

CHAPTER 20

QUALIFICATION OF ENERGETIC MATERIALS FOR PROCUREMENT OF MUNITIONS

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20 QUALIFICATION OF ENERGETIC MATERIALS FOR PROCUREMENT OF MUNITIONS

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20.1 AWARENESS SUMMARY

This chapter explains the procedures required to qualify energetic materials in accordance with the requirements of STANAG 4170⁽¹⁾ and AOP-7⁽²⁾ for the purposes of the UK MOD. The Defence Ordnance Safety Group (DOSG) Science and Technology (ST1) section is the UK National Authority for the assessment of the safety and suitability for introduction into military service of energetic materials in compliance with STANAG 4170.

Explosive Qualification is defined as the assessment of the explosive material by the National Authority according to STANAG 4170 and AOP-7 to determine whether it possesses properties which make it safe for consideration for use in its intended role.

Explosive Qualification provides evidence to answer the following questions early in the CADMID cycle⁽³⁾ of the munition:

- How does the munition work?
 - Chemistry, Physics, Engineering, Statistics etc.
- What is the munition 'safe working envelope'?
- What are the safety critical failure modes that might exist/develop?
 - How can these be quantified or demonstrated during Qualification/Surveillance process?
- What other service life limiting failure modes might exist?

Qualification of an explosive for use in a specific application or munition is a separate process, called Type Qualification. Type Qualification is given when the explosive has been assessed as part of a single design and shown to be safe and suitable in that munition. Explosive Qualification assessment should be completed prior to Type Qualification and selection for a particular application.

There is no pass/fail criterion for Explosive Qualification; judgement will be by comparison with explosives that have been used satisfactorily in the same role. Explosive Qualification provides essential data on mechanical, physical, chemical, sensitiveness (see 20.2.1) and explosiveness (see 20.2.1) properties, which:

- Enables quantitative risk assessment to be carried out
- Identifies safety critical failure modes
- Provides baseline data for service life prediction/surveillance
- Establishes explosive ageing characteristics
- Candidate energetic materials can be ranked in terms of their properties
- Provides data required for munitions disposal, hazard and environmental considerations

- Increases energetic materials knowledge and identifies areas where research is required

Qualification is a requirement for all explosives including; primary explosives, booster explosives, high explosives, solid gun and rocket propellants, liquid propellants and pyrotechnics compositions. Each of these types of materials and the roles in which they are used, require that their responses to different accident or threat stimuli be evaluated in order to assess whether the materials are likely to be safe and suitable for introduction into UK service.

20.2 STANAG 4170 and AOP-7

Under STANAG 4170 participating Nations agree the requirement for Qualification of energetic materials for the purposes of ensuring safety and suitability for service in ammunition. Both the mandatory data requirements and general test guidance are included in NATO AOP-7. This guidance includes the types of tests and ageing protocols to be carried out for the various classes of energetic material. The guidance allows for some variation of test and test procedures by participating Nations. Direct read across between data from different nations will not always be possible therefore specific UK testing may be required. AOP 7 Chapter 10.7.15 details UK specific national test requirements. The UK national section cross references STANAG test methods alongside specific UK small scale sensitivity tests. Specific UK tests are discussed at length in Appendix B.

It is recommended that material Qualification should be completed before a decision is made to use the explosive material in a weapon design. The Qualification of a new explosive does not imply Type Qualification (see 20.5) for use in a particular weapon.

20.2.1 Definitions

The definitions in AOP-7 ⁽²⁾ are to be used during Qualification. Specific attention should however be drawn to the following definition:

New Explosive. The term 'New Explosive' encompasses the following:

- An explosive material that has not been previously qualified
- An explosive material for which the existing specification defining its composition, its material constituents or the process by which the composition is prepared has been modified
- An explosive material resulting from a change in manufacturer or manufacturing location
- An explosive material used in a role for which the explosive has not already been qualified

The following definitions are used when considering safety and reliability of explosives:

- **Sensitiveness.** A measure of the minimum level of a prescribed stimulus, relevant to an accident situation, required to ignite or initiate an energetic material. Such stimuli include, among others, impact, friction, and electric spark.
- **Sensitivity.** A measure of a prescribed stimulus required to achieve reliable functioning of an energetic material in its designed mode.
- **Explosiveness.** This describes the degree of violence shown by an energetic material when it responds to a prescribed stimulus relative to an accident situation. It is dependent on the nature of the energetic material, its mass, physical state, configuration and confinement.
- **Compatibility.** Absence of reactions between explosives and other component within a munition, leading to unacceptable changes in physical properties, sensitiveness or sensitivity of explosives in the munition as detailed in STANAG 4147⁽⁴⁾.

20.2.2 General Advice

It should be noted that the safety and suitability characteristics assessed and interpreted for Qualification purposes relate to an energetic material or composition in a specific role and that the data presented from a Qualification programme may also assist the Regulatory Authority in its assessment for granting a storage and transport hazard classification certificate. It should also be noted that any estimation of service life and detailed performance parameters are not part of the Qualification process.

Qualification of a new composition does not imply any recommendation for use in a specific weapon system, but any evidence of significant change of properties on ageing will be included as a proviso, so that the change may be considered during subsequent assessment of the munition containing the composition.

A new energetic material having a characteristic rendering it unsuitable for Qualification in one role does not necessarily excluded its consideration for another. In these circumstances particular care will need to be taken in determining an appropriate trials programme in order to Type Qualify (see 20.5) the material in a particular munition.

The safety and suitability of the weapon system is demonstrated separately by relevant safety and environmental tests. This will include Type Qualification of the energetic material for the particular application in Accordance with the Allied Ammunition Safety & Suitability for Service Publication (AAS3P).

20.3 COMPOSITION DETAILS, USE AND SAFETY: MANDATORY REQUIREMENTS

When considering an energetic material for Qualification full details of the manufacturing specification and composition should be supplied and should include the percentage of individual ingredients, specifications and manufacturing source(s) of each of the ingredients, relevant toxicity and known

hazard data. These requirements are referenced and discussed at length in Appendix A and are STANAG 4170 Explosive Hazard Data Sheet requirements.

It is accepted that the composition of an energetic material, processing method and any controlling specifications may be modified during the material development programme. This may be necessitated by scale-up considerations or by changes required to accommodate issues identified during Type assessment trials. To enable the continued applicability of any existing Qualification certification the National Authority should be informed of any such changes and any additional testing requirement should be identified and undertaken.

Historically many explosive materials consisted of a well-defined formulation with reproducible micro-structure and physical properties. However, the drive to improve munition safety characteristics, particularly with respect to vulnerability, has led to the greater use of Polymer Bonded Explosive (PBX) type materials where a single explosive or a mixture of energetic components is bound in a polymer matrix that includes plasticisers. These compositions are necessarily highly heterogeneous. Widely differing mechanical properties (e.g. strain capability) can be produced by relatively minor modifications in cross-link agent concentration or cure conditions. Since material Qualification data must be representative of the properties of the energetic material as actually presented for service use Design Organisations should ensure that samples tested for the purposes of Qualification are materially representative throughout the explosive. It may be required to test samples extracted from munitions to verify that this requirement is fulfilled.

20.3.1 Origin of Energetic Materials

Historically the majority of UK energetic compositions were of UK design and manufacture, however this is no longer the case with the majority being supplied from abroad as are the explosive substances used in their manufacture such as RDX, HMX and nitrocellulose. Where an energetic composition or its key components are of overseas origin, the available test data for these materials may not entirely fulfil the requirements of DOSG ST1 as the National Authority. So far it has not been proven possible to fully standardise test procedures internationally. Those used by individual NATO nations are listed in AOP-7⁽²⁾ in the relevant Chapter 10 national sections. Some nations' tests are less well understood in the UK and where this presents difficulties for Qualification programmes, specific requests identifying the data shortfall will be made to the Design Organisation.

A programme of extra testing may be required should any Qualification data supplied be considered insufficient to establish material characteristics. If tests cannot be carried out in the country of origin, then it may be necessary to provide samples of the energetic composition for testing in the UK. This may be in the form of specifically manufactured test material or material extracted from actual munitions.

20.3.2 Change in Manufacture

Re-qualification of a material will also become necessary when there is a change of manufacturer and/or manufacturing site and using a particular manufacturing process. It is now quite normal for repeat production of explosives and munitions to be open to competition and obsolescence driven changes in manufacture are occurring with increased frequency. When this occurs, advice should be sought from the National Authority as to the extent of the re-qualification exercise.

In reaching a decision on whether their original advice for safety and suitability for service needs to be amended the National Authority will be guided by the principles that explosives and munitions subject to a change in manufacture:

- Must remain safe and suitable for service, and continue to have a service life acceptable to the MOD Project Team
- Materials are assumed not to have the same characteristics as the original

A change in manufacture will have occurred when there is a change in one or more of the following:

- The drawings or specification
- The manufacturing process
- The source of type of raw materials
- The source of components
- The manufacturer or manufacturing site
- A break in production which could have led to a loss of manufacturing expertise

Changes to drawings, specifications and manufacturing processes are normally recorded, assessed and approved by the MOD QA Authority and/or technical authority prior to implementation. On those occasions where safety may be affected advice should be sought from DOSG or the DE&S Weapons Operating Centre (WOC).

20.4 PRELIMINARY ASSESSMENT AND HAZARD DATA

Prior to Qualification a preliminary safety assessment is carried out which, in the UK, results in the issue of an Explosives Hazard Data Sheet (EHDS) conforming to the requirements of STANAG 4170, Defence Standard 07-85⁽⁵⁾ and Defence Standard 13-129⁽⁶⁾. DOSG ST 1 as the National Authority uses this information when qualifying energetic materials. A more detailed examination of the requirements for EHDS is covered in Annex A. Qualification of energetic materials new to UK service is determined by assessing the compositional details together with the physical, chemical, mechanical, sensitiveness (see 20.2.1) and explosiveness (see 20.2.1) properties. Properties are compared with those of energetic materials which have been used in a similar role in UK service and for which a high level of confidence has been established. Test data derived from energetic material Qualification

should be used to inform design of Type Qualification and Surveillance test programmes.

The extent of Qualification testing may range from nothing, where an explosive's characteristics can be read across from similar materials, to a full range of Qualification tests where a new explosive contains one or more of the following:

- New ingredients not previously used in explosives
- Ingredients new to this type of composition
- A substantially different ratio of major and minor ingredients
- Substantially different physical properties (e.g. particle size, density etc.)

20.4.1 Energetic Material Test Requirements

The testing required can be broadly categorised as follows: compositional; chemical; physico-mechanical; and hazard (small scale & charge scale) tests. Guidance on this is outlined below.

20.4.1.1 Chemical properties

The energetic material must demonstrate adequate chemical stability. Information from a variety of tests should have been obtained during a UK development programme and additional tests are not normally required by DOSG for Qualification purposes.

The overall thermal stability of the composition needs to be established for Qualification. Thermal stability can be determined relatively easily and data should normally have been obtained during the development programme. Techniques such as differential scanning calorimetry (DSC), differential thermal analysis (DTA), thermogravimetric analysis (TGA), Heat Flow Calorimetry (HFC) and Accelerating Rate Calorimetry (ARC) provide a great deal of information regarding the thermal behaviour of a composition and its constituents.

Properties such as melting points, phase changes, decomposition temperatures and ignition temperatures can be obtained from DTA and DSC, with the latter also providing the total energy output on decomposition. TGA on the other hand only indicates temperature of decomposition and changes, which affect mass, such as loss of water or volatiles.

Nitrate esters, for example in nitrocellulose based propellants, are known to degrade with time, and the products of the degradation catalyse further degradation, which can eventually lead to self-ignition of the propellant. In order to prevent this, sacrificial stabilisers are included in the formulation to remove some of the products of the degradation. The chemical stability of the explosives can thus be measured by monitoring the level of the stabiliser(s) and/or the presence of the degradation products.

In practice a variety of tests are used, assessing the range of decomposition modes. Together these indicate the safe chemical life of the explosive and may

be used as part of the determination of the service life. The methodologies as described in STANAG 4620⁽⁷⁾ are well established and strongly preferred as this enables comparison with many service compositions tested previously and the application of common sentencing criteria. Determination of chemical compatibility (see below) will be in accordance with the STANAG 4147⁽⁴⁾. Where specific STANAGs are not available, the chemical stability assessment regime will need to be agreed with the National Authority.

In addition to the risk of self-ignition, the gaseous degradation products of the nitrate esters can induce gas cracking in rocket propellants. The likelihood of this occurring for a given set of conditions is dependent on a number of parameters, including: the effectiveness of the stabiliser, the propellant formulation, the mechanical properties and the charge geometry. The vulnerability to gas cracking needs to be assessed using a test regime agreed with DOSG.

The overall chemical stability of composite propellants should also be assessed using vacuum stability and thermal stability tests, in accordance with STANAGs 4556⁽⁸⁾ (Explosives, vacuum stability tests) and STANAG 4515⁽⁹⁾ (Explosives, thermal analysis using DTA, DSC, HFC and TGA).

20.4.1.1.1 Compatibility

Stability testing of explosives is concerned with the inherent stability of the chemical components of the explosive against spontaneous decomposition. Compatibility testing considers the potential for a non-explosive material in contact or close proximity to the explosive material to cause a chemical reaction that compromises the stability of the explosive.

There are three issues to be considered when assessing the compatibility of materials with explosives:

1. Inert materials present in design interacting with the explosives and destabilising them causing an immediate safety issue.
2. Inert materials present in design interfering with some aspect of explosive performance without diminishing explosive safety characteristic causing an indirect safety issue.
3. Explosive material interacting with inert material to either increase hazard of inert materials or influence 'Type' performance.

When assessing these factors contact between the materials can be 'direct' or 'vapour path'. Current DOSG ST1 doctrine states that unless there are two reliable seals between materials their compatibility should be tested and they are included in the compatibility test matrix. Table 20-1 is an example compatibility matrix for the munition shown in Figure 20-1. Explosive to explosive compatibility must also be proven.

Most compatibility tests are modified stability tests. An example of compatibility testing is a variation of the Vacuum Stability Test, where an explosive test sample is held at a given temperature under a starting pressure no greater than 6.7 mbar for a given time period. Assessment of the pressure increase in a

known apparatus volume allows calculation of the volume of gas given off by the explosive. As a stability test this gas evolution is compared against a specification, for example in a Defence Standard. Typical test conditions are 40 hours at 100 °C for high explosives and 240 hours at 80 °C for double base propellants.

Table 20-1 Compatibility Test Matrix for the Munition Shown in Figure 20-1

Energetic Material (EM)	Inert Material (IM)	Contact Type	Test
Torpex 9	Polyurethane Type X	Contact	Vacuum Stability & HFC
Torpex 9	Heat Insulating Layer Type Y	Contact	Vacuum Stability & DSC
Torpex 9	Steel Body Grade 2568	Possible Vapour/ Manufacturing	None (by analogy), EM and IM common from Mark I, UK Service

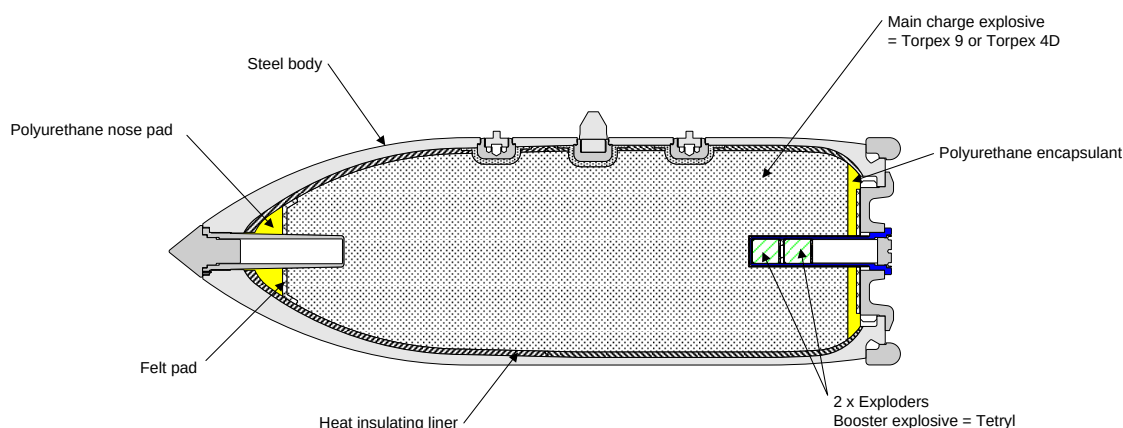


Figure 20-1 Example munition showing potential physical contact and vapour path contacts that require compatibility testing

To assess compatibility 2.5 g of the explosive is tested and gas evolution measured. Also 2.5 g of the material being assessed for compatibility is tested. This latter material is sub-divided in to small pieces. Finally 2.5 g of the material and 2.5 g of the explosive are intimately mixed and tested. If the gas evolved from the mixture is 5 cm³ or more compared to the addition of gas evolution from the material and explosive separately the combination is incompatible.

20.4.1.2 Physical and mechanical properties

In addition to the chemical deterioration/mechanical property interaction, other physical changes may occur. Any evidence of phase changes or exudation

needs to be considered. Plasticisers and desensitisers may become mobile under thermal or mechanical stressing conditions and transfer energetic material into screw threads or other close fitting joints. The likelihood of this occurring should be assessed.

Solid propellant must demonstrate satisfactory mechanical properties over the expected service temperature range. The suitability of the mechanical properties will depend on the intended role and the physical form (whether thick web charge, thin web charge, granular or powder, etc.). Most rocket motor propellants and many explosives in charge form will require both tensile and dynamic mechanical properties to be assessed across the expected service temperature and humidity range. Where it is relevant, mechanical property testing will be carried out in accordance with STANAG 4717⁽¹⁰⁾ (Energetic materials mechanical test methods). Other mechanical properties may need to be assessed and further mechanical testing may be required by DOSG to fulfil the requirements of the Qualification process.

Composite propellants also degrade with time; the rubbery matrix can be subject to oxidation which gives rise to a degradation of mechanical properties. In order to reduce this, sacrificial antioxidants can be included in the formulation. If this is the case, the level of these antioxidants will need to be monitored. The soluble fraction of the binder also provides a measure of the extent of its oxidation and hence it is necessary to determine this also, in accordance with the STANAG 4581⁽¹¹⁾ (Assessment of ageing characteristics of composite propellants containing an inert binder). Where specific STANAGs are not available, the assessment regime will need to be agreed with the National Authority.

Historically, secondary explosives were generally of very limited mechanical capability and mechanical testing was often only performed at ambient temperatures. Modern PBX compositions generally have much superior mechanical capabilities across wide temperature ranges and their characterisation for Qualification should include testing of relevant parameters across the range of service environments to be encountered. Variations in density may affect the overall hazard characteristics of High Explosive components manufactured by pressing and this should be quantified early in the development programme.

In addition, explosives will become brittle when the temperature falls below the glass transition temperature (T_g). It is therefore essential that relevant mechanical properties of PBXs be characterised both before and after ageing in order that these effects may be assessed.

By definition a booster composition is used as a component in the initiation train of a main High Explosive charge and will often be used in small quantities and with mechanical support. For this reason often only limited mechanical property testing is required.

The density, including percentage of theoretical maximum density, of the explosive material batch(es) used for the assessment is generally required by the National Authority.

20.4.1.3 Properties after ageing

Qualification requires demonstration that required properties are maintained throughout service life. AOP-7⁽²⁾ contains guidance on the UK preferred temperature/time storage requirements to demonstrate this. It is strongly recommended that these be agreed with DOSG prior to the start of the test programme.

The temperature used to promote artificial ageing of energetic compositions must be carefully selected. The majority of the DOSG ST1 national data base contains ageing data at 60 °C for 3, 6 and up to 12 months and use of these regimes makes comparison with in service compositions straightforward. Whilst selection of higher temperatures may enable more rapid achievement of 'age equivalence', it risks promoting ageing modes unrepresentative of those encountered in service and such test results may prove unsatisfactory and require repeat ageing at lower temperatures.

20.4.1.4 Sensitiveness and Explosiveness

The principle environments under which energetic materials are assessed are:

- Impact sensitiveness
- Friction sensitiveness
- Ignition temperature
- Electric spark (electrostatic discharge sensitiveness)
- Fragment attack
- Explosive shock
- Explosive response when ignited (confined and unconfined)

Subjecting the candidate material to small scale and charge scale tests assesses the response of energetic materials to abnormal environments. The difference in sensitiveness (see 20.2.1) and explosiveness (see 20.2.1) between loose and consolidated samples is reflected in the tests necessary to provide the required information for a Qualification assessment to be made. This methodology is discussed further in Appendix A.

The larger charge scale tests (See Appendix A) subject the energetic material to stimuli, where appropriate, comprising:

- Heating under confinement
- High velocity fragment impact
- Explosive shock
- Ignition under confinement

Test regimes have been devised for National Authority assessment of energetic materials and aim to satisfy the requirements AOP7⁽²⁾. Unless otherwise specified, each of the tests listed in a particular regime against a particular

serial should be conducted on the candidate material. If the National Authority is satisfied that there is sufficient evidence of equivalence, it may consider reading across some of the charge scale test results from relevant compositions. In these cases, the test programme will be reduced accordingly.

A booster needs to exhibit higher sensitivity than a main charge and thus will also invariably exhibit higher sensitiveness (see 20.2.1) than the associated main charge but it is considered unnecessary to determine some of the hazard characteristics of booster compositions in charge configurations when, by the very nature of the materials, they will not see service in that form. Booster materials are used in significantly smaller quantity than main charges and therefore their exposure to mechanical stress is much reduced. If the booster is likely to be used in pellet form then further mechanical and physical tests may be required to assess the likelihood of powdering and thus any consequent hazards.

20.5 APPROACH TO QUALIFICATION

Three approaches to qualifying explosives can be identified. Qualification followed by Type Qualification or Type Qualification alone. The third is a middle route.

Qualification of an explosive followed by Type Qualification of the explosive in the weapon is the standard route under which the National Authority provides advice, and is mandated in AOP 7⁽²⁾. This methodology has generally been the most cost effective route to provide advice when sought at a sufficiently early stage in the procurement process.

In some instances however it may be acceptable not to qualify the explosive in its own right but to undertake a more thorough Type Qualification instead. This is more likely to apply when good read across from similar explosives is available or when potential hazards have been assessed as low, for instance where weapons have only a small explosive content.

In other instances a limited amount of Qualification testing may be considered sufficient. These are when:

- Some, but insufficient Qualification data are available
- Limited read across is available
- The weapon or device in which the explosive is to be used is assessed as not presenting a significant hazard

In making a choice between the above approaches the risks of failure at a late stage in development must be considered, including the implications for the user of a weapon not being assessed as safe and suitable for service (S³), and the overall cost to the manufacturer or supplier of the weapon if not accepted in to UK service.

Guidance in the form of a report with test schedules, indicating the level of enhanced testing required for Type Q in the absence of material Qualification testing is required and the final decision as to whether the Qualification step can be omitted remains with the UK National Authority DOSG ST1 in the UK.

When an explosive composition has been produced and qualified to STANAG 4170⁽¹⁾ by another nation, due account should be taken, both of the Qualification methods and of the amount of data released to support it.

DOSG continues to request that other nations disclose Qualification data both to enable Qualification of new explosives to be completed with the minimum of extra testing and associated costs and to assist in the development of internationally agreed standard Qualification tests. Often further information cannot be made available by the National Authority due to manufacturer's commercial confidentiality issues or ITAR constraints. Until greater cooperation is achieved the full benefits in terms of risk reduction and time and cost saving of STANAG 4170⁽¹⁾ will not be fully realised.

In order to facilitate the maintenance of a UK database it is important that all Qualification reports and safety cases contain a formal statement regarding the Qualification status of all explosives used.

20.6 QUALIFICATION REPORTING

At the end of the Qualification programme the results of the small scale tests are normally presented in the form of an Explosive Hazard Data Sheet (EHDS) along with further trials reports documenting the results of the charge scale tests and any extra testing or analysis performed.

In the UK, after consideration of all the evidence in support of Qualification, the National Authority will issue a Qualification report detailing their consideration of the evidence and generally comparing the candidate energetic material with current or historic UK service materials used in similar applications. Since this will generally require inclusion of data from multiple sources, intellectual property and/or security considerations will prevent general issue. Where required, DOSG ST1 will issue a summary report suitable for more general distribution via the relevant project team.

An explosive Qualification certificate in accordance with the format suggested in AOP-7⁽²⁾ will be issued for each energetic material approval. The certificates will generally contain only limited formulatory information and are generally suitable for unrestricted distribution. The National Authority keeps a database of all UK Qualified energetic materials. To ensure control of intellectual property and adherence to security requirements the issue of this list is controlled by the Head of Energetic Materials in DOSG ST1.

The requirements for testing of in-service energetic materials to gain Qualification can be waived at the discretion of the National Authority DOSG ST1. This is specifically permitted within AOP-7⁽²⁾ providing there has been no

change to the specification, explosive material already in service for which there is a history of satisfactory application may be considered Qualified by the National Authority for use in the same role (e.g. main charge filling, booster, initiator, rocket motor propellant, gun propellant, pyrotechnic, etc.), without further testing. The National Authority will define the need for any restrictions of the role and for any further testing in specific cases. Given the significant changes to the UK MOD energetic material supply base it is unlikely that continuity of specification, process and expertise will have been maintained for significant numbers of explosives which have not already been presented for Qualification by DOSG ST1 in the past decade. Therefore, material Qualification requests which are not fully supported with specific and recent (i.e. within 5 years of date of request for Qualification) test data will not generally be considered for UK Qualification.

The Qualification status of energetic materials, when fully qualified, shall be reviewed on a five-yearly basis. The Design Organization or Project Team will be requested to confirm continuity of specification, supply and process should evidence not be fully available. Where evidence supporting continuing Qualification status beyond five years is not available for munitions and delivery to MOD is still underway or is planned, the National Authority DOSG ST1 will request the Design Organisation or Project Team ensure that the Munition Safety Case is amended to reflect the position.

20.7 PRIMARIES AND PYROTECHNICS

As the UK National Authority DOSG ST1 has not qualified primary explosives and pyrotechnic materials for many years, this is entirely due to the historic nature and formulation of these types of materials. Their current UK Qualification status can be considered satisfactory and they are included in the National Authority Qualified status list.

Many devices fitted to platforms include small quantities of primary explosives or pyrotechnics whose general use is not associated with weapon effect which include aircraft fire suppression, hydraulic activation and emergency escape systems. These devices are identified for consideration by DOSG ST1 though it is accepted that in many cases the quantities of energetic material employed are small and the safety risks associated are limited to the effectiveness of device.

20.8 REFERENCES

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Appendix A Qualification Methodology and Explosive Hazard Data Sheet (EHDS) Requirements under STANAG 4170

A.1 Part 1: Methodology and General Characteristics

STANAG 4170 outlines the requirements for material Qualification within NATO nations, and combined with the requirements of AOP 7 defines the tests requirements to provide small scale and charge scale hazard data for an EHDS.

STANAG 4170 contains the following terms and definitions which must be applied when qualifying explosive materials and selecting data for an EHDS:

- Assessment. The evaluation of the properties of an explosive material, including the results of appropriate tests, to determine its relationship, with particular regard to safety, to other known explosive materials already in service use.
- Explosive Material. An explosive material is a substance (or mixture of substances) which is capable by chemical reaction of producing gas at such a temperature and pressure as to cause damage to the surroundings. Included are pyrotechnic substances even when they do not evolve gases. This document will refer only to those explosive materials whose application requires that they shall react reliably on demand. The term “explosive” thus includes all solid and liquid materials variously known as high explosives and propellants, together with igniter, primer, initiatory and pyrotechnic (e.g. illuminant, smoke, delay, decoy, flare and incendiary) compositions.
- Qualification. This is the assessment of the explosive material by the National Authority according to this STANAG and AOP-7 to determine whether it possesses properties which make it safe and suitable for consideration for use in its intended role.
- Qualified Explosive Material. An explosive material which has successfully completed the Qualification process of a National Authority. This is an intermediate risk reduction stage prior to Final (or Type) Qualification.
- Final (or Type) Qualification. Final (or Type) Qualification relates to the use of the explosive material in a specific application or munition. Final Qualification is given when the explosive has been assessed as part of the design of the specific munition, and predicted to be safe and suitable for military operational or training use in that role. The database of results is a means of undertaking “Risk Assessment”.
- New Explosive Material. The term “New Explosive Material” encompasses:
 - An explosive material not previously Qualified;
 - An explosive material for which the existing specification defining its composition, its material constituents or the process by which the composition is prepared has been modified;

- An explosive material resulting from a change in manufacturer or manufacturing location;
 - An explosive material used in a role for which it has not already been Qualified.
- Comparison Explosive. An in-service explosive with proven safety characteristics whose properties are used to assess the relative safety and suitability of a new explosive intended for use in a similar role.
- Intended Role. The main roles are listed as follows. Nations may define other roles or add to specific information for the intended application of a particular explosive material.
 - Primary Explosive. Substance, or mixture of substances, used to initiate a detonation or a burning reaction. In their intended role these materials are sensitive to a range of thermal, mechanical and electrical stimuli.
 - Booster Explosive. Explosive material used to augment and transmit the reaction (initiated by the primary explosive) with sufficient energy to initiate a detonation reaction in the main charge.
 - High Explosive. Material which is used as a detonating final charge.
 - Solid Gun Propellant. Substance or mixture of substances which is required to burn in a controlled manner within a gun combustion chamber producing hot gases capable of propelling a projectile at high velocity. Combustible cases may also be included as they contribute to the overall energy of the propellant.
 - Solid Rocket Propellant. Substance, or mixture of substances, which is required to burn in a controlled manner within a rocket motor producing hot gases which are vented through a nozzle to propel the munition.
 - Liquid Propellant. Substance or mixture of substances, which is required to react in a combustion chamber in a controllable manner in order to generate propulsive force. These may be mono-propellants, bi-propellants or hybrids comprised of liquids and solids.
 - Pyrotechnic Compositions. Substances, or mixtures of substances, which when ignited undergo an energetic chemical reaction at a controlled rate intended to produce on demand and in various combinations, specific time delays or quantities of heat, noise, smoke, light or infrared radiation. Pyrotechnic compositions may be used to initiate burning reactions such as in igniters.

Note: Pyrotechnic compositions may be Qualified for specific role(s) such as time delay, illuminating flare etc. The Qualification status is not valid for other pyrotechnic roles for which the composition has not been assessed.

A.1.1 Methodology for Qualification of new explosive materials

For assessment of new explosive material leading to its Qualification, the following shall be identified and form the basis of an EHDS to support material Qualification;

- The explosive composition and its intended role;

- The characteristics of the explosive material which are relevant to its intended role and any specific application that is envisaged;
- The characteristics of the explosive material in its powdered state, as-used condition and after possible degradation due to ageing and the service environment;
- The effect of temperature, particularly on the safety and performance characteristics of explosive materials;
- Characteristics of particular interest include thermal, mechanical, and electrostatic discharge sensitiveness, rheological and physical properties (for example, for thermal sensitiveness), the ignition temperature and effects of confinement, charge size and heating rate should be known;
- In the case of mechanical sensitiveness, the sensitiveness to shock, friction, impact, or to a non-penetrating object, such as a crush or drop, or the effect of confinement and charge size on explosiveness should be known;
- For electrostatic discharge, the materials sensitiveness to static electricity should be assessed;
- Knowledge of the variation of rheological and physical properties with temperature and age of the material;
- The toxicity and disposal data on the explosive material, its components, and its reaction products, in-so-far-as possible.

The National Authority shall identify the minimum data requirements necessary to fulfil the conditions of the test programme. This shall include, but not be limited to, the mandatory tests (for the intended role) given in STANAG 4170.

Table 20-2 provides explosive material characteristics that need to be determined. Additional tests may also be further referenced in AOP-7. Procedures and equipment detailed in appropriate STANAGs shall be used to conduct the test methods.

AOP-7 contains guidance on procedures where STANAGs for explosive material properties are not available; this is referenced in AOP-7 Chapter 10 National sections where individual national test requirements are mandated.

STANAG and AOP methods define appropriate tests and procedures for Qualification, and are used in conjunction with and cross reference UK Energetic Material Testing and Assessment Policy (EMTAP) test procedures. These are described in AOP 7 UK Chapter 10 test methodology and the UK EMTAP manual of tests. Reference should always be made to STANAG 4170 and AOP 7 UK Chapter 10 when qualifying energetic materials for UK service.

Table 20-2 Composition Details, Use and Safety – Mandatory Requirements

Composition and Safety - Mandatory Requirements		Type of Information
1	Details of the composition and its ingredients shall be given.	Composition and specification. Method of manufacture. Source of ingredients.
2	Details of its intended role and explosive type shall be specified.	Main warhead filling, booster charge, primary explosive, pyrotechnic, propellant etc.
3	Details of its form as used in service shall be given.	Pressed, cast, extruded, machinability etc.
4	Chemical and physical properties shall be given.	Acidity, alkalinity, density (also as percentage of theoretical maximum density), melting/softening point, etc.
5	Comparisons to other similar compositions (if any) already in service shall be specified.	Service applications of closely related compositions and their status in regard to Qualification.
6	Information pertinent to material toxicity and disposal shall be specified.	Inherent toxicity data and information on the ability to be reused, recycled or destroyed with minimum effect on health and environment.
7	Compliance with National Health and Safety requirements shall be given.	Material Hazard Data Sheet, etc.

A.1.2 General characteristics

Any EHDS will only relate to an energetic material made by a given manufacturer, by a specific method of manufacture, to a given specification, and at a given place of manufacture.

Energetic materials will be supplied with an Explosive Hazard Data Sheet (EHDS) which consists of two parts,

Part 1: *Contains general characteristics that must include:*

- Designation
- Physical form
- Manufacturers name, address and contact details
- Ingredients
- Chemical stability
- Incompatibility with other materials
- Health hazards
- UN hazard division data
- Fire extinguishing agents

- Recommended method of destruction
- Packaging and storage methods

Part 2: *Contains the sensitiveness characteristics such as:*

- Sensitiveness to mechanical nipping / impact between hard surfaces
- Sensitiveness to friction
- Ignition by flash
- Behaviour on inflammation
- Ignition by electric spark
- Additional information
- Explosiveness and charge scale sensitiveness
- Name of test centre carrying out the assessment

In some cases the National Authority may be supplied with Material Safety Data Sheets (MSDSs) which do not contain specific small scale hazard test information contained in Part 2. MSDS data can be used when assessing an energetic material for Qualification but full Qualification will not be achieved without sufficient small scale hazard classification data being provided.

A.2 Part 2: Sensitiveness Characteristics.

Part two of the EHDS shall contain sensitiveness (see 20.2.1) characteristics of the energetic material and as outlined shall give the results of the tests for sensitiveness to impact, friction, and ignition by electric spark and igniferous flash. It shall also give the temperature of ignition of the energetic material, its behaviour on inflammation and any additional precautions that are considered necessary.

Sensitiveness is the UK term for a measure of the level of a prescribed stimulus, relevant to an accident situation, required to ignite or initiate an explosive material. Such stimuli include, among others, impact, friction and electric spark. The significance of the word is thus in terms of the hazard characteristics of the explosive material.

Sensitiveness and Explosiveness test methods and interpretation of results are specified in appropriate STANAGs and the UK Energetic Material Testing and Assessment Policy (EMTAP) test procedures, Manual of Tests. A fuller appreciation of the main tests required for Qualification is contained in Appendix B.

Table 20-3 Sensitiveness Characteristic Tests Described in the EMTAP Manual

EMTAP Test No.	Scale *	Major Stimulus	Duration of Stimulus	Confinement**	Info***
1a Rotter F of I	VS	Crushing a thin layer	ms	T Heavy	S
1c Grit sensitisation	VS	Crushing a thin layer	ms	T Heavy	S
2 Mallet friction	VS	Shearing a thin layer	ms	T Moderate	S
3 Temp of ignition	VS	Heat	seconds to mins	Light	Thermal Stability
4 Bickford fuze	S	Hot particles and gases	~100 ms	Light	S, X
5 Train	S/M	Flame	s	Light	S, X
6 + 7 Electric spark	VS	Electric spark	µs	Light	S
33 Rotary Friction	VS	Shearing a thin layer	ms	T moderate	S

* VS up to 0.5 g, S 0.5 – 25 g, M 25 g – 0.5 kg

** T = Transient, *** S = sensitiveness, X = explosiveness

A.3 Part 3: Explosiveness and Charge Sensitiveness Characteristics.

Information from larger scale tests for explosiveness (see 20.2.1) and charge sensitivity, where the energetic material is in charge form, is not normally required for an EHDS but will be required as a part of the final Qualification of the energetic material.

Explosiveness is the UK term which describes the degree of violence shown by an explosive material when it responds to a prescribed stimulus relevant to an accident situation. It is dependent not only on the nature of the explosive

material, but also on its mass, physical state, configuration and confinement. The significance of the word is thus in terms of the hazard characteristics of the explosive material.

The sensitiveness and explosiveness testing of materials in charge-form and in quantities greater than those used in Part 2 may highlight specific situations where the hazard is high, or may cause the apparent hazard derived from Part 2 results to be modified. Charge test results are more relevant to the hazard present when larger quantities of material are handled.

Table 20-4 Explosiveness and Charge Sensitiveness Characteristic Tests Described in the EMTAP Manual

EMTAP Test No.	Scale*	Major Stimulus	Duration of Stimulus	Confinement**	Info***
22 Large Scale gap	M	Shock	μs	Moderate	SDT
35 Tube test Internal ignition	M	Hot particles and gases	50 ms	Heavy	X
36 Fragment impact test	M	Fast fragments	μs to 100s μs	Moderate	S,X
41 Tube test-fast heating (Fuel fire)	M	Heat	seconds to mins	Heavy	X
42 Tube test (Electrically heated)	M	Heat	seconds to mins	Heavy	X

* M 25 g – 0.5 kg

** T = Transient, *** S = sensitiveness, X = explosiveness, SDT = sensitiveness to shock initiation (shock to detonation transition)

A.4 Explosive Hazard Measurement

Sensitiveness and explosiveness information is used to monitor the potential hazard. Data is obtained from the small-scale (powder) and larger-scale (charge) tests previously mentioned. Results from tests can be compared with those from well-known compositions for which considerable experience has shown to give long and satisfactory service. The information is used in a formal sense to produce the EHDS, for the Qualification of explosives for service use

and in determining the classification of the explosive for storage and transport under the UN International System.

Traditionally, it has been assumed that explosives intended to fulfil a specific function possessed broadly similar safety properties (i.e. the designations - propellant, high explosive, pyrotechnic, initiator or booster could imply various degrees of potential hazard). Today it is realised that this is no longer necessarily so, and sensitiveness and explosiveness should be evaluated for each material of interest. In the search for increased performance, propellants are being produced which, compared to their predecessors, have reduced sensitiveness but increased explosiveness.

In the search for high explosives with lower potential hazard, polymer-bonded and other new secondary explosives are being produced which, compared to their predecessors, have increased sensitiveness and reduced explosiveness. Thus in hazard terms, and as a consequence in formulations, are blurring the distinction between propellants and high explosives. Similarly, some modern pyrotechnics are sensitive enough to be classed as primary explosives. Figure 20-2 shows typical regions in the sensitiveness/explosiveness plane occupied by the various types of explosive. The most hazardous materials are those furthest from the origin.

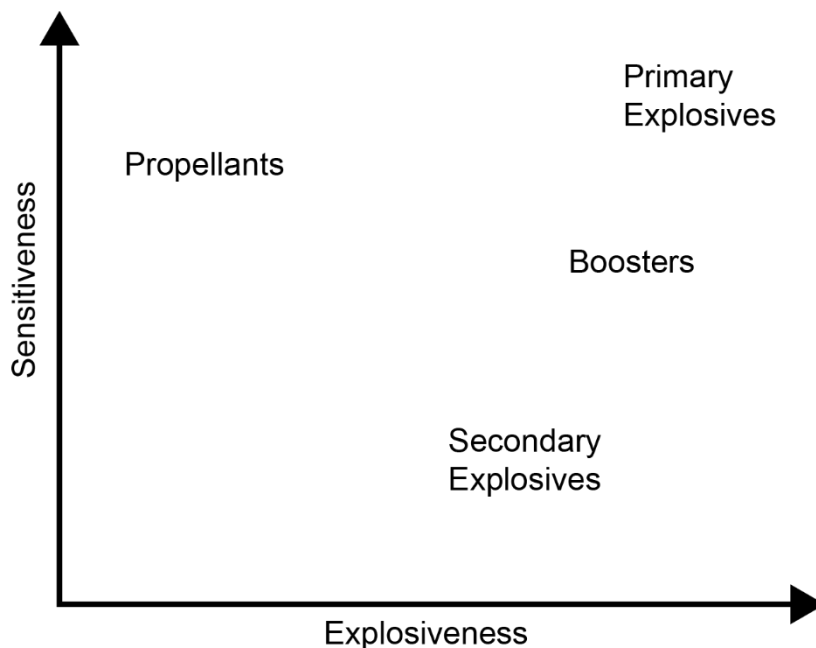


Figure 20-2 Sensitiveness/Explosiveness diagram

Apart from the monitoring of potential hazard, it is also desirable to gain a predictive capability in any hazard situation. It is clear that the probability of an event occurring is related to sensitiveness, whereas the magnitude of the event is related to explosiveness. Other factors such as explosive mass, confinement, physical form and environment also greatly affect hazard.

A.4.1 The Ideal stimulus/response curve

The ideal explosive would have a stimulus/response curve as shown by the dotted line in Figure 20-3 where no events occur below a critical stimulus level and above that level events occur in all tests. In a real situation, random variations in the explosive samples, in the state of the test machine and in the operator result in an 'S' shaped (sigmoid) stimulus/ response curve as shown by the solid line. Often data are analysed to give the '50% level', the value of the stimulus at which events would occur in 50% of the tests, e.g. Rotter Impact, Gap and Friction tests, using a technique such as the Bruceton Staircase (EMTAP Manual of tests). Although this is a useful concept for comparing explosives, it is often essential to have some idea of the probability of an event at low levels of stimulus. In this situation it is useful to analyse data by Probit Analysis so that extrapolation to low probabilities can be made using reasonably small sample sizes.

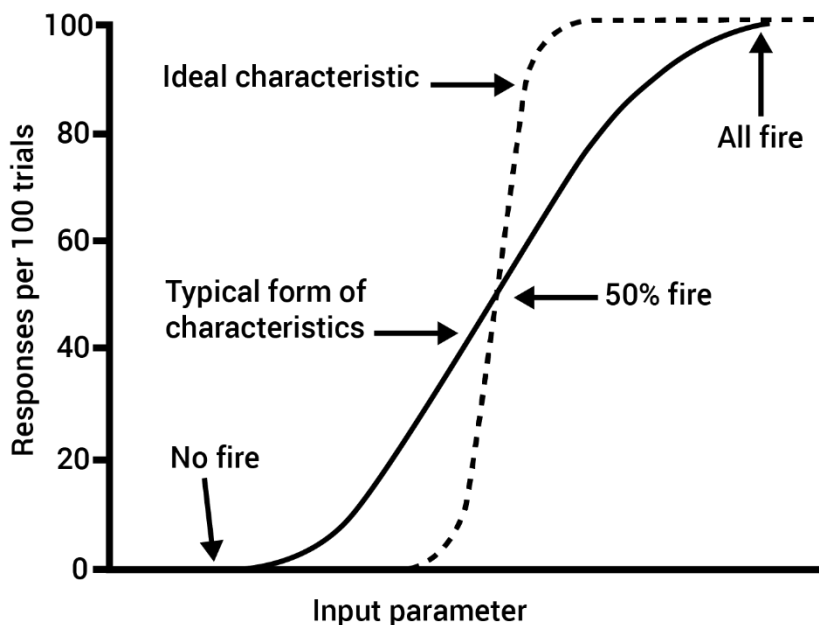


Figure 20-3 Basic stimulus-response characteristics of explosives

A.4.2 Probit analysis

In theory the sigmoid curve does not attain zero or 100 % response except at infinitely low or infinitely high stimuli respectively, whereas in practical tests it is usually found that there is a realistic lower limit at or below which no explosive substance or article will fire and a similar upper limit at or above which all explosive substances or articles will fire. The problem confronting the experimentalist with samples of limited size is the realistic evaluation of these limits. To obtain the limits it is usually necessary to extrapolate beyond the range of experimental observations.

Appendix B Material Qualification Tests from the EMTAP Manual

B.1 Introduction

The methods are specified in the Energetic Materials Testing and Assessment Policy Committee Manual of Tests (2016) (hereafter referred to as EMTAP Manual).

B.2 Safety tests

B.2.1 EMTAP Manual Test No 1: Figure of insensitiveness (F of I)

The Rotter Impact test assesses the impact sensitiveness of solid energetic materials. This is achieved by the transfer of energy from a falling drop-weight into a small test sample held within a gas tight system whilst monitoring the sample response. The drop-weight is 5 kg and drop heights vary between ~ 5 and 240 cm dependent on the susceptibility to ignition that the sample exhibits. Ignition is monitored by measurement of the evolved gas volume from reaction or from secondary ignition criteria (e.g. smoke, sample discoloration etc.).

The Rotter Impact test was originally developed to study the impact sensitiveness of crystalline secondary explosives. In the course of time and with appropriate modifications in procedure the test has been applied to all types of solid energetic materials and is now considered to be of universal application. It is a reproducible and versatile test, requiring only small quantities of explosive, and the basic action of the test is readily understood. The test can be used to quantify sensitiveness to impact, to assess relative sensitiveness and to investigate the effect of gritty contaminants on sensitiveness. A major advantage of the Rotter Impact test apparatus over other impact machines is the availability of gas volume data from ignition events and this gas volume data can be used to quantify small scale explosiveness.

The sensitiveness trials result in the calculation of a value termed the Figure of Insensitiveness (F of I). Ranking of F of I results from a range of explosives allows an order to be found that has been shown to correspond with practical experience in the handling of explosives. Surprise is often expressed at the relatively high sensitiveness (low F of I) to impact of double based propellants containing nitrocellulose and nitroglycerine where impact initiation is perceived to be Comparatively Insensitive. However accident data from such materials have shown that they are very sensitive when impacted in thin layers as is the case within the Rotter Impact test.

For the purposes of the UK MOD Explosives Hazard Data Sheets F of I results are divided into three categories shown in Table 20-5.

Table 20-5 Categories of Rotter Impact Sensitiveness

Figure of Insensitiveness (F of I)	Sensitiveness level
≤30	Very Sensitive
35-90	Sensitive
>90	Comparatively Insensitive

Grit sensitisation

This test assesses the sensitiveness of the explosive in the presence of grit and indicates the degree of hazard that would arise should an explosive filling become accidentally contaminated with hard foreign matter.

This method is as described in the Figure of Insensitiveness Rotter Impact Test No 1C with 0.1 % w/w and 1.0 % w/w grit by weight added to and intimately mixed with the test material. The EMTAP Committee on request will provide information on the nature and consistency of the grit to be used in this test and other details of the test.

B.2.2 EMTAP Manual Test No 2: Mallet Friction Test

The basic purpose of the Mallet Friction test is to provide part of the data needed to classify explosives into three main sensitiveness groups and this is achieved by the use of a series of mallets and anvils to obtain a graded response. Results from the mild steel surfaces may be taken to apply to any metal/metal combination. Results from the Yorkstone anvil may be taken to apply to concrete flooring.

Explosives giving ignitions on wooden surfaces are either inherently very sensitive to friction or sensitised by the addition of abraded wood/plastic particles which act as fuel. As a general rule, if 0 % ignitions are obtained on the mild steel anvil/mallet combination, the composition should not present handling difficulties from a frictional viewpoint, but care should be taken to avoid contamination from potential fuels.

In this test a small sample of explosive is spread onto an anvil and struck a glancing blow with a standard wood, steel-tipped, or nylon-tipped mallet held in the operator's hand. Approximately 0.1 ml of the test material is spread to give an area of about 6 mm by 12 mm on various anvils made of softwood, hardwood, Yorkstone and of mild steel, aluminium bronze and naval brass. The test material is struck up to 5 times using a standard wood mallet (for the wood and stone anvils) or a mild steel mallet (for the metal anvils). When an ignition takes place (shown by a crack, spark or flash), a fresh trace is tested. The operation is performed 10 times on each of the anvils.

The three main categories are classified as shown in Table 20-6.

Table 20-6 Categories of Mallet Friction Test Sensitiveness

A	Very Sensitive	100 % ignitions on metal and at least 50 % ignitions on Yorkstone and hardwood
B	Sensitive	All results excepting those in A & C
C	Comparatively Insensitive	No ignitions on any surface combination

Extreme care is required in handling compositions classed as “very sensitive”. Friction and impact between materials harder than softwood for those compositions classed as “sensitive” must be avoided. For “comparatively insensitive” compositions it is usual to recommend that blows between hard surfaces be avoided, but simple transfer between metal containers through pipes and valves is permitted.

B.2.3 EMTAP Manual Test No 3: Temperature of ignition

The actual values of “Temperature of Ignition” are meaningful only under the particular conditions used, as alterations in the rate of heating or quantity of explosive will give different results. Small weighed quantities of the explosive under test are heated in borosilicate test tubes placed in a steel block, the temperature of which is controlled to rise at a steady rate, until an event occurs. In this test a sample of the test explosive, 0.2 gram (or 0.05 gram for initiators) is heated in a test-tube held in an electrically heated metal block. The rate of heating is controlled to obtain a temperature rise of 5 °C per minute. The temperature of ignition is the temperature at which the sample inflames, explodes or undergoes rapid combustion.

Explosive samples giving results in the range 140 °C to 170 °C are found, in general, to have short service lives. Such samples need to be subjected to regular surveillance testing and must not be stored in close proximity to steam-heated pipes and radiators. A negative result, i.e. that reported as “not under 400 °C” should be treated with caution. It does not mean that the material has not decomposed, nor does it mean that it cannot be ignited, e.g. Pyrotechnic composition SR 371C does not inflame below 400 °C but is readily ignited in the Ease of Ignition Test (EMTAP Test No 4). Samples containing organic compounds that appear to give a negative result would normally be reported as “decomposes slowly”.

This test is also useful for:

- Screening candidate explosives (e.g. those with a “T of I” of less than 140 °C are not usually stable enough for Service use and can be ruled out);
- Distinguishing primary explosives from other classes (primary explosives explode at their Temperature of Ignition).

It is not a substitute for systematic stability testing.

B.2.4 EMTAP Manual Test No 4 Ease of Ignition (Safety Fuze) test

This test, previously called the Bickford Fuze Test, is used to assess the ease of ignition of an explosive by incandescence sparks and to ascertain whether it burns or explodes. The hazard category is affected by these two observations.

If a material ignites under the conditions of light confinement of this test it must be assumed that any non-electric spark will ignite it, the electrostatic risk being determined separately. Many flammable materials are not ignited by the short flash from the Safety Fuse so flammability, as opposed to ease of ignition, should be determined from a consideration of both this test and the Train Test. The physical form of the material can affect the results of the test, e.g. with certain pyrotechnic compositions, changes in the grain size of the individual ingredients, coupled with small changes in formulation, are enough to cause a change from “burning vigorously” to “explosion”.

If a material explodes in this test, it must be assumed that the material will explode on inflammation. A negative result must be supported by data from larger-scale tests, e.g. the sealed vessel tests and the tube tests.

In this test a sample of the powdered test material (3 gram) is put into a test-tube with a length of Bickford fuze resting on the surface of the material. The behaviour of the material is noted when the gunpowder core of the Bickford fuze burns through and the brief flash output impinges on the test material. The result is categorised as follows:

- fails to ignite
- ignites and burns quietly
- ignites and burns vigorously
- explodes

B.2.5 EMTAP Manual Test No 5: Train Test

This test determines whether an unconfined material in powder form will ignite if a flame is applied to it and assesses the degree of hazard arising from the ignition. An unconfined train of material is heated at one end by a naked flame for up to 2 minutes, and observations are made of the liability of the sample to burn and how vigorously it does so.

Many materials that are not ignited in the Ease of Ignition Test (EMTAP Test No 4) will burn when subjected to a naked flame of several seconds duration. Therefore, this test is used to determine whether a loosely or lightly confined material will ignite if a flame is applied, and to assess the hazard arising from an ignition.

The physical form of the material can affect the results of this test though the effect on the results is less than that experienced in EMTAP Test No 4. If an explosion is obtained under the conditions of this test then the material must be treated as an explosive risk. If the material burns vigorously, i.e. burns rapidly with or without sparks, or more slowly with copious sparks, it will obviously lead to a rapidly spreading fire. However, such a reaction does not necessarily

mean that the material will not exhibit an explosive risk when stored in bulk. The test is incapable of deciding this, and differences between explosive and fire risks are best made by means of sealed vessel tests, and/or tube tests. This aspect must also be considered in relation to materials placed in other categories by this test, since it has to be remembered that this test is carried out at virtually atmospheric pressure, whereas, in the real case, sufficient confinement may allow an increase of pressure leading to an increase in rate of burning.

The resultant behaviour of the explosive is observed and recorded as follows:

1. Fails to ignite (FI)
2. Ignites but fails to support train for more than XX mm
3. Ignites but supports train fitfully (ISTF)
4. Ignites and supports train steadily throughout (ISTST)
5. Ignites and supports train vigorously throughout (ISTVT)
6. Explodes (E)

B.2.6 EMTAP Manual Test No 6 and 7: Ignition by Electric Spark

This test has been devised to provide a fairly simple means of distinguishing between those explosive materials which need a relatively high energy for ignition by electrostatic sparks and those which need a relatively low energy. The dividing line between two such groups of risk was suggested by the maximum amount of electrostatic energy that can be accumulated on a person in the UK which is believed to be 0.02 J. In this test the lowest energy was fixed at 0.045 J, i.e. a safety factor of approximately two. Energies of ten times and one hundred times this were chosen for the higher levels, i.e. 0.45 J and 4.5 J. However, it has been found that this energy of 0.02 J is rarely attained and it is more realistic to regard 0.001 J as being the likely maximum. Consequently, a subdivision into three groups of risk is more appropriate for the UK, but this will not necessarily apply in other countries as the ability to store and discharge an electrostatic charge depends on several factors including humidity.

The highest energy group comprises those compositions needing energies in excess of 0.45 J and for which First Degree Precautions are sufficient. Compositions in this group are classified as "Comparatively Insensitive" to electrostatic spark. Full Second Degree Precautions are necessary for those compositions that have a 'minimum ignition energy of less than 0.001 J, i.e. those classified as "Very Sensitive".

Those compositions having ignition energies in the range 0.45 J to 0.001 J, are classed as "Sensitive", and form an intermediate class in which the precautions to be applied are gradually increased from First Degree towards Full Second Degree Precautions as the minimum ignition energy decreases from 0.45 J to 0.001 J.

The tests are carried out in two stages. The first stage is to determine whether an electrostatic spark with an energy of 0.045 J ignites the material. If the material is sensitive to a spark of that energy, then the second more searching

test is carried out to determine the minimum energy required for ignition. In each test the discharge from a charge capacitor is passed through the material, which is normally tested in a powdered or granular state.

B.2.7 EMTAP Manual Test No 22A. (The Large Scale Gap Test).

The Large Scale Gap Test is used to compare the shock detonation sensitiveness of a wide range of explosive materials having critical diameters up to approximately 100 mm. A witness plate is used to distinguish detonation of the test explosive from sub-detonative reactions.

The test uses a reproducible attenuator material accurately calibrated in terms of attenuator thickness versus shock pressure using a standard explosive donor. Following the US convention, the results are expressed as the pressure just inside the attenuator at the attenuator/acceptor interface at the gap thickness which produces a 50 % probability of detonation in the test explosive.

The test is based on the US Naval Ordnance laboratory (NOL) Large Scale Gap Test, and results obtained can be compared with the published US data. The material under test, in the form of a cylindrical pellet or casting (140 mm x 36 mm) is subjected to the shock generated by the detonation of a standard donor pellet. The donor pellet is Rowanex 3601 (50 mm x 50 mm) pressed to a density of 1600 kg m³. The test explosive is confined in a steel tube which is inserted into a cardboard tube containing the Rowanex 3601 pellet. Interposed between the donor or acceptor pellet is a barrier of polymethylmethacrylate (PMMA) (50 mm diameter) of density 1186 ± 1 kg m⁻³ and thicknesses ranging from 5 mm to 70 mm in 1 mm increments.

A steel witness block is used to assess the reaction of the test material. The thickness of the barrier is varied to determine the thickness at which there is a 50 % probability of detonation of the material under test. The criterion used to distinguish between a detonation and a non-detonation is that for detonation a clean hole is punched in the witness plate.

B.2.8 EMTAP Manual test No 33: Rotary Friction Test

This test is designed to provide a quantitative assessment of the friction sensitiveness of a wide range of solid explosives. Unlike the Mallet Friction Test (EMTAP Test No. 2) there is no element of impact in the stimulus. Although any wheel and block materials of interest can be used, the standard test uses mild steel blocks and wheels of standard surface roughness. Results are expressed relative to those for standard RDX by assigning a value of 3.0 to the Figure of Friction for the standard RDX. This value was chosen because it is close to the actual median peripheral wheel velocity (m/s) when testing the standard RDX.

Energy stored in a flywheel imparts an impulse to the wheel assembly, causing it to rotate. This rotation generates high shearing forces in the sample, which is under a pre-set load between the wheel and the stationary block

In this test a Bruceton Staircase procedure is used over 50 trials to establish a median flywheel angular velocity for the test material. Results are expressed in terms of Figures of Friction relative to standard RDX.

For MOD Explosives Hazard Data Sheets the Figure of Friction is normally quoted to two significant figures. Broad sensitiveness categories can be assigned as shown in Table 20-7 to conform to those used in the Explosives Hazard Data Sheet.

Table 20-7 Categories of Rotary Friction Test Sensitiveness

Very sensitive	Cat A	FoF < 3
Sensitive	Cat B	FoF >3 but <6
Comparatively insensitive	Cat C	FoF > 6

B.2.9 EMTAP Manual test No 35: Tube Test – Internal Ignition

A sample of explosive, confined in a steel tube, is ignited by means of a charge of propellant. The degree of fragmentation of the tube is used to assess the relative explosiveness of the composition under test.

The internal ignition version of the Tube Test can be used to evaluate the explosiveness of solid high explosives and propellants. It assesses the behaviour of explosive charges when ignited under confinement. The level of violence of this reaction, and its potential to cause damage to the surroundings, is referred to as "explosiveness".

Explosiveness is influenced by several variables including the nature of the stimulus, the temperature and physical form of the explosive and its confinement. The Internal Ignition Tube Test has been developed to assess explosiveness under one set of well-defined conditions. These conditions are more representative of a moderately confined explosive in munitions such as shells but the data generated form a basis for comparing the relative explosiveness of energetic materials. Because the test is relatively cheap and simple to perform it is used routinely in assessing the potential hazards of new explosive formulations.

In this test the explosive may be cast, pressed, extruded, or pre-machined to shape and fitted into a mild steel tube 253 mm long, i.d. of 31 mm and a wall thickness of 6 mm. The ends of the tube are sealed with heavy, mild steel, screw caps. The charge is ignited at one end using an electric match head and a 1 g charge of Bullseye or M9 propellant.

The number of fragments recovered provides the most useful indication of the relative explosiveness of the material under test. In those experiments where less than 100 % of the test vehicle is recovered, the number of body fragments may be divided by their fraction of the intact vehicle to give a "normalised number of body fragments". This number is then used to assign a reaction

category to the event using the descriptions listed in Table 20-8. For clarity, the reaction description should be included as well as the category when reporting the results

Table 20-8 Description of Tube Test Reaction Categories

Category	Reaction Description	Observation
0	No Reaction	From weighting
0/1	Burning decomposition	No disruption of test vehicle
1	Pressure burst due to burning/decomposition	Assembly ruptured but one fragment approximates to original weight
2	Deflagration	2 to 9 body fragments
3	Explosion	10 to 100 body fragments
4	Detonation	>100 test vehicle body fragments showing evidence of detonation

B.2.10 EMTAP Manual Test No 36: Fragment Attack Test

The fragment attack of confined explosive charges invariably produces disruptive events at velocities much below those required to initiate detonation via a shock mechanism. It is important to assess the vulnerability of explosives to fragment attack in this lower velocity region since the occurrence of a violent deflagrative event in a munition may pose a hazard equal to that of a detonation and in some cases it may be possible for an initial deflagration to burn to detonation ([link to 5.1.2.1](#)). The processes which control the response of an explosive to fragment attack are complex and are very susceptible to changes in test parameters such as case thickness, charge size and fragment mass. However, it is possible to obtain a preliminary assessment of the vulnerability of explosive compositions to fragment attack by selecting a standardized set of test parameters. The Fragment Attack Test has been designed for this purpose and allows the study of fragment-initiated reactions to be undertaken at velocities up to 2500 m s⁻¹.

The projectile employed is a 13.15 mm diameter, 25.4 mm long mild steel cylinder. It is fired from a smooth bore gun at velocities in the range 400 to 2000 m s⁻¹ to impact normally on the septum of the assembled test vehicle. Greatest control of fragment impact velocity is achieved by employing a modified 0.5 in (12.7 mm) Browning gun for velocities up to 1000 m s⁻¹ and a modified RARDEN (30 mm) gun with a sabot projectile for higher velocities. However, a gas gun or other gun giving similar control to the above over velocity and fragment orientation may be used.

The response of the explosive charge to the projectile impact is assessed in terms of the damage done to the test vehicle according to the categories listed in Table 20-9.

Table 20-9 Description of Fragment Attack Test Reaction Categories

Category	Reaction Description	Observation
0	No Reaction	No visible sign of reaction after penetration of the septum by the projectile.
1	Burning	Hint of a burning reaction which has faded rapidly, no obvious consumption of explosive.
2	Deflagration	Detachment of the septum, up to 20 % of the explosive consumed.
3	Explosion	Septum detached, vehicle intact or broken into large fragments, more than 20 % of explosive consumed.
4	Detonation	100 % of explosive consumed in a very violent reaction characterised by breaking up of vehicle into very many small fragments showing evidence of shear failure.