microscopy of NaN_3 observed that crysts from soln go through a change from needle and hexagonal forms, respectively, to microcrystalline aggregates. Both KN_3 and NaN_3, as a result of ageing, will form etch pits, oriented overgrowths and other surface defects. It is therefore necessary in interpreting physical data obtd with azide crysts to know the preparative method and history of the material examined

Toxicity. Sodium azide is more acutely toxic than LA and is considered 3 times as potent as org azides (Ref 192). According to Sax (Ref 194) NaN_3 is classed by ICC and Coast Guard regulations as a poison B subst and must be packed in wooden boxes, with inside containers of securely closed paper bags placed within a waterproof duplex bag. Net wt of material must not exceed 100 lbs. The minm lethal dose of NaN_3 following injection (Ref 103) is 35 to 38 mg/kg of body wt, while up to 150 mg/kg of body wt (ca 66 mg NaN_3) can be injected intraperitoneally without causing death. When a 0.005 to 0.010 g tablet was swallowed (Ref 36), there ensued within 5 min violent heart stimulation, throbbing at the base of brain and loss of consciousness for 10 mins followed by rapid recovery. Less severe attacks recurred during the following hour. The symptons are similar to those caused by strychine. According to Hurst (Ref 90), a single dose of NaN_3 was more lasting in effect than KCN. Repeated doses of NaN_3 produced necrosis or demyelination in optic nerves of the brain (Ref 179). Black et al (Ref 159) reported that an oral dose of NaN_3 had a rapid hypotensive effect and produced a sustained lowering of blood pressure

Other effects due to intoxication by NaN_3 (Ref 34) include respiratory arrest, development of convulsions, at first clonic, later

tetanic, and finally heart failure (Ref 174). Biehler (Ref 45) noted that NaN_3 when injected stimulated respiration and acted as a central irritant. Small doses decreased blood press, while repeated dosage increased it and caused a transient decrease in body temp. The final effect can result in rigor mortis although the onset is earlier in case of exposure to NaF (Ref 163). The reaction between hemoglobin and NaN_3 has been discussed by Sjöstrand (Ref 148) and by Kikuchi et al (Ref 176). According to Ponz (Ref 158) NaN_3 partially inhibits absorption of glucose thru the intestines. Neither HN_3 nor NaN_3 is excreted unchanged in the urine (Ref 116). The effect of NaN_3 on muscular activity is similar to that produced by 2,4-dinitrophenol (Ref 198). Death due to intoxication with NaN_3 can be prevented by prophylactic ingestion of methemoglobin-forming agents, such as $NaNO_2$ (Ref 74)

Preparation. Sodium azide was first prepd in 1891 by Curtius (Ref 1) by saponification of benzoylazide with an alc or aq soln of NaOH. Wislicenus (Ref 2) prepd NaN_3 by passing a mixt of NH_3 and N_2O over molten Na or by treating sodium amide at 150°-250° with nitrous oxide:

$$2NaNH_2 + N_2O = NaOH + NaN_3 + NH_3 \uparrow$$

In 1898 Dennis & Benedict (Ref 3) and Curtius & Rissom (Ref 4) independently made NaN_3 by evapg a soln of NaOH neutralized with HN_3. The prepn of NaN_3 has been described also by Dennis & Browne (Ref 5), Thiele (Ref 6), Orelkin et al (Ref 19), Browne & Wilcoxon (Ref 28), Wilcoxon & Grotta (Ref 32), Moldenhauer & Möttig (Ref 43), Dynamit-AG (Ref 65), Newman (Ref 63), Westfälisch-Anhaltische Sprengstoffe-AG (Ref 64), Acken & Filbert (Ref 106), Abe et al (Refs 138, 142 & 169), Wehrle et al (Ref 135), Funaoka & Iwanaga (Ref 167), Clusuis et al (Refs 181 & 189), Dreyfus & Levy (Ref 200) and others (Refs 37,38,62,84,107,109,113,133, 143,144, & 201)

Sodium azide is manufd in France, Germany, Italy and the USA by the sodamide process which is the one generally employed commercially. (See under Manufacture of Sodium Azide) This reactn discovered by Wislicenus (Ref 2) and known as the Wislicenus Method gives a 90% yield in lab prepn. The method was investigated thoroughly by Dennis & Browne (Ref 5). A later modn of this process is based on the catalytic conversion of Na to $NaNH_2$ in liq NH_3 and treatment of the resulting suspension with N_2O under press at RT (Ref 133). The reaction between hydrazine, ethyl nitrate and caustic soda (or sodium ethylate) in alc soln also forms NaN_3:

$$N_2H_4 + C_2H_5ONO + C_2H_5ONa = NaN_3 + 2C_2H_5OH + 2H_2O$$

This process is considered particularly attractive for coml development as it requires no particular precautions in handling the reactants during the manuf of NaN_3 (Ref 133)

According to Schlenk & Wichselfelder (Ref 14) when thin slices of Na were gradually added to free hydrazine in pure, dry N_2, a col cryst ppt formed which, after excess N_2H_4 was distd off in vacuo exploded violently when removed from the vessel or when brought in contact with moisture. On similarly treating hydrazine, entirely free from the hydrate, and evapg the yel soln, brilliant cryst lfts of sodium hydrazide, NH_2NHNa were identified. This compd exploded violently at the least breath of air or trace of moisture. It could be decompd without expln by soln in benz contg a little alc (Ref 37)

Manufacture of Sodium Azide was conducted at the Kankakee Ordnance Works, Joliet, Illinois, (Ref 144) by the "liquid phase process" as follows: For this five 12-lb bricks of sodium were melted in an electrically heated melter and the molten Na at 350°F (176.7°) dropped to a high-pressure autoclave contg 375 lbs liq ammonia and 1 lb ferric nitrate catalyst. The Na reacted to form Na amide and hydrogen:

$$2Na + 2NH_3 \rightarrow 2NaNH_2 + H_2,$$

the latter being vented out of the autoclave

along with some ammonia at a gage press 300 psi. The temp was held below 105°F (40.6°) by cold w in the autoclave jacket. When this reaction had subsided the remaining hydrogen was vented and about 55 lbs of nitrous oxide gas was added to the charge through a standpipe the end of which was directed beneath the gas disperser agitator in the autoclave. The following reaction took place: $2NaNH_2 + N_2O \rightarrow NaN_3 + NaOH + NH_3$. In this operation as much of the N_2O as possible was fed continuously and at such rate that the concn of N_2O in the vapor space was less than 25% by vol, to prevent forming an expl mixt with NH_3. When no more N_2O was absorbed the charge was blown into a 247 gal drowning tank which contained enough w to give a final soln strength of 8% NaN_3. The yield from Na to NaN_3 in the *crude solution* was 87%

The next step was the removal of ammonia from the "crude soln", which was done by steam-stripping in an evaporator to an ammonia recovery system, where NH_3 was absorbed in w. The resultant NH_3-free crude soln was filtered through a Nutsch type filter to remove the catalyst and other insol impurities. The filtrate referred to as *clear liquor* was stored in a 1000 gal tank from which it could be transferred by suction into either of two 280 gal jacketed evaporators. The evaporation was conducted under 24" vacuum with 50 lb steam press in the jacket. A total of 425 gal of "clear liquor" was concentrated until a sample of its "mother liquor" showed the strength of 35% NaOH. During this operation the bulk of NaN_3, being less sol in w than NaOH, pptd. Then the mixt was cooled to 80-90°F (27-32°) (to cause the pptn of addnl NaN_3) and dropped to a wringer. The yield was ca 75% NaN_3 and the overall cycling time was 5-6 hrs

The "mother liquor" was wrung out of the slurry and drained from the wringer to a catch tank from which it was pumped to a storage tank to be reworked. The crystals of NaN_3 were washed on the wringer with 100 lbs of treated w, which was pumped through spray nozzles inside the basket, and the washings sent to the *mother liquor storage tank.* (The total, about 350 gals, was referred to as the *first mother liquor).* Then the crysts were dissolved directly in the wringer with about 65 gals of treated w, which yielded the *refined solution* of 27% NaN_3 strength. The "refined soln" was pumped to a weigh tank from which it was dropped (after it was sampled and the wt recorded) to a 1000 gal storage tank. When required the "refined soln" was pumped to the LA manufg plant (See under Lead Azide)

The "first mother liquor" (see above), max 350 gals, was evapd until the NaOH concn reached 50%. After cooling to 80-90°F, the slurry was dropped to a wringer where the *second mother liquor* was wrung out to the catch tank from which it was pumped to a 240 gal storage tank. The second crop of crysts remaining in the wringer was not washed but dissolved in treated w and delivered to a 240 gal *receiver tank* from which the soln was sucked into the *clear liquor evaporator* to be reworked. The refining process gave an overall recovery yield of 94-96%

The "second mother liquor" was analyzed for its NaN_3 content and if it was over 2%, the liquor was reworked in the *mother liquor evaporator.* If the liquor contained less than 2% NaN_3 it was sent to the 710 gal *killing tank,* where the azide was destroyed by a calcd amt of Na nitrate and concd sulfuric acid to produce the reaction:

$$NaN_3 + NaNO_2 + H_2SO_4 + N_2O + N_2 + Na_2SO_4 + H_2O$$

After destroying the azide, the waste liquor of the killing tank was siphoned to a settling pond from which it was sent to a stream

Explosive Properties. According to Curtius (Ref 1) NaN_3 is neither volatile nor hygr, its solns may be evapd to dryness without undergoing any change, and it explodes only when heated to a high temp. This stability to heat was confirmed by Curtius & Rissom

(Ref 4), Dennis & Benedict (Ref 3) and Dennis & Browne (Ref 5). Wöhler & Martin (Ref 18) exploded NaN_3, without true deton, by heating a 0.02 g sample but did not explode it by impact on a compressed 0.01 to 0.05 g sample. Bowden & Williams (Ref 132) were unable to propagate deton in NaN_3

Thermal Decomposition. Wattenburg (Ref 48) observed that NaN_3 must be heated to 250° before decompn starts and that formation of Na_3N is a necessary stage in the decompn of NaN_3 to N_2 and Na. According to Garner & Marke (Ref 69) the decompn in a vac at 257-365° was similar to that of KN_3, but the catalytic effect of Na vapor was small. Decompn followed an induction period and then occurred in two or three steps (Refs 89 & 108). Audubert (Refs 72 & 80) noted that intense UV radiation was emitted during slow thermal decompn of NaN_3 and KN_3 (Ref 71). In a closed system the nature of gas or its absence had no effect but in a moving current of gas UV radiation was more intense (Ref 76). Bonnemay (Ref 100) detd the effect of bases and neutral salts on decompn rate. Thermal decompn of NaN_3 has been studied extensively by Yoffe (Ref 131) and by Jacobs & Tompkins (Ref 139)

Effects of Radiation. The threshold for decompn of NaN_3 by electron bombardment is at 11.65 ± 0.2 volt-electrons (Ref 52). Groocock & Tompkins (Ref 160) studied the decompn with 100 and 200 v-electrons at RT and found theoretical calcns agreed with exptl results. Muller & Brous (Ref 52) found that photochemical decompn occurred at wave lengths below 405 mμ at a rate directly proportional to the UV intensity. No relation between energies necessary for electronic and photochemical decompn was apparent. NaN_3 turned brown under X-ray and slow neutron bombardment (Refs 149 & 184). When irradiated NaN_3 was dissolved in H_2O, N_2 gas was evolved and OH^-, NH_3 and N_2H_4 were present. Heal (Ref 196) showed also that NaN_3 irradiated at or below RT was initially pale green and later became brn on standing a few hrs at RT. Irradiated NaN_3 dissolved in liq NH_3 gave a blue soln similar to that obtd from Na in liq NH_3. By irradiation of NaN_3 with alpha rays from Po or Rn, a new radioisotope was produced (Ref 61). Rosenwasser et al (Ref 184) investigated the induced coloring of NaN_3 exposed to gamma rays and to neutron bombardment. Gamma-rays produced a broad band at 3600 Å and a band at 6000 Å when gamma-irradiated NaN_3 was heated above 90°

Absorption and Emission of Radiation. Raman spectra of NaN_3 have been detd by Petrikalns & Hochberg (Ref 41), Kahovec & Kohlrausch (Ref 111) and by Sheinker & Syrkin (Ref 120). Moler (Ref 99) expressed the absorption spectra of aq NaN_3 soln as log of extinction coeff vs wave length. Sheinker (Ref 127) noted that the UV absorption spectra of aq NaN_3 soln were markedly different from those of typical aliphatic azides. Infrared absorption spectra were reported by Lieber et al (Ref 129) and by Delay et al (Ref 105) in the range 3 to 19μ. From the intensities of bands observed, it was concluded by Delay et al that the sym form was more abundant than the unsym form in azides of Na, Cu, Aq and Hg but the reverse was true for azides of Tl and Pb

The optical absorption and UV absorption of aq NaN_3 were detd by Bonnemay & Verdier (Refs 92 & 104). In both cases Beer's law was not obeyed except for narrow regions of concns and wave lengths. The kinetics of the photochemical effects on NaN_3 decompn has been discussed in detail by Bonnemay (Ref 94)

Chemical Reactions. Forster (Ref 8) described the interaction of benzhydroximic chloride with NaN_3 to give wh ndls (mp 124°) of 1-hydroxy-5-phenyltetrazole which decompd spontly. The reaction of acid chlorides with NaN_3 was described by Schrocter (Ref 9) Waltmann (Ref 83) and Kiss & Vinkler (Ref 124). While Schroeter found that coml NaN_3 reacted smoothly and rapidly with acid chlorides to form isocyanates (Also see Refs

63 & 83), Naegeli et al (Ref 39) obtd good results only when pure NaN_3 was used. Nelles (Ref 54) observed a similar diff in reactivities of pure and coml NaN_3. At the time of Schroeter's investigation coml NaN_3 was prepd by the Thiele-Stollé method (Refs 6 & 7), but later was manufd from N_2O & $NaNH_2$, and contd impurities which hindered its reaction with acid chlorides. These impurities were not removed even by liberating the acid from the salt and neutralizing with the purest NaOH. However, Nelles (Ref 54) found that if NaN_3 were rubbed with a trace of $N_2H_4 \cdot H_2O$ and pptd from a little w with a little acet, it was not only as reactive as pure NaN_3 made from N_2H_4 and NH_4NO_2, but because of its greater surface area it was even more reactive (See also Ref 197 for prepn of activated NaN_3). According to Stollé (Refs 22 & 57) the reaction between NaN_3 and benzalbenzhydrazide, dibenzhydrazide or diacylhydrazide chlorides gives various tetrazole and hydrazide azide derivs, some of which are expls. Goyal & Saxena (Ref 173) also studied the reactions of acid chlorides with NaN_3 and reported that dry NaN_3 and CrO_2Cl react explosively in CCl_4 below 0° probably forming a solid, $CrO_2(N_3)_2$

The interaction of nitrosates with NaN_3 was described by Forster & vanGeldern (Ref 10). Sommer (Ref 15) treated aq NaN_3 with CS_2 and obtd, on cooling to 0°, yel prisms of sodium azidodithiocarbonate, $N_3CS_2Na \cdot H_2O$, stable below 10° and only moderately expl but defgrg violently on Pt foil. The anhyd salt exploded on impact and detonated on gentle heating. NaN_3 with $AgNO_3$ gave a white Ag salt, insol in HNO_3 and NH_4OH, and explosive at the slightest touch when dry (see Silver Azide). On adding 3 moles of NaN_3 in alc to $Cr(N_3)_3$, Oliveri-Mandalà & Comella (Ref 21) sepd grn crysts of $Cr(N_3)_3 \cdot 3NaN_3$ which in aq soln with $AgNO_3$ gave an expl complex salt. Currier & Browne (Ref 23) absorbed CS_2 vapors in 5% satd aq solns of NaN_3 and formed an azido salt which possessed no dangerous expl props but decompd slowly on long standing. According to Spencer (Ref 26) dry bromine reacted with NaN_3 to give the highly unstable expl *bromazide:*

$$NaN_3 + Br_2 = NaBr + BrN_3$$

and bromine water reacted instantly with NaN_3 to give a mixt of hydrazoic and hypobromous acids:

$$NaN_3 + Br_2 + H_2O = NaBr + HN_3 + HBrO$$

Hofmann & Hofmann (Ref 31) found that NaN_3 reacted with molecular H_2 and O_2 to form $NaNH_2$ and $NaNO_2$, resp. NaN_3 with colloidal Pd and CO_2 was unchanged (Ref 53)

Anhyd HF decomp NaN_3, evolving HN_3 (Ref 47). Hoffmann (Ref 51) studied the mechanism of reaction of various types of ultramarines on NaN_3 and observed that rose ultramarine gave the most vigorous reaction, evolving N_2. Stollé et al (Refs 55 & 56) reacted 1,4-dichloro or 1,4-dibromophthalazine and dichloroquinazolines with NaN_3. The synthesis of aromatic nitro compds with an azide group in the side chain was accomplished by Yushchenko (Ref 67) by reacting either NaN_3 or AgN_3 with the appropriate chloride or iodide in alc or acet soln. Kuz'min & Zemlyanskii (Ref 66) prepd the monoazides of Ph styryl and Ph 3-nitrostyryl ketones by reacting NaN_3 with the appropriate starting materials. According to Labruto & Landi (Ref 73), the reaction of benzoyl chloride with NaN_3 without solv and in the presence of NaOH or KOH was spontaneous and violent, evolving suffocating and lachrymous vapors. The reaction between phenylcarbylamine chloride, $PhN{:}CCl_2$, and NaN_3 is quant in acet giving a product $Ph\,N \cdot N{:}N \cdot N{:}C\,N_3$, mp 99° (Ref 85)

By dissolving $Cu(N_3)_2$ in aq or alc solns of NaN_3 Straumanis & Cirulis (Ref 98) obtd sodium triazido-cuprate $Na\,[Cu(N_3)_3]$, anhyd after heating above 120°. This compd exploded at 216-23° and under impact of drop hammer. Explosion also took place when $Hg(NO_3)_2$ was added to $Na[Cu(N_3)_3]$ (Ref 98). The addn of NaN_3 to an amine soln of Cu^{++} salts pptd complex non-electrolytes (Ref 97). Wiberg &

Michaud (Ref 164) prepd a wh solid complex salt, sodium hexazidostannate, $Sn(N_3)_4 \cdot 2NaN_3$ or $Na_2Sn(N_3)_6$, from $SnCl_4^=$ in tetrahydrofuran soln heated with an excess of NaN_3. After filtration and evapn of solv, the solid complex was sol in tetrahydrofuran, insol or only sl sol in ether or benz and was hydrolyzed by moist air. In tetrahydrofuran soln the salt detonated on boiling. In the reaction betwn $FeCl_3$ and NaN_3 no complex Fe azides were formed (Ref 150). The prepn of azido compds by the reaction of NaN_3 with epoxides was described by Vander Werf et al (Ref 166) and by Ingham et al (Ref 190). Vander Werf et al (Ref 166) found that $(N_3CH_2)_2 \cdot CHOH$ when hydrogenated catalytically gave $(H_2NCH_2) \cdot CHOH$ (bp 3 mm Hg 93-5°) which decompd violently above 150°. The di-HCl salt and the picrate both melted with decompn at 184° and 233.6° respectively

Adamson (Ref 110) described the prepn of $NaC^{14}N$ from $BaC^{14}O_3$ and NaN_3 in a N_2 atm to yield 75-80% of prod after 30 min heating. Henneberry & Baker (Ref 118) modified Adamson's method, to prevent explns, by fusing NaN_3 and $BaCO_3$ at 630° for 20 min to form NaCN. The reduction of NaN_3 by amalgamated Al was reported by Labruto (Ref 125). Other reactions involving NaN_3 include: isomerization of halohydrins (Ref 152), reaction with anthraquinone derivs (Ref 154) and monobromomalonic ester to form diazidomalonic ester (Ref 155), synthesis of phenanthridines by interaction with fluoren-9-ols (Ref 168), conversion of secondary nitro compds to amides or lactams (Ref 178) and the prepn of toxic fluorine compds by using "activated" NaN_3 (Ref 197). Miller et at (Ref 195) discussed the role of NaN_3 in S_N mechanisms involving aromatic substitutions. Werle et al (Ref 161) reported that NaN_3 was decompd by animal or plant tissues, such as liver & kidney extracts and potato, sugar beet or apple extract. Levey (Ref 165) noted that a small amt of NaN_3 inhibited the anthrone reaction used for detn of total carbonates, but NaN_3 did not affect the ferricyanide test for reducing sugars or the skatole test for fructose

The Iodine-Sodium Azide Reaction. This reaction:

$$2NaN_3 + I_2 = 2NaI + 3N_2$$

has been used for the gasometric detection of thiocyanates, sulfides and thiosulfates (Refs 35,40,101,117,122,126 & 128). According to Senise (Ref 126) at pH 5.8 as little as 0.035 microgram can be detected by a spot test at a concn limit of 1×10^6. When N_2 was measured with a Lunge nitrometer, the vol showed a linear relationship to the amt of $S^=$, $S_2O_3^=$ and SCN^- present (Ref 122). LeRosen et al (Ref 117), by using equal vols of a satd soln of I_2 in 1% KI and of 10% NaN_3 in 1% starch, obtd a new streak reagent to detect CS_2 by bleaching of the blue color. The effect of catalysts on the iodine-azide reaction has received considerable attention from many investigators (Refs 35,40,70,91, 121,141,145,153 & others). Sulfhydryl compds as catalysts were listed by Friedmann (Ref 70),and thioureas were given by Feigl (Ref 171) and Kayama (Ref 121). The kinetics of the catalytic activity of cysteine and related compds was studied by Whitman & Whitney (Ref 153). Awe & Naiyoks (Ref 141) detd the catalytic order of effectiveness of S compds and noted that the reaction did not take place in acid media. Feigl & Chargov (Ref 35) used the iodine-azide reaction to detect small quants of CS_2 and for the detn of azides. According to Moss (Ref 147) Niello, the blk metallic-like mixt of the sulfide of Cu, Ag & Pb, used to inlay ornamental designs engraved in metal, can be identified by its ability to decomp catalytically a soln of NaN_3 in iodine

Uses of Sodium Azide. The principal use of NaN_3 in the expl ind is in the prepn of alkali alkaline earth and other azides (Refs 37,38, 42,96,113,201,% others) (See Lead Azide, Plant Manufacture, etc). Meissner(Ref 44) used equiv quants of NaN_3 and a heavy metal salt, such as Pb acetate, for the continuous prepn of LA. Matter (Ref 33) found that NaN_3 was freed from carbonates by the addn of aq

solns of hydoxides or salts of alkaline earth metals, such as these of Ba, and this NaN_3 was then suitable for use in the prepn of other metal azides. Buell (Ref 16) proposed as a priming chge for expls a mixt of NaN_3/-$KClO_3/Sb_2S_3$ - 35/30/35%

NaN_3 is used as an initiator for emulsion polymerization (Ref 137), as a cellulating agent (Ref 134) and as a retarder (Ref 185) in the manuf of sponge rubber. The addn of NaN_3, an alkali bicarbonate and an alkali to form a compn of pH 9-12 prevents or reduces plating out or coagulation of styrene and butadiene latexes stored in contact with metals (Ref 162). NaN_3 is used also to decomp nitrites in the presence of nitrates (Ref 172). The rate of nitrite decompn is increased with an increase in azide concn. Acosta (Ref 172) detd the optimum ratio to be C_{NaN_3}/C_{NaNO_2} = 3.9. Compds of the structure R_2R (→O) (→NH) have been prepd from the corresponding sulfoxide and NaN_3 + H_2SO_4 in chlf soln (Ref 171):

$$(CH_3)_2S{\rightarrow}O + HN_3 \rightarrow (CH_3)_2S\begin{matrix}\nearrow O\\ \searrow NH\end{matrix} + N_2$$

According to Black & Kleiner (Ref 112), encouraging therapeutic effects, without toxicity, were exerted by NaN_3 and some other compds in 31 cases of advanced cancer and leukemia in man. Cudkowicz (Ref 175) found that NaN_3 and hydroxylamine reduced growth of transplantable tumors up to 50%. NaN_3 has been used to some extent in the treating of wounds under conditions where the slow oxidizing action is considered desirable in inhibiting growth of anaerobic bacteria (Ref 114). Jones (Ref 182) reported that NaN_3 and merthiolate solns will preserve blood typing serums if they are kept sterile, whereas, under ordinary storage conditions untreated serums deteriorate

NaN_3 prevented fungus growth (Ref 193) which caused darkening of sake cake (Japanese beer). Fales (Ref 146) noted that the effect of NaN_3 on alcoholic fermentation was to increase efficiency in the conversion of glucose to fermentation products. NaN_3 is also useful as a wine preservative, inhibiting or preventing growth of microorganisms (Ref 177). The enzymic oxidation of polyphenols, causing red stain in pulp wood (Ref 170), and enzymic oxidation of lignin can be inhibited or prevented by NaN_3 (Ref 180). Since NaN_3 inhibits microbiological reactions in soil (Ref 123), its use in soil has had the following effects: a)inhibiting biological oxidation of iron (Ref 119) b) inhibiting oxidation of manganese (Ref 186) c)markedly inhibiting pyruvic oxime oxidation (Ref 136) d)completely inhibiting biological oxidation of arsenite to arsenate (Ref 151) and e) useful in the treatment of tobacco shank in soil but with some other toxic effects (Ref 191). Hill et al (Ref 157) proposed using NaN_3 to control the growth of weeds in plant beds while Wesenberg (Ref 49) used NaN_3 in a mixt with mashed potatoes to combat pests, such as insects or rats

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& CA **11**, 2176(1917) 18)L.Wöhler & F. Martin, ZAngChem **30** I, 33-9(1917); JSCI **36**, 570(1917) & CA **11**, 3432(1917) 19)B. P.Orelkin et al, ZhFizChem **49**, 82-7(1917) & CA **18**, 1254-5(1924) 20)E.Moles, JChim-Phys **16**, 401-4(1918) & CA **13**, 1665-6(1919) 21)E.Oliveri-Mandalà & G.Comella, Gazz **52** I, 112-5(1922) & CA **16**, 2089(1922) 22) R.Stollé & A. Netz, Ber **55**, 1297(1922) & CA **16**, 3899(1922) 23)A.J.Currier & A.W. Browne, JACS **44**, 2849-54(1922) & CA **17**, 501(1923) 24)E.Briner & P.Winkler, JChim-Phys **20**, 201-16(1923); Helv **6**, 429-35(1923) & CA **17**, 2841(1923) 25)E.Moles & J.M. Clavera, ZPhysChem **107**, 423-35(1923) & CA **18**, 1217-8(1924); E.Moles, AnalesSoc-EspañFisQuím **26**, 133-5(1928) & CA **22**, 3072(1928) 26)D.A.Spencer, JCS **127**, 216-24(1925) & CA **19**, 1106(1925) 27)S.B. Hendricks & L.Pauling, JACS **47**, 2904-8 (1925) & CA **20**, 318(1926) 28)A.W.Browne & F.Wilcoxon, JACS **48**, 682-90 (1926) & CA **20**, 1185(1926) 29)R.Suhrmann & K.Clusius, ZAnorgChem **152**, 52-8(1926) & CA **20**, 1962 (1926) 30)J.A.Cranston & A.Y.Livingstone, JCS **1926**, 501 & CA **20**, 2439-40(1926) 31) K.A.Hofmann & U.Hofmann, Ber **59**, 2574-9 (1926) & CA **21**, 870(1927) 32)F.Wilcoxon & B.Grotta, USP 1628380(1927) & CA **21**, 2137(1927) 33)O.Matter, BritP 300401(1927) & CA **23**, 3933(1929) 34)W.Biehler, Arch ExptlPathPharmakol **126**, 1-9(1927) & CA **22**, 823(1928) 35)F.Feigl & E.Chargov, ZAnal-Chem **74**, 376-80,(1928) & CA **22**, 4083-4 (1928) 36)E.Kayser, ZAngChem **41**, 49(1928) & CA **22**, 4819(1928) 37)Mellor **8** (1928), 345-7 38)Gmelin, System No **21** (1928), 249-53 39)C.Naegeli et al, Helv **12**, 227-61 (1929) & CA **23**, 2418(1929); Helv **15**, 49-59 & 60-75 (1932) & CA **26**, 2430-1(1932) 40) F.Feigl et al, Mikrochemie **7**, 10-20(1929) & CA **23**, 4421(1929) 41)A.Petrikalns & J. Hochberg, ZPhysChem **3**, Abt B, 217-28(1929) & CA **23**, 3628(1929) 42)W.Hoth & G.Pyl, ZAngChem **42**, 881-91(1929) & CA **23**, 5547 (1929) 43)W.Moldenhauer & H.Möttig, Ber **62**, 1954-9(1929) & CA **24**, 1300(1930) 44) J.Meissner, BritP 359659(1929) & CA **26**, 4920(1932) 45)W.Biehler, KnollsMitt **1927**, 21-6; BerGesPhysiolExptlPharmakol **49**, 829 (1930) & CA **24**, 659(1930) 46)P.Günther & W.Perschke, JCS **1930**, 100-4 & CA **24**, 2659 (1930) 47)K.Fredenhagen & G.Candenbach, ZPhysChem **146**, AbtA, 245-80(1930) & CA **24**, 2938(1930) 48)H.Wattenberg, Ber **63B**, 1667-72(1930) & CA **24**, 4998(1930) 49)G. Wesenberg, USP 1819399(1931) & CA **25**, 5739(1931) 50)E.Justi, AnnPhys [5] **10**, 983(1931) & CA **26**, 24(1932) 51)J.Hoffmann, ZAnorgChem **201**, 175-6(1931) & CA **26**, 933 (1932) 52)R.H.Müller & G.C.Brous, JChem-Phys **1**, 482-9(1933) & CA **27**, 4481(1933) 53)H.Weinhaus & H.Ziehl, Ber **65B**, 1461-7 (1932) & CA **26**, 5911(1932) 54)J.Nelles, Ber **65B**, 1345-7(1932) & CA **26**, 5925(1932) 55)R.Stollé & H.Storch, JPraktChem **135**, 128-36(1932) & CA **27**, 725(1933) 56)R. Stollé & F.Hanusch, JPraktChem **136**, 9-14 & 120(1933) & CA **27**, 1632 & 1885 (1933) 57)R.Stollé et al, JPraktChem **137**, 327-38 (1933) & CA **27**, 4233(1933) 58)V.K. Semenchenko & V.V.Serpinskii, ZhObsch-Khim **3**, 470-7(1933) & CA **28**, 1605-6(1934) 59)C.D.West, ZKrist **88**, 97-115(1934) & CA **28**, 5734(1934) 60)J.Wohlgemuth, CR **199**, 601-3(1934) & CA **28**, 7129(1934) 61)M. Danysz & M.Zyw, ActaPhysPolon **3**, 485-92 (1934) & CA **31**, 3781(1937) 62)L.F.Audrieth, ChemRevs **15**, 169 & 202(1934) 63)M.S. Newman, JACS **57**, 732-5(1935) & CA **29**, 3665-6(1935) 64)Westfälisch-Anhaltische Sprengstoffe-AGChemFab, GerP 619017(1935) & CA **30**, 582(1936) 65)Dynamit-AG, vorm A.Nobel & Co, GerP 619753(1935) & CA **30**, 1527(1936) 66)V.O.Kuz'min & M.I.Zemlyan-skii, MemInstChemUkrainAcadSci **2**, 183-9 (1935) & CA **31**, 3467(1937) 67)Y.Yushchenko, MemInstUkrainAcadSci **2**, 195-205(1935) & CA **31**, 3467(1937) 68)L.K.Frevel, JACS **58**, 779-82(1936) & CA **30**, 4756(1936) 69)W.E. Garner & D.J.Marke, JCS **1936**, 657-64 & CA **30**, 6270-1(1936) 70)E.Friedmann, JPrakt Chem **146**; 179-92(1936) & CA **30**, 8158(1936) 71)R.Audubert & H.Muraour, CR **204**, 431-2 (1937) & CA **31**, 2518(1937) 72)R.Audubert, CR **204**, 1192-4(1937) & CA **31**, 4210(1937);

CR **206**, 748-50 (1938) & CA **32**, 3696(1938) 73)G.Labruto & A.Landi, Gazz **67**, 213-6 (1937) & CA **31**, 8517(1937) 74)V.N.Rosenberg, FiziolZh **22**, 896-900(1937) & CA **33**, 747(1939) 75)C.S.Venkateswaran, Proc IndianAcadSci **7A**, 144-5(1938) & CA **32**, 5703(1938) 76)R.Audubert & J.Mattler, CR **206**, 1639-41(1938) & CA **32**, 6155(1938) 77) J.Mattler, JChimPhys **35**, 277-85(1938) & CA **33**, 1217(1939) 78)K.Hojendahl, KglDanske-VidenskabSelskabMath-FysMedd **16**, No 2, 1-58(1938) & CA **32**, 7319-20(1938) 79)A. Petrikalns & B.Ogrins, Radiologica **3**, 201-37(1938); ChemZtr **1939** II, 327 & CA **35**, 3145-6(1941) 80)R.Audubert, TrFaradSoc **35**, 197-204(1939) & CA **33**, 2806-7(1939) 81)M.Bassiere, CR **208**, 659-61(1939) & CA **33**, 3229(1939) 82)R.Audubert & E.T. Verdier, CR **208**, 1984-6(1939) & CA **33**, 6162 (1939) 83)E.Waltmann, GerP 680749(1939) & CA **36**, 2096-7(1942) 84)A.W.Browne, InorgSynth **1** (1939), 79-81 & CA **36**, 2488 (1942) 85)P.S.Pel'kis & T.S.Dunaevs'ka, MemInstChemAcadSciUkrain, SSR **6**, No 2, 163-77 (Eng 179-80) (1940) & CA **34**, 5829-30(1940) 86)S.Sato & T.Sogabe, BullInst-PhysChemRes(Tokyo) **19**, 943-50(1940); Sci-PapersInstPhysChemRes (Tokyo) **38**, 174-82 (1941) & CA **35**, 3156(1941) 87)R.Audubert & C.Racz, CR **210**, 217-9(1940) & CA **34**, 2709(1940); Bull Fr **7**, 907-14(1940) & CA **36**, 2209(1942) 88)N.Yui, BullInstPhysChem Res(Tokyo) **20**, 390-8(1941); EngAbstr in SciPapersInstPhysChemRes (Tokyo) **38**, Nos 1034-5(1941) & CA **36**, 1230-1(1942) 89)W. E.Garner, Chim & Ind **45**, Suppl to No 3, 111-8(1941); ChemZtr **1942** II, 365-6 & CA **37**, 4571-2(1943) 90)E.W.Hurst, Australian JExptlBiolMedSci **20**, Pt 4, 297-312(1942) & CA **37**, 2074(1943); MedJAustralia **1941** II, 661-6 & CA **36**, 6646-7(1942) 91)F.Feigl, AnaisAssocQuim Brasil **1**, 234-42(1942) & CA **37**, 1916(1943); JChemEd **20**, 137-41 (1943) & CA **37**, 2294(1943) 92)M.Bonnemay & E.T.Verdier, CR **214**, 228-30(1943) & CA **37**, 3341(1943) 93)E.T.Verdier, CR **214**, 617-9(1942) & CA **37**, 6194(1943) 94)M. Bonnemay, CR **214**, 826-8(1942) & CA **38**, 3540(1944); CR **215**, 65-7(1942) & CA **38**, 5457(1944); CR **216**, 52-4, 154-6 & 230-2 (1943) & CA **38**, 4868(1944) & CA **39**, 1594 (1945); JChimPhys **41**, 18-41(1944) & CA **39**, 3205(1945) 95)P.Jolibois & J.Clérin, BullFr **9**, 840-1(1942) & CA **38**, 3912(1944) 96)Davis(1943), 428-9 97)M.Straumanis & A.Cirulis, ZAnorgChem **251**, 341-54(1943) & CA **37**, 6573(1943) 98)M.Straumanis & A.Cirulis, ZAnorgChem **252**, 9-23 & 121-5 (1943) & CA **38**, 1701-2 & 3563-4(1944) 99) H.Moler, Helv **26**, 121-9(1943) & CA **38**, 299 (1944) 100)M.Bonnemay, CR **216**, 52-4 (1943) & CA **38**, 4868(1944) 101)H.Goto & T.Shishiokawa, JChemSocJapan **64**, 515-20 (1943) & CA **41**, 3392(1947) 102)M.Bassière, MSCE **30**, 33-46(1943) & CA **41**, 5374(1947) 103)L.T.Fairhall et al, USPublicHealthRpt **58**, No 15, 607-17(1943) & CA **37**, 3833(1943) 104)M.Bonnemay & E.T.Verdier, JChimPhys **41**, 113-24(1944) & CA **40**, 2384(1946) 105) A.Delay et al, BullFr **12**, 581-7(1945); CR **219**, 329-33(1944) & CA **40**, 2386 & 4273-4 (1946) 106)M.F.Acken & F.W.Filbert, USP 2373800(1945) & CA **39**, 3129(1945) 107)J. D.Parsons, PBRpt 12663(1945), 20 108)R. Audubert & J.Robert-Lung, JChimPhys **43**, 127-33(1946) & CA **41**, 1916(1947); CR **222**, 1228-9(1946) & CA **40**, 5626(1946) 109)M. W.Miller & L.F.Audrieth, InorgSynth **2** (1946), 139-41 & CA **40**, 6356(1946) 110)A.W. Adamson, JACS **69**, 2564(1947) & CA **42**, 475(1948) 111)L.Kahovec & K.W.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666 (1948) 112)M.M.Black & I.S.Kleiner, Cancer-Res **7**, 717-8(1947) & CA **42**, 7868(1948) 113)Anon, PB Rpt 95613(1947) 114)Kirk & Othmer **2** (1948), 81 115)S.Lormeau, CR **226**, 247-9(1948) & CA **42**, 2874(1948) 116) J.D.Graham et al, JIndHygToxicol **30**, 98-102(1948) & CA **42**, 3571-2(1948) 117)A.L. LeRosen et al, AnalChem **22**, 809-11(1950) & CA **44**, 8816-7(1950) 118)G.O.Henneberry & B.E.Baker, CanJRes **28B**, 345-51(1950) & CA **45**, 56(1951) 119)H.Gleen, Nature **166**, 871-2(1950) & CA **45**, 3105-6(1951) 120)Yu N.Sheinker & Ya K.Syrkin, IzvestAkadN, Ser Fiz **14**, 478-87(1950) & CA **45**, 3246(1951)

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SODIUM AZIDE PLANT, ANALYTICAL PROCEDURES. The sodium azide plant of the Kankakee Ordnance Works (KOW), Joliet, Illinois, operated by the US Rubber Co, used as starting materials anhydrous ammonia, sodium ferric nitrate (catalyst) and nitrous oxide

Following are the analytical procedures used at the KOW plant, as described in Ref 7

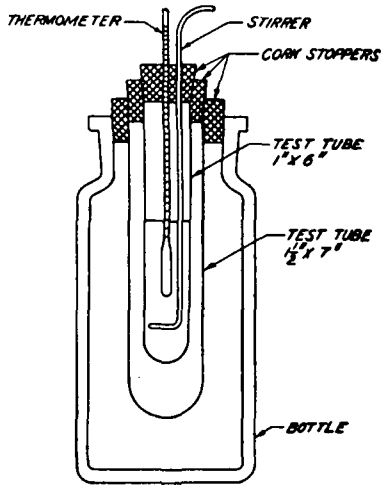
I)Anhydrous Ammonia Synthetic. The methods of analysis described in US Spec JAN-A-182 were used (See under Ammonia, Analytical Procedures.)

II)Sodium was in the form of bricks packed in barrels or tank cars. It had the following props: purity – min 99.95%, metallic Ca – max 0.04%, chlorides – max 0.005%, mp 97.6°, bp 880° and sp gr 0.970. The tests used at KOW are described in Ref 7, pp 25a to 25d. These tests are not included in US Spec JAN-S-328(1946) which requires only the following tests:

a)*Foreign Matter.* When examined visually the metal must be substantially free of foreign impurities

b)Solidification Point. The value must be 97.0 ± 2° when detd in the apparatus shown on fig(next page)

Procedure: Free the metal from any adhering oil or other impurities by shaving a thin layer. Fill a clean, dry, 1" × 6" Pyrex test tube, about ⅓ full with the clean sample and plunge the lower part of the tube in an oil bath at 105°. When the temp of molten sodium



reaches ca 105°, remove the tube, wipe it off and quickly assemble the apparatus. Stir continuously until the temp stops falling and then starts to rise. Stop stirring and note the max temp, which is known as "uncorrected setting point". After this the temp falls slightly and remains stationary for ca 1 min. Record this temp as the "solidifaction point" of Na

III)Ferric Nitrate(catalyst in reaction between Na and NH_3) was procured in No 50 drums. It was of analytical grade and had the following props: insoluble matter – max 0.01%, chloride – max 0.001% and sulfate 0.10%. No analysis was made at KOW (Ref 7)

IV)Nitrous Oxide was procured in car load lots of 200 50 lb cylinders. It was USP grade, free of impurities to the lowest practical amt and which were designated on the label of each container. No analysis was made at KOW (Ref 7)

V)Wringer Cake is a solid contg ca 99% Na azide,obtained after centrifuging the slurry obtained on evaporation of crude Na azide soln, as described under Manufacture of Sodium Azide. Basic unit weighs 100-250 lb and a 100-g sample is taken of each cake immediately after it is wrung and washed. A strip of cake of the width of the centrifuge was knocked down and sampled by means of a cup. The requirement was that the cake contain less than 1% NaOH and the test was as follows (Ref 7, p 26)

Procedure: Weigh a 10.00-g sample on a tared 4" watch glass and brush up all spilled crysts immediately, because they are very poisonous. Transfer the sample to a 400-ml beaker contg ca 150 ml distd w neutral to phpht and stir until completely dissolved. Titrate rapidly to colorless end-print with 0.1N sulfuric acid. Avoid overrunning the end point, which causes evoln of the very poisonous gas HN_3

$$\% \text{ NaOH} = \frac{A \times N \times 0.0401 \times 100}{W}$$

where A = burette reading, N = normality of H_2SO_4, and W = wt of sample

Note: The cake must contain at least 99% NaN_3 on the dry basis. A small amt of NaOH is not harmful but traces of Na carbonate produce Pb carbonate which inhibits the formation of desirable form of LA crysts. The presence of any appreciable carbonate will also tend to lower the purity of the final product. The cake must be practically free of NaCl

VI)First Mother Liquor is the liquid wrung from one evaporator chge (See under Manufacture of Sodium Azide) of normal or average analysis: NaOH (actual) 34.4%, NaN_3 (actual) 2.5% and Na_2CO_3 (actual) 0.2%. A 25-ml sample was dipped by hand from the catch tank after the charges had been centrifuged and all samples composited for one week. At the end of each month, an 8-oz sample was taken from the storage tank for monthly inventory and was analyzed in the same way as the weekly composites

Procedures:

a)NaN_3 Content. Pipette exactly 10.00 ml of sample into an accurately tared glass-stoppered weighing bottle and accurately weigh it. Use this weight for all 10.00-ml samples and save the contents of the bottle for the detn of NaOH (see proced c). Using the same

pipette, transfer another 10.00-ml sample to a Kieldahl distn flask and add 300 ml of freshly boiled distd w. Add a few boiling chips to prevent bumping and close the flask with a stopper equipped with a spray trap and a closed separatory funnel. Place the flask on a cold electric heating unit. Transfer exactly 50.00 ml of approx N/3 NaOH soln to a 1000-ml Erlen receiving flask and dilute with ca 200 ml of freshly boiled distd w. Assemble the flasks and a condensing apparatus, closing the receiving flask with a tight-fitting rubber stopper through which passes the tube (adapter) from the condensing app and a tube connected to a U-tube contg soda-lime. The tip of adapter must be slightly immersed in the contents of the receiving flask. Add 20 ml of 40% sulfuric acid to the separator funnel of Kjeldahl flask

Heat the liq in the Kjeldahl flask and continue boiling until most of the air has been driven from the apparatus and replaced with water vapor. This will be shown by the disappearance of air bubbles escaping from the tip of adapter tube in the receiver. Through the separatory funnel, slowly add 20 ml of 40% sulfuric acid, making sure that the resulting partial vacuum does not cause the receiver liq to back up more than half way in the condenser. Close the stopcock of the separatory funnel

Caution. Never add the acid before the system is filled with vapor, as high concns of hydrazoic acid may cause explns if air is present. Be careful to avoid leakage of acid vapor, as HN_3 is very poisonous

Continue the distn until ca 200 ml of the liq is distilled into the receiver

The following reactions takes place: in the Kjeldahl flask, $2NaN_3 + H_2SO_4 \rightarrow 2HN_3 + Na_2SO_4$, and in the receiver, $HN_3 + NaOH \rightarrow NaN_3 + H_2O$

Disconnect the inlet to the condenser and remove the heater. Elevate the adapter of the condenser about 2" out of the receiver, and with a stream of freshly boiled distd w, rinse the condenser and adapter 3 times into the receiver. Also, wash down the outside of the condenser end into the receiver.

Remove the receiver, rinse its inside and add 3 drops of phpht indicator. Titrate the excess of NaOH with approx N/3 HCl to just disappearance of pinkish coloration. The following reactions take place:

$$NaOH + HCl \rightarrow NaCl + H_2O$$

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

Save contents of the receiver for the next proced

Calculation:

$$\text{Apparent \% } NaN_3 = \frac{(AB - CD) \times 0.0650 \times 100}{W}$$

where A = ml NaOH soln used in receiver, B = its normality, C = ml HCl used in titration, D = its normality, W = wt of 10 ml sample in the weighing bottle, and $0.0650 = NaN_3/1000$ (Refs 4 & 7)

b)Na_2CO_3 Content. To the titrated distillate in the receiver add exactly 10.00 ml of approx N/3 NaOH (which makes a total of 60 ml NaOH in the receiver) and then 10 ml of neutral 10% $BaCl_2$ soln. Wash down with freshly boiled distd w, stopper, gently shake and allow to stand for 3 mins. Titrate dropwise with approx N/3 HCl using continuous swirling until pinkish color of phpht just disappears. The following reactions take place:

$$NaHCO_3 + NaOH \rightarrow Na_2CO_3 + H_2O$$

$$Na_2CO_3 + BaCl_2 \rightarrow BaCO_3 + 2NaCl$$

Run a blank detn using exactly 10.00 ml of N/3 NaOH, 10 ml of 10% $BaCl_2$ soln and ca 100 ml of freshly boiled distd w

Calculation:

$$\text{Actual \% } Na_2CO_3 = \frac{(B - S) \times N \times 0.1060 \times 100}{W}$$

where B = ml N/3 HCl required for blank, S = ml HCl required for distilled sample, N = normality of HCl soln, W = wt of 10-ml sample and $0.1060 = Na_2CO_3/1000$ (Refs 4 & 7)

c)NaOH Content. Transfer quantitatively the

contents of the weighing bottle (See beginning of proced a) to a 100 ml-vol flask and dil to the mark with freshly boiled distd w. Pipette a 10.00-ml aliquot into a 500-ml Erlen flask, dil with ca 100 ml w and titrate with N/3 H_2SO_4, using phpht as an indicator:

$$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

$$2Na_2CO_3 + H_2SO_4 \rightarrow 2NaHCO_3 + Na_2SO_4$$

Calculation:

$$\text{Apparent \% NaOH} = \frac{\text{ml } H_2SO_4 \times N \times 0.0400 \times 100}{\text{Wt of sample in 10 ml aliquot}}$$

N = normality of N/3 H_2SO_4, and 0.0400 = NaOH/1000

Actual % NaOH = Apparent % NaOH – (0.3774 × %Na_2CO_3), where 0.3774 = NaOH/Na_2CO_3

Actual % NaN_3 = Apparent % NaN_3 – (0.6134 × % Na_2CO_3), where 0.6134 = NaN_3/Na_2CO_3 (Ref 7)

VII)Second Mother Liquor, obtained as described under Manufacture of Sodium Azide, normally contained ca 46.3% NaOH, 1.0% NaN_3 and 0.15% Na_2CO_3. An 8-oz sample was taken of each chge from the catch tank when washing of the wringer cake was completed. If the NaN_3 content was 2.00% or less, the liq was pumped to a storage tank from which a 25-ml sample was taken and composited for 1 week. At the end of each month, an 8-oz sample was taken from the storage tank for monthly inventory

Procedures:

a)NaN_3 Content – *same* as in item VIa

b)Na_2CO_3 Content – same as in item VIb

c)NaOH Content – same as in item VIc

d)Specific Gravity. Fill a hydrometer jar about ½ full of sample and det sp gr using a hydrometer of range 1.4 – 1.6. The usual reading was ca 1.52 (Ref 7)

VIII)First Clear Liquor, obtained as described under Manufacture of Sodium Azide, normally contained 11-12% NaOH, 9-10% NaN_3 and 0.15% Na_2CO_3. A 25 ml sample was taken of every chge from the ammonia evaporator at the completion of evaporation and composited for one week. A month-end inventory sample (8 oz) was taken from storage at the end of each month

Procedures:

a)NaOH Content. Pipette exactly a 10.00 ml sample into an accurately tared glass-stoppered weighing bottle, close the bottle and accurately weigh the ensemble. Record the wt of the sample. Using the same pipette, transfer another 10.00-ml sample into a 500-ml Erlen flask contg some boiling chips to prevent bumping on boiling and add ca ¾" of neutral distd w. Boil on a hot plate under the hood with the suction fan on until all ammonia is driven off. (Test with moist red or neutral litmus paper held over the mouth of the flask – no change in color indicates the absence of ammonia fumes)

Wash down the sides of the flask, cool and titrate with N/3 H_2SO_4 in presence of 3 drops of phpht indicator

Calculation:

$$\text{Apparent \% NaON} = \frac{\text{ml } H_2SO_4 \times N \times 0.0400 \times 100}{\text{Wt of sample}}$$

where N = normality of N/3 H_2SO_4, and 0.0400 = NaOH/1000

b)NaN_3 Content. Alkalize the above sample with NaOH and transfer quantitatively to a Kjeldahl distillation flask. Dilute contents of the flask to 300 ml, add a few boiling chips and proceed as described in proced a) under item VI (First Mother Liquor)

Calculation – same as in proced a of item VI

c)Na_2CO_3 Content – same as in proced b) of item VI (Ref 7)

IX)Lime Treatment Tank contained liquor obtained from the wringer producing the second wringer cake. The liquor normally contained ca 9-10% NaN_3, 10-13% NaOH and up to 0.75% Na_2CO_3. Its sp gr by hydrometer was ca 1.2. An 8-oz sample was taken, and after analysis according to item VI (First Mother Liquor), the amt of lime necessary to ppt the carbonate as $CaCO_3$ was calcd and

added to the tank. Then the contents of the tank was filtered and the filtrate was the second clear liquor. (See next item.) (Ref 7)

X)Second Clear Liquor, obtained on filtering the contents of the lime treatment tank, had almost the same compn as before, except that its $\%Na_2CO_3$ was reduced to ca 0.25%. A 25-ml sample was taken after each lime treatment and filtered, and all samples were composited for one week. At the end of each month, an 8-oz sample was taken from storage tank for monthly inventory, and was analyzed in the same way as were the weekly composites:

Procedures:

a)NaN_3 Content – same as the proced a) of item VI (First Mother Liquor)

b)*Na_2CO_3 Content* – same as the proced b) of item VI

c)NaOH Content. Wash the contents of the weighing bottle contg the 10.00-ml sample into a 500-ml Erlen flask, dil with ca 75 ml dist w and titrate with N/3 H_2SO_4 in presence of phpht indicator

Calculation:

$$\text{Apparent \% NaOH} = \frac{H_2SO_4 \text{ soln} \times N \times 0.0400 \times 100}{\text{Wt of sample}}$$

where N = normality of H_2SO_4, and 0.0400 = NaOH/1000

Other calcns are the same as in item VI (First Mother Liquor) (Ref 7)

XI)Crude Sodium Azide Liquor was obtained from the drowning tank of the autoclave. It usually contained 7-10% NaOH, 9-11% NaN_3, 0.1-0.5% Na_2CO_3 and some ammonia. A 250-ml sample of each drowning tank chge was taken from the crude scale tank. The bottle was stoppered and kept in the lab's refrigerator until ready for analysis

Preparation of Samples. Prepare a composite of samples collected in a 24-hour period, taking for each increment of the composite a weight proportional to the total weight of the corresponding sample. The entire composite should be less than the volume of an 8-oz bottle. Tare the bottle on a torsion balance and add to the tare the weight desired for the first increment. Pour the corresponding sample into the bottle to balance, then increase the tare by the weight of the next increment, and so on. Conduct all this work very rapidly in order to prevent excessive loss of ammonia. Stopper the composite sample tightly, shake and place in the refrigerator until ready for use.

Procedures:

a)NaN_3 Content
b)NaOH Content
c)Na_2CO_3 Content
Same as in item VI (First Mother Liquor)

d)NH_3 Content. Add gradually 100 g of the composite sample to 40% sulfuric acid and dilute to 1 l. This must be done under a hood so as to prevent exposure to HN_3 fumes. Pipette a 10.00-ml aliquot into a Kjeldahl distilling flask and dil to 300 ml with w:

$$H_2SO_4 + 2NaN_3 \rightarrow 2HN_3 + Na_2SO_4$$

$$H_2SO_4 + 2NH_4OH \rightarrow (NH_4)_2SO_4 + 2H_2O$$

Arrange the apparatus as described in proc a) of item VI, add from a separator funnel 20.0 ml of 5% NaOH:

$$(NH_4)_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2NH_4OH$$

and distil into 80.00 ml N/3 HCl contained in a receiver, until ca 100 ml of liq is left in the Kjeldahl:

$$NH_4OH \rightarrow NH_3 + H_2O$$

$$NH_3 + HCl \rightarrow NH_4Cl$$

Break the connections, wash the app with neutral distd w into the receiver and titrate the excess of acid with N/3 NaOH, using methyl-red as indicator

Run a blank in exactly the same manner but without sample

Calculation:

$$\%NH_3 = \frac{(B-S) \times N \times 0.0170 \times 100}{\text{Wt of 10 ml aliquot}}$$, where

B = ml N/3 NaOH required for blank, S = ml N/3 NaOH required for sample, N = normality of NaOH and 0.0170 = NH_3/1000 (Ref 7)

XII)Refined Sodium Azide Liquor. See item IV under Lead Azide Plant, Analytical Procedures

XIII)Sodium Azide Feed Tank. See item V under Lead Azide Plant, Analytical Procedures

XIV)Sodium Azide Dilution Tank. See item VI under Lead Azide Plant, Analytical Procedures

XV)Sodium Azide, Crystalline. If NaN_3 was not manufd at KOW, but purchased outside, it had to comply with the following US Army requirements and tests as listed in the Pic Arsn Tentative Spec PXS-764(Rev 2), Jan 9, 1942, which became Military Spec MIL-S-20552(1951) (Ref 4):

a)NaN_3 Content. Same as proced a) in item VI, except that the sample introduced in the Kjeldahl flask is ca 1 g and the amts of reagents in the receiving flask (which has a capacity of 500 ml) are 50 ml of approx N/3 NaOH and 50 ml of freshly boiled distd w. This test gives % of apparent NaN_3

b)Na_2CO_3 Content – same as proced b) in item VI. This test gives % of actual Na_2CO_3. In order to calc % actual NaN_3 subtract from the % apparent NaN_3 the % actual Na_2CO_3 multiplied by 0.6134

c)Alkalinity of NaN_3. Dissolve a weighed 5 g portion of the sample in 100 ml of freshly boiled and cooled distd w which is contained in a 250 ml beaker. Add a few drops of phpht indicator and if the soln is alkaline, titrate with approx N/10 HCl and correct for a blank detn with the same quantities of w and indicator

Calculation:

$$\% \text{ Alkalinity as NaOH} = \frac{\text{ml HCl} \times N \times 0.040 \times 100}{W}$$

where N = normality of HCl, W = wt of sample, and 0.040 = NaOH/1000 (Ref 7)

Colorimetric Determination of Sodium Azide in Aqueous Ammonia (See under Manufacture of Sodium Azide)

After detg the sp gr of the aq ammonia sample with a hydrometer, place a 200-ml sample in a 400-ml beaker and boil on a hot plate in the hood until most of the NH_3 has boiled off (ca ½ hr). Add 2 drops of phpht and titrate with N/3 HCl until the pink color just disappears. Transfer to a 200-ml vol flask and adjust to the mark with neutral distd w. Add 1 ml of 10% $FeCl_3$ soln to each of two 100-ml Nessler tubes. Pipette 10-ml of the neutralized sample to one of the Nessles tubes and dil to 100-ml mark with w. Stopper and mix. Fill also the other tube (contg only $FeCl_3$) slightly below the mark and stopper and mix. Add to the 2nd tube dropwise, from a 10-ml burette, the standard soln contg 0.001 g NaN_3 per 1 ml (prepd by dissolving 1 g CP $NaNa_3$ in 1 l of distd w) until the intensity of coloration matches that of the sample. For this, stopper and mix well after each addn and compare the coloration by holding the tubes side by side against a white background

$$\% \text{ } NaN_3 = \frac{(\text{ml of std } NaN_3) \times 0.001 \times 100}{10 \times \text{Sp gr}}$$

(Ref 7, p 66)

XVII)Sodium Azide, Technical, Prepared From Hydrazine and Ethyl Nitrate

Impurities present in tech NaN_3 may be subdivided into w-insol, such as carbonates and oxides of heavy metals and w-sol, such as carbonates of Na & Ca, various nitrates and chlorides, hydrazine salts and ammonium salts

Barlot & Marsaule (Ref 6) recommended a method of analysis based on the following reaction:

$$NaN_3 + NaNO_2 + 2HCl \rightarrow 2NaCl + H_2O + N_2 + N_2O$$

. This method is a modification of method originated by Glen & Rvell

Procedures:

a)Insoluble Matter (IM). Dry the sample to const wt at 90°, weigh exactly 10.000 g and dissolve in 100 ml of distd w. Filter through a tared fritted glass crucible and transfer the

filtrate to a 250-ml vol flask (Filtrate No 1). Wash the residue in the crucible with dist w and transfer the washings to the same vol flask. Dry the crucible with residue to const wt at 100° and calc % IM

b)Calcium Carbonate Content. As $CaCO_3$ is appreciably sol in w contg azides, it might be present in the filtrate No 1 of proced a). In order to det $CaCO_3$, take 50 ml of Filtrate No 1 (which corresponds to 2 g of solid material) and dil it to 250 ml. Transfer a 50-ml aliquot (which corresponds to 2/5 = 0.4 g of solid) to a beaker, add 5 ml of satd aq Amm oxalate and 1 ml of glacial AcOH. The following reaction takes place:

$$(NH_4)_2C_2O_4 + Ca(CH_3COO)_2 \rightarrow 2NH_4(CH_3COO) + CaC_2O_4$$

Filter the ppt of CaC_2O_4 through paper, wash the ppt thoroughly with boiling w and transfer it quantitatively into a 250-ml beaker. Add 100 ml of boiling w and 20 ml of 1:3 sulfuric acid. Titrate the hot mixt with N/10 $KMnO_4$. The following reaction takes place:

$$5CaC_2O_4 + 2KMnO_4 + 8H_2SO_4 \rightarrow 5CaSO_4 + K_2SO_4 + MnSO_4 + 10CO_2 + 8H_2O$$

Calc % $CaCO_3$ from the following formula: $\frac{R \times N \times 0.0500 \times 100}{W}$, where R = ml $KMnO_4$ soln, N = its normality and W = wt of solid sample in aliquot (0.4g)

c)Sodium Carbonate Content. Transfer a 50 ml aliquot of filtrate No 1, corresponding to 2 g of solid sample, to a beaker; heat the liq to boiling and add 5 ml of 5% $BaCl_2$ soln. After keeping on a water bath for ½ hr, filter through a tared sintered glass crucible. Transfer the ppt ($BaCO_3$) quantitatively into the crucible and wash it with w. Dry the ensemble to const wt at 100° and calc % total carbonates as Na_2CO_3 from the formula:

$\frac{W_1 \times 106 \times 100}{197.37 \times W}$, where W_1 = wt of residue and W = wt of sample in aliquot (2g)

Actual % Na_2CO_3 is found by subtracting, from W_1, 1.059 times the amt of $CaCO_3$ found in proced b). Save the filtrate and washings (filtrate No 2) for proced h)

d)Nitrates Content. Take another 50 ml aliquot of filtrate No 1 (see proced a) add ca /g $NaNO_2$, then 5 ml glacial AcOH and heat at bp for ca 15 min to decomp azides (which otherwise interfere). After cooling the soln, add nitron acetate, filter through a tared sintered glass crucible and weigh the residue (nitron nitrate). Calculate % NO_3 from the formula

$$\frac{62 \times W_1 \times 100}{375.38 \times W}$$

where W_1 = wt of nitron nitrate (mw 375.38) and W = wt of solid sample in 50-ml aliquot (2 g)

e)Chlorides Content. Transfer 10 ml of filtrate No 1 (see proced a) to a small beaker, add an excess of 5% $AgNO_3$ soln, then 1 ml of concd HNO_3 and heat. If there is any ppt, filter dry the ppt, weigh and calc chlorides as %NaCl from the following formula

$$\frac{58.45 \times W_1 \times 100}{143.34 \times W}$$

where W_1 = wt of ppt (AgCl) and W = wt of solid sample in 10-ml aliquot (10/25 = 0.4 g)

If only turbidity is present repeat the operations, taking a larger sample

Note: Protect from direct light as much as possible the ppt of AgCl

f)Ammonium Salts Content. Take another 50 ml aliquot of filtrate No 1 (See proced a) and decomp the azides as described in proced d). Cool the soln, neutralize with NaOH and det % ammonia colorimetrically by means of Nessler's reagent

g)Hydrazine Salts are detd by colorimetric method of Pesez & Petit (Ref 2) based on the formation of intense orange-red azine when treated with p-dimethylaminobenzaldehyde. The reaction must be conducted in very dil soln in order to avoid secondary reactions

Procedure. Transfer ca 0.2-g accurately weighed sample to a 100-ml vol flask and add distd w to the mark. Stopper and shake until completely dissolved. Pipette out 5 ml and add 1 ml of the reagent (prepd by dissolving 1 g of p-dimethylaminobenzaldehyde in 50 ml absol alc and 5 ml concd HCl). If hydrazinium ion is present, the soln turns yel at first and then, after 10-15 mins, intensely red-orange. Compare the intensity of coloration of similarly treated solns (standards) contg 0.0001 mg to 1 mg of N_2H_4 per ml

b)Sodium Azide Content. Transfer the filtrate No 2 (see proced c) to a 200-ml vol flask and adjust to the mark. Pipette out 25 ml (which would correspond to 2/8 = 0.25 g of solid sample), dil with 50 ml distd w, add 0.5 g $NaNO_2$ and a few drops of phpht indicator. After making the soln neutral, add exactly 100 ml of N/10 HCl, shake for several minutes and titrate the excess HCl with N/10 NaOH (burette reading R_2). Det the NaOH equiv of the HCl by titrating 100 ml with NaOH (burette reading R_1). Calculate % NaN_3 from the formula:

$$\frac{(R_1 - R_2) \times N \times 0.0650 \times 100}{W \times 2}$$

where N = normality of NaOH, W = wt of sample in 25-ml aliquot, and 0.0650 = NaN_3/1000

It should be noted that if a solid tech Na azide is used as a starting material for the above test it is necessary to remove the carbonates prior to adding $NaNO_2$. This can be done by dissolving the sample in distd w and neutralizing the soln with HCl, using methyl-orange, methyl-yellow or equivalent test paper

Note. Barlot & Marsaule (Ref 6) also describe two other methods for detn of NaN_3:
a)The argentometric method of Volhard and
b)The iodometric method based on the reaction $2NaN_3 + I_2 \rightarrow 2NaI + 3N_2$
Some other methods of analysis of sodium azide are listed in Refs 1,3,5 & 8

Refs on Sodium Azide Plant, Analytical Procedures 1)J.W.Arnold, IEC, AnalEd **17**, 215-17(1945) & CA **39**, 2267(1945) (Assay of NaN_3 by cerate oxidimetry) 2)M.Pesez & A. Petit, Bull Fr **1947**, 122-3 & CA **41**, 5820 (1947) (Detn of hydrazine, applicable to analysis of SA) 3)J.H. van der Meulen, Rec **67**, 600-2(1948) & CA **43**, 1288(1949) (Detn of azides and hydrazoic acid with K permanganate) 4)US Military Spec MIL-S-20552 (29 Dec 1951) (Sodium azide, requirements and tests) 5)E.Werle & R.Fried, BiochemZ **321**, 500-7(1951) & CA **47**, 8125(1953) (A photometric method for detn of NaN_3) 6)J.Barlot & S.Marsaule, MP **35**, 7-13(1953) & CA **49**, 5843(1955) (Analysis of tech SA prepd from hydrazine and ethyl nitrate) 7)B.C.Carlson, "Lead Azide Laboratory Manual" USRubber Co, operator of Kankakee Ordnance Works, Joliet, Ill (1953) 8)Y.Mizushima & S. Nagayama, JIndExplsSocJapan **17**, 113-5(1956) & CA **50**, 16557(1956) (Microdetn of azides by ceric ammonium nitrate) 9)D.G.Young, formerly of Kankakee OW, Joliet, Ill; private communication, 1960 (info on manuf and analysis of NaN_3)

Strontium Diazide (Formerly called Strontium Azoimide or Strontium Trinitride), $Sr(N_3)_2$, mw 171.68, N 48.96%. Col rhmb, hygr crysts; mp decomps at 140° (in vacuo) with evolution of N_2 at 110° (Ref 3); Q_{expln} 295 cal/g (Ref 4), Q_{form} −1.72 to 0.1 kcal/mol (Refs 20 & 23); lattice energy 494 kcal/mol (Ref 23); ionic conductance of solid $Sr(N_3)_2$ obeyed equation $\log k = \log A - (E/2.303RT)$ in which $\log A = -10.70$ and $E = 5.1$ kcal/mol in the temp range 300 to 380°K (Ref 21). The low activation energy for the structure sensitive conductance in K, Ca & Sr azides was assocd with the mobility of surface lattice defects. Sr azide is sol in w (45.8 g/100 g w at 16°); sl sol in alc (0.095% at 16°) and insol in eth (Ref 2). The toxicity of $Sr(N_3)_2$ is not discussed by Sax (Ref 24) but its effects should be considered similar to those of the alkali and alkaline earth azides

Sr azide was first prepd in 1898 by Dennis & Benedict (Ref 1) and in the same year by Curtius & Rissom (Ref 2) by the action of HN_3 on the oxide, hydroxide or carbonate of Sr. Its prepn has also been described by Mellor (Ref 7), Gmelin (Ref 9), Audrieth (Ref 10) and others (Refs 11, 15, 18, 19 & 25). The cryst structure of $Sr(N_3)_2$ was investigated to a limited extent by A.C.Gill (cited in Ref 1) and in detail by Llewellyn & Whitmore (Ref 15) who established its orthorhmb nature as ionic, with a linear sym azide ion, N–N = 1.12Å, and Sr to N distance of 2.63 to 277Å. Kahovec & Kohlrausch (Ref 16) detd, from the Raman Effect, both on cryst powd and in soln, frequencies which corresponded to sym oscillation in a linear triatomic molecule. The spectra emitted during expln of Ca, Ba, Sr & Zn azides were photographed by Petrikaln(Ref 6). According to Garner & Maggs (Ref 13) the threshold for absorption of UV light by N_3 ions in soln and in the solid state is 2600-2700Å and that for photochemical reaction is in the same region

Explosive Properties. Curtius & Rissom (Ref 2) reported that Sr azide decompd violently at 194 to 196° while Wöhler & Martin (Ref 5) observed a temp of 169° for deton of a 0.02 g sample in 5 sec. Under impact of a 2 kg falling wt, a compressed Sr azide sample (0.01 to 0.05g) flamed but did not detonate. Later studies by Haid et al (Ref 8) showed that Sr azide, in contact with a direct flame, behaved similarly to Ca azide but it did not ignite as easily and it burned more slowly than Ca azide. On heating a 0.2g sample on molten Wood's metal, it ignited between 190 & 200° and then burned with expl violence. A 300 g sample, confined in an iron box 6 × 6 × 6 cm of 1 mm wall thickness, exploded in ½ to 1 min when heated in a flame. Haid et al (Ref 6) also reported that Sr azide ignited from friction on rubbing in a mortar, it exploded on impact with a hammer and in the Lead Block test gave a value of 30 ml. Its expl strength is considered approx equal to that of Ba azide

Thermal Decomposition. In a study of the thermal decompn of Sr azide between 99 and 124°, Maggs (Ref 12) observed a marked induction period, followed by an acceleration of the reaction. Decompn occurred in three stages, with an activation energy value of 20 kcal/mol causing the acceleration reaction. This process was unaffected by exposure of Sr azide to emission from Ra or UV light. Garner (Ref 14) listed the three stages of thermal decompn as: a)surface reaction from which alkali earth atoms result b)subsequent reaction within the crysts and c)finally the spreading of decompn from reaction centers. Experiments by Garner & Reeves (Ref 22) showed the thermal decompn of Ca and Sr azides obeyed a third-power law, whereas Ba azide obeyed a sixth-power law. Electrical conductivities of these azides were low and did not change during thermal decompn until the nuclei came into contact

Other Properties and Uses. Veenemans & Loosjes (Ref 17) proposed using Sr azide in mixt with Ba azide as the cathode for elec discharge tubes. However, Ficheroulle & Kovache (Ref 19) reported that due to its extreme sensitivity to hydrolysis and because Sr azide reacts with CO_2 to yield $SrCO_3$,

these props make it difficult to manuf and, therefore, Sr azide presents no advantage over Ba azide for use in vac tubes

Refs: 1)L.M.Dennis & C.H.Benedict, JACS **20**, 228 & 231 (1898); ZAnorgChem **17**, 22 (1898) & JCS **74** II, 426(1898) 2)T.Curtius & J.Rissom, JPraktChem **58**, 286 & 305(1898) & JCS **76** II, 91(1899) 3)E.Tiede, Ber **49**, 1742-5(1916) & CA **11**, 2176(1917) 4)L. Wöhler & F.Martin, Ber **50**, 595(1917); JCS **112** I, 383-4(1917) & CA **11**, 2900(1917) 5) L.Wöhler & F.Martin, ZAngChem **30** I, 33-9 (1917); JSCI **36**, 570(1917) & CA **11**, 3432 (1917) 6)A.Petrikaln, ZPhysik **37**, 610-18 (1926) & CA **20**, 2791(1926) 7)Mellor **8** (1928), 350 8)A.Haid et al, Jahresber CTR **8**, 102-8 (1931) & CA **26**, 3669(1932) 9) Gmelin, System No 29(1931), 88-9 10)L.F. Audrieth, ChemRevs **15**, 198(1934) 11)L.F. Audrieth & C.F.Gibbs, InorgSynth **1** (1939), 79-81 & CA **36**, 2488(1942) 12)J.Maggs, Tr FaradSoc **35**, 433-8(1939) & CA **33**, 4112 (1939) 13)W.E.Garner & J.Maggs, PrRoySoc **172A**, 299-314(1939) & CA **33**, 8501(1939) 14)W.E.Garner, Chim & Ind **45**, Suppl to No 3, 111-18(1941); ChemZtr **1942** II, 365-6 & CA **37**, 4571-2(1943) 15)F.T.Llewellyn & F.E. Whitmore, JCS **1947**, 881-4 & CA **41**, 7194 (1947) 16)L.Kahovec & K.W.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666-7 (1948) 17)C.F.Veenemans & R.Loosjes, USP 2463727(1949) & CA **43**, 4966(1949) 18)Kirk & Othmer **7** (1951), 593-4 19)H.Ficheroulle & A.Kovache, MP **33**, 17-9(1951) & CA **47**, 6617(1953) 20)S.M.Ariya & E.A.Prokof'eva, SbornikStateiObshcheiKhim, AkadNauk **1**, 9-18(1953) & CA **48**, 12522(1954) 21)P.W. Jacobs & F.C.Tompkins, JChemPhys **23**, 1445-7(1955) & CA **49**, 15336(1955) 22)W.E.Garner & L.E.Reeves, TrFaradSoc **51**, 694-704(1955) & CA **49**, 15398(1955) 23)P.Gray & T.C. Waddington, PrRoySoc **235A**, 106-19 & 481-95(1956) & CA **50**, 12627 & 15203(1956) 24) Sax(1957) 25)H.Rosenwasser, USArmyEngr-Res & DevelopLabsRpt 1551-TR, 12 & 26-30 (1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Sulfuryl Diazide, $SO_2(N_3)_2$, mw 148.11, N 56.7%, col liq first prepd in 1922 by Curtius & Schmidt (Ref 2) by the interaction of sulfuryl chloride and finely divided, sl moist Na azide: $SO_2Cl + 2NaN_3 \rightarrow SO_2(N_3)_2 + 2NaCl$. It explodes violently when heated and often spontaneously at RT. Sax (Ref 9) does not list this compd but it has a suffocating odor and the pronounced physiological effects of hydrogen azide (qv)

It is hydrolyzed slowly by cold alc or water, but more quickly on warming the solv. Alcoholic $AgNO_3$ reacts with $SO_2(N_3)_2$ instantaneously to form Ag azide (qv). When heated with org aromatic hydrocarbons, such as benz (Refs 3 & 4), p-xylene (Ref 2) and p-cymene (Ref 5), sulfuryl azide completely decomposed (Also see Refs 6, 7, & 8)

Related to sulfuryl azide are the salts of azidosulfonic acid, HSO_3N_3. Traube & Vockerodt (Ref 1) prepd the K salt by treatment of a concd aq soln of potassium nitrite with finely powdered hydrazine sulfonic acid:

$$NH_2NHSO_3H + KNO_2 \rightarrow 2H_2O + KSO_3N_3$$

It crystallized from soln as flat prisms which exploded on heating. The addn of mineral acid yielded hydrazoic acid and sulfuric acid. The NH_4, Na and Ba salts have also been prepd, but their props were not described

Refs: 1)W.Traube & A.Vockrodt, Ber **47**, 938-44(1914) 2)T.Curtius & F.Schmidt, Ber **55B**, 1571-81(1922) & CA **17**, 1000 (1923) 3)F. Schmidt, Ber **55B**, 1581-3(1922) & CA **17**, 1000(1923) 4)K.F.Schmidt, Ber **58B**, 2409-12(1925) & CA **20**, 1081(1926) 5)A.Bertho et al, Ber **60B**, 1717-20(1927) & CA **22**, 229 (1928) 6)P.Walden & L.F.Audrieth, Chem Revs **5**, 339-59(1928) & CA **22**, 4396(1928) 7)L.F.Audrieth, ChemRevs **15**, 216(1934) 8) Kirk & Othmer **7** (1951), 594 9)Sax(1957)

Thallium Azide (Formerly called Thallous Trinitride or Thallium Azoimide), TlN_3, mw 246.41, N17.05%, pal yel tetrag crysts which form as wh ppt; mp 330-40° (Refs 1 & 6), explodes 430° (Ref 6); Q_{expln} 232 cal/g or

55.60 kcal/mol (Ref 3), Q_{form} -55.45 kcal/mol (av value) (Refs 17,18,19 & 21); lattice energy 164 kcal/mol (Ref 19). It is easily sol in hot w, sl sol in cold w (Ref 1); the Soly Prod calcd by Suzuki (Ref 16) is 2.19×10^{-4} vs 2.88×10^{-9} for AgN_3. According to Sax (Ref 24) this compd is highly toxic

TlN_3 was first prepd in 1896 by Dennis & Doan (Ref 1) by adding a thallous sulfate soln to a concd soln of KN_3 contg a little free hydrazoic acid. Curtius & Rissom (Ref 2) used essentially the same procedure while Brouty (Ref 10) prepd the compd from equimolar proportions of NaN_3 & $TlNO_3$ and Rosenwasser (Ref 22) from $TlNO_3$ & KN_3 or TlCl & NaN_3 solns. Its prepn has also been described by Mellor (Ref 7), Gmelin (Ref 9) and others (Refs 13,20 & 25). According to Rosenwasser (Ref 22), Tl azide has numerous cryst habits depending on its method of prepn. Both perfect and imperfect Maltese crosses and rectangular plates, which transform by heat to acicular prisms, were obtd. TlN_3 forms as a fine wh cryst ppt and when crystd from a hot aq soln, it separates as lt-yel orthorhombic ndls which, on exposure to sunlight, assume a dk brn appearance (Ref 1) and form metallic Tl (Ref 18)

Delay et al (Ref 12) detd IR absorption spectra in the range 3 to 19μ and from the intensities of the bands concluded that the sym form was more abundant in the azides of Ag, Cu, Hg^+ & Na but the reverse was true for the azides of Pb & Tl. Gray & Waddington (Ref 18) stated that TlN_3 crysts are isomorphous with those of Na & Rb azides. The elec conductivity of TlN_3 is 5.9×10^{-5} mho at 275° (Ref 18). Brouty (Ref 10) detd the mean activity coefficient of TlN_3 by EMF measurements and calcd ionic radii of Tl^+ & N_3^-. Conductivity measurements by Brouty (Ref 11) did not agree with Onsager's theory; deviations were found at very high dilutions. An electro-chem cell used by Suzuki (Ref 16) gave a $\Delta F°$ 298° value of 59.17 kcal/mol for TlN_3 vs 78.69 kcal/mol for AgN_3. Nair & Nancollas (Ref 23) derived thermodynamic association constants for formation of the ion pairs TlN_3, and of other thallous ion pairs with univalent anions, at 10°, 25 & 40°

Explosives Properties Dennis & Doan (Ref 1) who first prepd TlN_3 state that it is not expl, resembling K and Na azides in this particular. However, just two years later Curtius & Rissom (Ref 2) reported that TlN_3 exploded when struck or heated strongly. Wöhler & Martin (Ref 4) detd an *Expln Temp* as low as 320° in 5 sec for a 0.02g TlN_3 sample and obtd deton of a 0.01 to 0.05g compressed sample under a 2 kg impact. These investigators also detd the *Temp Developed on Expln* on TlN_3 as 3307°, its *Press* (own volume) as 36900 kg/cc, *Work Density* as 72.0 kg/cc, *Loading Density* at 1100 kg/cc as 3.89 g/cc and its *Initiating Efficiency* for various HE's (see table under mercurous azide and Ref 5). Bowden & Williams (Ref 14) reported a *Rate of Detonation* of 1500 m/sec for TlN_3, in confined layers, when initiated at RT by impact on a grit particle. Grit particles melting above 500°, such as Pb chloride, borax, bismuthnite and chalcocite, markedly increased the impact sensitivity of TlN_3

Thermal Decomposition of TlN_3 in an atm of N_2 at any press, according to Audubert & Racz (Ref 8) was not accompanied by any UV radiation. UV light was emitted in O_2 or air at press from 2-10 mm. The energy of activation for thermal decompn at temp below 228 ± 8° is 43 ± 2.5 kcal/mol and above 228 ± 8° is 21.6 ± 2.0 kcal/mol. At press above 10 mm O_2 or air the value is 65.8 ± 2 kcal/mol. Audubert & Racz (Ref 8) suggested that Tl, like Na, can inhibit photogenic reaction and in O_2 there are two processes producing UV radiation. Yoffe (Ref 15) observed that there was very little decompn when TlN_3 was heated in a vacuum. Decompn occurred when TlN_3 was heated in an elec furnace. At 420° TlN_3 melted to a col liq, at 490° it decomposed rapidly and when the N_2 press was 17 cm some of the azide ignited with a green

flash after 2 sec. At 500° the time for expln was less than 1 sec and at 530° expln occurred instantaneously. At 530° expln occurred even when TlN_3 was dropped into an evacuated vessel. A bomb calorimeter was used by McEwan & Williams (Ref 17) to decomp TlN_3 at 24° under 30 atm of He. Gray & Waddington (Ref 18) found that addn of Tl_2S increased the sensitivity of TlN_3 to heat. Evans & Yoffe (Ref 20) attempted to correlate the expl sensitivity of the inorg azides with their cryst structure. They found that the decompn depended on the formation of neutral azide radicals. The expl sensitivity was therefore detd by the degree of elec neutrality possessed by the azide group in the cryst lattice. A consideration of the refractive indices and appropriate cryst structure led them to the conclusion that the expl sensitivity was dependent on the ionization potential of the metal forming the azide

Refs: 1)L.M.Dennis & M.Doan, JACS **18**, 970-5(1896) 2)T.Curtius & J.Rissom, J PraktChem **58**, 283 & 305(1898) & JCS **76** II, 90-2(1899) 3)L.Wöhler & F.Martin, Ber **50**, 595(1917); JCS **112** I, 383-4(1917) & CA **11**, 2900(1917) 4)L.Wöhler & F.Martin, ZAng Chem **30** I, 37-8(1917); JSC I **36**, 570(1917) & CA **11**, 3432(1917) 5)L.Wöhler & F.Martin, SS **12**, 1,18,39,54 & 74(1917) & CA **12**, 629 (1918) 6)A.R.Hitch, JACS **40**, 1202-3(1918) & CA **12**, 1951(1918) 7)Mellor **8** (1928),352 8)R.Audubert & C.Racz, CR **208**, 1810-11 (1939) & CA **33**, 6162(1939) 9)Gmelin, System No 38, Lieferung 2(1940), 234-5 10)M-L. Brouty, CR **214**, 258-61(1942) & CA **37**, 2640 (1943) 11)M-L.Brouty, JChimPhys **39**, 152-8(1942); CR **215**, 61-2(1942); ChemZtr **1943** I, 2073 & CA **38**, 4180-1(1944) 12)A.Delay et al, BullFr **12**, 581-7(1945); CR **219**, 329-33(1944) & CA **40**, 2386 & 4273-4(1946) 13) Kirk & Othmer **7**(1951), 594 14)F.P.Bowden & H.T.Williams, PrRoySoc **208A**, 176-88 (1951) & CA **46**, 5844-5(1952) 15)A.D.Yoffe, PrRoySoc, **208A**, 188-99(1951) & CA **46**, 5845 (1952) 16)S.Suzuki, JChemSocJapan, Pure ChemSect **73**, 150-2(1952) & CA **46**, 9952 (1952) 17)W.S.McEwan & M.M.Williams, JACS **76**, 2182(1954) & CA **48**, 9174(1954) 18)P.Gray & T.C.Waddington, Chem & Ind **1955**, 1555-6 & CA **50**, 6237(1956) 19)P. Gray & T.C.Waddington, PrRoySoc **235A** 106-19 & 481-95(1956) & CA **50**, 12627-8 & 15203 (1956) 20)B.L.Evans & A.D.Yoffe, PrRoy Soc **238A**, 568-74(1957) & CA **51**, 15129-30 (1957) 21)T.C.Waddington & P.Gray, Compt Rend 27e Congr Intern Chim Ind, Brussels **3** (1954) & CA **50**, 16328(1956) 22)H.Rosenwasser, USArmyEngrRes & DevelopLabsRpt 1507-TR, 24(1957) 23)V.S.Nair & G.H. Nancollas, JCS **1957**, 318-23 & CA **51**, 7111 (1957) 24)Sax (1957), 1174 25)H.Rosenwasser, US ArmyEngrRes & DevelopLabsRpt 1551-TR, 42(1958) "Hydrazoic Acid and the Metal Azides" (a literature survey)

Thallous-Thallic Azide, $TlN_3 \cdot Tl(N_3)_3$, mw 576.87, N29.12%, bright yel triclinic crysts highly expl by heat, percussion or even gentle friction. This compd as a double salt was first prepd in 1896 by L.M.Dennis & M. Doan (JACS **18**, 973-5) by dissolving thallic hydroxide in 1.6% hydrazoic acid at ca 0° and concg the soln in a vac desiccator. The thallium content was detd by dissolving some of the crysts in dil HCl, reducing the thallium to thallous by H_2SO_3, removing the excess H_2SO_3 by heating, and then titrating with $KMnO_4$. The nitrogen content could not be detd by the same method used for TlN_3 because the double salt, $TlN_3 \cdot Tl(N_3)_3$, could not be dissolved either in w or dil acids without evolution of HN_3. A few milligrams of sample spread in a long porcelain boat contg granular copper oxide, in an atm of CO_2, exploded violently when the temp of the boat had risen but slightly. Another portion when mixed with very fine, freshly ground copper oxide and heated as before decomposed quietly and gradually giving a nitrogen content of 29.3%. The supposition that this compd is a double salt was confirmed by its behavior when treated with hot water. No further work appears to have been

reported on this azide (Gmelin, System No 38, Lieferung **2** (1940), 235)

Tin Azide. Curtius & Rissom (Ref 1) prepd $Sn(N_3)_2$ only in a very impure state from a 17% HN_3 soln and tin-foil. A non-expl wh compd, insol in w, was separated. An aq soln of stannous chloride and NaN_3 gave a wh ppt which was thought to be a mixt of stannous azide & hydroxide which when heated with H_2SO_4 gave off HN_3 (Ref 3). Browne & Houhelan (Ref 2) obtd no reaction from tin in contact with NH_4N_3 in liq NH_3 after 4 hrs

Attempts by Wiberg & Michaud (Ref 4) to prepare $Sn(N_3)_4$ were unsuccessful. A tetrahydrofuran soln of $SnCl_4$ refluxed for 5 hrs with excess NaN_3 resulted in a wh, solid, complex salt, $Sn(N_3)_4 \cdot 2NaN_3$ or $Na_2SN(N_3)_6$, insol or only sl sol in eth or benz and hydrolyzed by moist air. A tetrahydrofuran soln of the complex salt, sodium hexazidostannate, detonated on boiling over a free flame but it was not sensitive to percussion (see Ref 5)

Refs: 1)T.Curtius & J.Rissom, JPraktChem **58**, 299(1898) & JCS **76** II, 91-2(1899) 2) A.W.Browne & A.E.Houlehan, JACS **33**, 1752 (1911) & CA **6**, 579(1912) 3)Mellor **8**(1928), 352-3 4)E.Wiberg & H.Michaud, ZNaturforsch **9b**, 500(1954) & CA **49**, 768(1955) 5)H.Rosenwasser, USArmyRes&DevelopLabsRpt 1551-TR, 48(1958), "Hydrazoic Acid and the Metal Azides" (a literature survey)

Zinc Diazide (formerly called Zinc Azoimide or Zinc Trinitride), $Zn(N_3)_2$, mw 149.43, N 56.25%, wh hygro powd, easily hydrolyzed. It was first prepd in 1892 by Wislicenus (Ref 1) by the action of nitrous oxide on Zn amide at 150-250°. Curtius & Rissom (Ref 2) obtd the basic zinc azide, $ZnOHN_3$, by dissolving the metal in dil HN_3 and allowing the soln to evap in air. The product, ill-defined anisotropic crysts, was insol in w. Dennis & Isham (Ref 3) prepd $Zn(N_3)_2 \cdot 2NH_3$ and Zn $(N_3)_2 \cdot 2C_5H_5N$ by dissolving Zn in alcoholic HN_3, adding dry NH_3 or pyridine in excess, and allowing the soln to evap. Both products were wh, crystalline ppts, insol in w, and decompd without expln when heated but when thrown on a hot plate they produced a bright flame and a sharp report. Zn in contact with NH_4N_3 in liq NH_3 did not react, except very slowly when Pt was present (Ref 4). By shaking together the finely divided, dry Zn carbonate or basic Zn azide with HN_3 in ether until the solid became entirely sol in H_2O, Wöhler (Ref 5) obtd $Zn(N_3)_2$ and detd some of its props. The toxicity of $Zn(N_3)_2$ is not discussed by Sax (Ref 16)

According to Wöhler & Martin (Ref 6), Zn $(N_3)_2$ is detonated under impact of a 2 kg wt and exploded in 5 sec at 289°. The heat of detonation is 360 cal/g (Ref 6) and Q_f −50.8 kcal/mol (Ref 14). These investigators consider $Zn(N_3)_2$ a rather weak expl approaching in its expl props, the alkaline earth azides which are not as powerful as the heavy metal azides

Petrikaln (Ref 7) photographed the spectra of $Zn(N_3)_2$ and other azides. With the azides of Ca, Sr and Ba, not only triplet system lines but also those of the singlet system were emitted. $Zn(N_3)_2$ emitted only triplet system lines of the diffuse and sharp series. In addn the oxide bands were present in all the spectra. Kahovec & Kohlrausch (Ref 13) detd the Raman spectra of basic zinc azide crysts.

An addn compd with mercuric cyanide, $Hg(CN)_2 \cdot Zn(N_3)_2$, which deflagrated but did not explode when heated, was prepd by Ricca & Pirrone (Ref 10) from aq solns of NaN_3, $ZnSO_4$ and $Hg(CN)_2$. Strecker & Schwinn (Ref 12) prepd some Zn azide complex salts, $[Zn(C_5H_5N)_2]$ $(N_3)_2$ and $[Zn(C_2H_4(NH_2)_2)_3]$ $(N_3)_2$, and detd their props. A group of mixed Zn halogen azides of the type $[ZnNI_2N_2]M$ were prepd by Vournazos (Ref 8).

For addnl information or discussion also see Mellor (Ref 9), Audrieth (Ref 11) and Gmelin (Ref 15)

Refs: 1)W.Wislicenus, Ber **25**, 2085(1892) & JCS **62** II, 1151(1892) 2)T.Curtius & J. Rissom, JPraktChem **58**, 292 & 305(1898) &

JCS **76** II, 92(1899) 3)L.M.Dennis & H. Isham, JACS **29**, 20-1(1907) & CA **1**, 528 (1907) 4)A.W.Browne & A.E.Houlehan, JACS **33**, 1751-2(1911) & CA **6**, 579(1912) 5)L.Wöhler, ZAngChem **27I**, 335-6(1914) & CA **9**, 1115(1915) 6)L.Wöhler & F.Martin, Ber **50**, 594-5(1917); JCS **112** I, 384(1917); ZAngChem **30** I, 33-9(1917); JCS I **36**, 570 (1917) & CA **11**, 2900 & 3432 (1917) 7)A. Petrikaln, ZPhysik **37**, 610-8(1926) & CA **20**, 2791(1926) 8)A.C.Vournazos, ZAnorg Chem **164**, 263-73(1927) & CA **21**, 3841 (1927) 9)Mellor **8** (1928), 350 10)B.Ricca & F.Pirrone, Gazz **59**, 564-8(1929) & CA **24**, 309(1930) 11)L.F.Audrieth, ChemRevs **15**, 199-201(1934) 12)W.Strecker & E.Schwinn, JPraktChem **152**, 205-18(1939) & CA **33**, 5314 (1939) 13)L.Kahovec & K.W.Kohlrausch, Monatsh **77**, 180-4(1947) & CA **42**, 6666-7 (1948) 14)F.D.Rossini, BurMinesCirc 500 (1952), 182 15)Gmelin, System No **32**(1956), 834-5 & **32** (1924), 148 16)Sax(1957)

AZIDES, ORGANIC

Azido or Triazo- Compounds, contg one or more $-N_3$ groups, are the org derivatives of hydrazoic acid, HN_3. These compds are usually unstable to heat and often decompose violently. They may be divided into four classes:

a)Alkyl Azides – obtd by reacting NaN_3 with alkyl sulfates or halides:

$$RSO_4 + NaN_3 \rightarrow RN_3 + R{\cdot}SO_4{\cdot}Na$$

The azide group in these compds usually hydrolyzes to form HN_3

b)Aryl Azides(Diazoimides) – obtd by reacting NaN_3 with aromatic diazonium salts in acid soln:

$$ArN_2Cl + NaN_3 \rightarrow ArN_3 + NaCl + N_2$$

Most of these azides are yel liquids, insol in w, more stable than the alkyl azides and do not hydrolyze

c)Acyl Azides – prepd by the action of HNO_2 on a hydrazide or of an acid chloride on NaN_3:

$$R{\cdot}CO{\cdot}NH{\cdot}NH_2 + HNO_2 \rightarrow RCON_3 + 2H_2O$$
$$R{\cdot}COCl + NaN_3 \rightarrow RCON_3 + NaCl$$

These compds are liquids or low melting solids, sl sol in w and sol in common org solvents

d)Azide Salts – obtd by treating org salts with metallic azides:

$$H_2N{\cdot}C{\cdot}(:NH){\cdot}NH_2{\cdot}HCl + AgN_3 \rightarrow$$
$$H_2N{\cdot}C{\cdot}(:NH){\cdot}NH_2{\cdot}HN_3 + AgCl$$

For purposes of org nomenclature the terms "azido" and "triazo" are used synomously in the literature. The aryl azides are often called in German diazoimides. Aliphatic diazo compds contain the group $=CN_2$, represented as a resonance hybrid of the structure

$$=C=N\rightarrow N \text{ and } =C\leftarrow N\equiv N$$

The monovalent azide group, $-N_3$, is closely related to the divalent diazo group and can be regarded as derived from the latter by replacement of a tetravalent carbon atom by a trivalent nitrogen atom. The structure may be represented as a resonance hybrid

$$R-N=N\rightarrow N \text{ and } R-N\leftarrow N\equiv N$$

The acyl derivatives of hydrazoic acid can be regarded as derivatives of acids in which the acidic hydroxyl group has been replaced by an azide group

The literature contains reviews of the chemistry of org azides (Refs 5 & 11). Smith (Ref 8) has reported on the acyl azides, Benson (Ref 9) on those α-azidoalkylidenimines which undergo cyclic isomerizations to tetrazoles and Boyer & Canter (Ref 15) made a thorough survey of the available information on alkyl and aryl azides. Cirulis & Straumanis (Ref 6) prepd a number of new azides of org bases but none of these azides showed expl props. Schaad (Ref 14) obtd a patent for the manuf of esters of hydrazoic acid based on the reaction of alkenes, cyclic olefins, arylalkenes & cycloalkylalkenes with HN_3 in the presence of an acid catalyst

The dipole moments and structures of the org azides and aliphatic diazo compds were studied by Sidgwick et al (Ref 4). Sheinker & Syrkin (Ref 13) made vibrational spectra measurements of org azides and deduced the configuration of azide compds. UV spectra confirmed the observations made on Raman and IR spectra. Heats of combustion and formation of org azides were detd by Murrin & Carpenter (Ref 16). Patterson et al (Ref 7) discussed the CA method of naming and indexing org azides. For addnl info and discussion of org azides see the general references (Refs 1,2,3,5,8,10 & 12)

This discussion or listing of org azides is based on the following conditions:

a)When an azide or azido compd is one of the derivatives (others are nitro, nitroso etc) of a parent compd, such as methane, the azide derivative, as well as others, may be located under the parent compd. For example, methyl azide is discussed under Methane and benzoyl azide under Benzoic Acid

b)When an azido compd is the only derivative

of a parent compd or if the compd is generally known as azido, for example azidodithiocarbonic acid, it is discussed in this section if it has expl props

c)Derivatives of acids in which the acidic hydroxyl group has been replaced by an azide group, for example acetazide, allophanylazide, crotonylazide, oxamylazide, phthalylazide etc, are placed in alphabetical order according to their first letter. Therefore, org azides or azido compds discussed or listed in this section all begin with letter A. Others will be found and discussed under their appropriate alphabet letter

d)Some azido compds, such as azidoaniline or azidoanisole, are also included in this section when considered more convenient than to list them under their parent compd

Refs on Org Azides: 1)Beilstein (see under individual compds) 2)T.Curtius et al, JPraktChem **50**, 275(1894) & **58**, 190(1898) 3)M.O. Foster & H.E.Fierz, JCS **93** I, 72-85(1908) 4)N.V.Sidgwick et al, JCS **1933**, 406-12 & CA **27**, 3459(1933) 5)Sidgwick, OrgChem of N (1942), 363-77 6)A.Cirulis & M.Straumanis, JPraktChem **161**, 65-76(1942) & CA **37**, 5022-3 (1943) 7)A.M.Patterson et al, CA **39**, 5917 (1945) 8)P.A.Smith, "The Curtius Reaction" in Organic Reactions, JWiley & Sons, NY, **3** (1946), 337 9)F.R.Benson, Chem Revs **41**, 1-61(1947) 10)Kirk & Othmer, **2** (1948), 213-4 11)V.Grignard & P.Baud, "Azides" in "Traité de Chimie Organique", Masson, Paris, **15** (1948), 714-91 12)Degering(1950), 258-91 13)Yu.N. Sheinker & Ya.K. Syrkin, IzvestAkadN, SerFiz **14**, 478-87(1950) & CA **45**, 3246 (1951) 14)R.E.Schaad, USP 2557924 (1951) & CA **46**, 1028(1952) 15)J.H.Boyer & F.C.Canter, ChemRevs **54**, 1-59(1954) 16) J.W.Murrin & G.A.Carpenter, USNavalPowder Factory, MemRpt 129(1957)

LIST OF ORGANIC AZIDES OR AZIDO COMPOUNDS

Acetonetetrazylazide. See under Acetonyltetrazole and Derivatives, p A47
Acetylglycineazide. See p A69
Acryloylazide. See under Acrylic Acid and Derivatives, p A97
Adipyldiazide. See under Adipic Acid and Derivatives, p A104
Aleuritylazide. See p A123-4
Alkylazides. See pp A129 & A130
Alkyltetrazylazides. See p A133
Allophanylazide. See p A133-4
Allylazide. See p A137
o-Aminoazidoacetophenone or **o-Aminophenylacylazide.** See under Aminoacetophenone and Derivatives, p A178
Aminobenzazide or **Aminobenzoylazide.** See under Aminobenzoic Acid and Derivatives, p A188-9
iso-Amylacetylazide. See under Amyl Acetate, p A394
Amylazide. See under Amylamine and Derivatives, p A395
Amylmalonylazide. See p A396
iso-Amylureidoacetyl Azide. See p A399
Anilinobenzenediazonium Azide. See under Anilinobenzenediazonium Hydroxide and Derivatives, p A421
Anilinodinitrobenzoyl Azide. See under Anilinobenzoic Acid and Derivatives, p A421
Anisalanishydrazide Azide. See p A444
Anisicazide or Anisoylazide. See p A456
Anthranilic Acid Azide. Same as Aminobenzazide (qv), p A188-9
Anthranoylazide. Same as 4-Aminobenzazide (qv), p A189
Anthraquinone Azide. See under Anthraquinone and Derivatives, p A459

Azidine or Acetazidin is listed in Ref 1 as a compd of formula $CH_3C(:NNH_2)\cdot N:NH$. No compd of this formula was found in CA through 1957

In English, azidine is the name suggested for the radical $-C(=NH)N_3$ by analogy with amidines which are derived from the amides by replacement of oxygen in $-CONH_2$ by the

divalent amido residue =NH or =NR. Thus, for the structural change $-CON_3 \rightarrow -C(=NH)N_3$ the nomenclature should be azide → azidine, according to Scott et al (Ref 4), except the term azidine does not appear to have been used previously in the literature

Carbamoyl azides (called by Scott et al carbamylazides), $R^1R^2NCON_3$, in which either R^1 or R^2 is H, form an enolic structure which contains the hydroxy-substituted azidine radical and thereby become resistant to the Curtius rearrangement: $RCON_3 \rightarrow N_2 + RCON \rightarrow RN = CO$. The carbamoyl azides, as a group, were classified by Bertho (Ref 3) as resistant to the Curtius rearrangement. However, other work by Stollé (Ref 2) showed that while some members of this group (R_1 or R_2 = H and R_2 or R_1 = alkyl/aryl) failed to become rearranged, others (R_1 or R_2 = C_6H_5) did so. Scott et al offered a different interpretation for the resistance of carbamoyl azide to such change

Refs: 1)Beil **2**, 4 2)R.Stollé, Ber **57**, 1063 (1924) 3)A.Bertho, JPraktChem **120**, 89 (1929) & CA **23**, 817(1929) 4)F.L.Scott et al, Nature **170**, 922-3 (1952) & CA **47**, 9923 (1953)

Azidoacenaphthene. See under Acenaphthene and Derivatives, p A12

Azidoacetaldehyde. See under Acetaldehyde and Derivatives, p A15

Azidoacetamide. See under Acetamide and Derivatives, p A16

Azidoacetamidophenol. See under Acetamidophenol and Derivatives, p A20 & 21

Azidoacetamidophenol, Dinitro. See under Acetamidophenol and Derivatives, p A21

Azidoacetanilide. See under Acetanilide and Derivatives, p A23

Azidoacetate (Triazoacetate) Salts. The normal Pb, Ag, K or uranyl triazoacetate salt has been proposed as an ingredient of priming compositions. For example, Pb triazoacetate/Pb styphnate/Pb thiocyanate/Pb nitrate/glass: 10/32/8/30/20%

Ref: P.H.Burdett & G.M.Calhoun, USP 2356211(1944) & CA **39**, 194(1945)

Azidoacetic Acid. See under Acetic Acid and Derivatives, p A27

Azidoacetic Anhydride. See under Acetic Anhydride and Derivatives, p A31

Azidoacetone. See under Acetone and Derivatives, p A39

Azidoacetoneacetylhydrazide. See under Acetone and Derivatives, p A39

Azidoacetonitrile. See under Acetonitrile and Derivatives, p A45

Azidoacetonylditetrazole. See under Acetonyltetrazole and Derivatives, p A47

Azidoacetonyltetrazole. See under Acetonyltetrazole and Derivatives, p A47

Azidoacetophenone. See under Acetophenone and Derivatives, pp A47-8

Azidoacetophenoneoxime. See under Acetophenoneoxime and Derivatives, p A49

Azidoacetoxime. See under Acetoxime and Derivatives, p A51

Azidoacetyl-dl-alanine. See under Acetylalanine and Derivatives, p A54

Azidoacetyl-dl-alanine Chloride. See under Acetylalanine and Derivatives, p A54

Azidoacetyl Chloride. See under Acetyl Chloride and Derivatives, p A57

Azidoacetyldiphenylamine. See under Acetyldiphenylamine and Derivatives, p A58

Azidoacetylglycine. See Acetylglycine Azide, p A69

Azidoacetylhydrazide, Acetone-. See under Acetone and Derivatives, p A39

Azidoacetylsalicylic Acid. See under Acetylsalicylic Acid and Derivatives, p A87

Azidoacrylic Acid. See Acryloylazide under Acrylic Acid and Derivatives, p A97

Azidoamide;(Carbamyl Azide). See under Carbamic Acid and Derivatives and Beil **3**, 129, (59) & [102]

Azidoaminoacetophenone. See under Aminoacetophenone and Derivatives, p A178

Azidoaminobenzoic Acid. See Aminobenzazide under Aminobenzoic Acid and Derivatives, p A188-9

Azidoaminoethane. See under Aminoethane and Derivatives, p A199

Azidoaminomethylguanidine. See under Aminomethylguanidine and Derivatives, p A232

Azidoaminopropane. See under Aminopropane and Derivatives, p A250

Azidoaminothiadiazole, Nitroso. See 5-Azido-2-nitrosamino-1,3,4-thiadiazole under Aminothiadiazole and Derivatives, p A262

AZIDOANILINE AND DERIVATIVES

Azidoaniline; Aminodiazobenzeneimide; Aminotriazobenzene or Triazoaniline, $N_3 \cdot C_6H_4 \cdot NH_2$, mw 134.14, N 41.77, OB to CO_2 -179%. The following isomers are known:

3-Azidoaniline, yel oil of unpleasant odor; mp-explodes on heating; volatile with steam; easily sol in alc or eth. It was prepd by heating 3-azido-phenyloxamic acid with concd KOH soln

Refs: 1)Beil **12**, 772 2)P.Griess, Ber **18**, 963(1885)

4-Azidoaniline, lfts (from eth), mp 62-5°; volatile with steam and puffs off on stronger heating; very sol in alc, eth or chlf & sl sol in w. It was first prepd by heating 4-azidophenyloxamic acid with concd KOH soln (Ref 2). Silberrad & Smart (Ref 3) prepd it by slowly distilling acetyl-p-azidoaniline in 40% KOH soln. This compd forms expl salts (Ref 4)

Refs: 1)Beil **12**, 772 2)P.Griess, Ber **21**, 1559(1888) 3)O.Silberrad & B.J.Smart, JCS **89** I, 171(1906) 4)S.Maffei & A.M.Rivolta, Gazz **84**, 750-2(1954) & CA **49**, 13925(1955)

Azidoaniline Perchlorate, $N_3 \cdot C_6H_4 \cdot NH_2HClO_4$, mw 234.61, N 23.88%, OB to CO_2 -78.4%; red plates, mp - puffs off without melting. It can be prepd by treating 4-azidoaniline with perchloric acid. It is very sensitive to expln by impact. This compd was prepd and investigated by the duPont Co during WW II

Refs: 1)Beil – not found 2)A.H.Blatt, OSRD Rpt 2014(1944), under Azides

p-Azidoaniline Picrate, $N_3C_6H_4NH_2 \cdot HOC_6H_2(NO_2)_3$, mw 363.26, N 26.99%, OB to CO_2 -107.9%; mp 64-5° (from MeOH). It was prepd in 75% yield from 4-azidoaniline and excess picric acid in MeOH, the soln freed of picric acid by treatment with aq Na_2CO_3. By the same method *p-azidodimethylaniline picrate,* mp 47% (from petr eth) was obtd in 78% yield. These compds were isolated as picrates to identify the respective azidoamines

Refs: 1)Beil – not found 2)S.Maffei & A.M.Rivolta, Gazz **84**, 750-2(1954) & CA **49**, 13925(1955)

AZIDOANISOLE AND DERIVATIVES

Azidoanisole or Methylazidophenylether, $C_7H_7N_3O$, mw 149.15, N 28.18%, OB to CO_2 -177.0%:

2-Azidoanisole (called o-Methoxy-diazobenzolimid in Ger), $N_3 \cdot C_6H_4 \cdot OCH_3$, yel oil. It was prepd from o-methoxybenzenediazoniumperbromide and NH_3

Refs: 1)Beil **6**, 293 2)H.Rupe & K. von Majewski, Ber **33**, 3405(1900)

4-Azidoanisole (p-Azidoanisole), $N_3 \cdot C_6H_4 \cdot OCH_3$; yel plates (from petr eth or eth), mp 35-36°; dec at 150° and 20 mm. It was prepd from p-methoxybenzenediazoniumperbromide and NH_3

Refs: 1)Beil **6**, 294 & (142) 2)H.Rupe & K.vonMajewski, Ber **33**, 3405-6(1900) 3) M.D.Forster, JCS **89**, 238(1906) M.O.Forster & H.E.Fierz, JCS **91**, 862(1907) 4)O.Dimroth & K.Pfister, Ber **43**, 2763(1910) 5)H. Bretschneider & H.Rager, Monatsh **81**, 970 (1950) & CA **45**, 7973(1951)

4-Nitro-2-Azidoanisole, $N_3 \cdot C_6H_3(NO_2) \cdot OCH_3$, mw 194.15, N 28.86%, OB to CO_2 −115.3; yel ndls having odor of bitter almonds. It was prepd by Griess

Refs: 1)Beil **6**, 294 2)P.Griess, JCS **20**, 88(1867)

Azidoanthraquinone. See under Anthraquinone and Derivatives, p *A459-60*

Azidoantipyrine. See under Antipyrine and Derivatives, p A471

Azidoazobenzene. See under Azobenzene, p A647

Azidoazomethine–Tetrazole Equilibrium. See under Tetrazole Equilibrium and J.H. Boyer & E.J.Miller, Jr, JACS **81**, 4671(1959)

p-Azidobenzaldehyde. See under Benzaldehyde and Beil **7**, 266 & (145)

Azidobenz-anti-aldoxime, Methyl Ester. See under Benzaldoxime and Beil **7**, 266

Azidobenzene; Triazobenzol; Diazobenzolimide or Phenyl Azide. See under Benzene and Beil **5**, 276, (141) & [207]

Azidobenzenediammoniumhydroxide. See under Benzenediammoniumhydroxide and Beil **16**, 493

Azidobenzenedioxime. See under Benzenedioxime and Beil **7**, 266 & (145)

Azidobenzenesulfonic Acid. See under Benzenesulfonic Acid and Beil **11**, 80 & [37]

Azidobenzfuroxan. See under Benzfuroxan and Derivatives

Azidobenzoic Acid. See under Benzoic Acid and Beil **9**, 418, (168-9) & [286]

Azidobenzoic Acid Amide. See under Benzoic Acid Amide and Beil **9**, 418

Azidobenzoic Acid Hydrazonium Hydroxide. See under Benzoic Acid Hydrazonium Hydroxide and Beil **16**, 548

Azidobenzoic Acid, Methyl Ester. See under Benzoic Acid and Beil **9**, 418

Azidobenzonitrile. See under Benzonitrile and Beil **9**, 418-9 & (169)

N-[(α-Azidobenzylidene)-N'-benzylidene]-hydrazide. See under Benzalazine and Derivatives

4-[4-Azidobenzylideneamino]-phenol. See under Benzylideneaminophenol and Beil **13**, (155)

Azidobromobenzene (1-Bromo-4-Triazobenzene). See under Bromobenzene and Beil **5**, 277, (142) & [208]

1-Azido-2-Bromoethane. See under Bromoethane and Beil **1**, (33)

Azido-5-Bromonicotinic Acid. See under Bromonicotinic Acid and (Beil – not found) R.Graf et al, JPraktChem **138**, 244(1933)

1-Azido-4(or 5)-Bromo-2-Nitrobenzene. See under Bromobenzene and Beil **5**, (143)

1-Azidobutane;(2-Triazobutane). See under Butyl Azide and (Beil – not found) J.H. Boyer & J.Hamer, JACS **77**, 951-4(1955)

1-Azidobutanone (1-Triazobutanone-2). See under Butanone and Beil **1**, 671

Azidobutanoneoxime. See under Butanoneoxime and Beil **1**, 671

Azidobuttersäure (Ger). Azidobutyric Acid

α-Azidobutyric Acid or α-Triazobutyric Acid. See under Butyric Acid and Beil **2**, 287,299, (126) & [257]

Azidobutyric Acid Amide. See under Butyric Acid Amide and Beil **2**, 287 & 299

Azidobutyric Acid Azide. See Azidobutyrylazide

Azidobutyric Acid, Ethyl Ester. See under Butyric Acid and Beil **2**, 287,299 & (126,130)

Azidobutyric Acid Hydrazide. See under Butyric Acid Hydrazide and Beil **2**, (126)

Azidobutyrylazide. See under Butyric Acid and Beil **2**, (126)

Azidocaffeine. See under Caffeine and Beil **26**, 477

3-Azido-d-camphor or Camphorylazide. See under Camphor and Beil **7**, 133

3-Azidocarbazole. See under Carbazole and Beil **20**, [290]

Azido Carbon Disulfide. See under Carbon Disulfide and Beil **3**, [160]

Azidocarbonyl Diazonium Hydroxide. See under Carbonyl Diazonium Hydroxide and (Beil – not found) R.Hofsommer & M.Pestemer, ZElectrochem **53**, 383-7(1949) & CA **44**, 4331-2(1950)

Azidocarboxyphenyloxamic Acid. See under Carboxyphenyloxamic Acid and Beil **14**, 418

Azidochloroethane (1-Azido-2-Chloroethane). See Chloroethyl Azide under Ethyl Chloride and Beil **1**, (33)

2-Azido-3-cumaranone. See under Cumaranone and Beil **17**, [127]

Azidocyanformamidine. See under Cyanformamidine and Beil **3**, [102]

Azidocyaniminoaminomethane. See under Cyaniminoaminomethane and Beil **3**, [102]

Azidocycloheptane. See Cycloheptyl Azide under Cycloheptane and J.H.Boyer et al, JACS **78**, 325-7(1956) & CA **50**, 12855(1956)

Azidocyclohexane. See Cyclohexyl Azide under Cyclohexane and J.H.Boyer et al, JACS **78**, 325-7(1956) & CA **50**, 12855(1956)

Azidocyclopentane. See Cyclopentyl Azide under Cyclopentane and J.H.Boyer et al, JACS **78**, 325-7(1956) & CA **50**, 12855(1956)

2-Azido-4-diazobenzenesulfonic Acid. See under Diazobenzenesulfonic Acid and Beil **16**, 565

1-Azido-1,2-dibromoethane. See under Dibromethane and Beil **1**, (33)

Azidodiethylether. See under Diethylether and Beil **1**, (171)

Azidodiethylsuccinate (Azidobernsteinsäurediäthylester in Ger). See under Diethylsuccinate and Beil **2**, (270)

Azido-3,4-dihydroxy-2,5-furandicarbonyl. See Dihydroxyfurandicarbonyl and A.Darapsky & M.Stauber, JPraktChem **146**, 209-18(1936) & CA **31**, 396(1937)

Azidodimethylaminobenzeneazotriazole. See under Dimethylaminobenzeneazotriazole and R.Stollé & W.Dietrich, JPraktChem **139**, 202(1934) & CA **28**, 2714(1934)

Azidodimethylazobenzene. See under Dimethylazobenzene and Beil **16**, 63,65 & 66

Azidodimethylbenzaldehyde. See under Dimethylbenzaldehyde and Beil **7**, 313

Azidodimethylbenzene. See Xylyl Azide under Dimethylbenzene (Xylene) and Beil **5**, 389 & (188)

Azidodimethylbenzoic Acid. See under Dimethylbenzoic Acid and Beil **9**, 538

Azidodimethyldinitrobenzene. See under Dimethylbenzene (Xylene) and Beil **5**, 382 & (188)

Azidodimethyldinitrobutylbenzene or Azidodinitrobutylxylene. See under Butylxylene and Beil **5**, 448 & [340]

Azidodimethylindazole. See under Dimethylindazole and Beil **23**, [166]

Azidodimethylnitrobenzene. See under Dimethylbenzene (Xylene) and Beil **5**, 381-2

4-Azido-3,5-dimethylpyrazole. See under Dimethylpyrazole and Beil **23**, (25)

Azidodinitroacetamidophenol. See under Acetamidophenol and Derivatives, p A21

Azidodinitrobenzene (Dinitrophenyl Azide). See under Benzene and Beil **5**, 279 & [209]

2-Azidodinitrobiphenyl. See under Biphenyl and P.A.Smith & B.B.Brown, JACS **73**, 2438 (1951) & CA **46**, 494-5(1957)

Azidodinitronaphthalene. See under Naphthalene and Beil **5**, 565-6 & [460]

2-Azido-4,6-Dinitrophenol. See under Phenol and (Beil – not found) A.H.Blatt, OSRD Rpt 2014(1944)

3-Azido-2,4-Dinitrophenylhydrazone Propionate. See under Phenylhydrazone Propionate and J.H.Boyer, JACS **73**, 5248(1951) & CA **47**, 489-90(1953)

Azidodinitrotoluene. See under Toluene and Beil **5**, 350-1, (174) & [350]

Azidodinitroxylene. See under Xylene and Beil **5**, 382 & (188)

Azidodiphenylamines. See under Diphenylamine and Beil **12**, [429]

AZIDODITHIOCARBONIC ACID AND DERIVATIVES

Azidodithiocarbonic Acid (Azidodithioformic Acid) (called Azidodithioameisensäure or Dithiokohlensäureazide in Ger), $H{\cdot}SC(=S)-N{=}N{\equiv}N$, mw 119.17, N 35.27%; wh or sl yel cryst, mp 50-65° (dec), explodes at 70°; sol in alc, methanol, eth, benz & AcOH and fairly sol in w. It was first prepd by Sommer (Ref 2) by treatment of a cold concd soln of Na azidodithiocarboxylic acid with concd HCl, but no evidence was offered to identify the compd. It's prepn has been described by Oliveri-Mandalà (Ref 3), Smith et al (Refs 4 & 9), Audrieth (Ref 8) and others. Azidodithiocarbonic Acid can be prepd, according to Smith et al (Refs 4 & 9), by refluxing for 48 hrs at 40° pure CS_2 and a concd aq soln of recrystd NaN_3, filtering the soln, washing the crysts with chilled concd HCl & ice w and drying the product. If $HSCSN_3$ is stored in a desiccator in the dark, below 10°, it is stable for several days

This compd is sensitive to light and heat. At RT it decomposes at a rate characteristic of monomolecular reactions. Decompn is catalyzed in the dry state, but not in aq solns, by an intermediate prod or by the thiocyanic acid formed:

$$HSCSN_3 \rightarrow HSCN + S + N_2 \uparrow$$

In aq soln the compd is much more stable (Ref 4). Elec conductivity, potentiometric titration and cryoscopic detns all show that $HSCSN_3$ is an acid comparable in strength to H_2SO_4 ($K \approx 2.14 \times 10^{-2}$) (Ref 7). The electrode potential of an azidocarbondisulfide-azidodithiocarbonate electrode was found to be 0.275V (Ref 10). The diln (in l/mol) at which the salt is completely dissociated in CH_3Cl, the ratio of this diln to that of AcOH taken as 1, and the ratio of the dissocn constant to that of AcOH taken as 1, was found by Hantzsch & Voight (Ref 6) to be as follows: 1450, 24160 & 1290

Dry azidodithiocarbonic acid is very sensitive to expln by friction, impact or heat. It detonates with a puff on contact with a hot wire below red heat. It is easily oxidized by various reagents to a more expl solid, $(SCSN_3)_2$, *azidocarbondisulfide* (Ref 4)

Browne & Smith (Ref 5) discussed several possible methods for detg the presence of $SCSN_3^-$ ion:

a)titration of the free acid using methyl red indicator

b)gravimetric detn as the Ag salt or as AgCl or equiv

c)titration with $AgNO_3$ as in the Gay-Lussac or Volhard method for titrating Cl^- ion and

d)titration with iodine dissolved in alc

Of these methods the Volhard titration method was preferred

Many derivs and salts of azidodithiocarbonic acid are known some of which are expl (see below)

Refs: 1)Beil **3**, (86) & [159] 2)F.Sommer, Ber **48**, 1833(1915) & CA **10**, 342-3(1916) 3)E.Oliveri-Mandalà, Gazz **52** II, 139 (1922) & CA **17**, 1642(1923) 4)G.B.Smith et al, JACS **45**, 2604(1923) & CA **18**, 27-8(1924) 5)A.W.Browne & G.B.Smith, JACS **47**, 2698 (1925) & CA **20**, 28(1926) 6)A.Hantzsch & W.Voigt, Ber **62A**, 975(1929) & CA **23**, 4200 (1929) 7)G.B.Smith et al, JACS **56**, 1116 (1934) & CA **28**, 3643(1934) 8)L.F.Audrieth, ChemRevs **15**, 196-7(1934) 9)G.B.Smith, Inorg Synth **1**(1939), 81-4 & CA **36**, 2488 (1942) 10)R.Ullman, G.B.Smith, JACS **68**, 1479(1946) & CA **40**, 6006(1946)

Acyl and Aryl Derivatives of Azidodithiocarbonic Acid:

Allyl Azidodithiocarbonate, $C_3H_5{\cdot}SCSN_3$, mw 159.24, N 26.39%; unstable and undergoes fairly rapid decompn. It was prepd by the interaction of allyl bromide and Na azidodithiocarbonate in acet. No careful study of the props of this compd was made

Benzohydryl Azidodithiocarbonate, $(C_6H_5)_2CH{\cdot}SCSN_3$, mw 285.40, N 14.72%; col crysts, mp 67°; very sol in acet, EtOAc, benz, CS_2, $CHCl_3$ or CCl_4, moderately sol in alc, MeOH or eth. It was prepd by the interaction of benzohydryl bromide and Na azidodithiocarbonate. After filtering to remove the pptd NaBr, the yel liq, separated by the addn of w, was dissolved in eth and benz which, after drying and cooling, separated in small col crysts

Benzoyl Azidodithiocarbonate, $C_6H_5CO{\cdot}SCSN_3$, mw 223.28, N 18.82%; col monocl plates, mp 92-4°(dec); very sol in $CHCl_3$, moderately sol in eth, benz, CS_2 or CCl_4 and sl sol in acet, EtOAc, alc or MeOH. It was prepd by the interaction of benzoyl chloride and Na azidodithiocarbonate, either in aq soln or in acet. The product was purified by crystn from chlf

Benzyl Azidodithiocarbonate, $C_6H_5CH_2{\cdot}SCSN_3$, mw 209.30, N 20.08%;col monoclinic prisms, mp 66; very sol in acet, EtOAc, CS_2 or $CHCl_3$; sl sol in alc or MeOH and moderately sol in eth or benz. It was prepd by the interaction of benzyl chloride and Na azidodithiocarbonate in acet at RT. The ppt NaCl was removed by filtration and the crude prod which separated on addn of water was purified by recrystn from chlf

p-Bromobenzoyl Azidodithiocarbonate, $BrC_6H_4CO{\cdot}SCSN_3$, mw 302.19, N 13.91%; col rhmb plates, mp 99-101°(dec); very sol in $CHCl_3$, moderately sol in CS_2 or CCl_4 and sl sol in acet, EtOAc, alc, MeOH, eth or benz. This compd was pptd, together with NaCl, by the interaction of p-bromobenzoyl chloride and Na azidodithiocarbonate in acet. The prod was purified by crystn from chlf

Methyl Azidodithiocarbonate, $CH_3{\cdot}SCSN_3$, mw 133.20, N 31.55%; col rhmb prisms, mp 34°; moderately sol in alc, MeOH, or eth and sl sol in acet, EtOAc, benz, CS_2, $CHCl_3$ or CCl_4. This prod was obtd by the interaction of methyl bromide and Na azidodithiocarbonate in acet and was purified by crystn from chlf. It is slowly attacked by concd aq alkalies

Triphenylmethyl Azidodithiocarbonate, $(C_6H_5)_3C{\cdot}SCSN_3$, mw 361.50, N 11.63%; col orthorhmb tablets or bipyramides; mp 102-4° (dec); very sol in $CHCl_3$; moderately sol in benz or CS_2; sl sol in acet, EtOAc, alc, MeOH or eth. It was prepd by the interaction of triphenylmethyl chloride and Na azidodithiocarbonate in acet. The ppt was recrystd from chlf

Remarks on Acyl and Aryl Derivatives

All of the above org azidodithiocarbonates were wh crystn compds which at RT undergo slow spontaneous decompn with ultimate quant formation of the corresponding thiocyanate or isothiocyanate, sulfur and nitrogen. The velocity of this decompn is sufficiently retarded at low temp to permit storage of samples at 0° for several days without deterioration

Unlike the inorganic salts of the azidodithiocarbonic acid, the org derivs are not particularly expl. They puff midly when held in a flame or when heated rapidly on a hot plate. On exposure to light they show no photosensitivity and undergo no coloration

Refs: 1)Beil – not found 2)L.F.Audrieth et al, JACS **52**, 1928-35(1930) & CA **24**, 3221 (1930)

Alkali and Alkaline Earth Salts of Azidodithiocarbonic Acid

The azidodithiocarbonates of the alkali and alkaline earth metals are all wh, deliq crystalline compds; sol in w, alc, eth or acet and insol in CS_2, CCl_4, chlf or benz. These salts usually contain w of hydration which makes them more stable than the anhyd heavy metal salts. However, they decomp slowly at RT and rapidly on heating. They have been prepd by three methods:

a)direct interaction of a metallic azide with carbon disulfide

b)action of the free azidodithiocarbonic acid upon the hydroxide or carbonate of a metal or

c)double decompn of Ba azidodithiocarbonate and an alkali sulfate

Their sensitivities to friction and their brisance increase with increasing atomic wt. The Amm, K, Rb & Cs salts are characterized by their peculiar sensitivity to light. All change color when exposed to sunlight. The Cs salt may even decomp violently during the process of crystn from aq soln. The alkali salts, especially Na, can be used to prepare the heavy metal salts, such as $Pb(SCSN_3)_2$, and the alkyl or aryl derivatives of azidodithiocarbonic acid

Although many of the alkali and alkaline earth salts have been prepd, studied and found to be explosive (Refs 1,2 & 3), only the more important ones are described here

Refs: 1)Beil **3**, (86) & [159-60] 2)F.Sommer, Ber **48**, 1833-41(1915) & CA **10**, 342-3(1916) 3)A.W.Browne et al, JACS **49**, 917-25(1927) & CA **21**, 1940(1927)

Ammonium Azidodithiocarbonate, NH_4SCSN_3, mw 136.21, N 41.14%; wh cryst, non-deliq solid; mp-turns red-org at 90°, begins to decomp at 110° and melts with gas evolution at 120°; readily sol in w, alc, MeOH or acet, somewhat sol in eth and practically insol in benz or xylene. It was prepd by:a)prolonged treatment of NH_4N_3 in aq soln with CS_2 b) neutralizing free azidodithiocarbonic acid with aq NH_4OH or c)bubbling NH_3 gas through an ethereal soln of the azido-acid. The third procedure was adjudged the most satisfactory

On exposure to direct sunlight the salt undergoes coloration, within a few minutes, to a very light orn tint. A partial reversal of this color change takes place slowly in the dark

When strongly heated the salt suddenly decomps with considerable flame and a puff of smoke. On heating in a sealed tube the salt explodes violently

Refs: 1)Beil **3**, [159] 2)L.F.Audrieth et al, JACS **49**, 2130(1927) & CA **21**, 3326(1927)

Potassium Azidodithiocarbonate, $KSCSN_3$, mw 157.27, N 26.72%; wh deliq crysts, mp 126° (dec with evolution of gas); very sol in w, fairly sol in acet or MeOH; very sl sol in eth and practically insol in alc, benz, CCl_4, CS_2 or chlf. It was prepd by the action of an aq soln of KN_3 on CS_2 at 40°. The concd clear filtrate was slowly cooled over ice until crystals formed

The K salt is rather sensitive to expln by shock, heat or friction. Crysts may expl violently when broken or rubbed in an agate mortar. When heated rapidly in air the subst detonates with a sharp expln, but less violently than the heavy metal azides. On expln in air, a spectacular flame is produced with the liberation of much heat and the formation of numerous products:

$$2KSCSN_3 + 5O_2 \rightarrow K_2S + 3SO_2 + 2CO_2 + 3N_2$$

Slow thermal decompn takes place in accordance with the equation:

$$KSCSN_3 \rightarrow KSCN + S + N_2$$

Aq solns of K azidodithiocarbonate are quite stable at 10° or lower, as is the dry salt itself. At somewhat higher temps the aq solns gradually become turbid. Samples of the dry salt stored in a desiccator at RT gradually turn yel. Solns of the K azido salt, when treated with various oxidizing agents or when subjected to electrolysis, yield the more expl wh ppt, *azidocarbondisulfide,* $(SCSN_3)_2$. An important catalytic effect is exerted by the K azido salt in the reaction between aq KN_3 and I_2 in the presence of CS_2

Refs: 1)Beil **3**, [160] 2)F.Sommer, Ber **48**, 1837-8 (1915) & CA **10**, 342-3(1916) 3)A.W. Browne & A.B.Hoel, JACS **44**, 2109-13 & 2315-20(1922) & CA **16**, 4154(1922) & **17**, 65-6(1923)

Sodium Azidodithiocarbonate, $NaSCSN_3$, mw 141.16, N 29.77%; col powd of no distinct cryst form; mp explodes between 139-43°. It was prepd by dehydration of either the tetrahydrate or the dihydrate at RT over P_2O_5 in a vac desiccator. Sl decompn takes place as evidenced by the development of a pink color on the cryst surface. This color disappeared on exposure to air

The anhyd Na salt explodes violently when rubbed vigorously on a porous plate or when thrown upon a hot plate. It is not very sensitive to shock (Ref 4)

Sodium Azidodithiocarbonate Dihydrate, $NaSCSN_3 \cdot 2H_2O$, mw 177.19, N 23.72%; col orthorhmb prisms, mp 75°(dec). It was obtd by crystn from aq soln of the anhyd Na salt at RT

This compd is very similar to the tetrahydrate salt in its lack of sensitivity to friction or impact, and in its behavior on a hot plate (Ref 4)

Sodium Azidodithiocarbonate Tetrahydrate, $NaSCSN_3 \cdot 4H_2O$, mw 213.26, N 19.71%; col ndls, mp 45-50° (dec with evolution of gas). It was first prepd by Sommer (Refs 1a & 1b) on treating aq NaN_3 at 40-50° with CS_2, filtering, concdg over P_2O_5 and slowly cooling to 0°. Although fairly stable at RT, the tetrahydrate salt gradually assumes a yel tint on long standing (See also Refs 2 & 3)

It does not det when rubbed on a porous plate or when struck with a hammer. When thrown upon a hot plate it explodes with a sl puff (Ref 4)

Kuznetsov (Ref 5) used Na azidodithiocarbonate and its derivs to increase the sharpness of color tests: for example, in neutral soln the anhyd Na salt gives a yel color in the presence of Cu. Hofman-Bang (Ref 6) found that dil solns of I_2 & NaN_3 were catalyzed by Na azidodithiocarbonate and proposed a chain reaction mechanism. The Na salt is used primarily to prepare the heavy metal salts of azidodithiocarbonate, which are very sensitive expl compds

Refs: 1a)Beil **3**, (86) & [160] 1b)F.Sommer, Ber **48**, 1837-8(1915) & CA **10**, 342-3(1916) 2)E.Oliveri-Mandalà, Gazz **52** II, 139(1922) & CA **17**, 1642(1923) 3)A.J.Currier & A.W. Browne, JACS **44**, 2849-54(1922) & CA **17**, 501-2(1923) 4)A.W.Browne & L.F.Audrieth, JACS **49**, 919-20(1927) & CA **21**, 1940(1927) 5)V.I.Kuznetsov, DoklAkadN **50**, 233-9(1945) & CA **43**, 4174-5(1949) 6)N.Hofman-Bang, ActaChemScand **4**, 856-60(1950) & CA **45**,936 (1951)

Bromine Azidodithiocarbonate, $BrSCSN_3$, mw 198.08, N 21.22%; wh amor prod, unstable above −5°, turns yel to brn on standing or when rapidly heated to 200°. Attemps to prep Br azidodithiocarbonate by interaction of liq Br_2 and solid azidocarbondisulfide at RT resulted in violent explosions. In org solvs more controllable reactions take place with the probable formation of $BrSCSN_3$. Bromine reacts with Ag azidodithiocarbonate in eth to form a *tribromo azidodithiocarbonate,* Br_3SCSN_3, and in chlf or CCl_4 to form a mixt of the monobromo and tribromo compds

Due to the extreme instability of these compds, their complete isolation, exact detn of their compn and props were not accomplished

Refs: 1)Beil **3**, [159] 2)W.H.Gardner & A.W.Browne, JACS **49**, 2761-3(1927) & CA **22**, 200(1928)

Chlorine Azidodithiocarbonate, $ClSCSN_3$, mw 153.62, N 27.35%; wh solid, changing gradually, even at temp below −15°, to a viscous yel oil; insol in w or chlf. The vapor of the oil affects the eyes, appears to act as a heart depressant, and causes blistering on contact with the skin. It was formed by passing Cl_2 gas through an anhyd soln of azidocarbondisulfide in chlf at −15°, after removal of excess Cl_2 and vapzn of part of the solv

Dry azidocarbondisulfide in contact at RT with Cl_2, either in gaseous form or in concd aq soln, causes a violent expln. Only a few degrees rise in temp can cause this reacting mass to explode, almost immedy, yielding the usual prods of decompn of the halogenoid compd

Refs: 1)Beil **3**, [160] 2)W.H.Gardner & A.W.Browne, JACS **49**, 2760-1(1927) & CA **22**, 200(1928)

Cyanogen Azidodithiocarbonate, $NC \cdot SCSN_3$, mw 144.19, N 38.86%; wh crysts, stable at 0°; a large sample explodes at 65-70°; a small sample becomes yel at 60° and at 81° fusion takes place, with decompn and formation of a yel-orn residue; very sl sol in w, CCl_4 or CS_2, sol in most org solvs, especially acet or ethyl acetate at 0°. It is formed by the

interaction of azidocarbondisulfide and mercuric cyanide in acet, but is best prepd by reacting cold aq soln of Na azido dithiocarbonate with an etheral soln of cyanogen bromide

Cyanogen azidodithiocarbonate is insensitive to impact and is much more stable than azidocarbondisulfide. On expln by heat it develops a dk orn vapor comparable in odor to thiocyanogen and to cyanogen thiocyanate. It undergoes spontaneous decompn at RT, with the liberation of N_2, leaving a solid residue of sulfur, thiocyanogen and cyanogen thiocyanate. As the temp is raised, the velocity of decompn undergoes marked acceleration. At 40°, for example, complete decompn takes place within 80 hrs

When a current of NH_3 gas is brought into contact with the dry solid compd, expl decompn occurs immedy. Bubbled through an ethereal soln of the CN salt, NH_3 gas ppts a wh solid consisting chiefly of NH_4 azidodithiocarbonate. The CN salt and concd H_2SO_4 react with explosive violence. Other dil acids react more slowly than concd acids but all eventually effect soln of cyanogen azidodithiocarbonate

Refs: 1)Beil – not found 2)L.F.Audrieth & A.W. Browne, JACS **52**, 2799-2805(1930) & CA **24**, 3963(1930)

Guanidine Azidodithiocarbonate, $HNC(NH_2)_2 \cdot HSCSN_3$, mw 178.25, N 47.15%; col prisms, mp 88-90° (dec); readily sol in w or acet, sol in alc and almost insol in eth. It was prepd by interacting aq guanidine carbonate with freshly prepd solid azidodithiocarbonic acid or by reacting aq guanidine azide, $HNC(NH_2)_2 \cdot HN_3$, with purified CS_2

Like the inorg compds of the azido acid, and unlike the alkyl and aryl derivs, the guanidine salt is photosensitive. In the dark it may be stored below 10°, in vacuo, for days without appreciable decompn. On long standing at RT, the salt decomps quanty, yielding N_2, sulfur and guanidine thiocyanate. In aq soln the azido salt reacts with $AgNO_3$ to form the insol Ag azidodithiocarbonate

When heated on a Pt foil, guanidine azidodithiocarbonate decomposes rapidly, with evolution of much gas, but without deton

Refs: 1)Beil – not found 2)J.Craik et al, JACS **56**, 2380-1 (1934) & CA **29**, 700(1935)

Heavy Metal Salts of Azidodithiocarbonic Acid. Some heavy metal salts of azidodithiocarbonic acid, such as Ag, Cu^{++}, Hg^{+}, Hg^{++}, Cd, Bi, Fe^{+++} & Zn, were prepd and studied in 1915 by Sommer (Refs 1a & 1b) before the acid was identified. He obtd these salts by reacting the appropriate aq metal azide with CS_2 at 40-50° or by reacting Na azidodithiocarbonate with the metal nitrate or chloride. No ppts were obtd with Sn, Al, Cr, Fe^{++}, Ni, Co & Mn salts. The heavy metal salts prepd by Sommer (Refs 1a & 1b) were considered to be of a complex nature and fearfully expl. Many were sol in org solvents, insol in acids and gave a color in solns different from that of the metallic ion

Rathsburg (Ref 2) obtd some of these salts by the procedures of Sommer and proposed their use, when they were phlegmatized with resin or paraffin solns, in priming compns. The Pb azidodithiocarbonate was considered of special importance and was prepd by treatment of the Na salt with $Pb(NO_3)_2$. Smith et al (Ref 3) prepd and studied the chem and expl props of the azidodithiocarbonates of Cu, Ag, An, Zn, Cd, Hg^{+}, Hg^{++}, Tl, Pb & Bi for their value as primer or detonator expls

These heavy metal salts were prepd by treating a fresh aq azidodithiocarbonic acid soln with an aq soln of the respective metal ion. The following list of salts studied includes the props which were detd by Smith et al (Ref 3):

Bismuth, formula not established; yel to reddish orn ppt, highly sensitive and may det under water

Cadmium, $Cd(SCSN_3)_2 \cdot 2H_2O$; long, fine ndls which undergo slow, spontaneous thermal decompn. The anhyd salt is very sensitive and may det, even under water on sl mechanical shock

Copper, a mixt of cupric & cuprous

azidodithiocarbonates; yel ppt which undergoes slow thermal decompn, forming an inert prod. This salt may det violently under sl mechanical shock

Gold, a mixt of aurous azidodithiocarbonate & azidocarbondisulfide; bulky wh fibrous ppt which changes to orn on standing

Lead, $Pb(SCSN_3)_2$, lt grn-yel crysts, fairly stable but may det under sl mechanical shock

Mercuric, $Hg(SCSN_3)_2$; silky ppt which undergoes spontaneous thermal decompn at RT, yielding mercuric thiocyanate. This salt may det violently under sl mechanical shock

Mercurous, $HgSCSN_3$; dk brn when first formed but becomes wh when the suspended ppt is stirred. This salt may det violently under sl mechanical shock

Silver, $AgSCSN_3$; wh photosensitive compd, either in aq suspension or in the dry state, which darkens on exposure to light. It may detonate violently under sl mechanical shock

Thallous, $TlSCSN_3$; lt yel crysts which may det violently under sl mechanical shock

Zinc, formula not established; yel, reg octahedra crysts which are exceedingly sensitive to friction and shock, even under water

Remarks: Azidodithiocarbonates of the heavy metals in the first and second groups of the periodic system show a sensitivity to mechanical impact that varies inversely with atomic wt, while the brisance of expln varies directly with the atomic wt. The sensitivity of compds of metals in the series Hg to Bi varies directly and the brisance inversely with the atomic wt. Certain of these compds, suitably stabilized and partially desensitized, should prove useful in primer or detonator compns

Refs: 1a)Beil **3**, (86-7) & [160] 1b)F. Sommer, Ber **48**, 1833-41(1915) & CA **10**, 342-3(1916) 2)H.Rathsburg, BrtiP 188302 (1922); CA **18**, 588(1924) & JSCI **42**, 688A (1923) 3)G.B.Smith et al, JACS **52**, 2806-10 (1930) & CA **24**, 3963(1930)

Tetramethylammonium Azidodithiocarbonate, $(CH_3)_4N{\cdot}SCSN_3$, mw 192.32, N 29.13; wh crystalline tablets (from w), mp 95-8° (with color change to dk grn and then dec); very sol in w, sol in MeOH or acet; sl sol in eth and insol in CCl_4, CS_2 & chlf. It can be prepd by three methods: a)neutralization of azidodithiocarbonic acid with aq tetramethylammonium hydroxide b)double decompn of Ba azidodithiocarbonate and tetramethylammonium sulfate and c)digestion of aq tetramethylammonium azide with a sl excess of CS_2 as reqd by the equation

$$N(CH_3)_4N_3 + CS_2 \rightarrow (CH_3)_4N{\cdot}SCSN_3$$

The third method of prepn was considered most satisfactory

This salt does not explode when struck with a hammer; when thrown on a hot plate or heated directly in a flame, it suddenly dec with a puff. The salt gradually dec at RT with the formation of the thiocyanate, sulfur and nitrogen.

Refs: 1)Beil – not found 2)L.F.Audrieth et al, JACS **49**, 2131-2(1927) & CA **21**, 3326 (1927)

Triethyllead Azidodithiocarbonate, $(C_2H_5)_3$-$PbSCSN_3$, mw 412.56, N 10.19%; mp (ignites without detonation). It can be prepd by the action of aq Na azidodithiocarbonate on triethyllead acetate, yielding about 80% prod

This compd may be used as a component of ignition compns for elec blasting caps

Refs: 1)Beil – not found 2)L.A.Burrows et al, USP 2105635(1938) & CA **32**, 2357-8 (1938)

Azidoethane (Triazoethane). See Ethyl Azide under Ethane and Beil **1**, [71]

Azidoethanol (2-Triazoethanol). See under Ethanol and Beil **1**, 340 & (171)

Azido-p-ethoxyphenyl Tetrazole. See under Ethoxyphenetyl Tetrazole and R.Stollé et al, JPrakt Chem **134**, 282-3 & 303-4(1932) & CA **26**, 5565 (1932)

Azidoethylalcohol or Azidoethanol (Triazoethanol). See under Ethanol and Beil **1**, 340 & (171)

Azidoethylamine. See under Aminoethane and Derivatives, p A199

Azidoethylazidoacetate (Triazoethyl Triazoacetate). See under Ethyl azidoacetate and Beil **2**, 229

Azidoethylbenzamide. See under Ethylbenzamide and Beil **8**, (97)

Azidoethylbenzene, $N_3C_6H_4 \cdot C_2H_5$. See under Ethylbenzene, and P.A.Levene et al, JBiol Chem **120**, 777(1937) & CA **32**, 484-5(1938)

Azidoethylene (Vinyl Azide), $CH_2 \cdot CH \cdot N_3$, mw 69.07, N 60.84%; yel liq, bp 26° at 760 mm Hg; it was prepd by the action of alcoholic KOH or NH_3 on 2-iodoazidoethane

Refs: 1)Beil **1**, (82) 2)M.O.Forster & S.H. Neuman, JCS **97** II, 2574(1910)

Azidoethylenediurethane. See under Ethylenediurethane and Beil **4**, (450)

Azidoethylmethylketone–Semicarbazone. See under Ethylmethylketone–Semicarbazone and Beil **3**, 102

Azidoethylnitrate (Triazoethanolnitrate). See under Ethyl Nitrate and (Beil – not found) A.H. Blatt, OSRD Rpt 2014(1944)

N-[β-Azidoethyl]-N'-Phenylurea. See under Ethylphenylurea and Beil **12**, (23)

Azidoethylurea. See under Ethylurea and Beil **4**, (360)

[α-Azidoethyl]-Urethane. See under Ethylurethane and Beil **3**, (11)

[β-Azidoethyl]-Urethane. See under Ethylurethane and Beil **4**, (360)

Azidofluorenone. See under Fluorenone and Beil **7**, [410]

Azidoformamide. See Carbamyl Azide under Formamide and Beil **3**, 129 & (59)

Azidoformamidine or Guanyl Azide. See under Formamidine and Beil **3**, 130 & (60)

Azidoformamidine Dinitrate. See under Formamidine

Azidoformamidine Nitrate. See under Formamidine

Azidoformadine, N-Nitro. See under Formadine

Azidoformamidine Perchlorate. See under Formamidine

Azidoformamidine Picrate. See under Formamidine

Azidoformic Acid. See under Formic Acid

Azidoformic Acid Dimethylamide. See under Formic Acid Dimethyl Amide and Beil **4**, [575]

Azidoformic Acid, Ethylester. See under Formic Acid and Beil **3**, 129 & [101]

Azidoformic Acid, Methyester. See under Formic Acid and Beil **3**, 129 & [101]

α-Azidoformylbutyrylglycine Azide, (formerly called in Ger "Azidoglutarsäureglycinazid), $N_3 \cdot CO(CH_2)_3 \cdot CO \cdot NH \cdot CH_2 \cdot CO \cdot N_3$, mw 239.21, N 40.99%; thick oil which explodes when touched with a flame

Refs: 1)Beil **4**, [791] 2)T.Curtius et al, JPraktChem **105**, 324(1922)

2-Azidoformylphenylisocyanate (2-Isocyanate Benzoyl Azide), $OC:N \cdot C_6H_4 \cdot CON_3$, mw 188.15, N 29.78%; Needles (from benz), mp 60° (dec) on careful heating; explodes violently on rapid heating or on impact. It can be prepd by heating phthalic acid diazide in benz

Refs: 1)Beil **14**, [225] 2)H.Lindemann & W.Schultheis, Ann **464**, 250(1928)

β-Azidoformylpropionylglycine Azide (formerly called in Ger "Azidosuccinylglycinazid"), $N_3 \cdot CO \cdot CH_2 \cdot CO \cdot NH \cdot CH_2 \cdot CON_3$, mw 211.15, N 46.44%; leaflets which explode when touched with a flame

Refs: 1)Beil **4**, [791] 2)T.Curtius et al, JPraktChem **105**, 305(1922)

Azidogallacetophenone. See under Gallacetophenone and Beil **8**, (686)

Azidoglutaricacideglucine Azide. Same as α-Azidoformylbutyrylglycine Azide (qv)

Azidoglycolic Acid. See under Glycolic Acid and Beil **3**, 244, (94) & [175]

Azidoguanidine Perchlorate. See under Guanidine

Azidoguanidine Picrate. See under Guanidine

3-Azidoheptane (3-Triazoheptane). See under Heptane and P.A. Levene et al, JBiolChem 20, 759(1937) & CA **32**, 486-7(1938)

6-Azido-2,3,5,2'4',5'-Hexamethylazobenzene. $(CH_3)_3(N_3)\cdot C_6H\cdot N{:}N\cdot C_6H_2(CH_3)_3$, mw 307.39, N 22.79%; red needles (from eth), mp 90-1° (dec); explodes mildly on rapid heating or on contact with concd H_2SO_4. It can be prepd from 6-amino-2,3,5,2',4',5'-hexamethylazobenzene as described in Ref 2

Refs: 1)Beil **16**, 76 2)T.Zincke & H. Jaenke, Ber **21**, 546(1888)

Azidohemimellitine or Azido-1,2,3-trimethylbenzene. See under Hemimellitine and Derivatives

Azidohexane (Triazohexane). See under Hexane and P.A.Levene et al, JBiolChem **120**, 759(1937) & CA **32**, 486-7(1938)

Azidohydrin. See individual azido derivatives of the hydrogen acid ester of a polyatomic alcohol, such as glycol glycerol, etc

Azidohydrocinnamic Acid. See under Hydrocinnamic Acid and Beil **8**, (205-6)

Azidohydroquinone. See under Hydroquinone and Beil **6**, (419)

Azidohydroxyacetophenone. See under Hydroxyacetophenone and Beil **8**, [87]

Azidohydroxymethoxybenzaldehyde. See under Hydroxymethoxybenzaldehyde and Beil **8**, 262

5-Azido-3-[(2-Hydroxy-1-Naphthyl)-azo]-1,2,4-Triazole. See under Hydroxynaphthylazotriazole and R.Stollé & W.Dietrich, J PraktChem **139**, 193-210(1934) & CA **28**, 2714(1934)

Azidohydroxyphenylpropionic Acid. See under Hydroxyphenylpropionic Acid and Beil **10**, [152 & 156]

Azidohydroxytetrazole. See under Hydroxytetrazole and A.H.Blatt, OSRD Rpt 2014 (1944)

Azidoiminomethanediazonium Hydroxide. See under Iminomethanediazonium Hydroxide and R.Hofsommer & M.Pestemer, ZElectrochem **53**, 383(1949) & CA **44**, 4331(1950)

1-(Azido-iminomethyl)-4-Guanyl-1-Tetrazene. See under Iminomethylguanyltetrazene and R.Hofsommer & M.Pestener, ZElectrochem **53**, 383(1949) & CA **44**, 4331(1950)

1-Azido-2-iodoethane. See under Iodoethane and Beil **1**, (33)

Azido-4-iodopicolinyl. See under Iodopicolinic Acid and R.Graf et al, Ber **64B**, 21-6(1931) & CA **25**, 2429(1931)

Azidoiodosobenzene (Triazoiodosobenzene). See under Iodosobenzene and Beil **5**, (142)

Azidoiodoxybenzene (Triazoiodoxybenzene). See under Iodoxybenzene and Beil **5**, (142-3)

Azidolactic Acid. See under Lactic Acid and Beil **3**, (110)

Azidomethane (Methyl Azide). See under Methane and Beil **1**, 80 & [48]

Azidomesitylene or Azido-1,3,5-Trimethylbenzene. See under Mesitylene and Derivatives

Azidomethoxyacetophenone. See under Methoxyacetophenone and Beil **8**, [87]

Azidomethoxytoluene. See under Methoxytoluene and Beil **6**, (181,195 & 207)

4-Azidomethylaniline (4-Methyl-4-azidoaniline). See under Methylaniline and Beil **12**, [429]

Azidomethylanisole. See under Methylanisole and Beil **6**, (181, 195 & 207)

Azidomethylbenzene (Azidotoluene). See Tolyl Azide under Toluene and Beil **5**, 349, (174) & [273]

Azidomethylbutane. See Amylazide under Amylamine and Derivatives, p A395

Azidomethylbutanone. See under Methylbutanone and Beil **1**, (353)

Azidomethylcarbimide. Same as Azidomethyl Isocyanate (qv)

Azidomethyldinitrobutylbenzene. See under Methylbutylbenzene and Beil **5**, 439

Azidomethylcarbamyl Azide. See under Methylcarbamic Acid and Beil **3**, (17)

Azidomethylethylketone Semicarbazide. See under Methylethylketone Semicarbazide and Beil **3**, 102

Azidomethylethyl Ketoxime; (Triazomethylethyl Ketoxime). See under Methylethylketoxime and Beil **1**, 671

Azidomethylformate (Azidoformicacid, Methyl Ester) (called Carbazidsauremethylester in Ger), $N_3 \cdot CO_2 \cdot CH_3$, mw 101.07, N 41.58%; col liq, bp 102-3°, explodes on heating near bp. It can be prepd by the action of ammonium nitride on chloromethylformate in ether

Ref: 1)Beil **3**, 129 & [101] 2)T.Curtius & K.Heidenreich, JPraktChem **52**, 480(1895) & JCS **70** I, 143(1896)

Azidomethylfuroyl. See Methylfuroylazide under Methylfuroic Acid and H.B.Stevenson & J.R.Johnson, JACS **59**, 2528(1937) & CA **32**, 937-8(1938)

Azidomethyl Isocyanate; Azidomethyl Carbimide or Triazomethyl Carbimide (called Azidomethylcarbonimid in Ger), $N_3 \cdot CH_2 \cdot N{:}CO$, mw 98.07, N 57.14%, OB to CO_2 −65.3%; col sharp smelling oil, bp 44-5° at 32 mm, d 1.2580 at 18°. It can be prepd from azidoacetyl chloride and NaN_3 in ether. Explosions often happened during its prepn

Refs: 1)Beil **3**, (17) & **26**, (77) 2)M.O. Forster & R.Müller, JCS **97**I, 1062(1910)

1-Azido-3-Methylpentane or 3-Methyl-1-triazopentane. See under Methylpentane and P.A.Levene & A.Rothen, JChemPhys **5**, 985(1937) & CA **32**, 1151(1938)

N-Azidomethyl-N'-phenylurea. See under Methylphenylurea and Beil **12**, (233)

Azidomethylurea or Triazomethylcarbamide (called Azidomethylharnstoff in Ger), $H_2N \cdot CO \cdot NH \cdot CH_2 \cdot N_3$, mw 115.10, N 60.85%, OB to CO_2 −76.5%; cryst (from acet), mp 56°, easily sol in acet, insol in benz & chlf. It can be prepd from azidomethylisocyanate and NH_3 in dry ether. Its expl props were not investigated

Refs: 1)Beil **3**, (27) 2)M.O.Forster & R. Müller, JCS **97** I, 1065(1910)

Azidonaphthalene. See under Naphthalene and Beil **5**, 565, (265) & [459]

Azidonitroacetyl Chloride. See under Acetyl Chloride and Derivatives, p A56

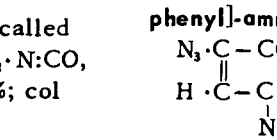
Azidonitrobenzene. See under Benzene and Beil **5**, 278, (143) & [209]

2-Azido-4-nitrobenzenesulfonic Acid. See under Benzenesulfonic Acid and Beil **11**, 81

5-Azido-6-Nitro-Benzofurazan-3-oxide. See under Benzofurazan and R.J.Gaughran et al, JACS **76**, 2233 (1954) & CA **49**, 6238(1955)

5-Azido-6-nitrobenzfuroxan. See under Benzfuroxan and J.R.Gaughran et al, JACS **76**, 2235(1954)

2-Azido-6-nitro-1,4-benzoquinone-4-trimethylimide or Trimethyl-[3-azido-5-nitro-4-hydroxyphenyl]-ammonium Hydroxide,



mw 255.23, N 27.44%,

OB to CO_2 −128.5%; red needles or scales (from w), mp becomes brn ca 100° and then explodes. It can be prepd from 2,6-dinitro-1,4-benzoquinone-4-trimethylamine by reduction [with $(NH_4)_2S$], diazotization and treatment with NaN_3

Refs: 1)Beil **13**, (198) 2)R.Meldola & W.F. Hollely, JCS **105** I, 1477(1914); PrRoySoc **30**, 159-60(1914) & CA **8**, 3026-7(1914)

1-Azido-N-Nitroformamidine. See under Formamidine

Azidonitromesitylene. See under Mesitylene and Beil **5**, [316]

Azidonitronaphthalene. See under Naphthalene and Beil **5**, [459]

Azidonitrophenol. See under Phenol and Beil **6**, 294

Azidonitrotoluene. See under Toluene and Beil **5**, 350 & (174)

6-Azido-2-nitro-4-trimethylammonium-1-benzoquinone. Same as 2-Azido-6-nitro-1-benzoquinone-4-trimethylimide (qv)

Azidoöxalicacid Ethylester or Oxalylethylester Azide (called Azido-oxalsäure-äthylester or Oxalsäure-äthylester-azid in Ger), $N_3 \cdot CO \cdot CO_2 \cdot C_2H_5$, mw 143.10, N 29.37%, OB to CO_2 −83.9%; col oily liq, bp explodes very violently on heating; sol in eth, insol in w and decompd by hot alc. It can be prepd from the hydrazide of oxalylethylester and $NaNO_2$ in well cooled aq soln

Refs: 1)Beil **2**, (244) 2)T.Curtius & K. Hochschwender, JPraktChem **91**, 434(1915) & JCS **108** I, 787-8(1915)

Azidoöxamate. See Oxamylazide under Oxamic Acid and Beil **2**, (244)

Azidoöxanilic Acid. See Oxanylazide under Oxanilic Acid and Beil **12**, 772

Azidoöxomethane Diazonium Hydroxide. See under Oxomethanediazonium Hydroxide and (Beil − not found) R.Hofsommer & M.Pestemer, ZElectrochem **53**, 383(1949) & CA **44**, 4331 (1950)

Azidoöxytetrazole. See under Oxytetrazole and (Beil − not found) A.H. Blatt, OSRD Rpt 2014 (1944)

Azidopentane. See Amyl Azide, p A395

Azidophenol (Hydroxyphenylazoimide). See under Phenol and Beil **6**, 293-4

Azidophenol, Methyl Ester. Same as Azidoanisole (qv)

Azidophenylacetic Acid. See under Phenylacetic Acid and CA **51**, 17816 (1957)

Azidophenylacyl Azide. See under Aminoacetophenone and Derivatives, p A178

5-Azido-1-Phenyl-5-Azidobenzene (1-Phenyl-5-Azidotetrazole). See under Phenyltetrazole

5-Azido-2-phenyl-1,3,5-oxadiazole (2-Azido-5-phenyl-1,3,4-furodiazole) $N_3 \cdot C \cdot O \cdot C \cdot C_6H_5$ (N—N)

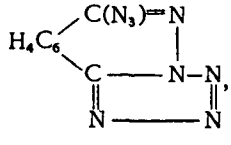
mw 187.16, N37.42%; leaflets (from alc), mp 89°, decomp at high temp and sometimes explodes midly; easily sol in alc or eth, insol in w. It can be prepd by reduction of 5-nitrosoamino-2-phenyl-1,3,4-oxadiazole

Refs: 1)Beil **27**, [633] 2)R.Stollé & K. Fehrenbach, JPraktChem **122**, 315(1929) & CA **24**, 115(1930)

Azidophenyl Propionic Acid (α-Azidohydrocinnamic Acid). See under Hydrocinnamic Acid and Beil **9**, (205)

5-Azido-1-phenyl-tetrazole (Phenyl-1-azido-5-tetrazole). See under Phenyl Tetrazole and R.Stollé, JPraktChem **134**, 282-3 & 297(1932)

(1'-Azidophthalazine-4',5')-5,1-tetrazole; (Tetrazolo-1,2-azido-4-phthalazine-1,2-dihydride),



mw 212.18, N52.81%; needles, mp 152°; easily sol in alc, diff sol in eth, nearly insol in w. It can be prepd by boiling for 3 hrs under reflux, 5 g of 1,4-

dichlorophthalazine (mp 163°) dissolved in eth, with 5 g of NaN_3

Refs: 1)Beil – not found 2)R.Stollé & H.Storch, JPraktChem **135**, 128 & 131(1932) & CA **27**, 725(1933)

Azidopropane (Propyl Azide). See under Propane and R.E.Schaad, USP 2557924(1951) & CA **46**, 1028(1952)

Azidopropanedicarbonic Acid. See under Propanedicarbonic Acid and Beil **2**, (276)

3-Azido-1,2-propanediol Dinitrate. See under Propanediol

Azidopropane Oxime. See under Propaneoxime and Beil **1**, 661

Azidopropanol. See under Propanol and C.A. Vander Werf et al, JACS **76**, 1231-5(1954) & CA **49**, 5284-5(1955)

Azidopropanone. See Azidoacetone under Acetone and Derivatives, p A39

3-Azido-1-propene. See Allylazide, p A137

Azidopropionaldehyde. See under Propionaldehyde and J.H.Boyer, JACS **73**, 5248-52 (1953) & CA **47**, 490(1953)

Azidopropionic Acid (α-Triazopropionic Acid). See under Propionic Acid and Beil **2**, 263 (114 & 115) & [234]

Azidopropionic Acid Amide. See under Propionic Acid Amide and Beil **2**, 263-4 & (114)

Azidopropionic Acid, Ethyl Ester. See under Propionic Acid and Beil **2**, 263-4 & (114)

Azidopropionic Acid, Methyl Ester. See under Propionic Acid and J.H.Boyer, JACS **73**, 5248-52(1951) & CA **47**, 489-90(1953)

Azidopropionitrile. See under Propionitrile and J.H.Boyer, JACS **73**, 5248-52(1951) & CA **47**, 489-90(1953)

Azidopropionylazide. See under Propionic Acid and Beil **2**, (115)

Azidopropylamine. Same as Azidoaminopropane described under Aminopropane and Derivatives, p A250

Azidopropyleneglycol Dinitrate. See under Propyleneglycol and A.H.Blatt, OSRD Rept 2014(1944)

1-[Azido-iso-propylidene]-semicarbazide. See under Propylidenesemicarbazide and Beil **3**, 102

Azidopropylurea. See under Propylurea and Beil **4**, (368)

1'-Azidopseudocumene or Azidotrimethylbenzene. See under Pseudocumene and Beil **5**, 405

Azido-2,5-pyrazinedicarbonyl. See under Pyrazinedicarbonyl and P.E.Spoerri & A. Erickson, JACS **60**, 400-2(1938) & CA **32**, 2535(1938)

Azidoquinazolinetetrazole. See under Quinazolinetetrazole and R.Stollé & F. Hanusch, JPraktChem **136**, 10,12 & 120(1933)

Azidosalicylic Acid. See under Salicylic Acid and M.J.Sullivan & C.K.Banks, USP 2633470 (1953) & CA **48**, 2107(1954)

Azido-iso-succinic Acid. See under Succinic Acid and Beil **2**, (272)

Azido-iso-succinic Acid Diamide. See under Succinic Acid Diamide and Beil **2**, (272)

Azido-iso-succinic Acid, Diethylester. See under Succinic Acid, and Beil **2**, (272)

Azidosuccinic Acid, Diethylester. See under Succinic Acid and Beil **2**, (270)

Azidosuccinic Acid, Dihydrazide. See under Succinic Acid and Beil **2**, (271)

Azidosuccinyldiazide. See under Succinic Acid and Beil **2**, (271)

Azidosulfonic Acids. See under names of individual sulfonic acids

Azidotetramethylazobenzene. See Tetramethylazobenzene and Beil **16**, 74

Azidotetrazole. See Tetrazolylazide under Tetrazole and Beil **26**, 347, (110) & [197 & 361]

ω-Azidotoluene. See Benzylazide under

Toluene and Beil **5**, 350, (174) & [274]

Azidotoluene. See Tolylazide under Toluene and Beil **5**, 349(174) & [273]

Azidotriazole. See under Triazole and Beil **26**, 21

2-Azido-3,5,6-trichloro-1,4-benzoquinone. See under Trichlorobenzoquinone and A. Korczynski & St. Namyslowski, BullFr **35**, 1186(1924) & CA **19**, 644(1925)

Azido-1,2,3-trimethylbenzene or Azidohemimellitene. See under Hemimellitene and Derivatives

Azido-1,2,4-trimethylbenzene or Azidopseudocumene. See under Pseudocumene and Derivatives

Azido-1,3,5-trimethylbenzene or Azidomesitylene. See under Mesitylene and Derivatives

1-Azido-2,4,6-trinitrobenzene; (Trinitrophenyl Azide or Picryl Azide). See under Benzene and Beil **5**, 279, (144) & [209]

Azidotrinitromethane. See under Methane

Azido-iso-valeric Acid. See under Valeric Acid and Beil **2**, 318

Azido-iso-valeric Acid Amide. See under Valeric Acid Amide and Beil **2**, 318

Azido-iso-valeric Acid, Ethylester. See under Valeric Acid Ethyl Ether and Beil **2**, 318 & (139)

Azido-iso-valeryl Azide. See under Valeric Acid and Beil **2**, 316

Azidoxylene or Dimethylphenyl Azide. See under Dimethylbenzene and Beil **5**, [296 & 303]

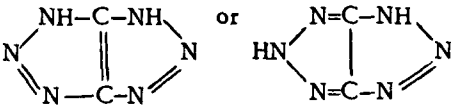
Aziethane. Same as Diazoethane

Aziethylene. Same as Diazoethane

Azimethane. Same as Diazomethane

Azimethylene. Same as Diazomethane

Azimid. A heterocylic compd, $C_2H_2N_6$,



listed in Beil **26**, 601 as Triazole-4'.5':4.5-triazol or 4.5-Azimino-1.2.3-triazol

Azimide. Same as Benzazide or Benzoyl Azide

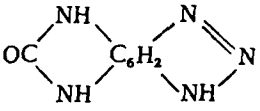
Azimido- or Azimino-. The bivalent group, –NH·N:N–, called triazene in this work (See Nomenclature, p III)

Azimidobenzene, $C_6H_5N_3$. The p-amidobenzene is the parent compd of anhydro-p-amino-diazo-compds (Beil **26**, 55)

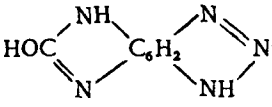
Azimido-; Azinitroso-; Oxazimido-; Oxaznitroso- and Nitrosoazimido Compds. See H. Conrad & C. Willgerodt, JPraktChem **55**, 375-98(1897) & JCS **72** I, 518(1897)

Azimidol-4,5-dicarboxylic Acid or 1-Hydroxy-1,2,3-triazole-4,5-dicarboxylic Acid [called 1-Oxy-1.2.3-triazol-dicarbonsaure-(4,5) in Ger]. See under Triazoles

5,6-Azimino-benzimidazole or 2-Hydroxy-5,6-azimino-benzimidazole (called 5,6-Azimino-benzimidazolon or 2-Oxy-5.6-azimino-benzimidazol in Ger);



or

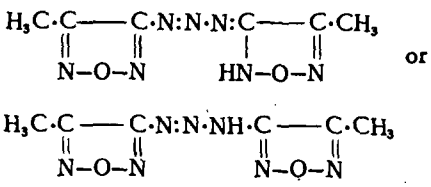


; mw 175.15, N 39.99%;

yel lfts, mp >300°; sol in cold dil NaOH or concd NH_3 with lt yel col; diffc sol in alc; insol in water; prepn described in Ref 2

Refs: 1)Beil **26**, (193) 2)O.Kym & L.Ratner, Ber **45**, 3249(1912) & CA **7**, 1184-5(1913)

3,3'-Aziminobis [4-methyl] furazan (called 1,3-Bis-[4-methyl-1.2.5-oxidiazolyl-(3)]-triazen or 4.4'-Dimethyl-[3.3'-diazoamino-furazan] in Ger),



or

mw 209.17, N 46.88%; yel lfts, hyd (from dil alc) decompg in light; mp 114°, decomg at a higher temp; the anhyd subst is sol cold alc, eth or acet and in hot ligroin, chlf or benz. The *silver salt*, separating from the alcoholic soln on adding $AgNO_3$, explodes violently on heating. It is sol in NH_3, pptd by dil nitric acid and is insol in org solvs

Refs: 1)Beil **27**, [868] 2)G.Ponzio & G. Ruggeri, Gazz **53**, 305(1923) & CA **17**, 3873-4 (1923)

Azine. This term has the following meanings: a)Pyridine (Ref 2) b)Sym di-ylidene derivs of hydrazones of ketones or aldehydes, such as *acetone azine*, $(CH_3)_2C{:}N{\cdot}N{:}C(CH_3)_2$ (Ref 3) c)The group $(N_3)_2$ is called free "azine" or nitrine. According to Walden & Audrieth (Ref 1), the *halides of azine* are extremely expl substs which undergo spontaneous decompn at RT

Refs: 1)P.Walden & L.F.Audrieth, ChemRevs **5**, 354(1928) & CA **22**, 4396(1928) 2)Hackh's (1944),90 3)CA **39**, 5925(1945) (Nomenclature)

Azinepurine (A term applied to a hypothetical subst); one deriv is called 6-Oxo-2-imino-5.6-dimethyl-tetrahydro-[pyrimidino-4'.5':2.3-pyrazin] in Ger , $C_8H_9N_6O$, mw 205.20, N 40.96%; yel ndls, sublimes without melting. An isomeric compd which forms a yel crystn Ag picrate salt, $AgC_8H_8N_5O$

Refs: 1)Beil **26**, 494 & 586 2)F.Sachs & G.Meyerheim, Ber **41**, 3965(1908) & CA **3**, 436-7(1909)

Azine of Tetrabromodintraminobenzaldehyde. See 3,3',5,5'-Tetrabromo-4,4'-dinitraminobenzalazine

Azino. The tetravalent radical, =N·N=

Azione dirompente o Azione frantumante (Ital). A shattering, fragmenting or brisant action

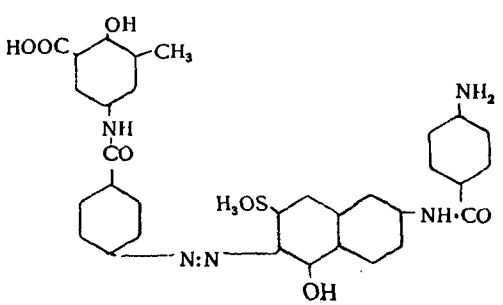
Azirane. Same as Ethylenimine

AZOCOMPOUNDS

Org compds having two hydrocarbon radicals attached to the azo group, –N=N–, and having the general formula $R{\cdot}N{:}N{\cdot}R'$ are called azocompds. If the radicals R and R' are the same, azo is usually prefixed to the name of the compd from which the radical is derived: for example, $CH_3{\cdot}N{:}N{\cdot}CH_3$ is called azomethane. However, there appears to be no uniform and consistent system of naming azocompds, especially when the radicals R and R' are identically substituted aromatic or aliphatic derivatives. For example, the prefix "bis" or "di" is used in the literature for both aromatic and aliphatic azocompds in addition to the azo designation

In this work we have usually listed the azocompds without any prefix, as a first name, unless the compd is known and found in the literature only as an azobis-or azodi-derivative, for example azobis-formic acid

When the radicals R and R' are different substituents, azo is placed between the names of the compds from which the radicals are derived: for example, $C_6H_5{\cdot}N{:}N{\cdot}CH_3$ is called benzene-azo-methane. This system suffices for naming the simpler compds, but is impracticable for the complicated dyestuff compds which are commonly known by trivial names. For example, amino-cresotinic acid can be converted into its p-nitrobenzoyl deriv, the nitro group reduced, and the resulting amino group diazotized and coupled with p-amino-benzoyl-J acid. The resulting dye, called "Diazo Light Scarlet 5BI" can be coupled with β-naphthol to give a complex azo dye-stuff which can be given no simple name (Ref 6, p 450):



The azocompds differ markedly from the diazocompds. The latter also contain doubly linked nitrogen atoms but they are attached to the same carbon atom, as represented by the characteristic diazo structural formula, >C=N⇄N or >C←N≡N. As a group, azo compds are more stable and less reactive than the diazo derivs

Azo compds may be divided into the following classes:

a)Aliphatic – in which both radicals are aliphatic

b)Mixed – in which one radical is aliphatic and the other aromatic

c)Aromatic – in which both radicals are aromatic

d)Hydroxy – aromatic compds contg a hydroxyl substituent and

e)Amino – aromatic compds contg an amino substituent

The last two classes include the largest group of synthetic org compds known, the azo dyes. The aromatic azocompds (class c, above) which are all colored solids, ranging from red to violet, are the most important to the expl industry.

Aromatic azocompds can be prepd by:

a)reduction of aromatic nitro derivatives in alkaline soln

$$\phi\cdot NO_2 \xrightarrow[\text{Alkali}]{\phi NH\cdot NH\phi} \phi\cdot N{:}N\cdot\phi$$

b)oxidation of the corresponding hydrazo compd

$$Ar\cdot NH\cdot NH\cdot Ar \longrightarrow Ar\cdot N{:}N\cdot Ar$$ and

c)condensation of an amine with a nitroso compd

$$Ar\cdot NO + H_2N\cdot Ar \longrightarrow Ar\cdot N{:}N\cdot Ar$$

These compds can be oxidized by peracetic acid to azoxy compds. They are easily reduced, first to hydrazo compds and then to two primary amines. Azocompds are unaffected by aqueous acids and alkalies. They are sol in concd HCl or HF acids and compds can be obtd from these solns which contain the acid

Rathsburg (Ref 5) proposed the use of nitrated aromatic azocompds and their heavy metal salts as a top-charge, and singly or together with other ingredients for priming compns in blasting detonators. Clarke (Ref 11) sensitized Mg pdr by coating it with azocompds to increase flammability. The list of azocompds and their metal salts given after these refs includes the more important ones from the viewpoint of their possible use in the expl industry

Refs: 1)P.Lemoult, AnnChimPhys [8] **14**; 289-310(1908) & CA **2**, 3298-9(1908) 2)W. Swietoslawski, ZhRusFiz-KhimObshch **41**, 920-5(1909) & CA **5**, 1414-5(1911) 3)W. Swietoslawski, Ber **43**, 1479-88, 1488-95 & 1767-73(1910) & CA **4**, 2488-90, 2638-9 & 2801(1910) 4)W.Swietoslawski, Ber **44**, 2429-45(1911) & CA **5**, 3816-7(1911) 5)H. Rathsburg, BritP 177744(1921) & CA **16**, 3399(1922); BritP 185555(1921) & CA **17**, 147-8(1923); BritP 190215(1921) & CA **17**, 3101(1923); GerP 411574(1923) & JSCI **44**, B739(1925); USP 1511771(1925) & CA **19**, 178-9(1925) and USP 1580572(1926) & CA **20**, 1907(1926); and with W.Friederich, BritP 195344(1922) & CA **17**, 3609(1923) 6) Sidgwick, OrgChem of N (1937),431 7)Davis (1943),127 8)V.O.Lukashevich, ZhObshch-Khim **17**, 808-22(1947) & CA **42**, 6763(1948) 9)Kirk & Othmer **2** (1948), 224-6 10)Degering (1950), 414,839,908,1128,1159,1192,1441 & 1896 11)R.G.Clarke, USP 2718463(1955) & CA **50**, 2174(1956)

LIST OF AZOCOMPOUNDS

Azoaminobiphenyl. See Azobis(p-amino-biphenyl)

Azo-aminotetrazole or 5-Azo-5'-aminotetrazolyl. See Di(tetrazolyl-5):N^1,N^3-triazene

Azoaniline; Azobisaniline or Azodianiline, $H_2N{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}NH_2$, mw 212.25, N 26.40%. Three isomers: o,o'-, m,m'- and p,p'- are described in Beil **16**, 303,305,334(309,319) & [148,149,174]. There are also isomers, *diaminoazobenzenes*, $C_6H_5{\cdot}N{:}N{\cdot}C_6H_3(NH_2)_2$. They are described under D

No azido-, diazido-, nitro-, dinitro-, etc derivs of azoaniline were found in Beil or CA through 1956, but there exist nitrated compds of diaminoazobenzenes

AZOANISOLE AND DERIVATIVES

Azoanisole or Azobisanisole (called Dimethoxy-azobenzol in Beil), $CH_3{\cdot}O{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}O{\cdot}CH_3$, mw 242.27, N 11.56%. Three isomers are described in the literature: o,o' - Azoanisole (Ref 1); m,m' – Azoanisole (Ref 2) and p,p' – Azoanisole (Ref 3). The nitro derivs of Azobisanisole may be of interest as expl ingredients

Refs: 1)Beil **16**, 92,(233) & [33] 2)Beil **16**, 95 & [37] 3)Beil **16**, 112,(237) & [43]

Dinitroazoanisole or 4,4'-Azobis-(3-nitroanisole), $CH_3{\cdot}O{\cdot}C_6H_3(NO_2){\cdot}N{:}N{\cdot}(NO_2)C_6H_3{\cdot}O{\cdot}CH_3$, mw 332.27, N 16.86%; bright orn prisms (from nitrobenzene), mp 259°. It was prepd by diazotizing 3-nitro-p-anisidine, $O_2N(CH_3O)C_6H_3NH_2$, and treating the diazonium soln with aq CuOH. This action yielded 2,2'–dinitro-p,p'-bisanisole and a considerable amt of the azobisnitro-anisole obtained from the acetic acid insol residue. See also Ref 2 for prepn by oxidation of 4-methoxy-2-nitroaniline

Refs: 1)W.C.Lothrop, JACS **64**, 1700(1942) & CA **36**, 5805(1942) 2)K.H.Pausacker & J.G.Scroggie, JCS **1954**, 4501 & CA **49**, 13226(1955)

Tetranitroazoanisole, $CH_3{\cdot}O{\cdot}C_6H_2(NO_2)_2{\cdot}N{:}N{\cdot}(NO_2)_2C_6H_2{\cdot}O{\cdot}CH_3$, – not found in Beil or CA through 1956

Hexanitroazoanisole or 3,3'-Azobis-(2,4,6-trinitroanisole) (called 2.4.6.2'.4'.6'-Hexanitro-3.3'-dimethoxy-azobenzol in Ger), $CH_3{\cdot}O{\cdot}C_6H(NO_2)_3{\cdot}N{=}N{\cdot}C_6H(NO_2)_3{\cdot}O{\cdot}CH_3$; mw 512.28, N 21.88%; dk red-yel cryst (from AcOH) or red-brn cryst (from MeOH), mp 126-7°, explodes on strong heating; easily sol in glacial acetic acid, sol in cold alc or eth, insol in w. It was prepd by nitration of m,m'-azoanisole with KNO_3 and concd H_2SO_4

Refs: 1)Beil **16**, [38] 2)K.Elbs & O.H. Schaaf, JPraktChem **120**, 2,3 & 11(1928) & CA **22**, 4508(1928)

Azobenzaldehyde; Azobisbenzaldehyde or Azodibenzaldehyde, $OHC{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}CHO$, mw 238.24, N 11.76%. Two isomers are described in the literature: m,m'-, orn-colored plates, mp 150° and p,p'-, red-orn crysts, mp 237-9°. No azido-, diazido- and nitrated derivs were found in Beil or CA through 1956

Ref: Beil **16**, 209-10

AZOBENZENE AND DERIVATIVES

Azobenzene; Azobenzole or Diphenyldiimide (also called Benzeneazobenzene in CA), $C_6H_5{\cdot}N{:}N{\cdot}C_6H_5$; mw 182.22, N 15.37%, OB to CO_2 -254.6%; orn monoclinic crysts, mp 68°, bp 297° and d 1.203 at 20°/4°; Q_c^p 1544.6 kcal/mol (Ref 4); Temp of Expln 540° (Ref 3); Vapor Press at various temps (Ref 9); sl sol in eth, ligroin & alc (4.2 g in 100 g alc at 20°). Azobenzene is quite toxic to animals (Refs 6 & 8) but its effect on humans is not known (Ref 10). It can be prepd by reduction of nitrobenzene with Na stannite, Fe in aq NaOH or by other methods (Ref 1). The industrial prepn is described in Ref 7

There are many other references in the literature on the physical & chemical props of azobenzene

Azobenzene was used in France in "Cheddite" type expls (Ref 2)

Cheddite Type Explosives

Composition	(% by weight) 1	2	3	4
Azobenzene	11.17	13.89	7.68	17.5
$KClO_3$	66.66	66.66	79.12	–
$NaClO_3$	–	–	–	75.0
Azobenzene picrate	16.61	–	–	–
NG	2.78	13.89	–	–
Castor oil	2.78	2.78	1.10	7.5
Dinitrocellulose	–	2.78	1.10	–
Mononitrobenzene	–	–	11.00	–

It was also proposed as a sensitizer for AN expls (Ref 5). Many of the salts and other derivs of azobenzene are expl (see below)

Refs: 1)Beil **16**, 8-12,(218-20) & [4-7] 2) Daniel (1902), 742 3)R.L.Datta & N.R. Chatterjee, JCS **115**, 1008(1919) 4)W. Swietoslawski & J.Bobinska, RoczCh **9**, 723 (1929) & CA **24**, 1790(1930) 5)W.O.Snelling & J.A.Wyler, USP 1827675(1932) & CA **26**, 601(1932) 6)M.I.Smith et al, USPublic Health Rpt **1943**, 304 7)Kirk & Othmer **2** (1948), 224 8)H.B.Elkins, "The Chemistry of Industrial Toxicology," Wiley, NY(1950), 168 9)T.E.Jordan, "Vapor Pressure of Organic Compounds," Interscience,NY (1954), Chapt 7, pp 178, 199 & plate 11 10) Sax (1957), 326

4-Azidoazobenzene, $N_3 \cdot C_6H_4 \cdot N{:}N \cdot C_6H_5$; mw 223.23, N 31.38%; lt yel needles (from dil alc), mp 91-93°; sol in most org solvents; can be prepd by treating diazotized 4-aminoazobenzene with NaN_3 soln. Its explosive properties were not investigated

Refs: 1)Beil **16**, 60 & [19] 2)A.Korczynski & S.Namyslowski, BullFr [4] **35**, 1192(1924) & CA **19**, 644(1925)

Azobenzene Addition Compounds and Salts

Azobenzene-Dipotassium Hydrazobenzene, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + C_6H_5 \cdot NK \cdot NK \cdot C_6H_5$; dk violet crysts; prepd from azobenzol and 4-phenyl benzophenone K salt in ether. It is readily oxidized in air to form azobenzene. This compd is probably very unstable although no expl props are given

Refs: 1)Beil **16**, (220) 2)W.Schlenk et al, Ber **47**, 485(1914) & CA **8**, 1580-1(1914)

Azobenzene Nitrate, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + HNO_3$, red crysts, very unstable

Refs: 1)Beil **16**, (219) 2)G.Reddelien, JPraktChem **91**, 241(1915) & CA **9**, 1910-11 (1915)

Azobenzene Perchlorate, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + HClO_4$; yel plates with bluish tinge; explodes ca 208°; easily hydrolyzed

Refs: 1)Beil **16**, (219) 2)K.A.Hofmann et al, Ber **43**, 1083(1910) & CA **4**, 2464(1910)

Azobenzene Picrate, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + HO \cdot C_6H_2(NO_2)_3$; red crysts, very unstable; explode on strong heating (Refs 1 & 3). It was used in some "Cheddite" type composite expls in France (See under Azobenzene) (Ref 2)

(Refs: 1)Beil **16**, (219) 2)Daniel (1902), 742 3)G.Reddelien, JPraktChem **91**, 242 *(1915) & CA* **9**, *1910-11 (1915)*

Azobenzene-1,3,5-Trinitrobenzene, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + 2C_6H_3(NO_2)_3$; orn plates, explode on strong heating; sol in alc, eth, & benz

Refs: 1)Beil **16**, (219) 2)K.A.Hofmann & H.Kirmreuther, Ber **43**, 1767(1910) & CA **4**, 2801(1910)

Azobenzene-2,4,6-Trinitrotoluene, $C_6H_5 \cdot N{:}N \cdot C_6H_5 + 2CH_3 \cdot C_6H_2(NO_2)_3$; crysts, mp ca 65°, explodes on strong heating

Refs: 1)Beil **16**, [7] 2)M.Giua & G. Reggiani, Gazz **55**, 654(1925) & CA **20**, 1062(1926)

Nitroso- and Nitrocompounds of Azobenzene

Mononitroazobenzene, $C_6H_5 \cdot N{:}N \cdot C_6H_4 \cdot NO_2$; mw 227.22, N 18.49%, OB to CO_2 -186.6; three isomers are described in the literature:

2-Nitroazobenzene. Blood-red crysts (from alc or eth), mp 105-6°; readily sol in chlf, benz, hot alc, hot ligroin & hot glacial acetic acid

Ref: Beil **16**, 50-1 & [16]

3-Nitroazobenzene. Orn-red ndls (from alc); mp 95.5-96° (sinters ca 94°, prior to melting; readily sol in hot alc, hot ligroin & hot glacial acetic acid

Ref: Beil **16**, 52 & [17]

4-Nitroazobenzene. Reddish-orn lfts or ndls (from ligroin), mp 135°; readily sol in chlf, acet, benz, hot alc, hot ligroin & hot glacial acetic acid and very sl in cold ligroin

Ref: Beil **16**, 54, (226) & [17]

Nitrosonitroazobenzene (called 2-Phenyl-5-nitro-benztriazol-3-oxyd in Ger), $C_6H_5 \cdot N{:}N \cdot C_6H_3(NO) \cdot (NO_2)$; mw 256.22, N 21.87%; yel ndls, mp 175°; its sublimate melts at 160°. It was obtd by heating 2,4-dinitrohydrazobenzene with glacial acetic acid

Refs: 1)Beil **26**, 45 2)C.Willgerodt & B. Hermann, JPraktChem **40**, 254(1889) & JCS **56** II, 1160-1(1889)

Dinitroazobenzene, $NO_2 \cdot C_6H_4 \cdot N{:}N \cdot C_6H_4 \cdot NO_2$; mw 272.22, N 20.58%, OB to CO_2 -141.0%; five isomers are described in the literature:

2,2'-Dinitroazobenzene. Yel crysts (from toluene), mp 209-10°; readily sol in hot glacial acetic acid or toluene, diffc sol in ether, petr eth, alc or carbon tetrachloride

Ref: Beil **16**, 51, (225) & [16]

3,3'-Dinitroazobenzene. Orn ndls, mp 153°; 100 g alc dissolves 0.1 g at 15° and 2.2 g in boiling alc, 100 g ether dissolves 0.5 g at 15° and 0.8 g in boiling eth, 100 g benz dissolves 3.8 g at 15° and 36.4 g in boiling benz

Ref: Beil **16**, 52 & [17]

2,4'-Dinitroazobenzene. Orn-red lfts (from alc), mp 131-2°;

Ref: Beil **16**, 54

4,4'-Dinitroazobenzene. Orn-red ndls (from xylene) & scarlet ndls (from glacial acetic acid) mp 222-3°; readily sol in hot glacial acetic acid, moderately sol in hot acet, 100 g benz dissolves 1.8 g at 15° and 5.2 g in boiling benz; nearly insol in eth, petr eth and cold alc

Ref: Beil **16**, 54, (226) & [17]

2,4-Dinitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_5$; red ndls (from alc) mp 116-9°; prepd by reacting yel mercuric oxide with a hot alcoholic soln of 2,4-dinitro-hydrazobenzene. Treatment with fuming nitric acid gives, according to the exptl conditions, either 2,4,4'-trinitro-azoxybenzene or 2,4,4'-trinitro-azobenzene

Ref: Beil **16**, 58

Nitrosodinitroazobenzene (called 2-phenyl-4.6-dinitro-benztriazol-1-oxyd in Ger), $C_6H_5 \cdot N{:}N \cdot C_6H_2(NO) \cdot (NO_2)_2$; mw 301.22, N 23.25%; golden-yel lfts, mp 249-50°; prepd by boiling picrylhydrazobenzene with alc or acetic acid. According to Freund (Ref 3) this compd is identical with the dinitrosonitroazobenzene (qv) of Willgerodt & Ferko (Ref 2) to which the formula $C_{12}H_7N_5O_4$ had been erroneously assigned

Refs: 1)Beil **26**, 50-1 2)C.Willgerodt & M.Ferko, JPraktChem **37**, 347(1888) & JCS **54** II, 829(1888) 3)M.Freund, Ber **22**, 1664 (1889) & JCS **56** II, 977(1889)

Dinitrosoazobenzene, $(ON) \cdot C_6H_4 \cdot N{:}N \cdot C_6H_4 \cdot (NO)$; mw 210.33, N 19.98%; wh crysts, mp – sublimes at 140° and melts at 178°. It can be prepd by heating 2,4-dinitrochlorobenzene with phenylhydrazine and alc in a sealed tube at 120-30° or by heating dinitrohydrazobenzene with alc and also by mixing a soln of phenylhydrazine hydrochloride in dil alc with caustic alkali and adding an alcoholic soln of 2,4-dinitrochlorobenzene

Refs: 1)Beil – not found 2)C.Willgerodt & M.Ferko, JPraktChem **37**, 345(1888) & JCS **54** II, 830(1888) 3)C.Willgerodt & B. Hermann, JPraktChem **40**, 252(1889) & JCS **56** II, 1160-1(1889)

Dinitrosonitroazobenzene, $C_6H_5 \cdot N{:}N \cdot C_6H_2(NO)_2 \cdot NO_2$; mw 285.22, N 24.56%; golden-yel scales, mp 247.5°; prepd by boiling picrylhydrazine with acetic acid, strong HCl or dil H_2SO_4 for a short time. A subst of brownish color, melting at 145°, was also formed (See Nitrosodinitroazobenzene)

Refs: 1)Beil – not found 2)C.Willgerodt & M.Ferko, JPraktChem **37**, 345(1888) & JSC **54** II, 829(1888)

Trinitroazobenzene, $C_{12}H_7N_5O_6$; mw 317.22, N 22.08%, OB to CO_2 -108.5%; five isomers are described in the literature:

2,4,2'-Trinitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_4 \cdot (NO_2)$; reddish ndls (from alc), mp 173°; readily sol in hot benz or chlf, sol in alc or acet, diffc sol in eth and insol in petr eth
Ref: Beil **16**, 58

2,4,3'-Trinitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_4 \cdot (NO_2)$; red ndls or plates (from benz), mp 172-3°; sol in hot glacial acetic acid, benz or chlf
Ref: Beil **16**, 58

2,4,4'-Trinitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_4 \cdot (NO_2)$; red ndls (from alc), mp 170-2°; readily sol in hot alc, eth, chlf, benz or glacial acetic acid
Ref: Beil **16**, 58

2,6,4'-Trinitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_4 \cdot (NO_2)$; red-yel ndls (from chlf + alc), mp 168°; readily sol in glacial acetic acid or chlf, diffc sol in alc or eth
Ref: Beil **16**, (227)

2,4,6-Trinitroazobenzene, $(NO_2)_3 \cdot C_6H_2 \cdot N{:}N \cdot C_6H_5$; dk-red prisms (from alc), mp 142°; readily sol in chlf or benz and diffc sol in hot alc; prepd by reacting yel mercuric oxide with a hot alcoholic soln of 2,4,6-trinitrohydrazobenzene
Ref: Beil **16**, 59

Tetranitroazobenzene, $C_{12}H_6N_6O_8$, mw 362.22, N 23.20%, OB to CO_2 -83.9%; two isomers are described in the literature:

2,4,2',4'-Tetranitroazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot N{:}N \cdot C_6H_3(NO_2)_2$; pale orn ndls (from acet + alc) or orn colored tablets (from glacial acetic acid), mp 220-2°; readily sol in benz, chlf or glacial acetic acid; diffc sol in alc or ether; the alc or acetone soln turns blue on treatment with caustic soda soln. Green & Rowe (Ref 2) prepd this compd both by nitration of 2,2'-dinitroazobenzene with mixed nitric-sulfuric acid and by oxidn of 2,4-dinitroaniline in tetrachloroethane soln with an aq soln of hypochlorite. Elderfield et al (Ref 3) investigated the latter method and found that the yields were low (40% max) in small runs and even lower in larger runs

Refs: 1)Beil **16**, 59 & (227) 2)A.G.Green, JCS **101**, 2450(1912) 3)R.C.Elderfield et al, OSRD Rpt No **158**(1941) or PBL Rpt No **31094**(1941), 25

2,4,6,4'-Tetranitroazobenzene, $(NO_2)_3 \cdot C_6H_2 \cdot N{:}N \cdot C_6H_4 \cdot (NO_2)$; orn-yel ndls (from concd HNO_3), mp 163-4; diffc sol in alkalies, turning the soln red. Ciusa (Ref 2) prepd this compd by treating the dipotassium salt of 2,4,6,4'-tetranitrohydrazobenzene, $C_{12}H_6O_8N_6K_2$, with concd nitric acid

Refs: 1)Beil **16**, 59 & (227) 2)R.Ciusa, AttiAccadLinceiRend **18** II, 66(1909) & CA **4**, 1740(1910); Gazz **41** I, 694(1911) & CA **5**, 3804(1911)

Pentanitroazobenzene, $C_{12}H_5N_7O_{10}$; mw 407.22, N 24.08%, OB to CO_2 -64.8%; one isomer is described in the literature:

2,4,6,2',4'-Pentanitroazobenzene, $(NO_2)_3 \cdot C_6H_2 \cdot N{:}N \cdot C_6H_3 \cdot (NO_2)_2$; orn colored ndls (from glacial acetic acid; mp 213°, explodes on heating above its mp; readily sol in acet, ethyl acetate or nitrobenzene; diffc sol in alc, eth or benz. It was prepd by heating an acetonic soln of 2,4,6,2',4'-pentanitrohydrazobenzene, $[(NO_2)_2 \cdot C_6H_3 \cdot NH \cdot NH \cdot C_6H_2 \cdot (NO_2)_3]$, with an excess of lead peroxide for ¼ hr

Refs: 1)Beil **16**, 60 2)H.Leemann & E.Grandmougin, Ber **41**, 1307(1908) & CA **2**, 2257(1908)

Hexanitroazobenzene, $C_{12}H_4N_8O_{12}$; mw 452.22, N 24.78%, OB to CO_2 -49.5%; one isomer is described in the literature:

2,4,6,2',4',6'-Hexanitroazobenzene, $(NO_2)_3 \cdot C_6H_2 \cdot N{:}N \cdot C_6H_2 \cdot (NO_2)_3$; blood-red prisms (from NB, glacial acetic acid or concd HNO_3); mp 215-6°; diffc sol in eth, alc or benz. It can be prepd by oxidn of 2,4,6,2',4',6'-hexanitrohydrazobenzene with HNO_3 (d 1.3) or N oxide gases (obtd from HNO_3 and As_2O_3) in glacial acetic acid soln. The

hexanitrohydrazobenzene can be obtd by treating picryl chloride, $(NO_2)_3 \cdot C_6H_2 \cdot Cl$, with hydrazine, $H_2N \cdot NH_2$, or in two stages: a)by suspending a mixt of dinitrochlorbenzene, $(NO_2)_2 \cdot C_6H_3 \cdot Cl$, with hydrazine in hot w contg Na or Ca carbonate to form tetranitrohydrazobenzene, $(NO_2)_2 \cdot C_6H_3 \cdot NH \cdot NH \cdot C_6H_3(NO_2)_2$ b)on treating the latter compd with concd HNO_3, two addnl NO_2 groups are introduced and the $-NH \cdot NH-$ group is oxidized to a $-N:N-$ group

Hexanitroazobenzene is a very powerful and brisant high expl. Its *Power* as measured by the *Trauzl test* is 113% PA or 123% TNT and its *Impact Sensitivity* is between that of tetryl and PA; the FI value is 67% PA. According to Davis (Ref 3) this expl is suitable for boosters and compound detonators. The presence of the azo group in hexanitroazobenzene makes it more powerful and brisant than hexanitrodiphenylamine (qv)

Refs: 1)Beil **16**, 60 2)E.Grandmougin & H.Leemann, Ber **39**, 4385(1906) & CA **1**, 861 (1907); Ber **41**, 1295(1908) & CA **2**, 2256-7 (1908) 3)Davis(1943), 189-90 4)A.H.Blatt, OSRD 2014(1944)

AZOBENZENEDIAZONIUM DERIVATIVES

Azobenzene-4-diazonium Chloride, $C_6H_5 \cdot N:N \cdot C_6H_4 \cdot N_2Cl$; mw 244.68, N 22.90%; orn prismatic crysts, mp dec; explodes when touched with a red hot rod; sol in w, sl sol in alc. It can be prepd by several methods, one of which is diazotization of p-aminobenzene with $NaNO_2$ in HCl (Refs 1,3&4)

Azobenzene-4-diazonium Dichromate, $[C_6H_5 \cdot N:N \cdot C_6H_4 \cdot N_2]_2Cr_2O_7$; mw 624.47, N 17.94%; yel solid, mp explodes ca 134°. It can be prepd by treating azobenzene-4-diazonium chloride with K dichromate (Refs 2 & 3)

Azobenze-4-diazonium Hydroxide, $C_6H_5 \cdot N:N \cdot C_6H_4 \cdot N(:N)OH$; known only in soln and in the form of salts, many of which are expl. One expl deriv of the hydroxide is the compd called *anhydro-(4-hydroxybenzene-3-carboxylic acid-4'-diazonium hydroxide)* (qv)

An isomer of the hydroxide, called *azobenzene-4-isodiazohydroxide,* $C_6H_5 \cdot N:N \cdot C_6H_5 \cdot N:NOH$; is known only in the form of its *sodium salt* (Ref 1)

Refs: 1)Beil **16**, 616(374 & [310] 2)R. Meldola & L.Eynon, JCS **87**, 4(1905) 3)J.T. Hewitt & F.B.Thole, JCS **97**, 514(1910) 4) A.C.Sircar & E.R.Watson, JSCI **31**, 970(1912)

AZOBENZENECARBOXYLIC ACIDS AND DERIVATIVES

Azobenzenecarboxylic Acid or Benzeneazobenzoic Acid, $C_6H_5 \cdot N:N \cdot C_6H_4 \cdot COOH$, mw 226.23, N 12.38%. Three isomers: o-, m- and p- are described in Beil **16**, 225,229, 235,(287,289) & [97-8]

Nitrobenzenecarboxylic Acids, $C_{13}H_9N_3O_4$, mw 271.23, N 15.49%. Various isomers are described in the literature, but none of them seems to be expl

Refs: 1)Beil **16**, 238 2)L.Chardonnens & P.Heinrich, Helv **23**, 1410 & 1416(1940) & CA **35**, 2122(1941) 3)E.Hecker, ChemBer **88**, 1673(1955) & CA **50**, 10028(1956)

Note: No higher nitrated derivs of azobenzenecarboxylic acid as well as azido- and diazidocompds were found in Beil or CA through 1956

Azobenzenedicarboxylic Acids. See Azobenzoic Acids

AZOBENZOIC ACIDS AND DERIVATIVES

Azobenzoic Acid; Azodibenzoic Acid or Azobenzenedicarboxylic Acid, $HOOC \cdot C_6H_4 \cdot N:N \cdot C_6H_4 \cdot COOH$, mw 270.24, N 10.37%. Three isomers: o,o'-, m,m'- and p,p'- are described in Beil **16**, 228,232,233,236 & (287). Of these only the para-isomer is of interest because its nitro- and dinitro- derivs form expl salts (see below)

Azobenzene-4,4'-dicarboxylic Acid or p-Azobenzoic Acid (called p,p'-Azobenzosäure

in Ger), $HO_2C{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}COOH$; mw 270.24, N 10.37%; orn or red crysts; mp dec ca 330°, without melting. It can be nitrated to mono and dinitro-derivs (Ref 1, pp 236-7)

x-Mononitroazobenzene-4-4'-dicarboxylic Acid or Nitro-p-azobenzoic Acid, $HO_2C{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_3(NO_2){\cdot}CO_2H$; mw 315.24, N 13.33%; lt yel platelets (from alc); mp dec ca 270°, without melting. Its *silver salt,* $Ag_2C_{14}H_7N_3O_6$, yel amor powdr, insol in w, was reported to explode on heating (Refs 1, p 238 & 2)

x,x'-Dinitroazobenzene-4,4'-dicarboxylic Acid or Dinitro-p,p'-Azobenzoic Acid, $HO_2C{\cdot}C_6H_3(NO_2){\cdot}N{:}N{\cdot}C_6H_3(NO_2)CO_2H$; mw 360.24, N 15.55%; yel ndls (from alc); mp dec ca 257°, without melting. Beil (Ref 1, p 238) lists several of its salts without mentioning whether or not they are expl. Its *silver salt,* $Ag_2C_{14}H_6N_4O_8$, dk yel amor powdr, insol in w, is undoubtedly an expl compd

Refs: 1)Beil **16**, 236-8 2)A.Rodzianko, ZhRusFiz-KhimObshch **20**, 20 & 25(1888) & JCS **56**, 141-2(1889)

Azobenzol. Same as Azobenzene

Azobis-(p-aminobiphenyl) (called 4'-Azo-4-amino-biphenyl or Diaminoazobiphenyl in Ger), $H_2N{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}NH_2$, mw 364.43, N 15.38%; yel-red ndls (from benz), mp 287; sol in hot acet, hot benz or chlf; sl sol in eth and insol in w. Its prepn and props are described in Refs 1 & 2. The chloride salt, $(ClN_2C_6H_4C_6H_4N{:})_2$, called *azodiphenyldiazonium chloride* by Willstätter & Kalb (Ref 2), prismatic crysts with a violet lustre (orn-red when powd) expl at ca 95°

Refs: 1)Beil **16**, 380 2)R.Willstätter & L.Kalb, Ber **39**, 3480(1906) & CA **1**, 300-1 (1907)

Azobisanisole. See Azoanisole and Derivatives

Azobis-(ethylenenitrosonitrate), $O_2NO{\cdot}\overset{O}{\underset{\uparrow}{C}}H_2CH_2\overset{\uparrow}{\underset{O}{N}}{:}N{\cdot}CH_2{\cdot}CH_2{\cdot}ONO_2$, mw 240.14, N 23.33%; mp 126-8° (after washing with petr eth). It was prepd by treating ethylene in CCl_4 & dioxane with nitrogen tetroxide at 0-10°

In a similar manner there were prepd: *bis(propylenenitrosonitrate),* $[H_3C{\cdot}CH(ONO_2){\cdot}\overset{O}{\underset{\uparrow}{C}}H_2{\cdot}\overset{\uparrow}{\underset{O}{N}}H]_2$, mp 131-2°, from propylene and *bis(amylenenitrosonitrate)* $[H_5C_2{\cdot}CH(ONO_2){\cdot}\overset{CH_3O}{\underset{\uparrow}{C}}H{\cdot}\overset{\uparrow}{\underset{O}{N}}H]_2$, mp 102-4°, from amylene. He also claimed the prepn of *bis(isobutylene)* and *bis(1-octene)* derivs; no props are given

Because of their reactivity, these nitroso nitrates are useful in the prepn of compds contg OH, NH_2, COOH and NOH groups

Refs: 1)Beil – not found 2)J.A.Crowder, USP 2402315(1946) & CA **40**, 6092(1946)

AZOBISFORMIC ACID OR AZODIFORMIC ACID AND DERIVATIVES

Azobisformic Acid; Azodiformic Acid or Azodicarboxylic Acid (called Diimiddicarbonsäure, Azoameisensäure or Azodicarbonsäure in Ger); $HOOC{\cdot}N{:}N{\cdot}COOH$, mw 118.05, N 23.75%. Its prepn and props are described in Beil. The potassium salt, $K_2C_2N_2O_4$, a yel powd, explodes when heated above 100°

Refs: 1)Beil **3**, 122, (58) & [97] 2)J.Thiele, Ann **271**, 130(1892)

Azobis-(methylformate) or Azobisformic Acid, Dimethylester, $H_3COO{\cdot}N{:}N{\cdot}COOCH_3$, mp 122.08, N 22.95%; orn-yel oil, bp 96° at 25 mm; explodes on rapid heating. It can be prepd by treating the dimethylester of hydrazodicarboxylic acid with fuming nitric acid

Refs: 1)Beil **3**, (58) & [97-8] 2)O.Diels & P.Fritzsche, Ber **44**, 3026(1911)

Azobisformamide or Azodicarboxamide (called Azodicarbonsäurediamid or Azodicarbonamid in Ger), $H_2N{\cdot}CO{\cdot}N{:}N{\cdot}CO{\cdot}NH_2$, mw 116.08, N 48.27%; orn prismatic ndls, mp 225-30°

(dec-depending on rate of heating); Picard & Boivin (Ref 4) reported a mp of 180° with decompn; Q_c (av) 254.83 kcal/mol (Ref 5) Q_f 69.91 (Ref 5). It was prepd by oxidg hydrazodicarboxamide, $(NHCONH_2)_2$, with $K_2Cr_2O_7$ in dil H_2SO_4. This compd when heated with aniline-HCl to 220° gives 4-phenylurazole, mp 202-3° (Ref 3)

Refs: 1)Beil **3**, 123, (58) & [99] 2)J.Thiele, Ann **271**, 127(1892) 3)A.T.d'Arcangelo, RevFacultadCiencQuím (La Plata) **18**, 81-93(1943) & CA **41**, 948(1947) 4)J.P.Picard & J.L.Boivin, CanJChem **29**, 223-7(1951) & CA **45**, 9469(1951) 5)M.M.Williams et al, JPhysChem **61**, 264(1957) & CA **51**, 9284 (1957)

Azobisformamidine or Azodicarboxamidine (called Azoformamidin Azodicarbonsäure-diamidin or Azodicarbonamidin in Ger), $H_2N{\cdot}C(:NH){\cdot}N:N{\cdot}C(:NH){\cdot}NH_2$, mw 114.12, N 73.65%. Its prepn and props are described in Refs. Some of its salts are explosive. The *dinitrate*, $C_2H_6N_6 + 2HNO_3$, yel platelets (from warm w) explodes at 180-4° without melting. It can be prepd by treating a nitric acid soln of aminoguanidine nitrate with an aq $KMnO_4$ soln. The *picrate* is also explosive

Refs: 1)Beil **3**, 123 2)J.Thiele, Ann **270**, 39(1892) 3)E.Lieber et al, AnalChem **23**, 1594(1951) & CA **46**, 3857-8(1952) 4)G.F. Wright, CanJChem **30**, 62-70(1952) & CA **47**, 3793-4(1953) 5)J.C.Mackenzie et al, JOrgChem **17**, 1666(1952) & CA **48**, 686(1954)

Azobis(nitroformamidine) or 2,5-Dinitroazobisformamidine, $H_2N{\cdot}C(:NNO_2){\cdot}N:N{\cdot}C(:NNO_2){\cdot}NH_2$, mw 204.12, N 54.89%; red or orn-yel cryst which decomp explosively at 165°. This compd was obtd by Wright (Ref 2) on nitrating azobischloroformamidine, $H_2N{\cdot}C(:NCl){\cdot}N:N{\cdot}C(:NCl){\cdot}NH_2$, with 98.8% HNO_3, in the presence of acetic anhydride. The latter compd was obtd by treating azobisformamidine dinitrate, $O_3N{\cdot}H_2N{\cdot}C(:NH){\cdot}N:N{\cdot}C(:NH){\cdot}NH_2{\cdot}NO_3$, with aq NaOH. Azobis(nitroformamidine) seems to be identical with the product prepd by Thiele (Ref 1). Henry et al (Ref 3) prepd azobis-nitroformamidine by oxidation of 1,6-dinitrobiguanidine with calcium permanganate and obtd an orn-yel prod of mp 171-2° (decomp)

The expl props of this compd were not detd but Kumler (Refs 4 & 5) established its structure, dipole moment, UV and IR spectra

Refs: 1)J.Thiele, Ann **270**, 39(1892) 2) G.F.Wright, CanJChem **30**, 65-8(1950) & CA **47**, 3793-4(1953) 3)R.A.Henry et al, JACS **75**, 958(1953) & CA **48**, 2050(1954) 4)W.D. Kumler, JACS **75**, 3092-3(1953) & CA **48**, 6962-3(1954) 5)W.D.Kumler, JACS **76**, 814-6 (1954) & CA **48**, 8051(1954)

α,α'-Azobis-(chloroformamidine); Chloroazidine; Azochloramide or N,N'-Dichloroazodicarboxamidine, $H_2N(ClN:)C{\cdot}N:N{\cdot}C(:NCl)NH_2$, mw 183.02, N 45.92%; bright yel ndls or plates; mp 152° (from H_2O) (Ref 8), 155.5° (from EtOAc) (Ref 2) and 157-8° (from dioxane) (Ref 8); soly in w at 1°-150, 20°-280, 40°-610 and 61°-1490 mg/l and in many org solvs was detd (Ref 2); triethylacetylacetate is a suitable solvent (Ref 9); insol in dil alkali (Ref 8); its toxicity and expln hazards are discussed by Sax (Ref 11)

Azobischloroformamidine was first prepd in 1934 by Schmelkes & Marks (Refs 2 & 3) by careful chlorination of azodicarboxamidine or hydrazodicarboxamidine with NaOCl or Cl gas. Braz et al (Ref 5) and Takagi et al (Ref 6) prepd this compd by converting guanidine nitrate into nitroguanidine, reducing it to aminoguanidine, oxidg to azodicarboxamidine, $[:NC(:NH)NH_2]_2$, and finally chlorinating with NaOCl. Braz et al obtd a 42-5% yield of prod, 96-8% pure, melting at 146-7° while Takagi obtd yel ndls decomg at 155°

Explosive and other props of a pure sample were detd at PicArsn in 1936 by Aaronson (Ref 4):

Brisance (using tetryl and MF as initiator) 29.7 g sand crushed vs 43g for TNT

Explosion Temp, °C (5 sec) 183°

Hygroscopicity at 31°C and 90% RH 0.10%

Ignitibility by flame–flame of match causes burning at point of contact but does continue to propagate flame

Sensitivity to detonation–not detond by spit of BkPdr fuze, 0.4 MF in a No 6 blasting cap caused only partial deton

Sensitivity to impact	5" vs 14" for TNT (2 kg wt)
Solubility at 25°	*g/100 g solvent*
water	0.03
alcohol	1.25
ether	0.4
acetone	4.0
Stability Tests:	
65.5° KI Heat test	4 min
100° Heat test	
% Loss 1st 48 hrs	47.7
% Loss 2nd 48 hrs	47.9
120° Vac Stab test	11^+ cc in 3 hrs

Azobischloroformamidine is sensitive to expln by impact and has a fairly high brisance value but due to its poor stability at elevated temps, it appears unsuitable for use in military expls

Kumler (Ref 10) detd the dipole moment, UV & IR spectra and structure of azobischloroformamidine; Wright (Ref 8) studied its X-ray diffraction pattern

Most of the studies reported in the literature have been directed to the antiseptic, disinfectant, dosage or sterilizing action of azobischloroformamidine. Galvin (Ref 7) froze the compd with H_2O into a cake, sheet or film for application as a disinfectant or germicidal agent

Refs: 1)Beil – not found 2)F.C.Schmelkes & H.C.Marks, JACS **56**, 1610-2(1934) & CA **28**, 5046(1934); USP 1958371(1934) & CA **28**, 4074(1934); USP 2016257(1935) & CA **29**, 8008(1935) and BritP 436093(1935) & CA **30**, 1520(1936) 3)Wallace & Tiernan Prods,Inc, FrP 765611(1934) & CA **28**, 6948 (1934); FrP 804450(1936) & CA **31**, 3641 (1937); GerP 633561(1936) & CA **31**, 507-8 (1937) and GerP 657378(1938) & CA **32**, 4180 (138) 4)H.A.Aaronson, PA Chem Lab Rpt 47658, Nov 1936 5)G.I.Braz et al, ZhPrikl-Khim **17**, 565-9(1944) & CA **40**, 2267-8(1946) 6)S.Takagi et al, JapanJPharm & Chem **20**, 132-4(1948) & CA **45**, 5628(1951) 7)T. Galvin, USP 2521358(1950) & CA **44**, 11042 (1950) 8)G.F.Wright, CanJChem **30**, 62-70 (1952) & CA **47**, 3793-4(1953) 9)R.L.Evans & E.G.McDonough, USP 2618584(1952) & CA **47**, 11245(1953) 10)W.D.Kumler, JACS **75**, 3092-3(1953) & CA **48**, 6962-3(1954) and JACS **76**, 814-6(1954) & CA **48**, 8051-2(1954) 11)Sax (1957), 326

Azobis(ethylformamide) or Azodicarboxyethylamide (called Azodicarbonsäure-bis-äthylamid in Ger), $H_5C_2{\cdot}NH{\cdot}CO{\cdot}N{:}N{\cdot}CO{\cdot}NH{\cdot}C_2H_5$, mw 172.19, N 32.54%; orn-red lfts, mp 133°. Its *silver salt*, $Ag_2C_6H_{10}N_4O_2$, red-yel powd, explodes mildly ca 144°. It was prepd by the action of ammoniacal $AgNO_3$ on azodicarboxyethylamide in alc

Refs: 1)Beil **4**, (354) & [609] 2)O.Diels & M.Paquin, Ber **46**, 2000-7(1913)

Azobis(methylformamide) or Azodicarboxymethylamide (called Azodicarbonsäure-bismethylamid in Ger), $H_3C{\cdot}NH{\cdot}CO{\cdot}N{:}N{\cdot}CO{\cdot}NH{\cdot}CH_3$, mw 144.14, N 38.87%; yel powd, mp 170° (decomp). Its salts are not mentioned in Refs 1 & 2 (below) but it seems possible that its Ag salt might be prepd in the manner described for the prepn of the Ag azobisethylformamide salt in Ref 2, p 2006 (above). Since the Ag salt of the ethylamide is expl, it is likely that the Ag salt of the methylamide would also be expl

Refs: 1)Beil **4**, [572] 2)H.E.Cooper & E.H.Ingold, JCS **1926**, 1895

Azobis-(isobutyronitrile) (called α,α'-Azoisobuttersäure-dinitril in Ger), $NC{\cdot}C(CH_3)_2N{:}N{\cdot}C(CH_3)_2CN$, mw 164.21, N 34.12%; wh ndls or prisms (from eth); mp 103-4.5° (decomp); readily sol in alc or eth, insol in w; the rate of decompn in toluene, isobutyl alcohol, t-amyl alcohol and aniline (all at 80.2°) has been shown by Overberger et al (Ref 4) to be independent of solvent type;

$Q_{activation}$ 34 kcal/mol (Ref 4); Q_c 1217 kcal/mol and Q_f 54.63 kcal/mol (Ref 6). This compd was first prepd in 1896 by Thiele & Heuser (Refs 1 & 2) on treating a cold soln of hydrazoisobutyronitrile with Br_2 water in HCl. Rohm & Haas (Ref 7) obtd a patent for the simple and inexpensive prepn of this compd and related derivatives from the reaction of Na, Ca or alkyl hypochlorites on the corresponding amino compd. Thus, by adding $(CH_3)_2C(NH_2)CN$ with stirring at 5-10° to a soln of NaOCl and pptg with H_2O, α,α'-azobisbutyronitrile was prepd

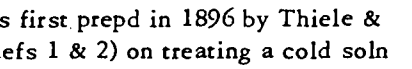
Carlisle (Ref 3) reported an expln occurred when its soln in acet was concd in a glass-lined steam jacketed vessel. The cause of this expln was not detd but examination of the subst showed that it was slightly flammable but did not explode when unconfined. Therefore, on heating in a closed system a pressure release valve should be provided

On heating azobis-isobutyronitrile in water, gas is evolved and there is formed tetramethylsuccinonitrile, mp 170° (Refs 2 & 7). This decompn of azobisnitriles, as shown by Thiele & Heuser (Ref 2) is a convenient synthetic method for obtg tetrasubstituted succinonitriles. Three new such compds, decompn products of azonitriles, were prepd and characterized by Overberger et al (Ref 4)

Azobisisobutyronitrile is reported useful as a blowing agent for the production of polymer foams and as a polymerization catalyst (Refs 4 & 7). The tetramethylsuccinonitrile product of its decompn is toxic (Ref 5)

Refs: 1)Beil **4**, 563 & (566) 2)A.Thiele & N.Heuser, Ann **290**, 30(1896) 3)P.J.Carlisle, ChemEngNews **27**, 150(1949) & CA **43**, 8681-2 (1949) 4)C.G.Overberger et al, JACS **71**, 2661-8(1949) & CA **44**, 5799(1950) 5)H.A. Watson et al, USBurMinesRptInvest No **4777** (1951) & CA **45**, 3190-1(1951) 6)W.S. McEwan & M.W.Rigg, JACS **73**, 4726(1951) & CA **46**, 4350(1952) 7)Rohm & Haas Co, BritP 672106(1952) & CA **47**, 3868-9(1953)

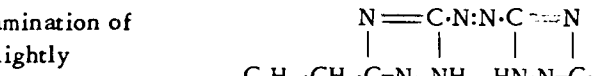
3,3'-Azobis-(4-methyl) furazan (called 4.4'-Dimethyl-[3.3'-azo-1.2.5-oxdiazol or 4.4'-Dimethyl-3.3'-azofurazan in Ger),



, mw 194.16, N 43.29%; orn-yel lflts, mp 107°; sol in common org solvs, insol in w. It can be prepd by treating methylaminofurazan, $H_3C{\cdot}C{:}N{\cdot}O{\cdot}N{:}C{\cdot}NH_2$, in 30% H_2SO_4 with dry $KMnO_4$ at 60-70°

Refs: 1)Beil **27**, [866] 2)G.Ponzio & G. Ruggeri, Gazz **53**, 304(1923) & CA **17**, 3873-4(1923)

5,5'-Azobis-(3-propyl-sym-triazole) or 3.3'-Dipropyl-5,5'-azo-1,2,4-triazole,



mw 248.29, N 45.13%; pale yel crysts; mp-dec without melting; easily sol in alk and repptd by weak acids; sol in concd H_2SO_4, giving a yel soln, and pptd by w. It was prepd by oxidg 5-amino-3-n-propyl-1,2,4-triazole with $KMnO_4$ in alk soln. Reduction of azobispropyltriazole with $SnCl_2$ in acid soln gave a col soln of the hydrazo-compd which easily reverted to the azo-compd on oxidn

From a concd soln of chloroauric acid and diazotized aminopropyltriazole, there was separated: *5-diazo-3-n-propyl-1,2,4-triazole chloroaurate,* $[(C_5H_7N_5 \cdot AuCl_3)_2 \cdot H_2O]$, which darkened on heating and melted at 135° (dec) and *5-diazo-3-isopropyl-1,2,4-triazole chloroaurate,* $[C_5H_7N_5 \cdot AuCl_3]$, as a yel ppt, stable at RT but exploded violently on heating and decomp in warm alc soln

Refs: 1)Beil **26**, [190] 2)J.Reilly & P.J. Drumm, JCS **1926**, 1733 & 1735 & CA **20**, 3293-4(1926)

Azobis-(stilbene). See Azostilbene and Derivatives

Azobutyronitrile. See Azobis-(isobutyronitrile)

Azoderivatives of Eulite. See under Eulite and Derivatives

Azodicarboxamide. See Azobisformamide under Azobisformic Acid and Derivatives

Azodicarboxamidine. See Azobisformamidine under Azobisformic Acid and Derivatives,

Azodicarboxyethylamide. See Azobisethylformamide under Azobisformic Acid and Derivatives, p A653

Azodicarboxyhydrazide (called 4-Amino-3.5-dioxo-1.2.4-triazolin in Ger), $O{=}C(-N{-}NH_2){-}N{=}N{-}C{=}O$, mw 114.07, N 49.12%; violet powd, very unstable, exploding ca 72°

Refs: 1)Beil **26**, (64-5) 2)R.Stollé, Ber **45**, 288(1912) & CA **6**, 1005(1912)

Azodicarboxylic Acid. See Azobisformic Acid, p A651

Azodicarboxymethylamide. See Azobismethylformamide under Azobisformic Acid and Derivatives, p A653

Azodiformic Acid. See Azobisformic Acid and Derivatives

Azodimethylbenzene. See Azoxylene and Derivatives

Azodiphenyldiazonium Chloride. See under Azobisaminobiphenyl

Azoethane(called in Ref 3 *Azobisethane),* $C_2H_5{\cdot}N{:}N{\cdot}C_2H_5$, mw 86.14, N 32.53%. Col liq, bp 58°, n_D 1.3852 at 20°. Can be prepd from 1,2-diethylhydrazine (Ref 3) or its dihydrochloride (Ref 2). Its expl props were not examined

Refs: 1)Beil – not found 2)J.L.Weininger & O.K.Rice, JACS **74**, 6216(1952) & CA **47**, 3704(1952) 3)R.Renaud & L.C.Leitch, Can JChem **32**, 549(1954) & CA **49**, 4503(1955)

Azoformic Acid. See Azobisformic Acid and Derivatives

Azoimide. Same as Hydrazoic Acid (qv). See under Azides, Organic

3,3'-Azoindazole (called Indazolinyliden-indiazenyliden-hydrazin in Ger), $C_{14}H_{10}N_6$. Three structural formulae are given in Beil mw 262.27, N 32.05%; dk-red brn crysts, with green luster (with 1 C_2H_6O from alc); mp 229.5°; sol in alc or chlf; diff sol in common org solvs and in w. Its prepn and props are described in Refs

Refs: 1)Beil **24**, 141 2)E.Bamberger et al, Ann **305** 347(1899) & Ber **39**, 4279(1906)

Azomethane (called Dimethyldiimid in Ger), $H_3C{\cdot}N{:}N{\cdot}CH_3$, mw 58.08, N 48.23%, OB to CO_2 -192.8%; col gas which can be liquefied and then solidified at -78° to col leaflets; bp 1.5° at 751 mm, d of liq 0.744 at 0°/15°. This compd can be prepd by the action of HNO_2 on N,N'-dimethylhydrazine, $H_3C{\cdot}H{\cdot}N{:}N{\cdot}H{\cdot}CH_3$, or by other methods (Refs 1,3 & 6). Its expl props have been reported by Allen & Rice (Ref 3)

The thermal expln of gaseous azomethane (Ref 3) occurs in accordance with the Semenov theory of thermal explosions as described in Ref 2. This process was studied also by Taylor & Jahn (Ref 4)

Kodama et al (Ref 5) studied the reaction of azomethane with methanol at ca 300°, measured the press change and analyzed the reaction products resulting from the decompn

Refs: 1)Beil **4**, 562, (566) & [966] 2)N. Semenov, ZPhysChem, Abt B, **2**, 161(1929) & CA **23**, 2870(1929) 3)A.O.Allen & O.K. Rice, JACS **57**, 310-7(1935) & CA **29**, 2359 (1935) 4)H.A.Taylor & F.P.Jahn, JChem Phys **7**, 470-3(1939) & CA **33**, 6691(1939) 5)S.Kodama et al, JChemSocJapan, Pure ChemSec **71**, 173(1950) & CA **45**, 6567(1951) 6)J.P.Picard & J.L.Boivin, CanJChem **29**, 223(1951) & CA **45**, 9469(1951) 7)R.Renaud & L.C.Leitch, CanJChem **32**, 549(1954)

Azomethines or Schiff Bases. See under Aldehyde-Amine Condensation Products, pp A120-1

Azomethylbenzene. See Azotoluene and Derivaties

Azomethylfurazan. See 3,3'-Azobis (4-methyl) furazan

AZONAPHTHALENE AND DERIVATIVES

Azonaphthalene (called Azonaphthalin or Dinaphthyl-diimid in Ger), $C_{10}H_7{\cdot}N{=}N{\cdot}C_{10}H_7$, mw 282.33, N 9.92%. Three isomers are described in the literature: 1.1'- or α,α'-Azonaphthalene (Ref 1); 1,2'- or α,β'-Azonaphthalene (Ref 2) and 2,2'-orβ,β'-Azonaphthalene (Ref 3). The nitro derivs of

azonaphthalene may be of interest as expl ingredients

Refs: 1)Beil **16**, 78, (231) & [26] 2)Beil **16**, 80 3)Beil **16**, 80, (231) & [26]

Dinitroazonaphthalene or Azobis-(nitronaphthaline), $C_{10}H_6(NO_2)\cdot N{:}N\cdot C_{10}H_6(NO_2)$, mw 372.33, N 15.05%. Several dinitro-derivs are described in the literature:

1,1'-Dinitro-2,2'-azonaphthalene; orn-red rods (from benz), mp 305-6 (Ref 8); yel ndls (from benz), mp 315° (Ref 7); prepd by oxidn of 1-nitro-2-naphthylamine with phenyl iodosoacetate yielding also some naphthofurazan oxide

4,4'-Dinitro-2,2'-azonaphthalene (called 2,2'-azobis [4-nitronaphthalene in CA); brn-red crysts, mp 315°. Prepd from the action of hydrazine in alc on 1-chloro-2,4-dinitronaphthalene, warmed 6 hrs on a w bath. The product was a mixt of the dinitro compd, the di-NH_4 salt of 2,4-$C_{10}H_6(NO_2)_2$, m-nitronaphthylazimidole and some 2,4-dinitro naphthalene. The reaction of anhyd hydrazine with 1-chloro-2,4-dinitronaphthalene gave only the 4,4'-dinitro-2,2'-azonaphthalene and 2,4-dinitronaphthalene (Refs 1 & 2)

2,2'-Dinitro-1,1'-azonaphthalene; orn-red rods (from benz), mp 219°; prepd by oxidn of 2-nitro-1-naphthylamine with phenyl iodosoacetate yielding also naphthofurazan, identical with the product obtd by oxidg 1-nitro-2-naphthylamine (Ref 8)

3,3'-Dinitro-1,1'-azonaphthalene; red-brn ndls, mp 315°; sparingly sol in acet; in concd H_2SO_4 gave a Prussian-blue color which was permanent for days, and changed to bright yel on dilution with w; soln in aq NaOH gave an intense emerald color; prolonged boiling with alcoholic KOH produced a brn powdr. It was prepd by the action of sodium sulfite on 3-nitronaphthalene-1-diazoacetate, previously obtd by diazotizing 3-nitro-1-naphthylamine and treating the diazonium chloride with aq sodium acetate (Ref 5)

4,4'-Dinitro-1,1'-azonaphthalene; red crysts (from nitrobenz), mp 334° (Ref 4) to 310° (from toluene) (Ref 6); sl sol in boiling glacial acetic acid to give a yel soln; sparingly sol in acet, boiling benz or chlorobenz; almost insol in boiling alc; and gave a bright blue color in concd H_2SO_4 soln which changed to bright yel on dilution with water. It was prepd by the action of sodium sulfite on 4-nitronaphthalene-1-diazonium chloride (or sulfate) (Refs 4 & 6)

5,5'-Dinitro-1,1'-azonaphthalene; yel-brn ndls (from nitrobenz) orn-yell ndls (by subln), mp 322-3° (Ref 3) & 280° (from toluene) (Ref 6); sparingly sol in boiling alc, glacial acetic acid or benz; the color in concd H_2SO_4 soln was reddish-violet, which changed to yel on heating. It was prepd by the reaction of 5-nitro-1-naphthalenediazonium sulfate with cuprous hydroxide (Refs 3 & 6)

Refs: 1)Beil **16**, [26] 2)E.Müller & K.Weisbrod, JPraktChem **111**, 309(1925) & CA **20**, 750(1926) 3)H.H.Hodgson et al, JCS **1942**, 746 & CA **37**, 1422(1943) 4)H.H. Hodgson et al, JCS **1944**, 16 & CA **38**, 2030 (1944) 5)H.H.Hodgson & D.E.Hathway, JCS **1945**, 452 & CA **39**, 4863(1945) 6)B.M. Bogoslovskii & Z.S.Kazakova, ZhObshchKhim **22**, 1183-6(1952) & CA **47**, 6388(1953) 7) G.B.Barlin et al, JCS **1954**, 3123 & CA **49**, 11608(1955) 8)K.H.Pausaker & J.G.Scroggie, JCS **1954**, 4502 & CA **49**, 13226(1955)

Trinitro, $C_{20}H_{11}N_5O_6$, *Tetranitro,* $C_{20}H_{10}N_6O_8$, *Pentanitro-,* $C_{20}H_9N_7O_{10}$ and *Hexanitro-,* $C_{20}H_8N_8O_{12}$, *Derivatives of Azonaphthalene* were not found in Beil or in CA through 1956

AZOPHENETOLE AND DERIVATIVES

Azophenotole or 4,4'-Azodiphenetole (called Diäthoxy-azobenzol in Beil), $C_2H_5\cdot O\cdot C_6H_4N{:}N\cdot C_6H_4\cdot O\cdot C_2H_5$, mw 270.32, N 10.36%. Several isomers are described in the literature: 2,2'-or o,o'-azophenetol (Ref 1), 3,3'- or m,m'-azophenetol (Ref 2), 4,4'- or p,p'-azophenetol (Ref 3), 2.4' phenetol (Ref 4) and 3.4' phenetol (Ref 5). The nitro derivs

of azobisphenetol may be of interest as expl ingredients. No azido or diazido-derivs were found in Beil or CA through 1946

Refs: 1)Beil **16**, 92 2)Beil **16**, 95 & [37] 3)Beil **16**, 112, (238) & [44] 4)Beil **16**, 109 5)Beil **16**, 110

Dinitroazophenetole $[C_2H_5 \cdot O \cdot C_6H_3(NO_2)N:]_2$, mw 360.32, N 15.55%. Two dinitro derivs are described in the literature: *x.x'-Dinitro-2,2'-diethoxy-azobenzene;* bright red-yel ndls (from alc), mp 190°; sol in boiling alc. It was prepd by nitrating o,o'-azophenetol with cold, fuming nitric acid (Ref 1)

5,5'-Dinitro-2,2'-diethoxy-azobenzene; brownish-red ndls (from chlf); mp 284-5°, sublimes without decompn; sol in cold chlf or cold benz; insol in boiling alc; dissolves without decompn with a yel-red color in cold concd H_2SO_4. It was obtd together with the x,x'-dinitro compd on nitrating o,o'-azophenetol

Ref: Beil **16**, 92 & 94

Tetranitroazophenetol, $C_{16}H_{14}N_6O_{10}$, not found in Beil or in CA through 1956

Hexanitroazophenetole (called 2.4.6.2'.4'.6'-Hexanitro-3.3'-diäthoxyazobenzol in Ger), $[C_2H_5 \cdot O \cdot C_6H(NO_2)_2N:]_2$; mw 540.32, N 20.74%, OB to CO_2 −71.1%; light orn ppt, which on recrystn from alc or glacial acetic acid gives dk red-yel to red crysts; mp 138-9°; easily sol in glacial acetic acid, cold alc, benz or eth; insol in w. Its prepn and props are described in Ref 2

Refs: 1)Beil **16**, [38] 2)K.Elbs & O.H. Schaaf, JPrakChem **120**, 2-14(1928) & CA **22**, 4508(1928)

AZOPHENOL AND DERIVATIVES

Azophenol; Azodiphenol or Dihydroxyazobenzene (called Dioxy-azobenzol or Azophenol in Beil) $HO \cdot C_6H_4 \cdot N:N \cdot C_6H_4 \cdot OH$, mw 214.22, N 13.08%. Three isomers are described in the literature: o,o'-azophenol or 2,2'-dihydroxyazobenzol (Ref 1); m,m'-azophenol or 3,3'-dihydroxy-azobenzol (Ref 2) and p,p'-azophenol or 4.4'-dihydroxy-azobenzol (Ref 3). The nitro derivs of azobisphenol may be of interest as expl ingredients

Refs: 1)Beil **16**, 91 & [33] 2)Beil **16**, 95 & [37] 3)Beil **16**, 110 (237) & [43]

Note: No azido- or diazido-derivs were found in Beil or CA through 1956

Mononitroazophenol, $C_{12}H_9N_3O_4$, one isomer is listed in Beil **16**, 96 and in CA **21**, 1971 (1927)

Dinitroazophenol, $C_{12}H_8N_4O_6$, the 3.3'-dinitro-4.4'-dihydroxyazobenzene isomer is described in Beil **16**, (239) & [58] and in CA **34**, 392 (1940). The 4,6-dinitro-3,4'-azodiphenol is listed in CA **15**, 2844(1921)

Trinitroazophenol, $C_{12}H_7N_5O_8$, not found in Beil but the p,p'-azodiphenol tetranitro deriv is listed in CA **34**, 392(1940)

Tetranitroazophenol (called 3.5.3'.5'-Tetranitro-4.4'-dioxyazobenzol in Ger), $HO(NO_2)_2 \cdot C_6H_2 \cdot N:N \cdot C_6H_2(NO_2)_2OH$; mw 394.22, N 21.32%; crysts (from glacial acetic acid), mp 261-2° with decompn (Ref 3). This compd was first prepd by Robertson (Ref 2) by nitration of α-p-azophenol in acetic acid soln with a slight excess of HNO_3 in the same solvent. After recrystn from hot acetic acid, yel crysts melting sharply at 230° were obtd. Lauer et al (Ref 3) prepd the compd by slowly adding dinitro-p-azophenol to fuming HNO_3, cooled in an ice-salt bath. On purifying the crude product by crystn from glacial acetic acid, crysts melting at 261° were obtd. The expl props of the product were not examined

Hart & Detroit (Ref 4) measured the dissocn constant of 3,5,3',5'-tetranitro- 4,4'-dihydroxybiphenyl in methanol to detn the effect of NO_2 groups, ortho or para to the OH function, on the acidity of the compd

Jurisch (Ref 5) patented the use of 4,4'-dihydroxy-3,3'-dinitrobiphenyl or other o-nitrophenol-type dimers, trimers, etc, having recurring benzene nuclei contg OH & NO_2 groups in ortho position to each other for reducing Ca carbonate, Ca phosphate and Mg phosphate scales in steam boilers

Refs: 1)Beil **16**, (239) & [58] 2)P.W. Robertson, JCS **103**, 1476 (1913) & CA **7**, 3751(1913) 3)W.M.Lauer et al, JACS **61**, 2779(1939) & CA **34**, 391-2(1940) 4)H.Hart & W.J.Detroit, JACS **74**, 5214(1952) & CA **48**, 13664(1954) 5)M.J.Jurisch, USP 2749305 (1956) & CA **50**, 17269-70(1956)

Pentanitroazophenol, $C_{12}H_5N_7O_{12}$, not found in Beil or in CA through 1956

Hexanitroazophenol; 3,3'-Azobis-(2,4,6-trinitrophenol; Azopicric Acid or 2,4,6,2',4',6'-Hexanitro- 3,3'-dihydroxyazobenzene, $(O_2N)_3(HO)\cdot C_6H\cdot N{:}N\cdot C_6H(OH)(NO_2)_3$, mw 484.22, N 23.14%; OB to CO_2 -39.6%; yel-red powd, mp 238-9° (dec); explodes violently on rapid heating; readily sol in w, alc, eth or acet; sol in benz with formation of addn compd; insol in CS_2 or concd HCl. It is a strong acid which can be prepd by nitrating m,m'-azophenol with KNO_3 and concd H_2SO_4 under cooling (Refs 1,2 & 3)

Azopicric acid is a more powerful expl than PA; explosion proceeds according to the equation (Ref 3):

$$[(O_2N)_3C_6H(OH)N{:}]_2 \rightarrow 12\ CO + 2H_2O + 8N$$

Several of its salts are described in the literature but their expl props are not given

Refs: 1)Beil **16**, [37] 2)K.Elbs & F. Schliephake, JPraktChem **104**, 282(1922) & CA **17**, 738-9(1923) 3)K.Elbs & O.H.Schaaf, JPraktChem **120**, 35(1928) & CA **22**, 4508 (1928)

Azopiperidine (called N.N'-Azopiperidin; Dipiperidinodiimid or 11;4.4-Bis-pentamethylen-tetrazene-(2) in Ger) (called "Dipiperyl-terazone" by Knorr, and Angeli & Angelico), $C_5H_{10}\cdot N\cdot N{:}N\cdot N\cdot C_5H_{10}$; mw 196.29, N 28.55%, crysts (from dil alc), mp 45 (distills without decompn); readily sol in alc, eth, benz or ligroin; insol in w. Its prepn is described in Beil and in Refs 2,3 & 4. The *platinum chloride salt,* $(C_{10}H_{20}N_4)_2H_2PtCl_6$, an amorph powd, decomposes with deton at 70° (Ref 2)

Refs: 1) Beil **20**, 91 & (26) 2)L.Knorr, Ann **221**, 299 & 311-3 (1893) & JCS **46** I, 468 (1884) 3)A.Angeli & F.Angelico, AttiAccadLinceiMem [5] **10** I, 168(1901) & JCS **80** I, 322(1901); Gazz **33**, II, 244(1903) & JCS **86** I, 172(1904) 4)A.Angeli & V.Castellana, AttiAccadLinceiMem [5] **14** I, 272(1905) & JCS **88** I, 491(1905)

Azopropane; Azobispropane; Azoisopropane; Azodipropane or Dimethylazoethane (called 2.2'-Azopropan or Diisopropyldiimid in Ger), $C_6H_{14}N_2$, mw 114.19, N 24.53%. Two isomers are described in the literature:

1,1'-Azopropane, $H_3C\cdot CH_2\cdot CH_2\cdot N{:}N\cdot CH_2\cdot CH_2\cdot CH_3$; pale yel liq, bp 104° (Ref 8) to 113.5° (Ref 10), n_D at 20° 1.4053 (Ref 10) to 1.4060 (Ref 8). This compd was prepd by oxidn of 1,2-di-n-propylhydrazine by mercuric oxide in w

2,2'-Azopropane, $(H_3C)_2CH\cdot N{:}N\cdot CH(CH_3)_2$; faintly straw-colored oil, nauseatingly sweet; bp 88.5°, d_4 at 23° 0.7408, n_D at 20° 1.3899 (Ref 3); Q_c^P 1053.4 kcal/mol (Ref 7), $Q_{activation}$ 40.9 kcal/mol (Refs 4 & 11); insol in w, dil acid or alk, neutral to litmus (Refs 1 & 2). This compd is a powerful poison, especially injurious to the liver; and moderate red cell disintegration occurrs from azopropane intoxication (Ref 3)

It was prepd by oxidn of 1,2-diisopropyl-hydrazine·HCl with copper or mercurous oxide in water (Refs 2, 8 & 10); and is reduced by H & colloidal Pd to 1,2-diisopropylhydrazine. The thermal decompn of azopropane was studied by a number of investigators (Refs 4,5,6,9&11)

The expl props of this compd were not reported. No azido or nitro derivatives were found in the literature

Refs: 1)Beil **4**, [966] 2)H.L.Lochte et al, JACS **44**, 2561(1922) & CA **17**, 267(1923) 3)M.Bodansky, JPharmacol **23**, 127(1924); JBiolChem **58**, 799(1924) & CA **18**, 1532 & 1859 (1924) 4)H.C.Ramsperger, PrNatlAcad-Sci **13**, 849(1927); JACS **50**, 714(1928) & CA **22**, 713 & 1517(1928) 5)F.O.Rice & B.L.Evering, JACS **55**, 3898(1933) & CA **27**, 5057(1933) 6)H.Gershinowitz & O.K.Rice, JChemPhys **2**, 273(1934) & CA **28**, 4295 (1934) 7)G.E.Coates & L.E.Sutton, JCS **1948**, 1187 & CA **43**, 931(1949) 8)B.W.

Langley et al, JCS **1952**, 4196 & CA **48**, 4432-3(1954) 9)R.W. Durham & E.W.Steacie, CanJChem **31**, 377(1953 & CA **47**, 7328 (1953) 10)R.Renaud & L.C.Leitch, CanJ-Chem **32**, 545(1954) & CA **49**, 4502-3(1955) 11)S.G.Cohen & C.H.Wang, JACS **77**, 2457 (1955) & CA **50**, 3342-3(1956)

Azopropyltriazole. See 5,5'-Azobis (3-propyl-sym-triazole)

2,2'-Azopyridine or 2,2'-Azobispyridine [called in Beil Di-pyridyl-(2)-diimid] $(C_5H_4N)\cdot N{:}N\cdot(C_5H_4N)$, mw 184.20, N 30.42%. Red ndls or prisms. No expl derivs were reported in the literature

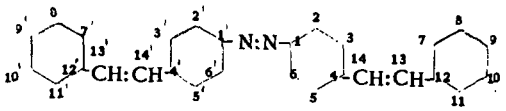
Ref: Beil **22**, [496]

AZOSTILBENE AND DERIVATIVES

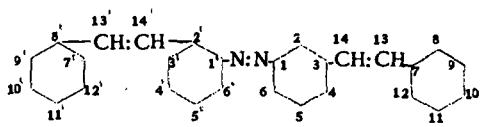
Azostilbene; Azobis(stilbene); Stilbene-azostilbene or Distyryl-azobenzene, $C_{28}H_{22}N_2$, although not described in the literature, may be considered as the parent compd of derivs described below

Three isomers are theoretically possible and it is proposed to name and to number them as follows, so that no confusion may arise with regard to the position of substituents:

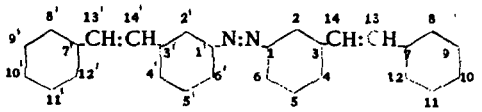
p-Azostilbene



o-Azostilbene



m-Azostilbene



Dinitroazostilbenes, $C_{28}H_{20}N_4O_4$, mw 476.47, N 11.76%. The following isomers are known:

3,3'-Dinitro-p-azostilbene or Azobis (3-nitrostilbene, called in Ref 2: **3,3'-Dinitro-4,4'-distyryl-azobenzene,** $C_6H_5\cdot CH{:}CH\cdot C_6H_3(NO_2)\cdot N{:}N\cdot C_6H_3(NO_2)\cdot CH{:}CH\cdot C_6H_5$; carmine-red crysts; mp 260-1°. Prepn and props are given in Ref 2

Refs: 1)Beil – not found 2)L.Chardonnens & P.Heinrich, Helv **23**, 1405(1940) & CA **35**, 2121(1941)

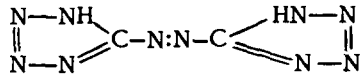
9,9'-Dinitro-p-azostilbene or Azobis-(9-nitrostilbene, called in Beil Bis-[4'-nitro-stilbenyl-(4)]-diimid or [4'-Nitro-stilben]-<4azo4>-[4'-nitro-stilben], $(O_2N)C_6H_4\cdot CH{:}CH\cdot C_6H_4\cdot N{:}N\cdot C_6H_4\cdot CH{:}CH\cdot C_6H_4(NO_2)$; yel-red crystsn mp 263°. It is listed in Beil **16**, 84 without reference to its source; was not found in CA through 1956

5,5'-Dinitro-o-stilbene, called in Ref 2: **5,5'-Dinitro-2,2'-distyryl-azobenzene,** $C_6H_5\cdot CH{:}CH\cdot C_6H_3(NO_2)\cdot N{:}N\cdot C_6H_3(NO_2)\cdot CH{:}CH\cdot C_6H_5$; brick red crysts, decmpg ca 265° with melting. Prepn and props are given in Ref 2

Refs: 1)Beil – not found 2)L.Chardonnens & P-Heinrich, Helv **23**, 1414(1940) & CA **35**, 2122(1941)

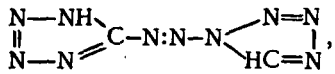
Note: No *dinitro-* deriv of *m-azostilbene* was found in Beil or CA through 1956. No *Azido-* $C_{28}H_{21}N_5$, *diazido-* $C_{28}H_{20}N_8$, *trinitro-* $C_{28}H_{19}N_5O_6$, or *tetranitro-* $C_{28}H_{18}N_6O_8$ *derivatives* of o-, m- and *p-azostilbenes* were found in Beil or CA through 1956

Azotetrazole, $C_2H_2N_{10}$, mw 166.12, N 84.33%: *5,5'-Azotetrazole* (called Di[tetrazolyl-(5)]-diimid in Ger);



. According to Beil (Ref 1) this compd was not prepd in the free state but its prepn by Rathsburg (Ref 4) by oxidg aminotetrazole in alc solns with permanganate or persulfates is described in CA **17**, 1147(1923). A diazotetrazole was prepd by diazotizing aminotetrazole

The British abstract of the patent of Rathsburg (Ref 4) shows an azotetrazole with the structure:

 , which we call *tetrazolyl-5'-azo-1-tetrazolyl;* but its method of prepn is not given

Salts of azotetrazole were prepd in 1898 by Thiele (Ref 2) and claimed for use in initiators, detonators and percussion caps by Rathsburg (Ref 4) and von Herz (Ref 3). The Ba, Ca, K Na and NH_4 salts of azotetrazole are described by Thiele (Ref 2) who noted that all of these were expl, especially those of the heavy metals, Ag, Hg & Pb. According to Rathsburg (Ref 4) the more important salts of azotetrazole are those of Cd and Pb. Impact sensitivity tests showed that the Pb salt fired once in six shots at 7 cm with a 100 g wt vs 12 cm for tech grade MF (Ref 5)

Blasting detonators proposed by Rathsburg (Ref 4) contd a top charge of Pb azotetrazole over Pb,or Cd tetrazolyl azide over TeNMA

Derivs of azotetrazole are described in Ref 6

Refs: 1)Beil **26**, 593 & [349] 2)J.Thiele, Ann **303**, 57-60(1898) 3)E. von Herz, GerP 37054(1920) & ChemZtr **1923** IV, 174 4) H.Rathsburg, BritP 185,555(1921); JSCI **41**, 880A (1922); ChemZtr **1923** II, 370 & CA **17**, 1147(1923) 5)H.Rathsburg, ZAnorgChem **41**, 1284(1928) 6)L.F.Audrieth & J.W. Currier, Univ of Ill Rpt, "Derivatives of 5-Aminotetrazole," pp 22-3 (1954)

AZOTOLUENE AND DERIVATIVES

Azotoluene; Azomethylbenzene or Dimethylazobenzene (called Ditolyldiimid; Dimethylazobenzol or Azototoluol in Ger), $H_3C{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4CH_3$; mw 210.27, N 13.32%. Seven isomers are described in the literature o,o'-azotoluene or 2,2'-dimethylazobenzene (Ref 1); m,m'-azotoluene or 3,3'-dimethylazobenzene (Ref 2); p,p'-azotoluene or 4,4'-dimethyl-azobenzene (Ref 3) o,m'-azotoluene or 2,3'-dimethyl-azobenzene (Ref 4); o,p'-azotoluene (?) or 2,4'-dimethyl-azobenzene (?) (Ref 5); m,p'-azotoluene or 3,4'-dimethyl-azobenzene (Ref 5) and ω,ω'-azotoluene or dibenzyldiimid, $C_6H_5{\cdot}CH_2{\cdot}N{:}N{\cdot}CH_2{\cdot}C_6H_5$ (Ref 6). The azido or nitro-derivs of azotoluene may be of interest as expl ingredients

Refs: 1)Beil **16**, 61, (227) & [19] 2)Beil **16**, 64 & [20] 3)Beil **16**, 66, (229) & [21] 4)Beil **16**, 63 & [20] 5)Beil **16**, 66 6)Beil **16**, (229)

Azidoazotoluene, $C_{14}H_{13}N_5$; mw 251.28, N 27.28%. Three isomers are described in the literature:

4-Azido-2,3'-dimethylazobenzene; dk-red prisms or small orn-red crysts (from alc), mp 65°; other props and prepn are given in Ref 1

4'-Azido-2,3'-Dimethylazobenzene; yel lfts (from alc), mp 58-60° or red-brn ndls (from alc), mp 67°; other props and prepn are given in Ref 2

6-Azido-3,4'-dimethylazobenzene; yel-red crysts (from alc), mp 85°; other props and prepn are given in Ref 3

Refs: 1)Beil **16**, 63 2)Beil **16**, 65 3)Beil **16**, 66

Nitroderivaties of Azotoluene

Mononitroazotoluene, $C_{14}H_{13}N_3O_2$; mw 255.27, N 16.46%. Four isomers are described in the literature:

x-Nitro-2,2'-dimethylazobenzene; ndls (from alc), mp 87°; prepd by treating o,o'-azotoluene with nitric acid (Ref 1)

x-Nitro-3,3'-dimethylazobenzene; crysts (from glacial acetic acid), mp 192-5°; prepd by treating m,m'-azotoluene with a mixt of cold nitric & sulfuric acids (Ref 2)

6-Nitro-2,4'-dimethylazobenzene; red oil, bp ca 215° at 11 mm press; prepd from 2-nitroso-3-nitrotoluene and aniline in glacial acetic acid (Ref 3)

2-Nitro-4,4'-dimethylazobenzene; orn-red triclinic crysts (from alc), mp 80°; prepd by treating p,p'-azotoluene with 5 parts nitric acid at a temp not exceeding 30° (Ref 4)

Refs: 1)Beil **16**, 63 2)Beil **16**, 65 3)Beil **16**, (228) 4)Beil **16**, 71

Dinitroazotoluene, $C_{14}H_{12}N_4O_4$; mw 300.27, N 18.66%. Five isomers are described in the literature:

3,3'-Dinitro-2,2'-dimethylazobenzene; brn crysts (from glacial acetic acid), mp 198°; prepd by diazotizing 6-nitro-2-aminotoluene in dil HCl and treating the diazonium chloride soln with a cuprous chloride soln in HCl (Ref 1)

4,4'-Dinitro-2,2'-dimethylazobenzene; red-brn lfts (from toluene), mp 258°; readily sol in boiling glacial acetic acid or nitrobenzene and in benz, diffc sol in alc; prepd by treating 5-nitro-1-methylbenzene-2-diazonium sulfate with a cuprous chloride soln in HCl (Ref 1)

5,5'-Dinitro-2,2'-dimethylazobenzene; red ndls (from toluene or nitrobenzene), mp 273°; sl sol (giving an orn color) in boiling alc, benz or toluene; prepd by treating the 4-nitrotoluene diazonium salt with cuprous chloride in concd HCl soln (Ref 1)

x,x-Dinitro-3,3'-Dimethylazobenzene; red ndls (from glacial acetic acid), mp 192-3°; prepd by treating m,m'-azotoluene with 4 parts nitric acid at a temp not exceeding 30° (Ref 2)

2,2'-Dinitro-4,4'-Dimethylazobenzene; prisms (from glacial acetic acid), mp 114°; readily sol chlf or eth; obtd by dissolving p,p'-azotoluene in 3 parts cold nitric acid (Ref 3)

3,3'-Dinitro-4,4'-dimethylazobenzene; red lfts (from benz), mp 149°; sol in boiling benz; prepd by treating 2-nitro-4-aminotoluene with a soln of the Na salt of chlorous acid (Ref 3)

Refs: 1)Beil **16**, 63 2)Beil **16**, 65 3)Beil **16**, 71

Trinitroazotoluene; $C_{14}H_{11}N_5O_6$; mw 345.27, N 20.29%. Two isomers are found in the literature:

x,x,x-Trinitro-4,4'-Dimethylazobenzene; *a* and *β*-trinitro derivs (*a*), mp 189° and (*β*), mp 138° were reported by Janovsky (Ref 2) on nitrating p,p'-azotoluene with nitric acid. On further treatment with HNO_3 both *a* and *β*-trinitro compds gave the same tetranitro compd, mp 198-200°

Refs: 1)Beil **16**, 71 2)J.V.Janovsky, Monatsh **9**, 836(1888) & JCS **56** I, 250-1 (1889); Monatsh **10**, 591 & 593 (1889) & JCS **58** I, 140 (1889)

Tetranitroazotoluene; $C_{14}H_{10}N_6O_8$; mw 390.27, N 21.54%. Three isomers are described in the literature:

x,x,x,x-Tetranitro-4,4'-Dimethylazobenzene; by further treatment of either *a* or *β*-trinitroazotoluene with nitric acid, Janovsky (Ref 2) reported a tetranitroazotoluene melting at 198-200°

Refs: 1)Beil **16**, 71 2)J.V.Janovsky, Monatsh **9**, 839 (1888) & JCS **56** I, 251(1889)

3,5,3',5'-Tetranitro-4,4'-dimethylazobenzene; orn colored ndls (from glacial acetic acid), mp 248-50°; prepd by treating 2,6-dinitro-4-hydroxylaminotoluene with PCl_5 in ether

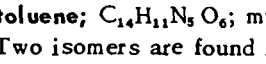
Ref: Beil **16**, (229)

4,6,4',6'-Tetranitro-2,2'-dimethylazobenzene; yel-red ndls (from glacial acetic acid), mp 218°; readily sol in acet, benz or boiling glacial acetic acid; diffc sol in alc or eth; prepd by treating the Na salt of 4,6-dinitro-2-methyl-phenyl-aci-nitramine, in the smallest possible amt of methyl alcohol, with an excess of bleaching powdr soln

Ref: Beil **16**, 63

Pentanitroazotoluene: $C_{14}H_9N_7O_{10}$; mw 435.27, N 22.53% and *Hexanitroazotoluene:* $C_{14}H_8N_8O_{12}$, mw 480.27, N 23.33% were not found in Beil or CA through 1956

3,3'-Azo-(1,2,4-triazole),



, mw 164.14, N 68.28%;

lt yel powdr; sl sol in alkali soln, and is

pptd again on acidifying this soln. It was prepd by the oxidn of 3-amino-1,2,4-triazole or 5-amino-1,2,4-triazole-3-carboxylic acid with permanganate in caustic soda soln. A red Ag salt is obtd by reaction of azotriazole with an ammoniacal silver soln

Refs: 1)Beil **26**, 340 2)J.Thiele & W. Manchot, Ann **303**, 48(1898)

AZOXYLENE AND DERIVATIVES

Azoxylene; Azodimethylbenzene or Tetramethylazobenzene (called Bis-[dimethylphenyl]-diimid; Tetramethylazobenzol or Azoxylol in Ger), $(H_3C)_2{\cdot}C_6H_3{\cdot}N{:}N{\cdot}C_6H_3{\cdot}(CH_3)_2$, mw 238.32, N 11.76%. Five isomers are listed in the literature: 4,4'-azo-o-xylene or 3,4,3',4'-Tetramethyl-azobenzene (Ref 1); 4,4'-azo-m-xylene or 2,4,2'4'-tetramethyl-azobenzene (Ref 2); 4,5'azo-m-xylene or 2,4,3'5'-tetramethyl-azobenzene (Ref 3); 5,5'-azo-m-xylene or 3,5,3',5'-tetramethyl-azobenzene (Ref 3) and 2,2'-azo-p-xylene or 2,5,2',5'-tetramethyl-azobenzene (Ref 4). The azido or nitro-derivs of azoxylene may be of interest as expl ingredients

Refs: 1)Beil **16**, 72 & [23] 2)Beil **16**, 73 & [23] 3)Beil **16**, 74 4)Beil **16**, 75 & [24]

2'-Azido-2,4,3',5'-tetramethylazobenzene, $(H_3C)_2C_6H_2{\cdot}(N_3){\cdot}N{:}N{\cdot}(N_3){\cdot}C_6H_2{\cdot}(CH_3)_2$; mw 279.34, N 25.07%; red ndls (from ligroin); mp 77°, explodes mildly on rapid heating or on contact with concd sulfuric acid, decompg into N_2 and 2-[2,4-dimethyl-phenyl]-4,6-dimethyl-benztriazole; readily sol in eth, more diffc sol in cold alc. It was prepd by the action of alcoholic NH_3 on the dry diazoperbromide, $C_{16}H_{17}N_4Br_3$, obtd as described in Ref 2, in the presence of excess ether

Refs: 1)Beil **16**, 74 2)T.Zincke & H. Jaenke, Ber **21**, 542(1888) & JCS **54** I, 469-70(1888)

Diazidoazoxylene, $C_{16}H_{16}N_8$ – not found in Beil or CA through 1956

Nitroderivatives of Azoxylene

Mononitroazoxylene, $C_{16}H_{17}N_3O_2$; *Dinitroazoxylene,* $C_{16}H_{16}N_4O_4$; *Trinitroazoxylene,* $C_{16}H_{15}N_5O_6$; *Tetranitroazoxylene,* $C_{16}H_{14}N_6O_8$; and higher nitroderivs of azoxylene were not found in Beil or CA through 1956

Azoles are heterocyclic compds characterized by a five membered ring contg nitrogen. They include diazoles, triazoles and tetrazoles, as well as compds contg other atoms such as O and S in the ring: oxazoles, dioxazoles, thiazoles, thiadiazoles etc. Some azoles or their derivs are expl, for example the triazoles and the tetrazoles

Refs: 1)K.A.Jensen & A.Friediger, Kgl DanskeVidenskabSelskab, Math-FysMedd **20**, No 20, 1-54(1943); ChemZtr **1944** I, 416-7 & CA **39**, 2068-70(1945) 2)A.A.Morton," The Chemistry of Heterocyclic Compounds," McGraw-Hill, NY(1946), 362-475 3)Kirk & Othmer **2** (1948), 269

Azon Guided Missile is one of the American weapons developed during WW II and consisted of a 1000-lb demolition bomb with a "radio brain" attached. This enabled the bombadier to sight the target in the Norden bombsight and allow the bomb to drop. Once the bomb was dropped, the bombadier guided its flight in azimuth (right or left of target) by remote control. A 1,000,000 candlepower flare on the tail of the Azon was automatically ignited after the bomb left the plane which permitted the bombadier to follow the Azon with his eye. More accurate guided missiles were developed in the USA after WW II (See also Razon)

Refs: 1)Anon, ArOrdn **30**, 160(1946) 2)G. Merrill, Edit, "Dictionary of Guided Missiles and Space Flight," VanNostrand, NY(1959), 73

Azot (Rus). Nitrogen

Azotate (ou Nitrate) (Fr). Nitrate

Azotate (ou Nitrate) d'ammoniaque (Fr). Ammonium Nitrate

Azotate (ou Nitrate) de plomb (Fr). Lead Nitrate

Azotate (ou Nitrate) de potasse (Fr). Potassium Nitrate

Azotate (ou Nitrate) de soude (Fr). Sodium Nitrate

Azote (Fr). Nitrogen

Azote Powder Company of Indianapolis, Ind patented in 1898 a method of nitrating starch (previously dried at 100-140° and then cooled) using 1 liter of mixed nitricsulfuric acid (1:2) per 200 g of starch. Nitration was done in a hermetically closed vessel at a temp below 4°

Ref: Daniel (1902), 48

Azothydrates (Fr). Azides

Azothydrique (Acide) (Fr). Hydrazoic Acid

Azotidrato (o Azotidruro) d'argento; Acido d'argento (Ital). Silver Azide

Azotidrato (o Azotidruro) di piombo; Acido di piombo (Ital). Lead Azide

Azotidrato (o Azotidruro) di sodio; Acido di sodio (Ital). Sodium Azide

Azotine. A blasting expl patented by A.Bercsey contd $NaNO_2$, sulfur, charcoal and petroleum residues

Ref: Daniel (1902), 48

Azotique (Acide) (Fr). Nitric Acid

Azotometer. An apparatus for detg gasometrically the nitrogen content of compds in soln. See also Nitrometer, described under Ammonium Nitrate, Analytical Procedures, p A373

Azotures (Fr). Azides

Azoxime. Same as 1,2,4-Oxdiazole

AZOXYCOMPOUNDS

Azoxy compds are a small class of stable substs which contain the characteristic azoxy group, represented by R–N(→O)=N–R'.

The azoxy group is not symmetrical and the oxygen atom is attached to only one nitrogen atom, but not to both. The link uniting nitrogen to oxygen is a co-ordinate (semi-polar) link as indicated. In the majority of azoxy compds known, the two groups R and R' attached to nitrogen are aromatic radicals.

The most common method of preparing azoxy compds is by the reduction of nitro compds:

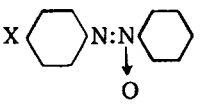
$$R{\cdot}NO_2 + R{\cdot}NO_2 \longrightarrow R{\cdot}NO + HONH{\cdot}R \longrightarrow R{\cdot}NO{:}N{\cdot}R + H_2O$$

A variety of reducing agents have been used but the usual ones are sodium methoxide in MeOH or sodium arsenite. The azoxy compd results from the condensation of the nitroso and the hydroxylamine compds produced during the reduction. The nitroso and hydroxyl amine compds can be prepd separately and condensed together to give the azoxy compd. Even when the nitroso and the hydroxylamine compds contain different substituted groups, with only one or two exceptions sym azoxy compds (R = R') are formed, and not the unsym azoxy compd, as expected. Another method for prepg azoxy compds is by the oxidn of azo compds:

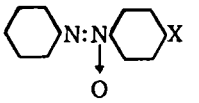
$$Ar{\cdot}N{:}N{\cdot}Ar \longrightarrow Ar{\cdot}NO{:}N{\cdot}Ar$$

The best oxidg agent for this purpose is 30% hydrogen peroxide dissolved in glacial acetic acid.

Nearly all aromatic azoxy compds crystallize well; they are completely stable towards strong HCl but if warmed with concd H_2SO_4 they undergo rearrangement. The true structure of azoxy compds was revealed by A. Angeli's discovery of isomerism in azoxy compds:



α-p-(x)azoxybenzene



β-p-(x)azoxybenzene

In nomenclature, the prefixes α and β are used to distinguish between the two structures; α indicates that the substituent is attached to the benzene ring which is linked to trivalent nitrogen

Refs: 1)P.Lemoult, AnnChimPhys [8] **14**, 184-90(1908) & CA **2**, 3298(1908) 2)D. Bigiavi, AttiAccadLinceiMem [6] **5**, 444-50 (1927) & CA **21**, 2123(1927) 3)H.E.Bigelow, ChemRevs **9**, 117-67(1931) & CA **25**, 4861 (1931) 4)Sidgwick, OrgChem of N (1937), 426-30 5)H.E.Bigelow & K.F.Keirstead, CanJRes **24B**, 232-7(1946) (Parachor of certain azoxy and related azo compds) & CA **41**, 406-7(1947) 6)V.O.Lukashevich & T.N.Sokolova, DoklAkadN **54**, 693-5(1946) (Action of chlorosulfonic acid on azoxy compds) & CA **41**, 5472(1947) 7)Kirk & Othmer **2**(1948), 270-1 8)V.O.Lukashevich & T.N.Kurdyumova, ZhObshchKhim **18**, 1963-76(1948) (Rearrangement of azoxy compds) & CA **43**, 3800-1(1949) 9)R.Gaudry & K.F. Keirstead, CanJRes **27B**, 897-901(1949) (Identification of azoxy compds) & CA **45**, 571(1951) 10)H.W.Galbraith et al, JACS **73**, 1323-4(1951) (Alkaline reduction of aromatic nitro compds with glucose to azoxy compds) & CA **45**, 8992-3(1951) 11)E.Macovski & A.Petrescu, AcadRepPopulareRomâne, Bul Stiint (U of Bucharest, Romania) **A1**, 485-500(1949) (Formation of azoxy derivs from nitro compds under influence of aromatic ketones) & CA **46**, 921(1952) 12)N.Campbell et al, MikrChem **38**, 376-80 (1951) (Polymorphism and liq cryst formation of some azo and azoxy compds) & CA **46**, 2867(1952) 13) B.W.Langely et al, JCS **1952**, 4191-8 (Prepn of primary aliphatic azoxy compds) & CA **48**, 4432-3(1954) 14)S.Kobayaski & Y.Aoyama, JapanP 4328(1953) (Electrolytic prepn of aromatic azoxy, azo and hydrazo compds) &

CA **48**, 9241-2(1954) 15)G.M.Badger et al, JCS **1953**, 2143-7, 2147-50 & 2151-5 (Oxidn of aromatic azo compds) and 2156-8 (Absorption spectra of azo and azoxy compds) & CA **48**, 9944-5(1954) 16)G.Costa, Gazz **83**, 875-83(1953) (Electrochemical reduction of azo and azoxy compds) & CA **48**, 10456 (1954) 17)S.Kobayashi & Y.Aoyama, JapP 4329(1953) (Prepn of aromatic nitroso, azoxy, azo and hydrazo compds) & CA **49**, 4712 (1955) 18)G.M.Badger & R.G.Buttery, JCS **1954**, 2243-5(Action of light on azoxy compds) & CA **49**, 9535-6(1955) 19)P.H.Gore, Chem & Ind **1954**, 1355 (Thiourea as a reducing agent for aromatic nitro, azoxy, azo and hydrazo compds) & CA **49**, 13106 (1955) 20) S.Jóźkiewicz and H.Kuczyński, Zeszyty-NaukPolitechWroclaw (Poland) No **4**, Chem No 1, 5-14 (1954) (Reduction of aromatic nitro compds by H_2S in pyridine) & CA **50**, 214-5(1956) 21)S.Carboni & G.Berti, Gazz **84**, 683-91(1954) (Transformation of nitramines into azoxy compds) & CA **50**, 991-2 (1956) 22)G.Costa, Gazz **85**, 548-60(1955) (UV spectra of azo and azoxy compds) & CA **49**, 14483(1955) 23)W.Kast, AngChem **67**, 592-601(1955) (Molecular structure of azo and azoxy compds) & CA **50**, 1398(1956) 24)J.F.Brown, Jr, JACS **77**, 6341-51(1955) (Identification of azoxy compds and others by their IR spectra) & CA **50**, 2297(1956)

AZOXYANILINE AND DERIVATIVES

Azoxydianiline or Diaminoazoxybenzene (called Azoxyanilin or Diaminoazoxybenzol in Ger), $H_2N{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_4{\cdot}NH_2$; mw 228.25, N 24.55%. Three isomers are described in the literature: *o,o'-azoxyaniline or 2,2'-diaminoazoxybenzene* (Ref 1); *m,m'-azoxyaniline or 3,3'-diaminoazoxybenzene* (Ref 2); and *p,p'-azoxyaniline or 4,4'-diaminoazoxybenzene* (Ref 3). Some of its nitro derivs may be of interest as expl ingredients

Refs: 1)Beil **16**, 652 & (392) 2)Beil **16**, 653, (392) & [338] 3)Beil **16**, 654

Azido, $C_{12}H_{11}N_7O$, and *Diazido*, $C_{12}H_{10}N_{10}O$, *Derivatives* were not found in Beil or in CA through 1956

Mononitroazoxyaniline, $C_{12}H_{11}N_5O_3$, was not found in Beil or in CA through 1956

4,4'-Diamino-3,3'-dinitroazoxybenzene, $H_2N{\cdot}C_6H_3(NO_2){\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}NH_2$; mw 318.25, N 26.41%; orn-red tabular ndls (from phenol + alc), mp 328-30(decompn). This compd was prepd by refluxing with acetic acid and concd HCl 4,4'-bis (acetamido)-3,3'-dinitroazoxybenzene, previously obtd by oxidn of 4-amino-2-nitroacetanilide in dioxane with Caro's acid soln. No other props of the dinitroazoxyaniline were given

Ref: 1)Beil – not found 2)C.M.Atkinson et al, JCS **1954**, 2026-7 & CA **49**, 5486-7 (1955)

Trinitro, $C_{12}H_9N_7O_7$, *Tetranitro*, $C_{12}H_8N_8O_9$, or higher nitro derivs of azoxyaniline were not found in Beil or in CA through 1956

AZOXYANISOLE AND DERIVATIVES

Azoxyanisole or Azoxydianisole (called Azoxyanisol or Dimethoxy-azoxybenzol in Ger), $CH_3{\cdot}O{\cdot}C_6H_4(N_2O){\cdot}C_6H_4{\cdot}O{\cdot}CH_3$; mw 258.27, N 10.85%. Three isomers are described in the literature: *o,o'-Azoxyanisole* (Ref 1); *m,m'-Azoxyanisole* (Ref 2) and *p,p'-Azoxyanisole* (Ref 3). The nitro derivs of Azoxyanisole may be of interest as expl ingredients

Refs: 1)Beil **16**, 635 2)Beil **16**, 636& [325] 3)Beil **16**, 637, (383) & [326]

Azido, $C_{14}H_{13}N_5O_3$, and *Diazido*, $C_{14}H_{12}N_8O_3$, *Derivs* of azoxyanisole were not found in Beil or in CA through 1956

Nitroderivatives of Azoxyanisole

Mononitroazoxyanisole, $C_{14}H_{13}N_3O_5$, *Derivs* were not found in Beil or in CA through 1956

Dinitroazoxyanisole, $H_3CO{\cdot}C_6H_3(NO_2){\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}OCH_3$; mw 348.27, N 16.09%. Two isomers are described in the literature:

5,5'-Dinitro-2,2'-dimethoxyazoxybenzene, lfts (from benz), mp 209°; was prepd by electrolytic reduction of 2,4-dinitroanisole in an alkaline soln with a Hg cathode or in a nearly neutral soln with a Cu or Ag cathode

Refs: 1)Beil **16**, (382) 2)K.Brand & T. Eisenmenger, JPraktChem **87**, 495 & 506 (1913) & CA **8**, 2154(1914)

5,5'-Dinitro-3,3'-dimethoxyazoxybenzene, mp between 170 & 200°; was formed in small quantity by the reduction of 3,5-dinitroanisole with Na_2S_2 in alc, in addn to a larger quantity of 5-nitro-3-aminoanisole

Refs: 1)Beil **16**, 636 2)J.J.Blanksma, Rec **28**, 111(1909) & CA **3**, 1746(1909)

Trinitro, $C_{14}H_{11}N_5O_9$, *Tetranitro,* $C_{14}H_{10}N_6O_{11}$ and higher nitro derivs of azoxyanisole were not found in Beil or in CA through 1956

AZOXYBENZALDEHYDE AND DERIVATIVES

Azoxybenzaldehyde; Azoxybisbenzaldehyde or Azoxydibenzaldehyde, $OHC{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_4{\cdot}CHO$, mw 254.24, N 11.02%. Its three isomers: o,o'-, m,m'- and p,p'- are described in Beil **16**, 640,641,(387) & [334]. Of these the para- is of interest because it flashes easily and because its nitrocompd is known:

p,p'-Azoxybenzaldehyde, yel ndls (from benz, alc or dil AcOH), mp 194° (Ref 2), 190° (Ref 3); decomp or flashes up at higher temp with evoln of flame; sol in benz or AcOH; sl sol in alc ligroin and hot w. Can be prepd by reducing p-nitrobenzaldehyde or by other methods

Refs: 1)Beil **16**, 641 & [334] 2)A.Kirpal, Ber **30**, 1598(1897) 3)Alway, AmChemJ **28**, 40(1902) & JCS **82** I, 697 (1902) 4)E. Bamberger & F.Elger, Ann **475**, 307(1929)

x-Nitro-p,p'-azoxybenzaldehyde, $OHC{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}CHO$, mw 299.24, N 14.04%. Yel ndls (from AcOH), mp 171-2°; sol in hot AcOH; sl sol in alc & ether; insol in w. Was obtained on heating p,p-azoxybenzaldehyde with fuming nitric acid

Refs: 1)Beil **16**, 643 2)F.J.Alway, AmChemJ **28**, 43(1902) & JCS **82** I, 697(1902)

AZOXYBENZENE AND DERIVATIVES

Azoxybenzene, $C_6H_5{\cdot}(N_2O){\cdot}C_6H_5$; mw 198,22, N 14.13%. Exists in two modifications: ordinary, mp 36°; and isoazoxybenzene; mp 84°. Snelling & Wyler (Ref 2) found that AN is sensitized by the addn of 1-6% azoxybenzene. Some of its azido derivs and high nitro compds may be of interest as expl ingredients

Refs: 1)Beil **16**, 621-4, (376) & [313-4] 2)W.O.Snelling & J.A.Wyler, USP 1827675 (1932) & CA **26**, 601(1932)

Azidoazoxybenzene, $C_{12}H_9N_5O$, not found in Beil or in CA through 1956

3,3'-Diazidoazoxybenzene, $N_3{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_4{\cdot}N_3$; mw 280.25, N 39.99%; ocher col crysts (from petr eth); mp 85-6°, explodes when heated in a tube above its mp. It was prepd by diazotizing 3,3'-diamino-azoxybenzene, pptg the perbromide of the tetrazo deriv, and treating the latter with NH_3

Refs: 1)Beil **16**, 629 2)R.Meldola & E.R. Andrews, JCS **69** I, 9(1896)

2-Nitroso-azoxybenzene, $ON{\cdot}C_6H_4{\cdot}N{:}(O{:})N{\cdot}C_6H_5$; mw 227.22, N 18.49%; sulfur-yel crysts, mp ca 106° with a blue-green color becoming orn-red. It was prepd by heating on a steam bath 2-hydroxyl-amino-azoxybenzene with freshly pptd mercury oxide in ether

Refs: 1)Beil **16**, [316] 2)G.Cusmano & L.Della Nave, Gazz, **51** I, 68(1921) & CA **15**, 2840(1921)

Nitroderivatives of Azoxybenzene

Mononitroazoxybenzene, $O_2N{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_5$; mw 243.22, N 17.28%. Three isomers are described in the literature:

2-Nitroazoxybenzene; yel ndls or prisms, mp 49°; readily sol in eth or benz, less sol in alc. Its prepn and other props are given in Ref 1

3-Nitroazoxybenzene α-form); yellowish ndls

(from acet), mp 120-1°; sol in 5 p boiling acet or in 25 p hot alc, diffc sol in boiling w. (β-form); lt yel ndls (from absol alc), mp 86-88°; nearly insol in w. The prepn and other props of both α & β forms are given in Ref 2

4-Nitroazoxybenzene (α-form); yellowish crysts (from alc or ligroin), mp 153°. (β-form); yel crysts (from benz), mp 149°. The prepn and other props of both α & β forms are given in Ref 3

Refs: 1)Beil **16**, 627, (377) & [316] 2)Beil **16**, [317] 3)Beil **16**, 627, (377) & [317]

Dinitroazoxybenzene, $O_2N{\cdot}C_6H_4(N_2O){\cdot}C_6H_4{\cdot}NO_2$; mw 288.22, N 19.44%. Seven isomers are described in the literature:

2,2'-Dinitroazoxybenzene; yellowish ndls, mp 175.5°; readily sol in acet, chlf or hot benz; less sol in alc or ligroin. Its prepn is given in Ref 1

3,3'-Dinitroazoxybenzene; yellowish ndls (from concd HNO_3), almost wh ndls (from concd formic acid), mp 143-6.5°; very sol in cold concd formic acid, sol in eth or carbon disulfide, moderately sol in benz or toluene, very diffc sol in cold alc. Its prepn and other props are given in Refs 2,7 & 8

2,4'-Dinitroazoxybenzene; mp 135°, obtd in addn to 4,4'-dinitroazoxybenzene on treating β-4-nitroazoxybenzene with HNO_3 (Ref 3)

4,4'-Dinitroazoxybenzene; sulfur-yel ndls (from benz) or lemon-yel crysts (from glacial acetic acid), mp 192-3°. Its prepn and other props are given in Refs 4 & 8

2,4-Dinitroazoxybenzene, $(O_2N)_2C_6H_3{\cdot}(N_2O){\cdot}C_6H_5$; lt yel ndls (from alc), mp 141°. Its prepn in given in Ref 5

2,6-Dinitroazoxybenzene; lfts (from benz), mp 172°. Its prepn is given in Ref 5

3,5-Dinitroazoxybenzene; nearly colorless crysts (from glacial acetic acid), mp 171-3°; sl sol in all solvs. Its prepn is given in Ref 6

Refs: 1)Beil **16**, 627 2)Beil **16**, 627,(377) & [317] 3)Beil **16**, (378) 4)Beil **16**, 628, (378) & [318] 5)Beil **16**, (379) 6)Beil **16**, [318] 7)R.C.Elderfield & E.F.Clafin, JACS **74**, 2953-9(1952) & CA **48**, 9370(1954) 8) P.H. Gore & O.H.Wheeler, JACS **78**, 2160-3 (1956) & CA **50**, 9873(1956)

Trinitroazoxybenzene, $(O_2N)_2{\cdot}C_6H_3{\cdot}(N_2O){\cdot}C_6H_4{\cdot}NO_2$; mw 333.22, N 21.02%. Four isomers are described in the literature:

2,4,2'-Trinitroazoxybenzene; nearly colorless tablets or prisms (from acet), mp 187-92°; readily sol in hot chlf, acet, glacial acetic acid, nitrobenz or hot HNO_3; diffc sol in alc, eth or ligroin. Its prepn and other props are given in Ref 1

2,4,3'-Trinitroazoxybenzene; sulfur-yel prisms or crysts (from acet), mp 175-8°; 100 g hot benz dissolves 4.5 g of compd. Its prepn and other props are given in Refs 1 & 2

2,4,4'-Trinitroazoxybenzene; sulfur-yel ndls (from HNO_3) or sulfur-yel prisms (from benz), mp 135-7°. Its prepn and other props are given in Ref 1

2,4,6-Trinitroazoxybenzene, $(O_2N)_3C_6H_2{\cdot}(N_2O){\cdot}C_6H_5$; lt-yel ndls (from glacial acetic acid), mp 170°; sol without change in HNO_3 (d 1.45), but after 12 hrs in contact with HNO_3 (d 1.52) there is formed *2,4,6,3' (?)-tetranitroazoxybenzene.* The trinitro azoxy-compd is formed by treating 2,4,6-trinitro-azobenzene with concd H_2O_2 in acetic acid soln (Refs 2 & 3)

Refs: 1)Beil **16**, 628 2)Beil **16**, (379) 3)A.Angeli & B.Valori, AttiAccadLinceiMem [5] **22** I, 139(1913) & JCS **104** I, 533-4(1913)

Tetranitroazoxybenzene, $C_{12}H_6N_6O_9$; mw 378.22, N 22.22%. Two isomers are described in the literature:

3,5,3'5'-Tetranitroazoxybenzene, $(O_2N)_2C_6H_3{\cdot}N(:O):N{\cdot}C_6H_3(NO_2)_2$; yel crysts (from HNO_3 or alc + acet), mp 185°; sol in benz or acet, the acet soln gives a violet color in the

presence of alkali; the addn salt with HCl is unstable. The prepn of the tetranitro compd is described in Ref 1. Gore & Wheeler (Ref 4) detd its absorption spectrum and some other props

2,4,6,3'(?)-Tetranitroazoxybenzene, $(O_2N)_3C_6H_2\cdot(N_2O)\cdot C_6H_4\cdot NO_2$; yellowish prisms (from benz), mp 192°. By prolonged treatment of 2,4,6-trinitroazoxybenzene with HNO_3 (d 1.52), the tetranitro compd was obtd (Refs 2 & 3)

Refs: 1)Beil **16**, 629 & [318] 2)Beil **16**, (379) 3)A.Angeli & B.Valori, AttiAccad LinceiMem [5] **22** I, 139-40(1930) & JCS **104** I, 534(1913)

Pentanitroazoxybenzene, $C_{12}H_5N_7O_{11}$, not found in Beil or in CA through 1956

2,4,6,2',4',6'-Hexanitroazoxybenzene, $(O_2N)_3C_6H_2\cdot(N_2O)\cdot C_6H_2(NO_2)_3$; mw 468.22, N 23.93%. This compd is listed in Refs 2 & 3 but no information is given concerning its method of prepn or expl props, except mention that the compd is reactive

Refs: 1)Beil – not found 2)A.H.Blatt & F.C.Whitmore, OSRD Rpt No 1085(1942), p 43 3)A.H.Blatt, OSRD Rpt No 2014(1944), p XVIII

AZOXYBENZENECARBOXYLIC ACID AND DERIVATIVES

Phenylazoxybenzoic Acid or Azoxybenzene-monocarboxylic Acid [called Azoxybenzol-carbonsäure-(2) or (4) in Ger]; $C_6H_5\cdot(N_2O)\cdot C_6H_4\cdot COOH$; mw 242.23, N 11.57%. Two isomers are described in the literature:

Azoxybenzene-2-Carboxylic Acid; yel prisms or small lfts (from benz); mp 105-6°, 110-11° & 118°; readily sol in common org solvs, diffc sol in w; the soln in concd H_2SO_4 rapidly becomes dk-red. Its prepn is described in Ref 1

Azoxybenzene-4-Carboxylic Acid. This compd exits in both *α* and *β* forms: *α-form*-yel lfts (from alc); mp 231°; readily sol in alc or glacial acetic acid; *β-form*-yel ndls (from alc); mp 241°; readily sol in alc or glacial acetic acid.

The prepn and other props of both *α*- and *β*-forms are described in Ref 2

Refs: 1)Beil **16**, 644, (388) & [335] 2)Beil **16**, (389) & [336]

Azido, $C_{13}H_9N_5O_3$, and *Diazido,* $C_{13}H_8N_8O_3$, *Derivatives* were not found in Beil or in CA through 1956

4'-Nitroazoxybenzene-4-Carboxylic Acid, $O_2N\cdot C_6H_4\cdot(N_2O)\cdot C_6H_4\cdot COOH$; mw 287.23, N 14.63%; yel cryst powd; mp ca 260 with decompn; prepd from *β*-azoxybenzene-4-carboxylic acid by treatment with HNO_3 (d 1.48) in glacial acetic acid

Ref: Beil **16**, (389)

Dinitroazoxybenzene-monocarboxylic Acid, $C_{13}H_8N_4O_7$, was not found in Beil or in CA through 1956

3,5,3'-Trinitroazoxybenzene-5'-carboxylic Acid, $HOOC\cdot C_6H_3(NO_2)\cdot(N_2O)\cdot C_6H_3(NO_2)_2$; mw 377.23, N 18.57%; col ndls (from alc), mp 216°; sol in NaOH, giving a red color in strong alkali; dissolves in Na_2CO_3 soln or 0.1N NaOH with no color change. This compd was obtd when 3,3'-dinitro-5,5'-dicarboxyazoxybenzene was boiled in HNO_3 (d 1.48) for 16 hrs. The *NH_4 salt* was prepd by adding strong NH_4OH dropwise to a suspension of the compd in warm w until the subst dissolved. Repeated crystn from w gave fine cream-col ndls which lost their w of crystn in vacuo at 60°

Refs: 1)Beil – not found 2)A.Bolliger & F.Reuter, JProcRoySocNSWales **73**, 74 (1939) & CA **34**, 5419-20 (1940)

Tetranitro, $C_{13}H_6N_6O_{11}$, and higher nitro derivs of azoxybenzene-monocarboxylic acid were not found in Beil or in CA through 1956

AZOXYBENZENEDICARBOXYLIC ACID AND DERIVATIVES

Azoxydibenzoic Acid or Azoxybenzenedicarboxylic Acid (called Azoxybenzoesäure or Azoxybenzol-dicarbonsäure in Ger), $HOOC{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_4{\cdot}COOH$; mw 286.24, N 9.79%. Three isomers are described in the literature: *o,o'-azoxybenzoic acid or azoxybenzene-2,2'-dicarboxylic acid* (Ref 1); *m,m' azoxybenzoic acid or azoxybenzene-3,3'-dicarboxylic acid* (Ref 2); and *p,p'-azoxybenzoic acid or azoxybenzene-4,4'-dicarboxylic acid* (Ref 3). The prepn and props of these isomers are described in the refs indicated

Refs: 1)Beil **16**, 644,(388) & [335] 2)Beil **16**, 646 & (388) 3)Beil **16**, 647 & [336]

Azido, $C_{14}H_9N_5O_5$, and *Diazido,* $C_{14}H_8N_8O_5$, *Derivatives* were not found in Beil or in CA through 1956

Mononitroazoxybenzoic Acid, $C_{14}H_9N_3O_7$, was not found in Beil or in CA through 1956

5,5'-Dinitroazoxybenzene-3,3'-dicarboxylic Acid or 3,3'-Dinitro-5,5'-dicarboxyazoxybenzene, $HOOC{\cdot}C_6H_3(NO_2){\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}COOH$; mw 376.24, N 14.89%; cream-col small ndls (from anisole + alc), mp 288; readily sol in alc but nearly insol in cold w, eth or benz. It was prepd by treating 3,5-dinitrobenzoic acid with NaOH soln and pptg the product by adding HCl. It can be recrystd from HNO_3 without change

Refs: 1)Beil **16**, 647 2)A.Bolliger & F.Reuter, JProcRoySocNSWales **73**, 74(1939) & CA **34**, 5419-20(1940)

Trinitro, $C_{14}H_7N_5O_{11}$, and *Tetranitro,* $C_{14}H_6N_6O_{13}$, *Derivatives* of azoxybenzoic acid were not found in Beil or in CA through 1956

AZOXYDICARBOXYLAMIDE DIOXIME AND DERIVATIVES

Azoxydicarboxylamide Dioxime (called Azoxy-carbonsäure-bisamidoxim in Ger), $HO{\cdot}N{:}C(NH_2){\cdot}N_2O{\cdot}C(NH_2){:}N{\cdot}OH$; mw 162.12, N 51.84%; red-orn crysts (from w at 60°), mp explodes ca 99° without leaving a residue; sol in hot w with slow decompn; insol in org solvs. This compd was obtained, in addn to other products, by Wieland (Ref 2) on carefully treating with HNO_3 the alkaline soln of dihydroxyguanidine hydrobromide, $HO{\cdot}NH{\cdot}C(:NH){\cdot}NH{\cdot}OH$, HBr. The latter compd was prepd by reacting at –20° cyanogen bromide and hydroxylamine dissolved in a mixt of methyl alc and anhyd eth. The reaction between cyanogen bromide and free hydroxylamine in alcoholic ethereal soln at RT is violent and almost expl

The *silver salt* of azoxydicarboxylamide dioxime, $Ag_2C_2H_4N_6O_3$, dk-brn flakes, was reported to detonate when heated on a Pt foil

Refs: 1)Beil **3**, 124 2)H.Wieland, Ber **38**, 1452-3(1905) & JCS **88** I, 421(1905)

Azoxydicarboxylamide Dioxime Dibenzoate, $C_6H_5{\cdot}CO{\cdot}O{\cdot}N{:}C(NH_2){\cdot}(N_2O){\cdot}C(NH_2){:}N{\cdot}O{\cdot}{-}OC{\cdot}C_6H_5$; mw 370.32, N 22.70%; yel ndls (from glacial acetic acid) mp – explodes at 155°. This compd was obtd, in addn to col ndls of benzoylguanidine benzoate [$C_6H_5{\cdot}CO{\cdot}NH{\cdot}C(NH_2){:}N{\cdot}O{\cdot}OC{\cdot}C_6H_5$], when dihydroxyguanidine and benzoyl chloride reacted in the presence of sodium bicarbonate. Attempts to methylate or benzoylate azodicarboxylamide dioxime by means of methyl sulfate or benzoyl chloride either in Na_2CO_3 or $NaHCO_3$ soln were without success. The yel material isolated was purified by repeated shaking with acet until it was sol in dil NaOH, which quickly caused decompn into benzoic acid, nitrogen, and hydroxycarbamide. The col purified crysts of the benzoylguanidine benzoate decompd at 162-3°

Refs: 1)Beil **9**, 300 2)H.Wieland & H. Bauer, Ber **40**, 1687-90(1907) & JCS **92** I, 492(1907)

Azoxyethane, $C_2H_5{\cdot}(N_2O){\cdot}C_2H_5$, as well as its *Azido, Diazido, Nitro, Dinitro, Nitroso,*

and Nitronitroso Derivatives were not found in Beil or CA through 1956

AZOXYMETHANE AND DERIVATIVES

Azoxymethane, $H_3C{\cdot}(N_2O){\cdot}CH_3$; mw 74.08, N 37.82%; col liq, bp 98° at 760 mm Hg; n_D 1.4300 at 17°. It was prepd by oxidg azomethane with perbenzoic acid. Azoxymethane was decompd by hot concd aq alkali with the formation of volatile base and was converted by hot HCl into formic acid and methylhydrazine. The UV and IR spectra were also reported

The combustion of azoxymethane must be carried out in a current of air as the use of oxygen led to explosions

Refs: 1)Beil – not found 2)B.W.Langley et al, JCS **1952**, 4191-5 & CA **48**, 4432-3 (1954)

No *Azido*, $C_2H_5N_5O$, *Diazido*, $C_2H_4N_8O$, *Mononitro*, $C_2H_5N_3O_3$, *Dinitro*, $C_2H_4N_4O_5$, or other nitro *Derivatives* of azoxymethane were found in Beil or in CA through 1956

AZOXYNAPHTHALENE AND DERIVATIVES

Azoxynaphthalene, $C_{10}H_7{\cdot}(N_2O){\cdot}C_{10}H_7$; mw 298.33, N 9.39%. Two derivs are described in the literature:

1,1'- or α-α'-Azoxynaphthalene (Ref 1) and *2,2'- or β-β'-Azoxynaphthalene* (Ref 2)

Refs: 1)Beil **16**, 632, (380) & [322] 2)Beil **16**, 633 & [322]

Azidoazoxynaphthalene, $C_{20}H_{13}N_5O$, or *Diazido Derivatives* $C_{20}H_{12}N_8O$, were not found in Beil or in CA through 1956

Mononitroazoxynaphthalene, $C_{20}H_{13}N_3O_3$, not found in Beil or in CA through 1956

Dinitroazoxynaphthalene, $O_2N{\cdot}C_{10}H_6{\cdot}(N_2O){\cdot}{\cdot}C_{10}H_6{\cdot}NO_2$; mw 388.33, N 14.43%. Two isomers are described in the literature:

5,5'-Dinitro-1,1'-azoxynaphthalene, orn-yel to brn-red powd (when dry); mp-decompg above 200°, slowly turning brn; sol in concd H_2SO_4 with a red color, turning blue-violet on heating. This compd was obtd by reducing 1,5-dinitronaphthalene with Zn dust and NH_4Cl or phenylhydrazine and NaOH

Refs: 1)Beil **16**, 633 2)L.Wacker, Ann **321**, 65(1902) & JCS **82** I, 506(1902)

4,4'-Dinitro-2,2'-azoxynaphthalene, yellowish brn-red iridescent ndls (from nitrobenzene), mp 305-6° & 315°; insol in w, alc, ether, benz or glacial acetic acid. This compd was obtd as one of the products from the reaction of hydrazine or hydrazine hydrate and 4-chloro-1,3-dinitronaphthaline in alc

Refs: 1)Beil **16**, [322] 2)A.K.Macbeth & J.R.Price, JCS **1937**, 983-4

Trinitro, $C_{20}H_{11}N_5O_7$; *Tetranitro*, $C_{20}H_{10}N_6O_9$; and higher nitro derivatives of azoxynaphthalene were not found in Beil or in CA through 1956

AZOXYPHENETOLE AND DERIVATIVES

Azoxyphenetole or Diethoxyazoxybenzene, $C_{16}H_{18}N_2O_3$; mw 286.32, N 9.78%. Three isomers are described in the literature: *o,o'-azoxyphenetole* (Ref 1), *m,m'-azoxyphenetole* (Ref 2), and *p,p'-azoxyphenetole* (Ref 3). The nitro derivs of azoxyphenetole may be of interest as expl ingredients

Refs: 1)Beil **16**, 635 & [324] 2)Beil **16**, 636 & [325] 3)Beil **16**, 638, (384) & [327]

Azido, $C_{16}H_{17}N_5O_3$, and *Diazido*, $C_{16}H_{16}N_8O_3$, *Derivatives* of azoxyphenetole were not found in Beil or in CA through 1956

Nitro Derivatives of Azoxyphenetole

3-Nitro-4,4'-diethoxyazoxybenzene, $C_2H_5{\cdot}O{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}O{\cdot}C_2H_5$; mw 331.32, N 12.68%; lt yel ndls (from alc), mp 153°; obtd by treating p,p'-azoxyphenetole with HNO_3 (d 1.45) in acetic acid soln

Refs: 1)Beil **16**, (385) 2)B.Valori, Atti AccadLinceiMem [5] **23** II, 285(1914) & CA **9**, 1475-6(1915)

3,3'-Dinitro-4,4'-diethoxyazoxybenzene, $C_2H_5 \cdot O(NO_2) \cdot C_6H_3 \cdot (N_2O) \cdot C_6H_3(NO_2) \cdot O \cdot C_2H_5$; mw 376.32, N 14.89%; yel cryst powd (from alc), mp 185°; readily sol in glacial acetic acid, diffc sol in alc; obtd by treating p,p'-azoxyphenetole with HNO_3 (d 1.48) in glacial acetic acid

Refs: 1)Beil **16**, (385) 2)B.Valori, Atti AccadLinceiMem [5] **23** II, 290(1914) & CA **9**, 1475-6(1915)

X,X,X-Trinitro-4,4'-diethoxyazoxybenzene, $C_{16}H_{15}N_5O_9$, mw 421.32, N 16.62%. Two isomers identified by mp have been reported:

I. mp 168°, sulfur-yel ndls (from hot ethyl acetate); readily sol in boiling eth, chlf, benz or glacial acetic acid; diffc sol in cold alc; insol in w.

II. mp 187°, lt yel ndls (from hot ethyl acetate); readily sol in boiling ethyl acetate; moderately sol in chlf or benz; diffc sol eth or hot glacial acetic acid; insol in alc.

The prepn and other props of these trinitroazoxyphenetoles is given in Beil

Ref: Beil **16**, 639

Tetranitro, $C_{16}H_{14}N_6O_{11}$, and higher derivs of azoxyphenetole were not found in Beil or in CA through 1956

AZOXYPHENOL AND DERIVATIVES

Azoxyphenol or Dihydroxyazoxybenzene, $C_{12}H_{10}N_2O_3$, mw 230.22, N 12.17%. Three isomers are described in the literature:

o,o'-azoxyphenol (Ref 1), *m,m'-azoxyphenol* (Ref 2), and *p,p'-azoxyphenol* (Ref 3). The nitro derivs of azoxyphenol may be of interest as expl ingredients

Refs: 1)Beil **16**, (381) 2)Beil **16**, 636 & [325] 3)Beil **16**, 637 & [326]

Azido, $C_{12}H_9N_5O_3$, and *Diazido*, $C_{12}H_8N_8O_3$, *Derivs* of azoxyphenol were not found in Beil or in CA through 1956

3-Nitro-4,4'-dihydroxy-azoxybenzene, $HO \cdot C_6H_4 \cdot N(:O):N \cdot C_6H_3(NO_2) \cdot OH$; mw 275.22, N 15.27%; orn-yel ndls (from alc), mp 195°; can be prepd by treating 4,4'-dihydroxyazoxybenzene with $NaNO_2$ in glacial acetic acid or by sapon of 3-nitro-4-hydroxy-4'-benzoylhydroxy-azoxybenzene with concd KOH soln

Ref: Beil **16**, [328]

Dinitro, $C_{12}H_8N_4O_7$, *Trinitro*, $C_{12}H_7N_5O_9$ and higher nitro derivs of azoxyphenol were not found in Beil or in CA through 1956

AZOXYPROPANE AND DERIVATIVES

Azoxypropane or Dimethylazoxyethane, $C_6H_{14}N_2O$; mw 130.19, N 21.52%. Two isomers were reported:

1,1'-Azoxypropane, $C_2H_5 \cdot CH_2 \cdot (N_2O) \cdot CH_2 \cdot C_2H_5$; col liq, bp 67° at 20 mm Hg; d 0.902 at 20°; and n_D 1.4365 at 20°. It was prepd by oxidg 1-azopropane, in dry methylene dichloride, with perbenzoic acid. This compd was decompd by hot concd aq alkali and converted by hot HCl into propionic acid and n-propylhydrazine. The UV and IR spectra were reported

2,2'-Azoxypropane, $(H_3C)_2 \cdot CH \cdot (N_2O) \cdot CH \cdot (CH_3)_2$; col liq, bp 38° at 14 mm Hg. It was prepd by oxidg 2-azopropane as described for 1-azopropane. Catalytic reduction of 2,2'-azoxypropane produced N,N'-di-2-propylhydrazine and with hot HCl the compd was converted into hydrazine hydrochloride. The UV and IR spectra were reported

Refs: 1)Beil – not found 2)B.W.Langely et al, JCS 1952, 4191-7 & CA **48**, 4432-3 (1954)

Azido, $C_6H_{13}N_5O$, and *Diazido*, $C_6H_{12}N_8O$, *Derivatives* were not found in Beil or in CA through 1956

Mononitro, $C_6H_{13}N_3O_3$, and *Trinitro*, $C_6H_{11}N_5O_7$, *Derivatives* were not found in Beil or in CA through 1956

2,2'-Dinitro-2,2'-Azoxypropane or **2,2'-Dinitro-2,2'-Dimethylazoxyethane** (called

sym-Dinitro-tetramethylazoxymethan in Beil), $(H_3C)_2{\cdot}C{\cdot}(NO_2){\cdot}(N_2O){\cdot}C(NO_2){\cdot}(CH_3)_2$; mw 220.19, N 25.45%; crysts (from ligroin), mp 97°, bp 100° at 25 mm press (with a little decompn); readily sol in alc, ether, chlf, benz, acet or glacial acetic acid. It is one of the products formed when acetoxime in eth soln was treated with nitrogen peroxide, N_2O_4. Other props are given in the Ref

Ref: Beil **1**, 651

AZOXYTOLUENE AND DERIVATIVES

Azoxytoluene or Dimethylazoxybenzene, $CH_3{\cdot}C_6H_4{\cdot}(N_2O){\cdot}C_6H_4{\cdot}CH_3$; mw 226.27, N 12.38%. Five isomers are described in the literature: *o,o'-azoxytoluene or 2,2'-dimethylazoxybenzene;* yel plates (from petr eth) or lt yel lfts (from dil alc); mp 59-60°, explodes and chars on rapid heating (Ref 1); *iso-o,o'-azoxytoluene;* lt yel long ndls (from petr eth), prisms (from benz) or little ndls (from methyl alc + w); mp 80-2°; readily sol in common org solvs, petr eth or cold benz; insol in w; its solns are lt yel in color (Ref 2); *m,m'-azoxytoluene or 3,3'-dimethylazoxytoluene;* lt yel ndls (from eth); mp 37-9°; readily sol in alc, eth, chlf, CS_2, ligroin or benz (Ref 3); *p,p'-azoxytoluene or 4,4'-dimethylazoxytoluene;* pale yel ndls (from alc) or sulfur-yel plates (from ligroin); mp 69-70°; readily sol in alc or eth (Ref 4); and *w,w'-azoxytoluene,* $C_6H_5{\cdot}CH_2{\cdot}(N_2O){\cdot}CH_2{\cdot}C_6H_5$ (Ref 5). The prepn and other props of these azoxytoluenes can be found in the refs indicated. Nitro or other derivs of azoxytoluene may be of interest as expl ingredients

Refs: 1)Beil **16**, 629, (379) & [318] 2) Beil **16**, 629 & [318 nm] 3)Beil **16**, 630 & [319] 4)Beil **16**, 630, (380) & [320] 5) Beil **16**, 631

Azido, $C_{14}H_{13}N_5O$, and *Diazido,* $C_{14}H_{12}N_8O$, derivs of azoxytoluene were not found in Beil or in CA through 1956

Nitroso, $C_{14}H_{13}N_3O_2$, and *Dinitroso,* $C_{14}H_{12}N_4O_3$, derivs of azoxytoluene were not found in Beil or in CA through 1956

Nitroderivatives of Azoxytoluene

Mononitroazoxytoluene, $C_{14}H_{13}N_3O_3$, was not found in Beil or in CA through 1956

Dinitroazoxytoluene or Dinitrodimethylazoxybenzene, $H_3C{\cdot}C_6H_3(NO_2){\cdot}(N_2O){\cdot}C_6H_3(NO_2){\cdot}CH_3$; mw 316.27, N 17.72%. Three isomers are described in the literature:

3,3'-Dinitro-2,2'-dimethylazoxybenzene; yel ndls (from benz) or nearly col crysts (from acet), mp 187-90°; sol in formic acid, diffc sol in alc; heated on a w bath with concd H_2SO_4 gives 3,3'-dinitro-4-hydroxy-2,2'-dimethylazobenzene. The prepn of dinitroazoxytoluene is described in Ref 1

4,4'-Dinitro-3,3' (or 2,2')-dimethylazoxybenzene, dk-brn prisms (from glacial acetic acid + benz), mp 188-9°; diffc sol in all solvs. Its prepn is described in Ref 2

3,3'-Dinitro-4,4'-dimethylazoxybenzene, lt yel ndls (from benz), mp 164°; sol in hot formic acid or in ethyl acetate; diffc sol in alc or cold formic acid. Its prepn is described in Ref 3

Refs: 1)Beil **16**, 630 & [318] 2)Beil **16**, (380) 3)Beil **16**, 631 & [320]

Trinitroazoxytoluene, $C_{14}H_{11}N_5O_7$, was not found in Beil or in CA through 1956 but this compd (no formula given) is listed by A.H. Blatt & F.C. Whitmore, OSRD Rpt No 1085 (1942), p 100

Tetranitroazoxytoluene, $H_3C{\cdot}C_6H_2(NO_2)_2{\cdot}(N_2O){\cdot}C_6H_2(NO_2)_2{\cdot}CH_3$; mw 406.27, N 20.69%. Two isomers are mentioned in the literature:

2,6,2',6'-Tetranitro-4,4'-dimethylbenzene was reported (Ref 3) isolated in pure cryst form as an intermediate compd during the reduction of TNT by partially purified xanthine oxidase. No props of this compd were given

3,5,3',5'-Tetranitro-4,4'-dimethylbenzene,

ndls (from benz or glacial acetic acid), mp 212-6°; was prepd by treating 2,6-dinitro-4-hydroxylaminotoluene with concd HCl (Ref 1)

Tetranitroazoxytoluene is a HE of power and brisance less than that of PA (Ref 2)

Refs: 1)Beil **16**, (380) 2)Blatt (1944) 3) E.Bueding & N.Jolliffe, JPharmacol **88**, 300-12 (1945) & CA **41**, 510(1947)

Pentanitroazoxytoluene, $C_{14}H_9N_7O_{11}$, and *Hexanitroazoxytoluene*, $C_{14}H_8N_8O_{13}$, were not found in Beil or in CA through 1956

AZOXYXYLENE AND DERIVATIVES

Azoxyxylene or Tetramethylazoxybenzene, $(CH_3)_2C_6H_3\cdot(N_2O)\cdot C_6H_3(CH_3)_2$; mw 254.32, N 11.02%. Five isomers are described in the literature: *3,3'-azoxy-o-xylene or 2,3,2',3'-tetramethylazoxybenzene* (Ref 1), *4,4'-azoxy-o-xylene or 3,4,3',4'-tetramethylazoxybenzene* (Ref 1), *2,2'-azoxy-m-xylene or 2,6,2',6'-tetramethylazoxybenzene* (Ref 2), *4,4'-azoxy-m-xylene or 2,4,2',4'-tetramethylazoxybenzene* (Ref 2), and *2,2'-azoxy-p-xylene or 2,5,2',5'-tetramethylazoxybenzene* (Ref 2)

Refs: 1)Beil **16**, 631 & [321] 2)Beil **16**, 632 & [321]

Azido, $C_{16}H_{17}N_5O$, and *Diazido*, $C_{16}H_{16}N_8O$, *Derivs* of azoxyxylene were not found in Beil or in CA through 1956

Mononitro, $C_{16}H_{17}N_3O_3$, *Dinitro*, $C_{16}H_{16}N_4O_5$, and higher nitro derivs of azoxyxylene were not found in Beil or in CA through 1956

TABLE I

Comparison of US, Tyler, British and German Sieve Series

US(NatlBurStds)			Tyler	BritStdsInstitution(BSI)			German DIN 1171		
Sieve No	Openings in mm	Openings in inches	Approximate Equiv Mesh	Mesh No	Openings in mm	Openings in inches	DIN No	Mesh per sq cm	Openings in mm
2½	8.00	0.315	2½	–	–	–	–	–	–
3	6.72	0.265	3	–	–	–	–	–	–
3½	5.66	0.223	3½	–	–	–	1	1	6.000
4	4.76	0.187	4	–	–	–	–	–	–
5	4.00	0.157	5	–	–	–	–	–	–
6	3.36	0.132	6	5	3.353	0.1320	2	4	3.000
7	2.83	0.111	7	6	2.812	0.1107	–	–	–
8	2.38	0.094	8	7	2.411	0.0949	2½	6.25	2.400
10	2.00	0.079	9	8	2.057	0.0810	3	9	2.000
12	1.68	0.066	10	10	1.676	0.0660	4	16	1.500
14	1.41	0.0557	12	12	1.405	0.0553	–	–	–
16	1.19	0.0468	14	14	1.204	0.0474	5	25	1.200
18	1.00	0.0394	16	16	1.003	0.0395	6	36	1.020
20	0.84	0.0331	20	18	0.853	0.0336	–	–	–
–	–	–	–	–	–	–	8	64	0.750
25	0.71	0.0278	24	22	0.699	0.0275	–	–	–
–	–	–	–	–	–				
30	0.59	0.0234	28	25	0.599	0.0236	10	100	0.600
–	–	–	–	–	–	–	11	121	0.540
35	0.50	0.0197	32	30	0.500	0.0197	12	144	0.490
40	0.42	0.0166	35	36	0.422	0.0166	14	196	0.430
45	0.35	0.0139	42	44	0.353	0.0139	16	256	0.385
50	0.297	0.0117	48	52	0.295	0.0116	20	400	0.300
60	0.250	0.0098	60	60	0.251	0.0099	24	576	0.250
70	0.210	0.0083	65	72	0.211	0.0083	30	900	0.200
80	0.177	0.0070	80	85	0.178	0.0070	–	–	–
100	0.149	0.0059	100	100	0.152	0.0060	40	1600	0.150
120	0.125	0.0049	115	120	0.124	0.0049	50	2500	0.120
140	0.105	0.0041	150	150	0.104	0.0041	60	3600	0.102
170	0.088	0.0035	170	170	0.089	0.0035	70	4900	0.088
200	0.074	0.0029	200	200	0.076	0.0030	80	6400	0.075
230	0.062	0.0025	250	240	0.066	0.0026	100	10000	0.060
270	0.053	0.0021	270	300	0.053	0.0021	–	–	–
325	0.044	0.0017	325	–	–	–	–	–	–
400	0.037	0.0015	400	–	–	–	–	–	–

Note: This table combines information given in 1)Lange's Handbook of Chemistry 2)Chemical Rubber Co's Handbook of Chemistry and Physics and 3)R. D. Cadle's "Particle Size Determination," Interscience, NY(1955). The IMM (Institution of Mining and Metallurgy, London) Series, given in Ref 3, has been largely replaced by the BSI Series, and is not included in this table. The Tyler screens are manufd by the W. S. Tyler Co, Cleveland, Ohio. The French use AFNOR screens which are similar to the German DIN Series

TABLE II

Calibers (Millimeter – Inch Equivalents)

Conversion Factors: 1 inch = 25.40005 mm; 1 mm = 0.03937 inch

mm	in	mm	in	mm	in	mm	in	mm	in	mm	in
5.	.19685	**7.9**	.31102	10.9	.42913	**25.**	.98425	107.	4.2126	300.	11.811
5.08	.20	**7.92**	.31181	10.922	.43	**25.4**	1.	110.	4.3307	304.8	**12.**
5.1	.20079	**8.**	.31496	**11.**	.43307	27.	1.063	114.3	**4.5**	305.	12.008
5.2	.20472	8.1	.31890	11.1	.43701	27.94	1.1	119.38	**4.7**	310.	12.205
5.334	.21	8.128	**.32**	**11.15**	.43898	**28.**	1.10236	**120.**	4.7244	320.	12.598
5.4	.21260	8.2	.32283	11.176	**.44**	**30.**	1.1811	122.	4.8031	330.2	13.
5.5	.21654	8.3	.32677	11.2	.44094	**37.**	1.4567	125.	4.9213	340.	13.386
5.588	**.22**	8.382	.33	11.3	.44488	40.	1.5748	127.	**5.**	**353.**	13.898
5.6	.22047	8.4	.33071	**11.35**	.44685	**42.**	1.65354	**128.**	5.0394	**355.**	13.976
5.7	.22441	8.5	.33465	11.4	.44882	**44.**	1.73228	130.	5.1181	355.6	**14.**
5.8	.22835	8.6	.33858	11.430	**.45**	**45.**	1.7717	135.	5.3150	370.	14.567
5.842	.23	8.636	.34	11.5	.45276	**47.**	1.8504	140.	5.5118	**380.**	14.961
5.9	.23228	8.7	.34252	11.6	.45669	**50.**	1.9685	145.	5.7087	381.	15.
6.	.23622	8.8	.34646	11.684	.46	50.8	2.	**149.**	5.8661	390.	15.354
6.096	.24	8.89	.35	11.7	.46063	55.	2.1654	149.1	**5.87**	400.	15.748
6.1	.24016	8.9	.35039	11.8	.46457	**57.**	2.2441	**150.**	5.9055	406.4	**16.**
6.2	.24409	**9.**	.35433	11.9	.46850	**58.**	2.2835	**152.**	5.9842	410.	16.1417
6.3	.24803	9.068	**.357**	11.938	.47	59.94	**2.36**	152.4	**6.**	420.	16.535
6.35	**.25**	9.1	.35827	12.	.47244	**60.**	2.3622	**155.**	6.1024	430.	16.929
6.4	.25197	9.128	.36	12.1	.47638	**65.**	2.5591	**157.**	6.1811	431.8	17.
6.5	.25591	9.2	.36220	12.192	.48	**70.**	2.7559	**160.**	6.2992	440.	17.3228
6.6	.25984	9.3	.36614	12.2	.48031	**73.**	2.874	**165.**	6.4961	450.	17.717
6.604	.26	9.398	.37	12.3	.48425	**75.**	2.9528	**170.**	6.6929	457.2	18.
6.7	.26378	9.4	.37008	12.4	.48819	**76.**	2.9921	**172.6**	6.7953	470.	18.504
6.8	.26772	9.5	.37402	12.446	.49	**76.2**	**3.**	177.8	7.	482.6	19.
6.858	.27	9.6	.37795	12.5	.49213	**76.5**	3.0118	182.88	7.2	500.	19.685
6.9	.27165	9.65?	**.38**	12.6	.49606	**77.**	3.0315	194.	7.6378	508.	20.
7.	.27559	9.7	.38189	**12.7**	.50	**80.**	3.1496	200.	7.874	5334.4	21.
7.01	**.276**	9.8	.38583	12.8	.50394	**81.**	3.1890	**203.**	7.9921	**540.**	21.2598
7.1	.27953	9.9	.38976	12.9	.50787	**82.**	3.2283	203.2	**8.**	550.	21.654
7.112	.28	9.906	.39	12.954	.51	**83.5**	3.2874	209.3	8.2401	558.8	22.
7.2	.28346	10.	.39370	13.	.51181	**85.**	3.3465	**210.**	8.2677	570.	22.441
7.3	.28740	10.1	.39704	**13.2**	.51968	**87.**	3.4252	**211.**	8.3071	584.2	23.
7.35	.28937	10.15	.39961	13.5	.53150	87.63	**3.45**	220.	8.6614	600.	23.622
7.366	.29	10.16	**.40**	13.9	.54724	**88.**	3.4646	228.6	9.	609.6	24.
7.4	.29134	10.2	.40157	**14.**	.55118	88.9	**3.5**	**234.**	9.213	635.	25.
7.5	.29528	10.3	.40551	14.5	.57087	**90.**	3.5433	**238.**	9.3701	650.	25.591
7.6	.29921	10.35	.40748	15.	.59055	93.98	3.7	**240.**	9.4488	660.4	26.
7.62	**.30**	10.4	.40945	15.24	.60	95.	3.7402	250.	9.8425	685.8	27.
7.63	.30039	10.414	.41	15.43	.60748	**100.**	3.9370	254.	10.	700.	27.559
7.65	.30118	10.5	.41339	17.78	.70	101.6	4.	**260.**	10.236	711.2	28.
7.696	**.303**	**10.6**	.41732	20.	.78740	**102.**	4.0157	270.	10.6299	736.6	29.
7.7	.30315	10.668	.42	20.32	.80	**104.**	4.0945	274.	10.7874	750.	29.528
7.8	.30709	10.7	.42126	22.86	.90	**105.**	4.1339	279.4	11.	762.	30.
7.874	.31	10.8	.42520	**23.**	.90551	**106.**	4.1732	**280.**	11.024	787.4	31.
						106.68	**4.2**	**283.**	11.142	**800.**	31.496
								290.	11.4173	915.	**36.**

Note: Bold figures represent standard and some obsolete US and Foreign calibers

Selected List of Books on Explosives and Propellants Published Since 1900

1)J.Daniel, "Dictionnaire des Matières Explosives," Dunod, Paris(1902) 2)L. Gody, "Traité Théorique et Pratique des Matières Explosives," Wesmael-Charlier, Namur(1907) 3) R.Escales, "Nitroglyzerin und Dynamit," Veit & Co, Leipzig(1908) 4)R.Escales, "Ammonsalpetersprengstoffe," Veit & Co, Leipzig(1909) 5)R.Escales, "Chloratsprengstoffe," Veit & Co, Leipzig(1910) 6)R.Escales, "Schwarzpulver und Sprengsalpeter," Veit & Co, Leipzig(1914) 7)R.Escales, "Nitrosprengstoffe," Veit & Co, Leipzig(1915) 8)R.Escales & A.Stettbacher, "Initialexplosivstoffe," Veit & Co, Leipzig (1917) 9)E.M.Weaver, "Notes on Military Explosives," Wiley,NY(1917) 10)A.Marshall, "Explosives," Churchill, London, v 1(1917), v 2(1917), v 3(1932) 11)E.deW.S.Colver, "High Explosives," VanNostrand,NY(1918) 12) E.deB.Barnett, "Explosives," VanNostrand, NY(1919) 13)H.Kast, "Spreng- und Zündstoffe," Vieweg, Braunschweig(1921) 14)H.Brunswig, "Das rauchlose Pulver," W.deGruyter, Berlin (1926) 15)Ph.Naoúm, "Schiess- und Sprengstoffe," Steinkopf, Dresden(1927) 16)Ph. Naoúm, "Nitroglycerine and Nitroglycerine Explosives," Williams & Wilkins, Baltimore(1928) 17)R.Molina, "Esplodenti e Modo de Fabricarli," Hoepli, Milano(1930) 18)P.Pascal, "Explosifs, Poudres, Gaz de Combat," Hermann, Paris(1930) 19)H.Vennin, E.Burlot et Lécorché, "Les Poudres et Explosifs," Ch. Béranger, Paris(1932) 20)A.Stettbacher, "Die Schiess- und Sprengstoffe," Barth,Leipzig(1933) 21) J.Pepin-Lehalleur, "Traité des Poudres, Explosifs et Artifices," Baillière, Paris(1935) 22)C.Beyling & K.Drekopf, "Sprengstoffe und Zündmittel," Springer, Berlin(1936) 23)J.Reilly, "Explosives, Matches and Fireworks," VanNostrand, NY(1938) 24)E.E.Sancho, "Química de los Explosivos," A.Aguado, Madrid (1941) 25)J.Bebie, "Manual of Explosives, Pyrotechnics and Chemical Warfare Agents," Wiley,NY(1943) 26)T.L.Davis, "The Chemistry of Powder and Explosives, "Wiley,NY(1943) 27)M.Meyer, "Explosives," Crowell,NY(1943) 28)H.Kast & L.Metz, "Chemische Untersuchung der Spreng- und Zündstoffe," Vieweg, Braunschweig(1944) 29)H.Blatt et al, "Compilation of Data on Organic Explosives," OSRD Report 2014(1944) 30)M.Vivas, R.Feigenspan & F.Ladreda, "Pólvoras y Explosivos Modernos," J.Morata, Madrid, Vols 1-5(1944-1948) 31) A.Perez Ara, "Tratado de Explosivos," Editorial Cultural, LaHabana(1945) 32)P.P.Karpov, "Initiating Agents," GosizdatOboronprom, Moscow(1945)(in Rus) 33)A.D.Shilling, "Explosives and Loading of Ammunition," Oborongiz, Moscow(1946) (in Rus) 34)H.Muraour, "Poudres et Explosifs," Presses Universitaires, Paris (1947) 35)A.Mangini, "Quaderni di Chimica Industriale No 14, Esplosivi," Pàtron, Bologna (1947) 36)A.Stettbacher, "Spreng- und Schiesstoffe," Rascher, Zürich(1948) 37)C.Belgrano, "Gli Esplosivi," Hoepli, Milano(1952) 38)A. Stettbacher, "Pólvoras y Explosivos," G.Gili, Buenos,Aires(1952) 39)E.I.duPont de Nemours & Co, "Blaster's Handbook," Wilmington(1952) 40)Anon, "Military Explosives," Dept of the Army TM 9-1910, Washington,DC(1955) 41)M.A. Budnikov et al, "Explosives and Propellants," Oborongiz, Moscow(1955)(in Rus) 42)A.G. Gorst, "Propellants and Explosives" Gosizdat-Oboronprom,Moscow(1957) (in Rus) 43)W.R. Tomlinson, Jr & O.E.Sheffield, "Properties of Explosives of Military Interest", Picatinny Arsenal Technical Rept No 1740, Revision 1 (1958) 44)M.A.Cook, "The Science of High Explosives," Reinhold,NY(1958) 45)G.Taylor & P.F.Gay, "British Coal Mining Explosives," G.Newnes, London(1958) 46)J.Taylor, "Solid Propellent and Exothermic Compositions," Interscience,NY(1959)

Selected List of Journals on Explosives and Related Items

1)Explosifs, Bruxelles,Belgium 2)Explosives Engineer, Wilmington,Del,USA 3)Explosivstoffe, Manheim,Germany 4)Mémorial de l'Artillerie Française,Paris,France 5)Mémorial des Poudres, Paris,France

INDEX

An alphabetical listing of items discussed in this volume, which may not necessarily begin with letter A, and which may represent alternate names of items or compounds already listed alphabetically in the text

A

Abbreviations, Code Names, etc Abbr 1 to 65
Abbreviations for Books, Periodicals, etc Abbr 66 to 76
Acetazidine or Azidine A24-R & A627-R
Acrylic Aldehyde or Acrolein A96-L
Note: It was used during WWI by the French under the name "Papite" as a CWA
Active Oxygen A101-L & A515-L
Actual Nitric as Nitric Acid .A89-L & A90-L
Actual Sulfuric as Nitric Acid A90-R
Actual Sulfuric as Sulfuric Acid A90-L
Acyl and Aryl Derivatives of Azidodithiocarbonic Acid A632-R
Aerojet Propellants A108-L
Aerojet Propellants, See under Ammonium Nitrate Blasting Explosives, High Explosives and Propellants A350(table)
Aitch-Tu-Ess. See under Asbestos A494-L
Akardit. See Acardite A7-R
Aldehyde. See Acetaldehyde A14-L
Aldolcondensation Product of 5-Aminotetrazole A260-R
Alkali and Alkaline Earth Salts of Azidodithiocarbonic Acid A633-R
Alkali Amides. See under Amides A168-R
Allylazidodithiocarbonate A632-L
Aluminum Acetylide A70-L
Aluminum Azide A521-L
Aluminum Carbide. See under Acetylides and Carbides A70-L
Aluminum Triazide A521-L
American Ammonium Nitrate Dynamites A355 (table)
American Ammonium Nitrate Gelatin Dynamites A368 (table)
Ammonalmatrit. See under Almatrites A140-L
Ammon- Gelatine Dynamite and Ammon-Gelignite (British Not-Permitted (Ammonium Nitrate Dynamites) A368 (table)
Ammonium Acetate A27-R
Ammonium-Aluminum Alum A156-L
Ammonium Azide A521-L
Ammonium Azide Ammonates A521-R
Ammonium Azidodithiocarbonate A634-L
Ammonium Chrome Alum A156-L
Ammonium-Iron Alum A156-R
Ammonium Nitrate A311 to A340
Introduction A311
Historical A312
Laboratory Preparation and Manufacture A313 & A340
Explosive and Other Properties A318
Uses A334
Analytical Procedures, General A369
Analytical Procedures, Spencer Chemical Co A379
Ammonium Nitrate Blasting Explosives, High Explosives and Propellants A341 to A354
Ammonium Nitrate Dynamite (AND)(American and European Types) A355 to A356
Ammonium Nitrate Explosions, Fires and Hazards A357 to A363
Ammonium Nitrate Explosives of Spencer Chemical Co A354 (table)
Ammonium Nitrate Explosives, Tests of Spencer Chemical Co [a)Cook-off temperature b)Detonation velocity c)Wax-gap and d)Impact-friction pendulum] A354 (notes)
Ammonium Nitrate Fertilizer Grade (FGAN) A364 to A367
Ammonium Nitrate Gelatin (ANG) A367 to A368
Ammonium Nitrate, US Military Specification Requirements and Tests A370 to A378
Analytical Procedures for:
Acardites A9-L

Acetal A13-L
Acetaldehyde A15-L
Acetic Acid A25-R
Acetic Anhydride A30-L
Acetins A32-L
Acetone A35-R
Acetylides A63-R
Aluminum A143-R
Amatol A164-L
Ammonal A292-R
Ammonia A303-L
Ammonium Nitrate A369-L
Aniline and Derivatives A415
Anisole and Derivatives A454
Lead Azide A563 to A576
Lead Azide Explosives A580 to A587
Sodium Azide A612-R to A619
Aniline, Azido Derivatives A629-L
Aniline, Azo Derivatives A646-L
Aniline, Azoxy Derivatives A665-L
Anisole, Azido Derivatives A629-R
Anisole, Azo-Derivatives A646-L
Anisole, Azoxy-Derivatives A665-R
Antigrisou (Explosifs) A466-R
Antimony, Metal A467-R
Antimony, Analytical Procedures A469 to A470
Antimony Triazide A522-R
Arsenic Azide A522-R
Arsenic Triazide A522-R
Arsenium Carbide. See under Acetylides and Carbides A70-R
Aryl and Acyl Derivatives of Azidodithiocarbonic Acid A632-R
Ascarite. See under Asbestos A494-L
Asphaltines. See under Asphalt A496-R
Auric Imidoamide A169-L
Aurous Azide or Gold Azide A536-L
Austrian Ammonals A289 & A290 (table)
Azacyclobutadiene. See Azete A518-R
Azeotropic Distillation Method for Moisture Determination A370-R to A371-L
Azide-Styphnate-Aluminum. See ASA A493-R
Azidine or Acetazidine A24-R & A627-R
Azodicarbonhydrazide A271-L
1,1'-Azo-5,5'-di(p-tolyl)-tetrazole A266-R

B

Ballistic Cap or Windshield A483-L
"Banana Oil." See Amyl Acetate A393-L
BARC. See under Amphibious Vehicles A393-L
Barium Acetylide A70-R
Barium Diazide A523-L
Barium Nitroaminoguanidine A212-L
Barium Picramate A242-L
Belgian Ammonium Nitrate Gelatin Dynamites A368 (table)
Benzalaminoguanidinium-1,6-di-nitro-2-(aminoguanyl)-biguanidine Benzalhydrazone A215-L
4-Benzeneazodiphenylamine or 4-Anilinoazobenzene A420-R
Benzeneazotrinitromethane and Derivatives A67-R
Benzodiazole, Benzopyrazole or Aminoindazole A224-L
Benzohydryl Azidodithiocarbonate A633-L
1,2,3-Benzothiazino[3,4-a] perimidine. See under Aminophenylperimidines A246-L
Benzoylacetylperoxide or Acetylbenzoylperoxide A54-R
Benzoyl Azidodithiocarbonate A633-L
Benzoylazidomethane or ω-Azidoacetophenone under Acetophenone A47-R
Benzozone or Acetylbenzoylperoxide A54-R
Benzyl Abietate. See under Abietic Acid A3-R
Benzylacetylperoxide or Acetylbenzylperoxide A55-L
1-Benzyl-5-amino-vic-tetrazole. See 5-Amino 1-benzyl-vic-tetrazole A191-L
Benzyl Azidodithiocarbonate A633-L
Benzylperacetate or Acetylbenzylperoxide A55-L
Beryllium Acetylide A70-R
Beryllium Azide A524-R
Beryllium Carbide. See under Acetylides and Carbides A71-L
Beryllium Diazide A524-R
Binding Energy. See under Atomic Energy A500-R
Biphenylamines. See under Aminobiphenyls A191-L

Biphenyldiazonium Perchlorate. See under Aminobiphenyls A191-L
p,p'-Biphenylenebisazotrinitromethane A67-R
Bis-(aminoguanidinium)-1,6-dinitro-2-(aminoguanyl)-biguanidine A214-R
Bis-(benzalaminoguanidinium)-1,6-dinitrobiguanidine A215-L
Bis(carboxamide)-acetylene. See Acetylenedicarboxamide A65-L
4,4'-Bis-dimethylamino-benzophenone. See Auramine A507-R
Bis(1,1-dimethyl-2-propynyl)-peroxide. See under Acetylene Hydroperoxides A66-R
Bis(hydroxylamino) Azide A525-L
Bis(hydroxymethyl)methylaminomethane. See Aminomethylpropanediol A232-R
4-[Bis(p-hydroxyphenyl)methylene]-2,5-cyclohexadien-1-one. See Aurine A508-R
Bis(1-methyl-1-ethyl-2-propynyl)-peroxide. See under Acetylene Hydroperoxides A66-R
Bis(3-methyl-2,4,6-trinitrophenyl)-amine. See 2,4,6,2',4',6'-Hexanitro-3,3'-dimethyldiphenylamine A443-R
Bismuth Azidodithiocarbonate A636-R
Bismuth Triazide A525-L
1,2-Bis(2-nitramino-2-imidazolin-1-yl)-ethane A220-R
Bis-1-(2-nitroamino-2-imidazolinyl)-ethane A220-R
1,2-Bis(2-nitrimino-3-nitro-1-imidazolidyl)-ethane A220-R
Bis-1-(3-nitro-2-imidazolidonyl)ethane A221-L
1,2-Bis(3-nitro-2-oxo-1-imidazolidyl)ethane A221-L
N^1,N^6-Bis[α-tetrazolyl-5]-hexazidiene A260-R
Bistriazomesidine. See 2-Amino-4,6-diazidomesitylene A224-R
Bitumen. See under Asphalt A496-R
Blasting Gelatin A147 (table)
Boron Azide A525-L
Boron Carbide. See under Acetylides and Carbides A71-L
Boron Triazide A525-L
British Ammonals. See under Ammonals A289, A290 & A291-R
British Ammonium Nitrate Gelatin Dynamites A368 (table)
Bromine Azide A525-R
Bromine Azidodithiocarbonate A635-R
p-Bromobenzoyl Azidodithiocarbonate A633-L
1,4-Butanedicarboxylic Acid. See Adipic Acid A104-L
Butanolaniline. See Anilinobutanol A422-R
Butylcarbinol. See Amyl Alcohol A394-R

C

Cadmium Acetylide A71-L
Cadmium Amide A169-L
Cadmium Azide A526-L
Cadmium Azidodithiocarbonate A636-R
Cadmium Diazide A526-L
Calcium Acetate A28-L
Calcium Acetylide A71-L
Calcium Azide A527-R
Calcium Carbide. See under Acetylides and Carbides A71-R
Calcium Carbide-Ammonia-Acetylene A72-L
Calcium Diazide A527-R
Calcium Diazide Dihydrazinate A528-L
Calcium Hydrogen Acetylide A72-L
Calibers(Millimeters vs Inches) A675 (Table II)
Calibers of US Ammunition and Weapons A386 to A387
Caliver. See under Arquebus A488-L
Carbenes. See under Asphalt A496-R
Carbides. See Acetylides and Carbides A69-R
Carbonyl Diazide A528-L
Cellulose Triacetate. See under Acetyl Celluloses A55-R
Cerium Azide A528-R
Cerium Hydroxydiazide A528-R
Cerium Triazide A528-R
Cesium Azide A528-R
Cesium Carbide A72-L
Chain Reaction. See under Atomic Energy A501-L
Chishokianin. See 2,3,4,6-Tetranitroaniline A411-L
Chlorine Azide or Chloroazide A529-L
Chlorine Azidodithiocarbonate A635-R

Chloroazidine. See Azobis-(chloroformamidine) A652-R
Chloronitroanilinopropanols A436-R
Chromium Azide A530-L
Chromium Azide Complexes A530-R
Chromium Carbide. See under Acetylides and Carbides A72-R
Chromylacetylacetone. See under Acetylacetone A53-R
Clean Bomb (Hydrogen Bomb). See under Atomic Bomb A499-R
Cluster, Aimable. See Aimable Cluster A114-L
Cobalt Azide A531-L
Cobalt Azide Complexes A531-R
Cobaltous Azetylide A72-R
Cobalt Triazide A531-L
Cold-Working. See Autofrettage A510-R
Complex of Trinitroanisole A453-R
Compound $C_6H_6N_3O_6P$, called in Ger "Salpetersäure-diazophosphenylsäure A246-R
"Cook-Off" Temperature Test, as conducted at the Spencer Chemical Co, Kansas City, Mo A354 (Note a)
Copper Acetylides, Analytical Procedures A74 to A76
Copper Azidodithiocarbonate A636-R
Copper Nitroaminoguanidine A212-R
Copper Picramate A242-L
Corallin. See Aurine A508-R
Critical Mass. See under Atomic Energy A501-L
Crossbow. See Arbalest A477-L
CSE Commission des Substances Explosives (Explosif) (1902). See under Aluminum-Containing Explosives A146-L
Cupric Acetylide A74-L
Cupric Amminoazide and Complexes A533-L
Cupric Azide A532-L
Cupric Azide, Basic A533-L
Cupric Azide Complexes A533-R
Cuprous Acetaldehyde Catalysts A74-L
Cuprous Acetylide A72-R
Cuprous Acetylide-Chloride A74-L
Cuprous Azide A534-L
Cuprous Hydrogen Acetylide A74-L
Curocellulose. See under Adipocellullose A104-A105
Cyanodiphenylamine. See Anilinobenzonitrile A422-L
Cyanogen Azidodithiocarbonate A635-R
Cyanomethane. See Acetonitrile A45-L
Cyclotrimethyleneimine. See Azetidine A519-L

D

"Davy Crocket" (Atomic Rifle) A504-R
DD Device. See under Amphibious Devices for Tanks A392-R
Destruction of Amatol A162-L
Destruction (Disposal) of Lead Azide A574 & A575
Detonation, Advance. See Advance Detonation A105-R
Diacetin. See under Acetins A31-R
Diacetone Diperoxide. See under Acetone Peroxides A41-R
Diacetophenone Diperoxide. See Acetophenoneperoxide, Dimeric A48-R
1,9-Diacetoxy-2-acetyl-4,6,8-trinitro-2,4,6,8-tetrazanone or H-16. See under Acetyldiacetoxytetrazanonane A57-R
1,2-Diacetylethane. See Acetonylacetone A46-R
Diacetylmethane. See Acetylacetone A53-L
Dialkyltetrazolylureas. See under Alkyltetrazolylureas A132-R
Diaminoazoxybenzene. See Azoxydianiline A665-L
Diaminohydroxytriazine Picrate. See Ammeline Picrate A274-R
4,6-Diamino-s-triazin-2-ol. See Ammeline A273-R
Diammonium-5-nitraminotetrazole A260-L
Diazoacetylacetoneanhydride. See 4-Acetyl-5-methyl-1,2,3-oxydiazole A84-L
Diazotetrazolephenylhydrazine. See N'-Amino-N'-phenyl-N^3-(tetrazolyl-5)-triazine A247-R
Dibenzopyridine. See Acridine A94-L

1,1-(N-Dichloramino)-5-(p-tolyl)-α-tetrazole A266-R
N,N'-Dichloroazodicarboxamidine. See α,α'-Azobis-(chloroformamidine) A652-R
Dicuproacetaldehyde A73-L
Diethoxyazobenzene. See Azophenetole A656-R
Diethoxyazoxybenzene. See Azoxyphenetole A670-R
1,1-Diethoxyethane. See Acetal A13-L
3-(β-Diethylaminoethyl)-α-sym-triazole Dipicrate A209-L
1,1-Diethyl-2-propynylhydroperoxide. See under Acetylene Hydroperoxides A66-L
7,8-Dihydroacenaphthylene. See Acenaphthene A12-L
Dihydro-diketo-anthracene. See Anthraquinone A459-R
5,6-Dihydro-6-imino-s-triazine-2,4(1H,3H)-dione. See Ammelide A273-L
Dihydroxyazobenzene. See Azophenol A657-L
1,1'-Dihydroperoxy-1,1'-dicyclohexylacetylene. See under Acetylene Hydroperoxides A66-R
Dihydroxyazobenzene. See Azophenol A657-L
Dihydroxyazoxyphenol. See Azoxyphenol A671-L
4,4'-Dihydroxyfuchsone. See Aurine A508-R
Dihydroxypropylamine. See Aminopropanediol A251-L
2,5-Diketohexane. See Acetonylacetone A46-R
Dimeric Acetone Peroxide A41-R
Dimeric Acetophenone Peroxide A48-R
Dimethoxyazobenzene. See Azoanisole A646-L
Dimethoxyazoxybenzene. See Azoxyanisole A665-R
Dimethylaniline. See Aminoxylene A272
Dimethylazobenzene. See Azotoluene A660-L
Dimethylazoxybenzene. See Azoxytoluene A672-L
Dimethylazoethane. See Azopropane A658-R
Dimethylazoxyethane. See Azoxypropane A671-R
2,5-Dimethyl-2,5-di-(t-butylperoxy)-3-hexyne. See under Acetylene Hydroperoxides A66-R
2,5-Dimethyl-2,5-dihydroperoxy-3-hexyne. See under Acetylene Hydroperoxides A66-R
Dimethyldiphenylamine. See under Anilinoxylene A443-L
Dimethylenemethane. See Allene under Allenic Compounds A133-R
Dimethylketone. See Acetone A33-R
Di-(3-methylpentynyl)-3-peroxide. See under Acetylene Hydroperoxides A66-R
2,3-Dimethyl-1-phenyl-3-pyrazolin-5-one. See Antipyrine A471-R
1,1-Dimethyl-2-propynylhydroxide. See under Acetylene Hydroperoxides A66-L
Diphenylaminocarboxylic Acid. See Anilinobenzoic Acid A421-R
Diphenylamino-4-diazonium Hydroxide. See p-Anilinobenzenediazonium Hydroxide A421-L
Diphenylcarbamylallylamine. See N-Allyl-N',N'-diphenylurea A137-R
Diphenyldiimide. See Azobenzene A646-R
Diphenylmethylamine. See under Anilinotoluene A438-L
N,N-Diphenylurea. See Acardite I A7-R
Dipotassium Hydrazobenzene-Azobenzene A647-L
Dipotassium Salt of Nitroacetic Acid A27-R
Disol. See 2,4-Dinitroanisole A448-L
Distyrylazobenzene. See Azostilbene A659-R
N^1,N^6-(Ditetrazolyl-5)-hexazadiene A260-R
Dithiocarbonic or Dithioformic Acid, Azido Derivatives A632
Ditolylamine. See Anilinoxylene A443-L
DUKW. See under Amphibious Vehicles A393-L
Dynamite O (French Ammonium Nitrate, Non-Permitted Gelatin) A368 (table)

E

Effective Oxygen. See under Available Oxygen A515-R

Elvanol. See under Aminoethylpolyvinyl Alcohol A205-R
Energies, Activation. See Activation Energies A100-R & A101-L
Enheptin. See 2-Amino-5-nitrothiazole A263-R
Erosive Action of ACT 5 Propellant A98-L
Erythric Acid of Brugnatelli. See Alloxan A134-R
Erythritetetranitrate A147 (table)
Ethanal. See Acetaldehyde A14-L
Ethanaloxime. See Acetaldoxime A16-L
Ethanamide. See Acetamide A16-R
Ethaneamidine or Ethenylamidine. See α-Amino-α-imidoethane A223-L
Ethanoic Acid. See Acetic Acid A25-L
Ethanoic Anhydride. See Acetic Anhydride A29-R
Ethanolamine. See Aminoethanol A200-L
β-Ethanolaniline. See under Aminophenylethanol and under Anilinoethanol A245-R & A424-L
Ethanoyl Bromide. See Acetyl Bromide A55-R
Ethanoyl Chloride. See Acetyl Chloride A56-R
Ethine. See Acetylene A58-R
Ethoxyacetanilide. See Acetamidophenetole A20-L
Ethoxyaminobenzene. See Aminophenetole A240-R
Ethoxyaniline. See Aminophenetole A240-R
Ethyl Abietate. See under Abietic Acid and Derivatives A3-R & A4-L
Ethylacardite. See Acardite III A8-R
Ethylaldehyde. See Acetaldehyde A14-L
Ethylamine. See Aminoethane A199-R
3-(β-Ethylaminoethyl-α-sym-triazole) Dipicrate A208-R
Ethylaminotetrazoles. See under Aminoethyltetrazoles A206-R
5-Ethylamino-α-tetrazole A207-L
Ethylaminotriazoles. See under Aminoethyltriazoles A207-R
Ethylazaurolic Acid. See under Azaurolic Acids A517-R
Ethylene Aldehyde. See Acrolein A96-L
Ethylenecarboxylic Acid. See Acrylic Acid A96-R
N,N'-Ethyleneguanidine. See under Aminoimidazolines A219-L
Ethylenenaphthalene. See Acenaphthene A12-L
Ethylidene Diethyl Ether. See Acetal A13-L
Ethylidene Oxide. See Acetaldehyde A14-L
Ethylidene-(2,4,6-trinitrophenylhydrazine). See Acetaldehydepicrylhydrazone A15-L
1-Ethyl-2-nitramino-Δ^2-imidazoline A220-R
1-Ethyl-2-nitrimino-3-nitroimidazolidine A221-L
3-Ethyl-1,2,4-triazole-4-diazonium Hydroxide A208-R
Ethyne. See Acetylene A58-R
Eulite and its mercuric salt A67-R
European Ammonium Nitrate Dynamites. See under Ammonium Nitrate Dynamites A356 (tables)
Explosif amylacé. See Amide (Explosif) A168-L
Explosifs antigrisouteux A467-L
Explosion and Ignition of Anesthetic Agents A402-L
Explosives Containg Aluminum A147 to A151
Explosophores. See under Auxoexplose A513-R

F

Fallout. See under Atomic Energy A502-L
False Ogive or Ballistic Cap A483-L
Felixdorf Factory Ammionals (Austrian) A289 (table)
Ferric Azide A543-L
Ferric Azide, Basic A543-R
Ferric Triazide. See Iron Azide A543-L
Ferrous Acetylide. See Iron Acetylide A76-R
Ferrous Azide A543-L
Fission Bomb, See under Atomic Bomb A499-L
Fission Reaction A501-L
Fluorine Azide A536-L
Forcites (Belgian and French) A368 (table)
Formula 226(Explosif). See under Aluminum-Containing Explosives A146-L

French Ammonals. See under Ammonals A290 (table)
French Ammonium Nitrate Gelatin Dynamites A368 (table)
Fulminating Gold. See Auric Imidoamide A169-L
Fulminating Silver of Berthollet. See Silver Amide under Amides A169-R
3-Furazanacetic-4-carboxylic Acid A67-R
Furylacetamide. See Acetamidofuran A19-L
Fusion or Hydrogen Bomb. See under Atomic Bomb A499-L
Fusion Reaction. See under Atomic Energy A501-L

G

Galcit. See under Asphalt-Perchlorate Castable Propellants A497-L
Gallium Triazide A536-L
Gas Explosions, Action on Solid Propellants of A98-L
Gasometric Method for Determination of Ammonium Nitrate Content by Means of Nitrometer A373 to A377
Gelatin. See under American Dynamites, Gelatinized A167-L
Gel-Coalites. See under American Dynamites, Gelatinized A167-L
German Ammonals. See under Ammonals A289 to A291
Gessner Projectile. See under Arrow Projectile A488-R
Glycerol-β-monoamine. See 2-Amino 1,3-propanediol A251-L
Glyceryl Diacetate. See Diacetin under Acetins A31-R
Glyceryl Monoacetate. See Monacetin under Acetins A31-R
Glyceryl Triacetate. See Triacetin under Acetins A31-R
Glycine or Glycocoll. See Aminoacetic Acid A178-L
Glycoluril. See Acetylenediurein A65-L
Gold Acetylide A76-R
Gold Amidoimide. See Auric Imidoamide A169-L
Gold Azide A536-L
Gold Azidodithiocarbonate A637-L
Gold Carbide A76-R
Goudronite, Ammonite. See under Ammonite A310-L
Grisonite or Mineral Rubber. See under Asphalt A496-R
Grisou-dynamite roche (French Non-Permissible Gelatin) A368 (table)
Grisounite couche and Grisounite roche A466-R (table)
Guanazine. See 4-Aminoguanazole A209-R
Guanidine Azidodithiocarbonate A636-L
Guanidinium-5-nitraminotetrazole A260-L
Guanidinoethylaminoimidazoline, Nitrated Derivatives. See under Aminoimidazolin-1-yl-ethylguanidine A222
Guanylaminotetrazole. See under 5-Amino-tetrazole A260-R
Guanylhydrazine. See Aminoguanidine A210-L

H

"H_2" Symbol for N-Acetylamidomethyl-hexamethylenetetraminemononitrate A54-L
H-16. Symbol for 2-Acetyl-1,9-diacetoxy-4,6,8-trinitro-2,4,6,8-tetrazanonane A57-R
Halogen Derivatives of Anilinoethanol A430 to A431
Harquebus. See Arquebus A488-L
H Bomb (Hydrogen Bomb). See under Atomic Bomb A499-L
Heavy Metal-Salts of Azidodithiocarbonic Acid A636-R
Heptryl. See N-(2,4,6-Trinitro-N-nitranilino) -trimethylolmethane Trinitrate under Anilinotrimethylolmethane and Derivatives A441-R to A442-R
Heptylamines. See Aminoheptanes A215-R
Hexahydro-2,4,6-triimino-s-triazine. See 1-Aminohexahydro-2,4,6-triimino-sym-triazine A216-L

2,5-Hexanedione. See Acetonylacetone A46-R
Hexanedionic Acid. See Adipic Acid A104-L
5-Hexen-2-one. See Allylacetone A135-R
Hexylamines. See Aminohexanes A215-R
High Pan Fires (in manuf of Ammonium Nitrate) A318-L
Homologs of Pentryl A429-R
Hybrid. See under Auxoexplose A514-R
Hydrazine Azide A536-R
Hydrazine Azide Hemiammonate A537-L
Hydrazine Azide Monohydrazinate A537-L
Hydrazoic Acid. See under Hydrogen Azide A539-L
Hydrogen Arsenide. See Arsine A491-L
Hydrogen Azide A537-R
Hydrogen Azide, Anhydrous A538-L
Hydrogen Azide, Aqueous or Hydrazoic Acid A539-L
Hydroperoxides and Peroxides of Acetylene Derivatives A66
Hydroxyamines. See Aminoalcohols A179-L
2-Hydroxy-2-aminoimidazolidine. See 2-Amino-2-imidazolinol A222-L
Hydroxyaminomethylpropane. See Aminomethylpropanol A233-L
Hydroxyaminopropane. See Aminopropanol A253-L
β-Hydroxybutyraldehyde. See Acetaldol (Aldol) A15-R
Hydroxydiphenylamine. See Anilinophenol A433-R
β-Hydroxyethylamine. See Aminoethanol A200-L
β-Hydroxyethylaminobenzene or β-Hydroxyethylaniline. See under Anilinoethanol A424-L
1-(2-Hydroxyethyl)-2-nitramino-Δ^2-imidazoline A220-R
N-(β-Hydroxyethyl)-N'-phenyl-1,2-diaminoethane. See Anilinoethylaminoethanol A431-L
1-Hydroxy-2-propanone. See Acetol A33-R
2-Hydroxy-3,4,6-trinitroacetanilide. See under Acetamidophenol A21-R
Hypnone. See Acetophenone A47-L

I

Ignition and Explosion of Anesthetic Agents A402-L
Iminodihydropurine. See Aminopurine A254-L
Iminodihydrotriazine. See Aminotriazine A267-L
2-Imino-1,3,4-thiadiazoline. See under Aminothiadiazole A262-R
Impact-Friction Pendulum Test as conducted at the Spencer Chemical Co, Kansas City, Mo A354 (Note d)
Inorganic Amides and Imides A168 to A170
Inorganic Azides A520 to A625
Introduction (to this volume) I-II
Iodine Azide or Iodoazide A542-R
Iron Acetylide A76-R
Iron Azide A543-L
Iron Carbide. See under Acetylides and Carbides A76-R
Iso-Amylpicrate A399-R
Iso-Amylureidoacetyl Azide A399-R
2-Isocyanate Benzoylazide. See 2-Azidoformylphenylisocyanate A638-R
Iso-Me-NENA. See Nitraminopropanol Nitrate under Aminopropanols A253-L
Isoöctoic Acids, Aluminum Soaps A155-R
Isopicramic Acid. See 2,6-Dinitro-4-aminophenol under Aminophenol A243-R
Isoxazole, Amino-Diazo-and Nitro-Derivatives A67
α-Isoxazoleazotrinitromethane A67-R
Isoxazolecarboxylic Acids A67
4-(3-Isoxalyl)-3-furazancarboxylic Acid, Silver Salt A67-R
Italian Ammonals A291R
Italian Military Aluminized Plactic Explosive A148-L

J

Japanese Explosives:
Ammonyaku A383-L
Angayaku A402-R
Chishokianin (2,3,4,6-Tetranitroaniline) A411-L

Type 91 (2,4,6-Trinitroanisole) A450-L
Type A or A(ko) Explosive A119-L
JATO. See under ATO A497-R

K

Kaliialmatrit. See under Almatrites A140-L
2-Ketotrimethyleneimine. See 2-Azetidinone A519-L
KI-Starch Test. See Abel's Test A2-L
Kreulen, Aluminum Block of A145-L

L

Lanthanum Triazide A544-R
Lead Acetates. See under Acetates A28 to A29
Lead Aceto-Bromate A29-L
Lead Aceto-Chlorate A29-L
Lead Aceto-Perchlorate A29-L
Lead Aceto-Sodium Perchlorate A29-L
Lead Acetylide A76-R
Lead Azide (Lead Diazide) (LA) A545 to A556
General Properties A545 to A550
Laboratory Preparation A546
Manufacture of Dextrinated Lead Azide A547
Explosive Properties A548
Destruction (Disposal or Killing) of Lead Azide A550
Uses of Lead Azide A551
Lead (IV) Azide A556-L
Lead Azide Basic A555-R
Lead Azide Explosive, Primer and Detonator Compositions A576 to A580
Analytical Prodedures:
Analysis of an Unknown Sample A580-R
Analysis of mixtures: LA, Sb_2S_3, $Pb(SCN)_2$ & $KClO_3$ A580-R to A584-R
Analysis of Mixtures: LA, Sb_2S_3, $KClO_3$, glass & shellac A585-R
Analysis of Mixtures: LA, $Ba(NO_3)_2$, basic LSt & Sb_2S_3 A586-L
Analysis of Mixtures: LA & Al A587-R
Lead Azide, Plant Analytical Procedures A563 to A576
Determination of Lead Nitrate A563
Detn and Tests for Dextrin A564
Analysis of Lead Nitrate Dilution Tank A565
Analysis of Sodium Azide Liquor A565
Analysis of SA Feek Tank A567
Analysis of SA Dilution Tank A567
Detn of Lead Azide by the US Military Specification Method A567 to A570
Detn of LA by the US Navy Method A570-L
Detn of LA by the British Method A570-R
Detn of Total Lead Content in LA A571-R
Detn of Acidity in LA A571-R
Detn of Solubility of LA in H_2O A572-L
Detn of Sand Test Value for LA A572-L
Detn of Moisture in LA A572-3
Detn of Ball Drop Test Value for LA A573-L
Analysis of Ethyl Alcohol Solution A573-R
Analysis of "Killing Tank" Liquid A573-R
Analysis of Nitric Acid Used for "Killing" LA A574-L
Disposal of Laboratory Samples Containing LA (by Various Methods) A574-R to A575
Laboratory Test for the Presence of LA A575-R
Lead Azide, Various Military Types A557 to A563
Dextrinated LA, Type I(US) A557 & A559
British(Service) LA A557 & A559
Colloidal LA, Type II(US) A558 & A559
PVA (Polyvinylalcohol) LA A558 & A559
Dextrinated Colloidal LA A558 & A559
RD-1333 LA A558 & A559
RD-1343 LA A558 & A559
RD-1352 LA A559 & A560
Lead Azidodithiocarbonate A637-L
Lead Imide A169-L
Lead Nitroaminoguanidine A212-R
Lead Picramate A242-R
LeRoux (Explosif) A148-L
Liardet Powder. See Acme Powder A93-R
Liquid Air(or Liquid Oxygen)-Aluminum Explosives A154-L

List of Abbreviations, Code Names, Symbols, etc Abbr 1 to Abbr 65
List of Abbreviations for Books, Periodicals, etc Abbr 66 to Abbr 76
Lithium Acetylides and Lithium Carbides A77
Lithium Aluminohydride. See Aluminum-Lithium Hydride A154-R
Lithium Azide A588

M

Magnesium Acetylide A77-R
Magnesium Arsenide A491-L
Magnesium Carbide A77-R
Magnesium Diazide A589-R
Magnesium-Methanol Explosives A155-L
Manganese Acetylide A78-L
Manganese Carbide A78-L
Manganese Diazide A589-R
Mercuric Azide A590-L
Mercuric Azidodithiocarbonate A637-L
Mercurous Azide A591-L
Mercury Acetylides A78
Mercurous Azidodithiocarbonate A637-L
Mesidine. See Aminomesitylene A224-R
N,N'-Mesoxalyl Urea. See Alloxan A134-R
Meta. See Metaldehyde under Acetaldehyde A14-R
Metalammonium. See Ammonium Metal A310-R
Metaldehyde. See under Acetaldehyde A14-R
Methanol-Aluminum (or Magnesium) Explosives A155-L
Methazonic Acid. See Mononitroacetaldoxime A16-L
Methoxyacetanilide. See Acetamidoanisole A17-L
Methoxyaminobenzenes. See Aminoanisoles A182-L
Methoxybenzaldehyde. See Anisaldehyde A444-R
Methoxybenzaldehyde-phenylhydrazone. See Anisaldehydephenylhydrazone A445-L
Methoxybenzene. See Anisole A448
Methoxybenzoic Acid. See Anisic Acid A446-R
Methoxybenzoylazide. See Anisoylazide A456-L
Methoxybenzyl Alcohol. See Anisyl Alcohol A456-R
Methoxyphenylaminotetrazoles. See under Aminomethoxyphenyltetrazoles A228-L
Methoxyphenyltetrazole. See Anisyltetrazole A457-L
Methyl Abietate. See under Abietic Acid A4-L
Methylacardite. See Acardite II A8-L
Methylacetanilide. See Acetamidotoluene A22-L
Methylacetyl Ether. See Acetone A33-R
Methylamine. See Aminomethane A225-R
Methylaminoguanidines. See under Aminomethylguanidines A231-L
Methylaminonitroform. See Aminomethane Nitroform A227-R
Methylaminophenols. See Aminocresols A193-R
Methylaminotetrazole. See Aminomethyltetrazole A233-R
Methylaminothiazole. See Aminomethylthiazole A234-R
Methylaminotriazole. See Aminomethyltriazole A235-R
Methylaminotriazole. See Aminomethyltriazole A235-L
Methylaniline. See under Aminotoluenes A264-R
Methylazaurolic Acid. See under Azaurolic Acids A517-R
Methyl Azidodithiocarbonate A633-L
Methylbiphenylamine. See Aminomethylbiphenyl A229-L
Methyl Cyanide. See Acetonitrile A45-L
Methyldiphenylamine. See under Anilinotoluene A438-L
N-Methyldipicrylamine. See 2,4,6,2',4',6'-Hexanitro-N-methyl-diphenylamine A440-R
3-Methyl-3-hydroperoxy-1-butyne. See under Acetylene Hydroperoxides A66-L
3-Methyl-3-hydroperoxy-1-pentyne. See under Acetylene Hydroperoxide A66-L
α-Methylisoxazolecarboxylic Acid. See under Acetylene-Nitric Acid Reaction Studies A67-L

1-Methyl-2-nitramino-Δ^2-imidazoline A220-R
1-Methyl-2-nitrimino-3-nitroimidazolidine A221-L
Methylnonylthiuronium Picrate A69-L
Methylphenylamines. See under Aminotoluenes A265
Methylphenylether. See Anisole A448
Methylphenylketone. See Acetophenone A47-L
Methylphenylketoxime. See Acetophenoneoxime A49-L
Methyl Picrate. See 2,4,6-Trinitroanisole A450-L
Methylpyrrylketone. See Acetylpyrrole A86-R
Military Nitrate of Ammonia. See Amatol A158-L & A163-R
Mineral Pitch. See Asphalt A496-L
Mineral Rubber or Gilsonite. See under Asphalt A496-R
Moisture Determination in Ammonium Nitrate by Azeotropic Distillation A370-R to A371-L
Monacetin. See under Acetins A31-R
Monoethanolamine. See Aminoethanol A200-L
Monolene. See under Aminoethylation of N-(2-Hydroxypropyl)-ethylenediamine A203-L
MSX. See 1-Acetoxy-2,4,6-trinitro-2,4,6-triazaheptane under Acetoxytriazaheptane A53-L
Mudcapping. See Adobe shooting under Agriculture and Forestry Use of Explosives A113-R
Musk Xylene. See Trinitroterbutylxylene under Alkyd Resins A128-L

N

p-Naphthalene. See Anthracene A457-R
Naphthyleacetamide. See Acetamidonaphthalene A19-R
Naphthylacetate. See Acetoxynaphthalene A52-R
Naphthylamine. See Aminonaphthalene A237-L
β-Naphthylazotrinitromethane A67-R
Natriialmatrite. See under Almatrites A140-L
NBSX. See ATX A507-L
Needle Point Projectiles. See Arrowhead Projectiles A489-L
Needle Shell. See Arrow Projectile A488-R
NENA. See 1-Nitramino-2-ethanol Nitrate under Aminoethanol A201-L
Nickel Acetylide and Nickel Carbide A78-R
Nickel Diazide A592-R
Nickel Nitroaminoguanidine A213-R
Nitramide or Nitroxylamide A170-L
Nitramite. See Avigliana 3 A516-R
Nitrogen Oxides, Absorbent Materials for A5-L
α-Nitro-α-isonitrosoacetone. See Acetylmethylnitrolic Acid A84-L
Nitrolit. See 2,4,6-Trinitroanisole A450-L
Nitrometer Method for Determination of Nitrogen Content in Nitrates A373 to A378
3-Nitropropene. See Allyl, Nitro A138-R
γ-Nitropropylene. See Allyl, Nitro A138-R
Nitroso(N-so) (Nitrosylsulfuric Acid) Determination in Acids A89-R
Nitrosyl Azide A594-L
Nn°30; Nn°31; Nn°32 and Nn°33 (Explosifs) A148-L
Nomenclature II-VI
Non-Permissible Ammonium Nitrate Gelatin Dynamites (American, Belgian and French) A368 (table)
Not-Permitted Ammonium Nitrate Gelatine Dynamites (British) A368 (table)
Nuclear Bomb. See Atomic Bomb A499-L
Nuclear Energy. See Atomic Energy A500-L
Nuclear Explosions. See Atomic Explosions A501-R
Nuclear Fission Weapons and Ammunition. See Atomic Weapons and Ammunition A504
Nuclear Reactions. See Atomic Reactions A501-L
Nuevo Anagon (Spanish Ammonal) A289 (table)

O

Octahydro-1-acetyl-3,5,7-trinitro-s-tetrazocine. See 1-Acetyl-3,5,7-trinitrooctahydro-s-tetrazine, designated QDX or SEX A49-R
Organic Amides and Imides A170 & A171
Organic Azides and Azido Derivatives A626 to A643
Oxalylethylester Azide. See Azidoöxalic acid Ethylester A641-L
Oxoethylpyrrole. See Acetylpyrrole A86-R
2-Oxo-4,4,6-trimethyltetrahydropyrimidine. See Anhydroacetoneurea A403-R
Oxygen Balance to CO(OB to CO) and Oxygen Balance to CO_2(OB to CO_2). See under Available Oxygen A515
Oxyliquit A147(table)

P

Papite. French for CWA Acrolein A90-L
Paraldehyde. See under Acetaldehyde A14-R
Paraldol. See under Acetaldol A15-R
Pentaerythritol-Acetone Compounds. See Acetone Compounds of Pentaerythritol A40-L
Pentaerythritolmonoallylether Trinitrate. See 2-Allyloxymethyl-2-hydroxymethyl-1,3-propanediol Trinitrate A138-R
2,4-Pentanedione. See Acetylacetone A53-L
Pentanol. See Amyl Alcohol A394-R
Penthrinit A147 (table)
Pentryl. See 2-(2',4',6'-Trinitro-N-nitranilino Ethanol Nitrate under Anilinoethanol and Derivatives A425-L to A429-R
Pentryl Homologs A429-R
Peracetic Acid, Benzylester. See Acetylbenzylperoxide A55-L
Phenacyl Azide. See ω-Azidoacetophenone under Acetophenone A47-R
Phenazone. See Antipyrine A471-R
Phenetidine. See Aminophenetole A240-R
Phenyl Abietate. See under Abietic Acid A4-L
N-Phenylacetaminde. See Acetanilide A22-R
Phenylallylamine. See N-Allylaniline A136-R
Phenylallylozonide. See Allylbenzeneozonide A137-L
Phenylalanine. See under Anilinopropionic Acid A436-R
Phenylamine. See Aniline A406
Phenylaminobutanol. See Anilinobutanol A422-R
Phenylaminobutyric Acid. See Anilinobutyric Acid A423-R
Phenylamino-4-diazonium Hydroxide. See p-Anilinobenzenediazonium Hydroxide A421-L
Phenylaminodihydroxypropane. See Anilinopropanediol A434-R
Phenylaminoethanol or Phenylethanolamine. See under Anilinoethanols A424-L
Phenylaminoguanidine. See Anilinoguanidine A431-R
2-Phenylamino-2-methyl-1,3-dihydroxypropane. See 2-Anilino-2-methyl-1,3-propanediol A433-L
Phenylaminopropanol. See Anilinopropanol A436-L
1-Phenyl-5-amino-tetrazole. See under Aminophenyltetrazoles A247-L
5-Phenylamino-tetrazole. See Anilinotetrazole A437-L
Phenylaminopropanediol. See Anilinopropanediol A434-R
Phenylaminopropanol. See Anilinopropanol A436-L
Phenylamino-trimethylolmethane. See Anilinotrimethylolmethane A441-L
Phenylaniline. See Aminobiphenyl A191-R
N-Phenylanthranilic Acid. See Anilinobenzoic Acid A421-R
Phenylazodiphenylamine A420-R
Phenylazobenzoic Acid. See Azobenzenecarboxylic Acid A650-R
Phenylazoxybenzoic Acid. See Azoxybenzenecarboxylic Acid A668-L
N,N-o-Phenyleneguanidine. See 2-Aminobenzimidazole A187-L
Phenylethanolamine. See under Anilinoethanol A424-L
N-Phenylglycine or N-Phenylglycocoll. See Anilinoacetic Acid A420-L

N-Phenyl-N',N'-phthalylhydrazine. See N-Anilinophthalimide A434-L
Phenyltoluidine. See under Anilinotoluene A438-L
Phenyltrimethylolmethylamine. See Anilinotrimethylolmethane A441-L
N-Phenyl-(tris-hydroxymethyl)-methylamine. See Anilinotrimethylolmethane A441-L
Phenylxylidine. See Anilinoxylene A443-L
Phosphorus Carbide A78-R
Phosphorus-Nitrogen Azide A594-R
Physical Tests Used to Determine Explosive and Other Properties VII
Picatinny Arsenal. See under Arsenals A489-R
Picramic Acid. See 2,6-Dinitro-2-aminophenol under Aminophenols A241-R
Picramide. See 2,4,6-Trinitroaniline A409-R to A411-L
Picric Powder. See Abel Powder A1-R
5-(Picrylamino)-α-tetrazole. See 5-(2',4',6'-Trinitroanilino)-α-tetrazole A437-R
N-Picrylglycine or N-Picrylglycocoll. See 2,4,6-Trinitroanilinoacetic Acid A420-L
2-(N-Picryl-N-nitramino)-1-butanol Nitrate. See under Anilinobutanol A423-L
2-(N-Picryl-N-nitramino)-1-butanol Nitrate. See 2-(N,2,4,6-Tetranitroanilino)-1-butanol Nitrate A423-L
α-Picrylnitramino-iso-butyric Acid. See under Anilinobutyric Acid A423-R
Picrylphenylenediamines. See Trinitroaminodiphenylamines under Aminodiphenylamines A197
Picryltoluidine. See Trinitroanilinotoluenes A438-R
Plaster Shooting. See Adobe Shooting under Agriculture and Forestry Use of Explosives A113-R
Plosophore. See under Auxoexplose A514-L
Polverifici Giovanni Stacchini SA (Esplosivo) A149-L
Polymer of Acetylacetone Peroxide A53-R
Polynitroalcohols, Ammonia Derivatives of A306-L
Polynitroderivatives of Abietic Acid A3-R
Potassium Acetylide A79-L
Potassium-Aluminum Alum A156-R
Potassium Amide or Potassamide A169-R
Potassium Azide A594-R
Potassium Azidodithiocarbonate A634-L
Potassium Carbide A79-L
Potassium Chrome Alum. See under Alums A156-R
Potassium Hydrogen Acetylide A79-L
Potassium-Iron Alum A156-R
Potassium-5-nitraminotetrazole A260-L
Potassium Picramate A242-R
Potatoes as a Source of Absorbent Materials A5-R
Propadiene. See Allene under Allenic Compounds A133-R
Propanolamine. See Aminopropanol A253-L
Propanolaniline. See Anilinopropanol A436-L
Propanolon. See Acetol A33-R
2-Propanone. See Acetone A33-R
Propenal. See Acrolein A96-L
Propeneamide. See Acrylamide A96-R
Propenenitrile. See Acrylonitrile A97-R
Propene-1,2,3-tricarboxylic Acid. See Aconitic Acid A93-R
Propylamine. See Aminopropane A250-L
Propenylanisole. See Anethole A402-R
Pulsometer. See under Air Lifts A118-L
Puriculine. See Azete A518-R
Pyridylamine. See Aminopyridine A254-R
Pyrimidinetetrone. See Alloxan A134-R
Pyroacetic Ether. See Acetone A33-R
Pyrotechnic Compositions Containing:
Aluminum and Alloys A145, A153 & A154
Antimony A468
Auramin O A508-L
Pyruvic Alcohol. See Acetol A33-R
Pyruvonitrolic Acid. See Acetylmethylnitrolic Acid A84-L

Q

QDX or SEX. See 1-Acetyl-3,5,7-trinitro-öctahydro-s-triazine A49-R
Quinolylamine. See Aminoquinoline A255-R

R

Radioactivation Analysis. See Activation Analysis A99-L
RATO. See under ATO A497-R
Ripping Ammonal A289 (table)
Röchling Anticoncrete Projectile. See under Arrow Projectile A488-R
Rubidium Acetylide A79-L
Rubidium Azide A596-R
Rubidium Carbide A79-L
Rubidium Hydrogen Acetylide A79-L
Russian Ammonals A292-L
Russian Ammunition and Weapons A385-R
Russian Mixture. See Ammontol A383-L

S

St Helen's Powder. See under Ammonals A289 (table)
Salicylic Acid Triazoacetate. See under Acetylsalicylic Acid A87-L
"Salpetersäure-Diazophosphenylsäure" of Michaelis. See Compound $C_6H_6N_3O_6P + 3H_2O$ A246-R
Screens. See Sieves A674 (Table I)
Self-Destroying Ammunition A391-R
Self-Hooping. See Autofrettage A510-R
Semi-Gelatine (A British Ammonium Nitrate Not-Permitted Dynamite) A368 (table)
Sevranite No 1 (Explosif) A148-L
SEX. See QDX A49-R
Sieves(Screens). Comparison of US, Tyler, British and German Systems A674 (Table I)
Silicon Carbide A79-L
Silicon Carbide-Aluminum Oxide Fiber A155-R
Silicon Tetrazide A597-R
Silver Acetylide, Analytical A81-R
Silver Acetylide, Destruction A81-R
Silver Acetylide or Silver Carbide A79-R
Silver Acetylide, Analytical and Destruction A81-R
Silver Acetylide Complexes A80-A81
Silver Amide A169-R
Silver Azide A597-R to A601-R
Silver Azidodithiocarbonate A637-L
Silver Nitroaminoguanidine A213-R
Silver Picramate A242-R
Smoke Compositions Containing Auramine O A508-L
Sodamide A170-L
Sodium Acetate A29-L
Sodium Acetylide A82-L
Sodium Aluminum Alum A157-L
Sodium Amide A170-L
Sodium Azide (SA) A601 to A612
  General Properties A601 to A603 & A605 to A607
  Laboratory Preparation and Manufacture A603 to A604
  Explosive Properties A604 to A605
  Uses A607 to A608
Sodium Azide, Plant Analytical Procedures A612-R to A619
  Analysis of Ammonia A612-R & A303
  Analysis of Sodium Metal A612-R
  Analysis of Wringer-Cake A613-L
  Analysis of First Mother Liquor A613-R
  Analysis of Second Mother Liquor A615-L
  Analysis of First Clear Liquor A615-L
  Analysis of Lime Treatment Tank A615-R
  Analysis of Second Clear Liquor A616-L
  Analysis of Crude Sodium Azide Liquor A616-L
  Analysis of SA, Crystalline A617-L
  Colorimetric Determination of SA in Aqueous Ammonia A617-L
  Analysis of Technical SA Prepared from Hydrazine and Ethyl Nitrate A617-R
Sodium Azidodithiocarbonate A634-R
Sodium Carbide A82-L
Sodium Hydrogen Acetylide A82-L
Sodium Picramate A242-R
Sofranex A (Explosif) A148-L
SP-42, SP-43, SP-45, SP-47 and SP-49 Propellants. See Aerojet Propellants A350 (table)
Space Travel. See Astronautics A498-L
Spanish Ammonals A292-L
Stabilit. See Acardite I A7-R
Stilbeneazostilbene. See Azostilbene A659-R
Storage Batteries. See Accumulators A12-L
Strontium Acetylide or Strontium Carbide A82-R

Strontium Diazide A620-L
Styrylamine. See Aminostyrene A257-L
Succinum. See Amber A165-R
Sulfurless Black Powder. See under Amide (Explosif) A168-L
Sulfuryl Diazide A621-R
Sylvic Acid. See Abietic Acid A2-R

T

TAX. See 1-Aceto-3,5-dinitro-1,3,5-triazacyclohexane under Acetotriazacyclohexane A50
α-Terpinene Peroxide. See Ascaridol A494-R
Tests, (Physical) Used to Determine Explosive and Other Properties VII
Tetrahydroxyanthraquinone. See Anthrachrysone A458-R
3,4,5,6-Tetrahydro-4,6-diimino-s-triazin-2 (1H)-one. See Ammeline A273-R
Tetrahydroimidaz[d]imidazole-2,5(1H,3H)-dione. See Acetylenediurein A65-L
Tetrahydro-3,3,5,5-tetrakis(hydroxymethyl)-4-oxypyrane. See Anhydroenneaheptitol A404-L
2,2,4,4-Tetrakis(hydroxymethylnitrate)-1-pyranol-1-nitrate. See Anhydroenneaheptitol Pentanitrate A404-L
Tetramethylammonium Azidodithiocarbonate A637-R
Tetramethylazobenzene. See Azoxylene A662-L
Tetramethylazoxybenzene. See Azoxyxylene A673-L
1,1,4,4-Tetramethyl-2-butynylenedihydroperoxide. See under Acetylene Hydroperoxides A66-R
Tetramethyl-p_2-diaminobenzophenone. See Auramine A507-R
Tetranitromethane, Manufacture from Acetylene A67-L
Tetrazolo-1,2-azido-4-phthalazine-1,2-dihydride. See (1'-Azidophthalazine-4', 5')-5,1-tetrazole A641-R
Thallium Azide A621-R
Thallium Diethyl Picramate A243-L
Thallium Dimethyl Picramate A243-L
Thallous Azidodithiocarbonate A637-L
Thallous-Thallic Azide A623-R
Thermonuclear or Fusion Bomb. See under Atomic Bomb A499-L
Thorium Dicarbide A82-R
Tin Azide A624-L
Titanium Carbide A82-R
Titanous Chloride Method for Determination of Nitrobenzene in Aniline A415-R
TNT Recovery from Scrap Amatol A161-L
Toluidine. See under Aminotoluenes A265-R
Tolylamine. See under Aminotoluenes A264-R
Tolyltetrazolonimide. See under Aminotolyltetrazoles A265-R
Total Acidity as Nitric Acid A88-R
Total Acidity as Sulfuric Acid A89-L
Total Actual Acidity A90-L
Total Nitric as Nitric Acid A88 & A89
Total Sulfuric as Sulfuric Acid A89
Transannular Photoperoxide of Anthracene A458-R
Triacetin. See under Acetins A31-R
Triacetone Triperoxide. See under Acetone Peroxides A42-R
Trialkyls of Aluminum A144-R
3,4,5-Triamino-α-sym-triazole. See 4-Aminoguanazole A209-R
2,4,6-Triaza-2,4,6-trinitro-heptan-1-ol Acetate. See 1-Acetoxy-2,4,6-trinitro-2,4,6-triazaheptane or MSX A53-L
4-Triazoacenaphthene. See under Acenaphthenes A12-R
Triazoates. See Azides, Inorganic A520-L
Triazo Compounds. See Azido Compounds A626ff
1H-1,2,3-Triazole-4-ethylamine. See 4-(β-Aminoethyl)-α-vic-triazole A208-L
3-Triazopropene. See Allylazide A137-L
Tricrotonylidenetriperoxide-tetramine. See under Amine Peroxides A178-L
Triethyllead Azidodithiocarbonate A637-R
4',5,7-Trihydroxyflavone. See Apigenin A473-R
4,5,2'-Trihydroxy-2-methylanthraquinone. See Aloeemodine A140-R
4',5,7-Trihydroxy-2-phenylchromone. See Apigenin A473-R

Trimeric Acetone Peroxide. See under Acetone Peroxides A42-R
Trimeric Methyleneaniline. See Anhydroformaldehydeaniline A404-R
Trimethylaniline. See Aminohemimellitene A215-R
Trimethylaniline. See Aminomesitylene A224-R
Trimethyl-[3-azido-5-nitro-4-hydroxy-phenyl]-ammonium Hydroxide. See 2-Azido-6-nitro-1,4-benzoquinone-4-trimethylimide A640-R
Trimethyleneimine. See Azetidine A518-R
N-(Trimethylolmethane)-aniline. See Anilinotrimethylolmethane A441-L
2,4,6-Trimethyl-1,3,5-trioxane. See Paraldehyde under Acetaldehyde A14-R
Trinitrides. See Azides, Inorganic A520 to A625
Triphenylmethyl Azidodithiocarbonate A633-R
1,3,5-Triphenyl-trimethylenetriamine. See Anhydroformaldehydeaniline A404-R
Tungsten (or Wolfram) Carbide A82-R
Type 91 Explosive (Japanese) A450-L
Type A Explosive (Japanese). See A(ko) Explosive A119-L

U

p-Urazine. See 4-Aminourazole A272-L
US Ammunition and Weapons (Calibers) A386 & A387

V

Vanadium Carbide A83-L
Vinylaniline. See Aminostyrene A257-L
Vinyl Carbinol. See Allyl Alcohol A135-R

W

Water(or Methanol)-Aluminum(or Magnesium) Explosives A155-L
Wax-Gap Test as conducted at the Spencer Chemical Co, Kansas City, Mo A354, Note c
White German Powder. See Augendre Powder A507-L
Windshield or Ballistic Cap A483-L
Wolfram Carbide. See Tungsten Carbide A82-R

X

Xylidene. Same as Aminoxylene A272
Xylylamine. See Aminoxylene A272-L

Z

Zinc Acetylide A83-L
Zinc Azide A624-L
Zinc Azidodithiocarbonate A637-L
Zinc Diazide A624-L
Zirconium Carbide A83-L

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