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Assessment of Self Ignition Temperature (Cook-Off) in Gun Barrels

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Supervisor: Dr Amer Hameed
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the degree of Degree of Master of Science

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ABSTRACT

When an explosive (gun propellant or combustible cartridge case) is brought in contact with a hot surface its temperature increases and there is a corresponding increase in chemical reaction rate (decomposition) where self heating may occur. If the surface temperature is sufficiently high then the reaction rate exceeds that of the heat lost to the surface and the material ignites or 'Cooks-Off'.

Cook-Off in gun barrels can lead to a violent and damaging response affecting operator safety and/or create a lack of capability in a battle fighting system. Numerous tests and experiments show that a time to cook-off varies with time vs. temperature where short durations of temperature will have higher cook-off assumptions. Extrapolation of these results predict that cook-off will occur in 1800s and that if a gun barrel does not exceed 180°C then the the propellant will not ignite spontaneously.

Gun systems may cool linearly or exponentially; decay varies from 200s for small calibre guns to about 20,000s for large calibre guns. This assessment will investigate a variation of time/temperature using small calibre 7.62mm cartridge cases filled with Double Base (DB) propellant using a locally manufactured experimental apparatus to measure cook-off temperatures of the cartridge case and propellant. In essence, the locally designed equipment will simulate the firing of a small calibre weapon and record thermal cook-off data. These results will be analysed and compared with existing data with the aim of using this supporting evidence of cook-off temperatures to underpin any increase to rates of fire in gun systems.

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LIST OF ABBREVIATIONS

AAAV	Advanced Amphibious Assault Vehicle
ACA	Ammunition Container Assembly
ARC	Accelerated Rate Calorimeter
APDS	Armour Piercing Discarding Sabot
AWE	Atomic Weapons Establishment
CCT	Confined Cook-Off Test
COTS	Commercial Off the shelf
DB	Double Base
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
EM	Energetic Materials
EOE	Explosive Ordnance Engineering
FAP	Frangible Armour Piercing
FCO	Fast Cook-Off
FFV	Forenda Fabrikssverken Karlsburg
FNH	Flashless, Non-Hygroscopic
FNM	Fabrica National de Municoes de Armes Lagerias
HEI-T	High Explosive Incendiary with Tracer
HITP	High Ignition Temperature Propellant.

HMX	High Melting Explosives (Cyclotetramethylenetetranitramine)
HPLC	High Performance Liquid Chromatography.
HT	Heat Transfer
H-W-S	Heat – Wait – Seek
MHDS	Material Hazard Data Sheet
MPLD-T	Multi-Purpose, Low Drag with Tracer
MRCA	Multi-Role Combat Aircraft
MSIAC	Munitions Safety Information Analysis Centre
N	Neolite
NATO	North Atlantic Treaty Organisation
NC	Nitrocellulose
NEQ	Net Explosive Quantity
NG	Nitro-glycerine
NH	Non-Hygroscopic
Nigu	Nitroguadine
NIMIC	NATO Insensitive Munitions Information Centre
NNN	Nobels Nitro Neolite
NRN	Nobel's Rifle Neonites
PETN	Penterythritol Tetranitrate
Q	Heat of Explosion
ODTX	One Dimensional Time to Explosion
RDX	Research Developed Explosives (Cyclotrimethylenetetranitramine)

S ³	Safe and Suitable for Service
SAA	Small Arms Ammunition
SB	Single base
SC	Solventless Cordite
SCB	Small-Scale Cook-Off Bomb
SCO	Slow Cook-Off
SEAP	Self Extinguishing at Ambient Pressures
SSCB	Super Small-Scale Cook-Off Bomb
STANAG	Standard NATO Agreement
STEX	Scaled Thermal Explosion Equipment
TA	Thermogravimetric Analysis
TAGN	Triaminoguanidine Nitrate
TC	Thermocouple
TGA	Thermal Gravimetric Analysis
THA	Threat Hazard Assessment
THT	Thermal Hazard Technologies
TMXR	Thermal Mechanical Explosive Response
W	Waltham Abbey propellant (now obsolete)
VCCT	Variable Confined Cook-Off Test

CHAPTER 1 Introduction

1.1 Introduction to Gun Propellants

Conventional gun propellants are mixtures of one or more energetic materials with additives fabricated into granular or cord like forms which in ideal conditions burn in a controllable way (relatively slow and in an even way when compared to primary and secondary explosives). The rate and pattern of burning may be varied to suit the internal ballistic requirements of a particular system by varying the chemical composition of the propellant, and the geometry of the grains or sticks in which the propellant is produced. The rate of propellant consumption is proportional to the surface area, and an increase in the total surface area of a charge increases the rate at which the gas is produced, and hence the rate at which the pressure increases. Therefore, charges consisting of small grains will burn faster because the greater the surface area in relation to the mass of the charge (Davies 2008).

Modern gun propellants are smokeless and have replaced gunpowder as a propellant for conventional guns. British service ammunition for both guns and small arms contains solid colloidal propellants in granular form which in essence have changed little in since the turn of the 20th century.

In theory, many stable explosive compounds of low volatility which do not very readily burn to detonation are candidate ingredients of gun propellants. During World War II the Germans used di-ethyleneglycol dinitrate (DEGDN) and the Americans tried tetramethylolcyclopentanone tetranitrate (fivonite) to replace Nitro-glycerine (NG). In more recent times countries have replaced NG in propellants with ethyleneglycol dinitrate (EGDN) which is more energetic and not having the drawback of being more volatile. As a rule of thumb most conventional gun propellants fall into one of three compositions; single, double or triple based.

- a. Single Based (SB) Propellants - NC is the only explosive ingredient and employed in all kinds of guns (pistols to artillery weapons). Their Heat of Explosive (Q) value is generally between 3100 Jg^{-1} and 3700 Jg^{-1} .
- b. Double Based (DB) Propellants - In addition to NC these propellants contain NG (or similar liquid nitrate ester). They are more energetic than SB propellants, having Q values between 4300 Jg^{-1} and 5200 Jg^{-1} depending on the percentage of NG present.
- c. Triple Based (TB) Propellants - In addition to NC and NG, Nitroguadine (Nigu) and/or RDX are the additional energetic constituents. TB propellants were developed during the World Wars to reduce muzzle flash and making gun positions less easy to locate during night firing (usually large calibre guns). The Q values between 3200 Jg^{-1} and 3700 Jg^{-1} but newer variants contain RDX have much higher values.

In addition to these energetic compositions there may be non-energetic additives which will act as a stabiliser, coolant, surface lubricant/moderant, plasticiser, anti-wear additive, decoppering agent and flash inhibitor. NC and NG are nitrate esters and decompose slowly at normal temperatures to form oxides of nitrogen, mainly di-nitrogen tetroxide (N_2O_4). If these oxides are not chemically removed from the system as they are formed, they catalyse further the decomposition rate and thus shorten the storage life of the propellant and can, in some cases, lead to auto ignition. It is stabiliser that is common to all conventional propellants and the most important on the propellants decomposition rate in relation to aging and elevated temperatures (JSP 333).

Increased temperatures are one of the many 'stimuli' that result in reaction outside its normal burning rate function inside the chamber of a gun system. NC has a relatively low ignition temperature of about 170°C and once ignition is caused by external stimuli the burning rate at atmospheric pressure is sufficiently high to ensure the propellant charge is rapidly and completely consumed, with highly incendiary effect (JSP 333).

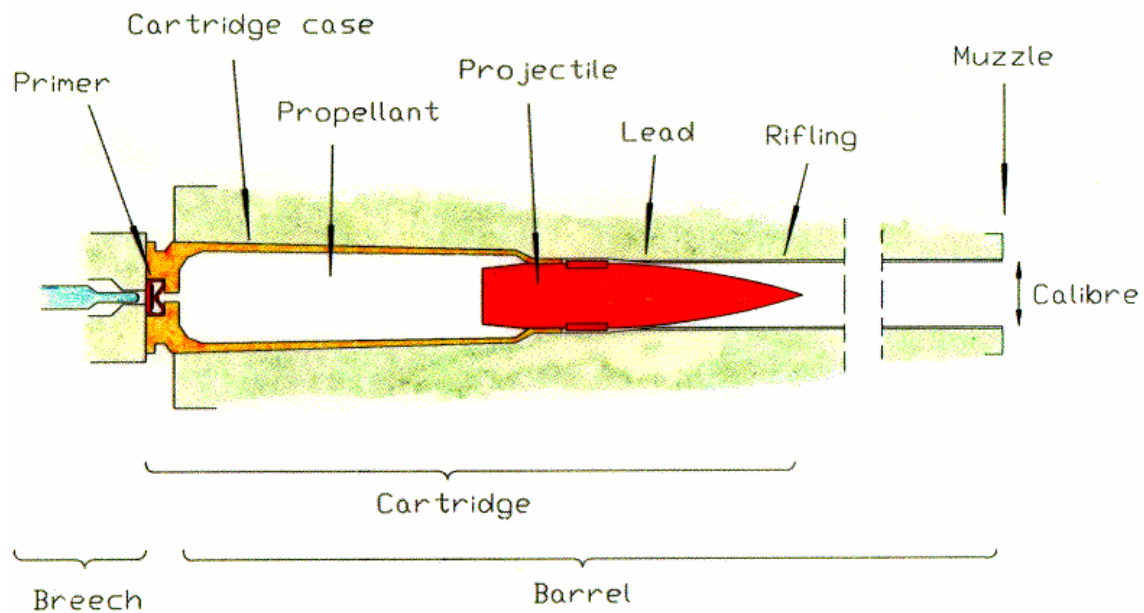


Figure 1.1 Example of a conventional round in a chamber of a gun (Knock 2009)

The normal ignition process in the gun chamber is the heat/flash mechanism from the primer to the propellant. Alternatively the surface temperature from the chamber (breech and cartridge case combination) can be sufficiently high that the reaction rate exceeds the heat lost to the surface and the propellant ignites. This is referred to as 'Cook-Off' – the steady temperature at which the heat generation and the heat loss are equal to the cook-off temperature of the propellant/cartridge case material.

Accidents involving cook-off have been well documented through research reports and data collection. Marchandin and Sharp (2002) describe many examples of cook-off accidents but many detailed within the study focus on 'fuel fires' from an external source rather than radiated heat accumulated from repeated firings from the parent gun which may eventually lead to cook-off its own ammunition when fed in to the chamber. However, the Ministry of Defence (MoD) MID (2012) highlights 277 of accidents and incidents from 2003 to 2012 there have been from gun barrel cook-offs. These include 156 serious accidents which include injury to personnel occurring from breech explosions resulting in facial lacerations and gas wash burns. Included within these

statistics are 49 major incidents which have resulted in damaged to equipment and the loss of capability.

This assessment will investigate the current tests available to aid in the assessment of cook-off prediction and devise an experimental method / mechanism that simulate a propellant in contact with a rising and decaying temperature similar to that found in guns.

CHAPTER 2 Literature Review of Cook-Off Testing

2.1 Theory of why Propellants Cook-Off

The rate at which gas is produced during the burning phase depends on the rate at which the mass is consumed, and this depends on the surface area of the propellant grain over which the consumption can occur. These rates can be:

- a. Progressive – mass flow rate increasing with time.
- b. Regressive - mass flow rate decreasing with time.
- c. Neutral – mass flow rate being constant.

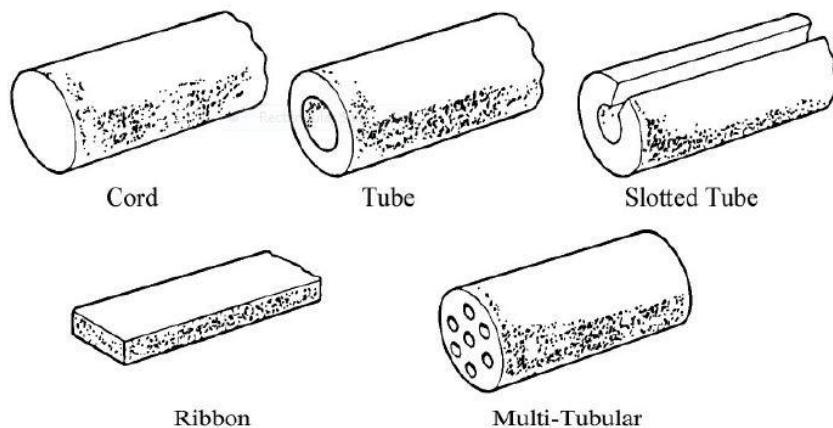


Figure 2.1 Typical propellant grain geometries (Davies 2008)

All conventional propellants contain NC, in the form of a gel, together with other ingredients which may be explosives or non-explosives. The compound is correctly named cellulose nitrate (not NC) since it contains the characteristic nitrate ester grouping (C-O-NO_2). All grades of NC have a negative oxygen balance, but the higher their percentage of nitrogen and oxygen the higher their Q value and the temperature achieved by their combustion.

2.1.1 The Ignition and Burning of Propellants

The effectiveness of gun propellants depends on two factors:

- a. The quantity of energy available.
- b. The rate at which this energy is released.

Although modern gun propellants are mixtures of explosives, the conditions in which they are used are designed to ensure that detonation does not occur.

This ignition process is essentially a thermal phenomenon, in which the propellant surface is heated from initial temperature (T_p) to the ignition temperature (T_i) which is typically in the range of 160°C - 175°C. At this point explosive burning develops with the rapid liberation of the Q of the propellant.

Propellant	Ignition Temperature (°C)	Propellant	Ignition Temperature (°C)
WM ¹	163	FNH (SB)	165
SC	165	NH (SB)	170
N (TB)	165	NRN (SB)	175
NQ ² (TB)	165	NNN (DB)	175

Table 1: Examples of ignition temperatures of propellants (JSP 333)

¹ A modified version of propellant 'W'

² A current UK gun propellant, initials including a hotter variant of the standard 'N' composition.

2.1.2 The Rate of Burning

The majority of propellants are non-porous to provide ballistic regularity but some types of propellant are designed to produce porosity in the grains (necessary for short barrelled weapons like pistols). When the propellant is ignited, the flame penetrates the pores of the grains and so the rate of burning increases rapidly. In 1839 Piobert showed that the burning of non-porous propellant is a surface phenomenon, in which the surface recedes layer by layer, in a direction normal to the surface at every point. The rate of burning (r) in mm^{-1} is the velocity at which the burning surface recedes along a line normal to the surface. The rate of burning of a propellant is governed by the instantaneous pressure (P) of the given propellant. In 1862 de Saint Robert carried out a number of experiments and expressed this in a burning rate equation:

$$r = \beta P^{2/3}$$

(where β is a constant for a particular propellant, or burning rate coefficient)

Equation 1: Rate of burning (de Saint Robert)

This equation was put into a more general form by Vieille in 1883 after experimenting with SB NC propellants:

$$r = \beta P^\alpha$$

(where α is the pressure index)

Equation 2: Rate of burning (Vieille)

In practice the 'strand' of propellant is inhibited by a coating of flame retardant material with only the ends exposed and the burning rate can be measured in a 'strand' burner. This consists of a closed, isobaric bomb, filled with an inert gas (i.e. argon), at a predetermined pressure and provided with electrical ignition and a means of measuring the time taken for the flame to travel down the

strand. The propellant should be conditioned to a standard temperature (298K or 25°C) before use. This is because the r has small but significant temperature dependence.

2.1.3 Effect of Propellant Temperature on Burning rate

From a discovery made by Muraour (JSP 333), the value of r is dependent on the initial temperature T_p of the propellant as well as the chamber pressure. The equation can now be written more correctly as:

$$^{(r)}T_p = \beta P^\alpha$$

Equation 3: Initial temperature of a propellant (Muraour)

As the flame front travels into the propellant, each successive layer ahead is heated by radiation, conduction and the heat of its own decomposition until ignition temperature is reached. The higher the initial temperature, the less heat is required to raise unit mass of propellant to its ignition temperature, values of r and mass burning rate (m^*) will increase. To determine the temperature dependence of the r an empirical equation is used:

$$r = \beta P^\alpha f(T_p)$$

(where $f(T_p)$ is an increasing function of T_p)

Equation 4: To determine the temperature dependence on r

This function is approximately linear over the range -40°C to +60°C, then:

$$f(T_p) = 1 + c(T_p - x)$$

(where T_p is in Kelvin, c is the temperature coefficient of the particular propellant composition and x depends on the reference temperature applying to β . Values of c are of the order of 0.002°C at 300 MN m^{-2})

Equation 5: Change of Equation 3 when linear temperature range exceeded

The effect of a change in temperature on burning rate is small in relation to the pressure dependence, but is large enough to cause appreciable differences in gun chamber pressures, and hence muzzle velocities, at extremes of charge temperature (this could be several hundred metres for a 105mm calibre at extreme range).

2.1.4 Availability of Heat Resistant Propellants

Most cartridges cases are made of 70/30 brass which have been used for over 150 years (although aluminium, steel, polythene and even paper are also in use). Brass has a number benefits over its contenders for its mechanical properties but costs have risen and being a dense material it accounts for 50% of the total weight of the cartridge. This led to research in the 1960's into the caseless round. They sought a propellant composition which would not self ignite in a chamber below 300°C. Such a composition was designated High Ignition Temperature Propellant (HITP), but this was not sufficient: it was required also to be resistant to accidental ignition outside the chamber (flash / sparks) and this is described as Self Extinguishing at Ambient Pressures (SEAP). A number of ideas were tried, including a resistant outer covering around an NC core, an HMX/Polyurethane propellant and combinations of HMX, Triaminoguanidine Nitrate (TAGN) and plastic matrix. Some of these succeeded the heat treatment requirements but failed on internal ballistic properties and residue-free combustion. The caseless round was never developed satisfactory and even with heat resistant materials the round cooked-off at circa 300°C when trials suggests that the chamber temperature of a light support weapon may reach 400°C. Although it is argued that any viable propellant could reach this degree of insensitiveness to heat while still remaining susceptible to ignition by the normal process, and retaining its ability, once ignited, to burn rapidly and completely. In defence of caseless cartridges, if cook-off did occur it would be less dangerous than brass cases because the latter would scatter bullets and brass fragments and caseless would just flare without any missile effect (JSP 333). However, research conducted by Spiegel

(2006) and Wando (2006) detail a renewed interest on improved caseless and telescopic HITP SAA with improved S^3 properties (i.e. prevention of accidental ignition from external stimuli). The technology drive is to support the decrease in weight that the modern combat soldier requires because of the extra technology (i.e. addition personal weapon fittings such as grenade launchers, thermal imaging scopes), extra ammunition and body armour he now carries compared to that of a soldier in the 1980s.

2.2 Propellant Cook Off in Guns

Lawton and Klingenberg (1996 pp443 - 446) stated the time to cook-off for the propellant charge of a 120mm tank APDS round is about 1 second (s) at about 250°C and increases to about 100s when the surface temperature is about 200°C. After a series of results have been obtained a curve is drawn and extrapolated to give a prediction of the surface temperature at which cook-off occurs in 1800s. This is taken to be the cook-off temperature and it is argued that if a gun barrel does not exceed this value (typically circa 180°C) then propellant cook-off will never occur. The time constant for exponential decay for typical guns varies from about 200s for small calibre guns to 20,000s for large calibre guns. Given the time constant, the initial contact temperature at which the time to cook-off is infinite is then determined.

This could turn out to be considerably higher than the conventional cook-off temperature and means that the gun may fire significantly more rounds before it reaches its cook-off limit without sacrificing the 1800s margin of safety.

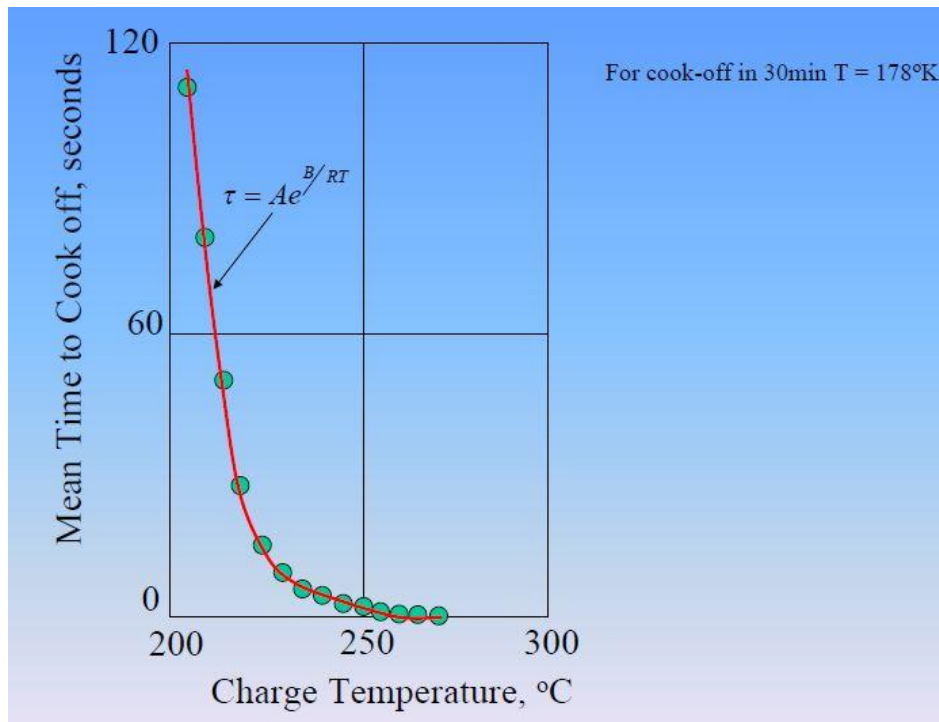


Figure 2.2 – Time to self ignition for combustible cartridge case material in contact with a hot surface (Lawton and Klingenberg 1996 p443)

There are a number of testing mechanisms of evaluating testing the cook-off of energetic materials which are covered in STANAGs 4240 and 4382 to comply with the NATO IM policy directed by STANAG 4439. The baseline heating trials are discussed in the next section and examples shown in an EOE presentation by Stennett (2009):

2.3 Slow Cook-Off Full Scale Trials (STANAG 4382)

This type of trial uses a controlled heating environment where the munitions being tested are heated at a rate of 3.3°C / hour. This rate simulates the heat increase from adjacent compartments (i.e. ships magazines during a fire situation). Grauw (1998) argued that this rate seemed low, however it is the test currently recommended in the STANAG 4439 test that NATO countries should abide to in assessing Insensitive Munitions (IM) response levels.



Figure 2.3 Slow cook-off (large)



Figure 2.4 Slow cook-off (small)

The Slow Cook-Off (SCO) for a large munition shown in figure 2.3 is a Rapier missile rocket motor and it would be heated from ambient temperature until a response is observed. For small SCO trials, figure 2.4 shows an example of a rapier missile warhead which is instrumented with thermocouples. The heating rate would typically be $3.3^{\circ}\text{C} / \text{hour}$ unless a THA indicates a more credible heating threat and a different rate is required.

2.4 Fast Cook-Off Full Scale Trials (STANAG 4240)

This method is used to assess the reaction of munitions from exposure of a fuel fire. The munition would be placed in an open container with several hundred litres of diesel applied and ignited. The temperature of the munitions and diesel flame temperature would be measured at the time of the first explosive event.



Figure 2.5 – Mini-Fuel Fire



Figure 2.6 – Large Scale Fuel Fire

The small scale fuel fire test illustrated in figure 2.5 would be suspended approximately 300mm above a pool of the hydrocarbon fuel, ignited and

expected to last for 150% of expected time to reaction. Sufficient space is required around the sides of the test item to ensure full development of a flame. In large scale tests the design can vary from a small open pit in the ground, a steel hearth with integral fuel pan or a large 'Pendine' block enclosure around a large pit (as shown in figure 2.6). The test sample would be instrumented with at least four thermocouples. The heating regime should reach 550°C within 30s of ignition and sustain at 800°C throughout the test.

2.5 Test Methods for Small Scale Fast and Slow Cook-Off

There are a number of Small Scale Trials available to establish cook-off and these have benefits to modellers, safety authorities and those conducting THAs. Peugeot and Sharp (2003) conducted a review of mechanisms and tests available for cook-off assessments and are explained and evaluated as follows:

2.5.1 Small Scale Cook-Off (SCB) Test

This test used to be one of the tests to reproduce the heating effects seen by an explosive or propellant in bombs, munitions and missiles in an aircraft fuel fire. An explosive mass of up to 700g is placed in a single steel sealed cylinder with a welded base and threaded screw-on cap, which is located between two 127mm square, 12.7mm thick steel plate and held together by four bolts (shown in figure 2.7). The substance to be tested is loaded into the SCB steel vessel to within 10mm (approx.) of the top. The space remaining above the substance of interest allows for thermal expansion. This test has been used to assist in modelling heat transfer to understand and simulate cook-off. An example of a SCB test was conducted by Pakulak (1984) and the apparatus heated the sample between 0.2°C/s and 3°C/s (using 110 and 220 heater voltages). A DB gun propellant under test cooked off (deflagration event) at 201°C in 4.2 mins using 110v heater and another SB NC propellant cooked off (burning event) at an unrecorded temperature in 1.3mins using a 220v heater.

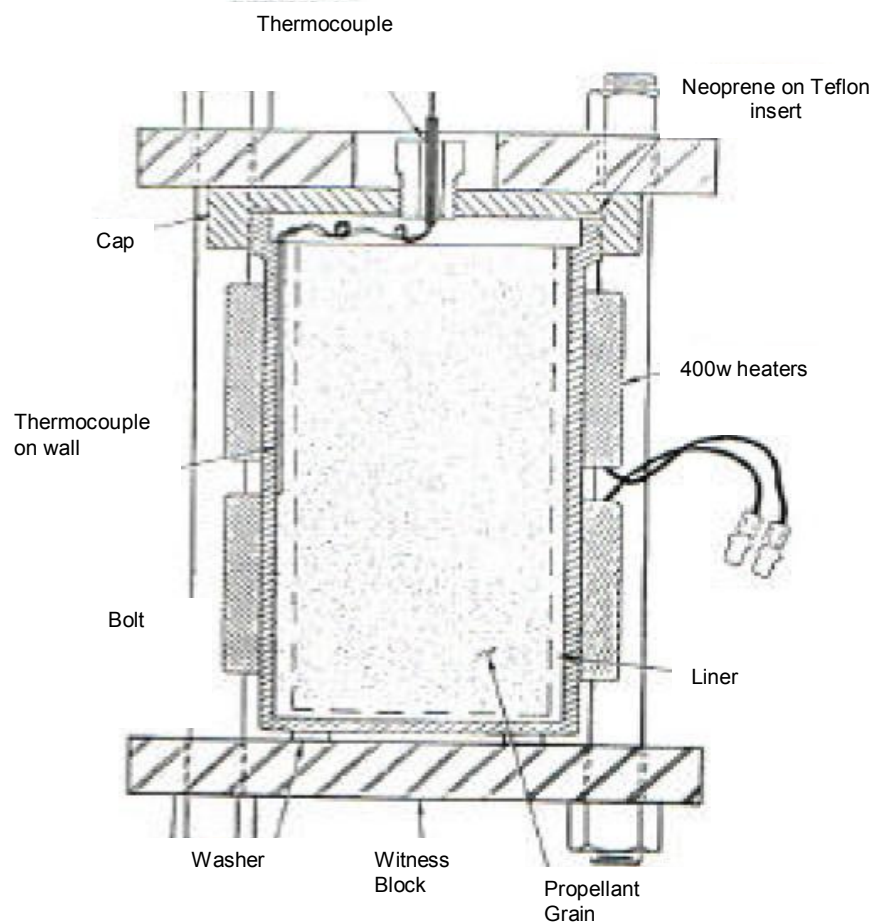


Figure 2.7 Typical SCB Set Up (Peugeot and Sharp)

When only one substance is being tested, the SCB is instrumented with one thermocouple spot welded to the centre of the vessel wall.

2.5.2 Super Small Scale Cook-Off Bomb (SSCB) Test

The test consists of an outer cylinder which is spot welded to a steel base plate. Two band heaters are clamped around the cylinder with hose clamps and an aluminium line with a slot for a thermocouple is placed inside the outer cylinder (this cylinder assists in evening out the heating delivered to the sample from the heaters). This test uses a sample of approximately 20g of Energetic Material (EM), in a cylinder measuring 15.9mm in diameter and 63.3mm in length. The EM is placed inside a confined metal tube that is heated by controllable electric

heating bands (figure 2.8). The test has been used to assist in determining the severity of the reaction in conjunction with slow and fast heating of EM.

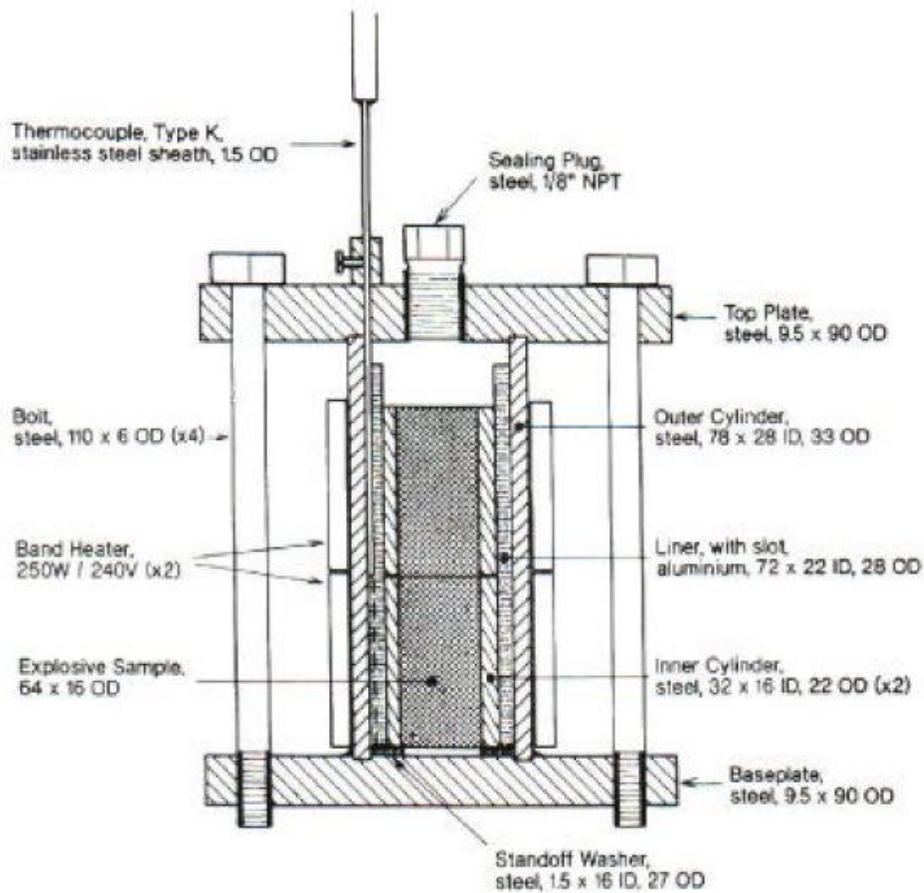


Figure 2.8 SSCB set up (Peugeot and Sharp)

The top plate incorporates a hole for the thermocouple and has a central hole which is closed with a steel plug. The temperature recorded during the test is that sensed by the thermocouple in the slot of the aluminium liner, located between the heaters (on the outside of the test assembly) and the explosive (within the liner cylinders). The temperature reached by the outer surface of the explosive sample during the test is obtained from a calibration chart produced by heating an inert-filled SSCB which has a second thermocouple welded to the inner surface of the cylinder. The two temperature-time curves obtained (as shown in figure 2.9) allow the temperature reached by the explosive to be estimated from the recorded temperature in the liner slot.

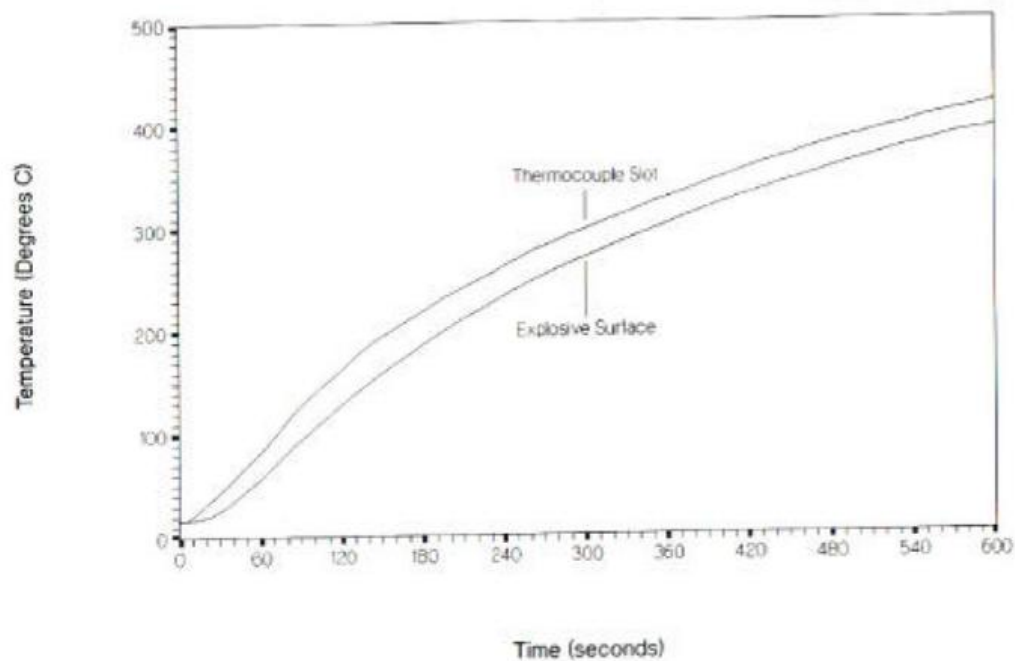


Figure 2.9 SSCB Calibration Curves, Fast Heating Rate (Peugeot and Sharp)

2.5.3 Cook-Off Test Tube

The EM sample is housed in a seamless steel tube between 200 and 250mm in length, with the outside diameter of 25mm wall thickness is varied to change the confinement (figure 2.10). Sealing plugs and a thermal barrier material is placed between the steel caps and the EM sample. The cook-off tube is then instrumented with externally mounted thermocouples and strain gauges are attached to evaluate wall motion during the cook-off event. The heating cable is wrapped around the tube, covering the portion of the tube between the steel retaining rings. Once the heater is in place, a thermal barrier material is wrapped around the heater to prevent heat loss (figure 2.11).



Figure 2.10 Diagrammatic view of assembled cook-off tube (Peugeot and Sharp)

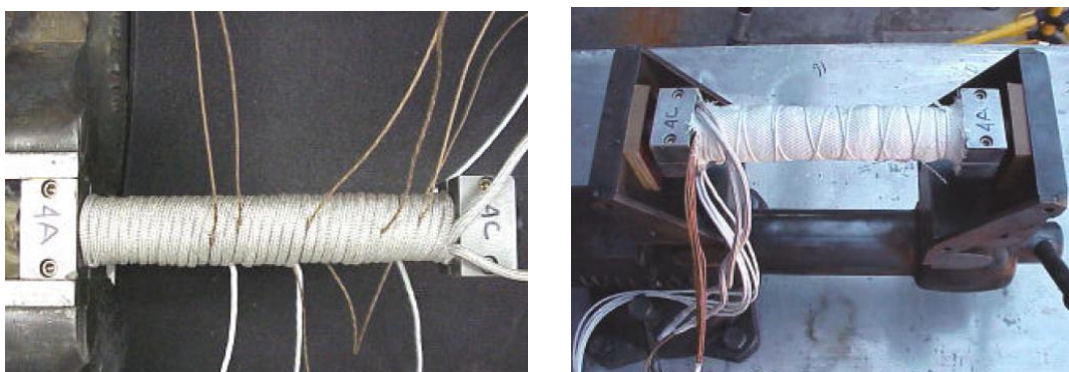


Figure 2.11 Photographs showing thermal wrap and heating cable around tube.

A heating profile where the tube is heated to 130°C at 10°C / min and then held at 130°C for 30minutes (to allow for thermal lag) and then ramped at 0.1°C / minute until cook-off is shown in the graph below (figure 2.12). The black line is the heater and the red line the sample under test. The heating rate changes the time to reaction, but does not seem to effect the violence of the reaction.

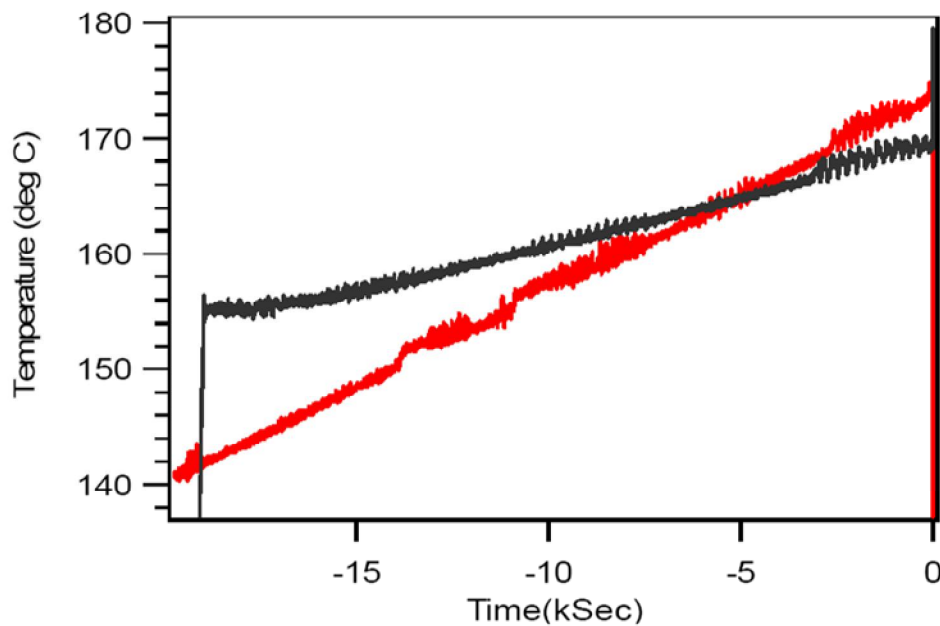


Figure 2.12 Tube test results graph showing the two heating rates (Peugeot and Sharp)

2.5.4 Variable Confinement Cook-Off Test (VCCT)

An inexpensive test with an aluminium sleeve filled with EM which is slid into a steel tube steel sleeve with the confinement pressure determined by the thickness of the sleeve. The sample is placed between two sets of steel washers having a hole in the middle to provide some space for thermal expansion. It is then tightened securely between two witness plates to provide extra confinement and preclude its exudation or venting (figure 2.13). The test is conducted by heating the sample at a specified rate until a reaction occurs. The steel sleeve thickness is increased until a more violent than a burn is observed. A graphical result is shown in figure 2.14 where internal and external thermocouple measurements are recorded throughout a test.

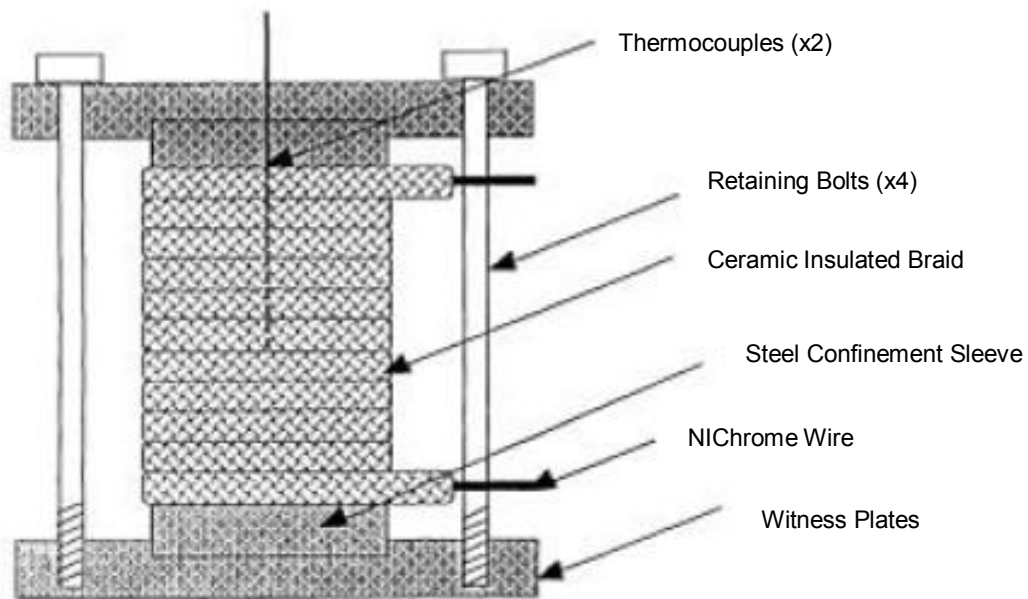


Figure 2.13 VCCT Fast Cook-Off Set-Up (Peugeot and Sharp)

For a fast cook-off set up the test unit should attain 540°C in 30s and maintain an average temperature of 870°C over the following 900s.

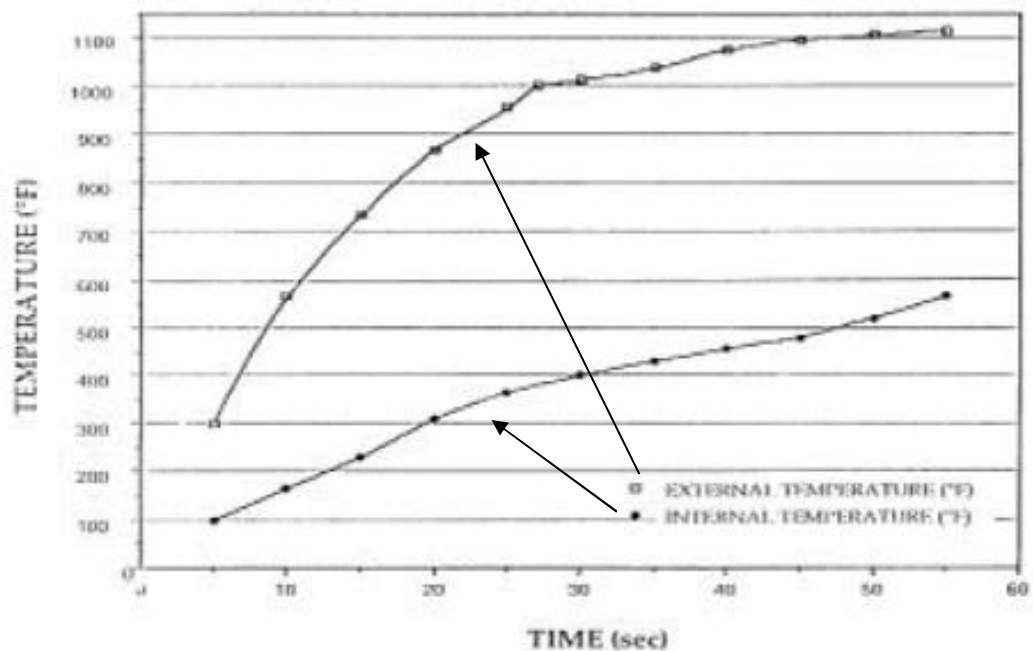


Figure 2.14 Fast VCCT temperature Profile (Peugeot and Sharp)

2.5.5 Confined Slow Cook-Off Test

The sample assembly is placed in an oven which provides a controlled thermal environment over a 40°C to 365°C temperature range. The sample is subjected to a gradually increasing air temperature at a rate of 3.3°C / hour until a reaction occurs throughout the temperature operating range. The steel test pipe can be between 40mm and 53mm in diameter, 200mm in length and the case 4mm thick. They are closed with steel or cast iron end caps (the torque strength should be able to withstand 45MPa).

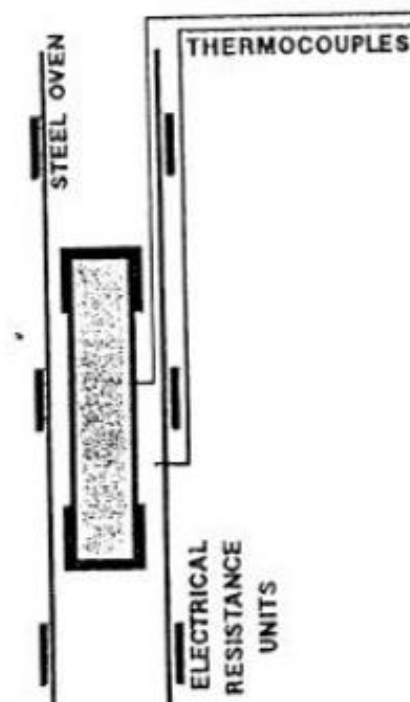


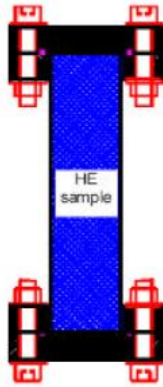
Figure 2.15 Set-Up for the Confined Slow Cook-Off Test (Peugeot and Sharp)

2.5.6 Scaled Thermal Explosion Equipment (STEX)

This test equipment is designed to quantify the violence of a thermal event under carefully monitored conditions, and to provide a database which can be used to validate predictive codes and models. It was developed with the following goals:

- Pre-determined reaction location away from end effects.
- A range of physical scales.
- Quantitative measurements of reaction violence
- A design to allow accurate simulations of a system whilst avoiding physical features that is difficult to model.

This test has been designed to ensure that the weak point in the system is the cylinder wall and not the end caps. Some venting is made possible through the top internal thermocouple stealth leakage or by eliminating the gasket sealing the top flange.



Figures 2.16 Diagrammatic and real time STEX Set-Up (Peugeot and Sharp)

Three radiant heaters are used to heat the cylinder wall. The non-contact heaters (positioned 140mm from the vessel wall) reduce temperature gradients and are controlled by one temperature controller. Each end cap is heated with a separately controlled heating element which is attached to an aluminium plate to provide more uniform end heating conditions (see figure 2.16).

2.5.7 One Dimensional Time to Explosion (ODTX) Apparatus

The ODTX apparatus (figure 2.17) fundamentally comprises of two aluminium or steel anvils that can be heated by a controlling unit from 100°C up to 400°C. The principle measurement is the time to explosion, which is represented by the

confinement failure. The anvils are held together under hydraulic pressure of 20.7 MPa, which allows for a confinement cavity pressure of 331.2 MPa (pressure exceeding this from an EM event will breach the cavity confinement). The anvils hold an explosive sample of approximately 2g in spherical shape, 12.7mm in diameter. This apparatus has the primary function of obtaining the time to explosion from a regulated temperature and can be used to analyse cook-off trends. It provides a simple, cost effective means to conduct cook-off experiments and Matei (2001) used conducted tests with several secondary High Explosives as part of a PhD thesis into Cook-Off with ODTX.

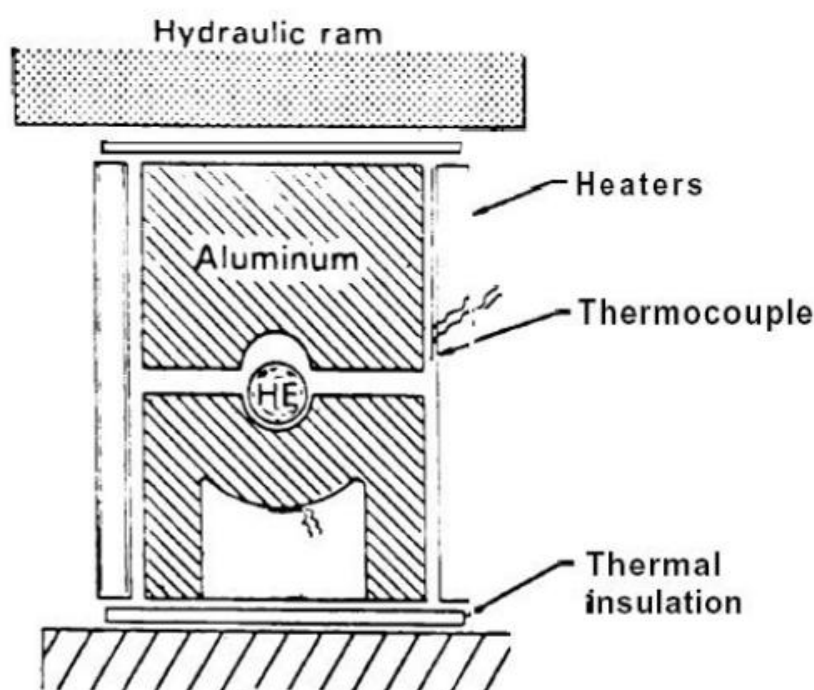


Figure 2.17 Set-Up for a TMXR Test (this is a modified ODTX).

2.5.8 Accelerated Rate Calorimetry (ARC)

The ARC is a highly adiabatic calorimeter designed to assess the safety of reactive chemicals and mixtures. It is a safety calorimeter designed originally for the chemical processing industry, it is fully automatic in use and safe for the normal laboratory environment. It provides a time, temperature and pressure

profile of a reaction within a period of 24 hours and the finished data is simple to interpret.



Figure 2.18: Photograph of complete ARC assembly



Figure 2.19: Photograph of ARC Titanium Bomb

Figure 2.18 shows the main assembly of the main test components and these are housed in the described containment vessel. Electronics control the experiment via a PC set up by an operator where the experimental conditions are input. Once the sample is in the bomb (figure 2.19) the lid is lowered and the containment vessel (see figure 2.20) is shut. Tests to be conducted must have a start / end temperature, values of heat steps, a wait time and sensitivity (a self heating rate which exothermic reaction is defined) must be selected. Typically, if nothing is known of the sample, the start and end temperature may be 50°C and 400°C respectively, the heat steps of 5°C, the wait time 900s and the sensitivity 0.02°C/min. Therefore, it is not difficult to define the experimental conditions.

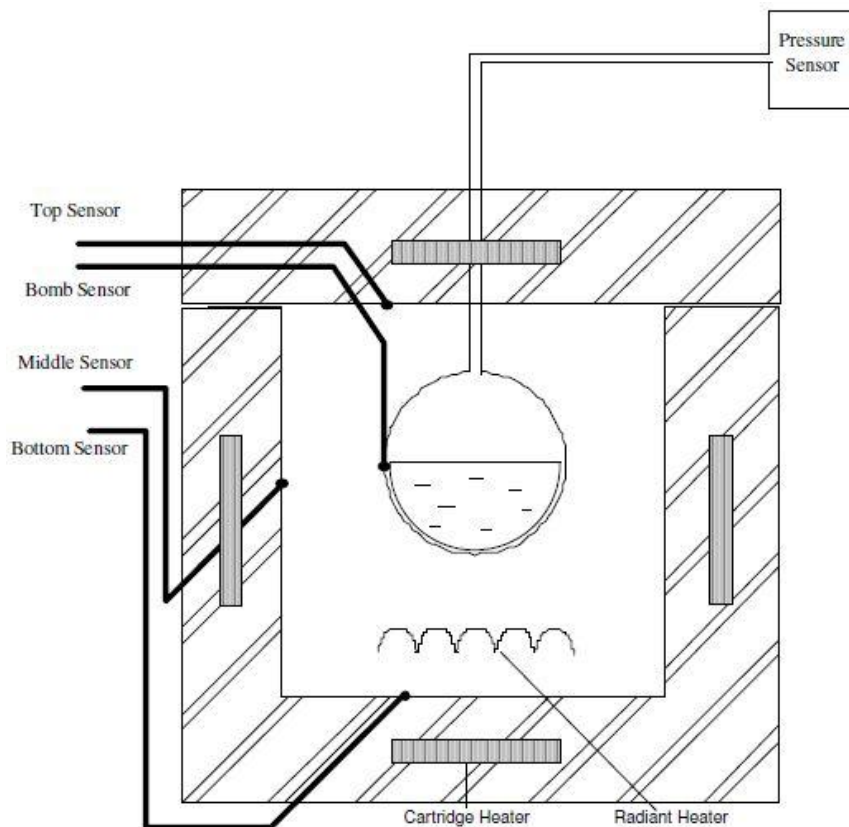


Figure 2.20: Diagram Detailing ARC heater, thermocouple and heater assembly

(ref: www.thermalhazardtechnology.com/presentations)

At the start, the system will be heated to the start temperature and the system will come to isothermic equilibrium during the wait time. The temperature differences between the bomb thermocouple and the three jacket thermocouples that control the heated zones of the calorimeter (figure 2.20) will be reduced towards zero. Then there will be a seek period where there is a temperature rise and an exothermic reaction will be detected. If there is no temperature rise (greater than selected sensitivity) the first small heat step will automatically occur. These steps are then repeated until an exothermic is determined. The detection of the exothermic is by a continuous rise in temperature at a rate greater than the selected sensitivity. This is called the

‘Heat-Wait-Seek’ (H-W-S) process. The system will enter the exothermic mode automatically and as the temperature of the bomb increases the calorimeter temperature will also increase (the system keeping the temperature differences between the sample bomb and the three separate calorimeter zones as small as possible). The system will continue in exothermic mode until the self heating rate decreases below the selected sensitivity, then the H-W-S process will commence again. When the temperature reaches the end temperature the test automatically terminates.

Throughout the test data comprising time, temperature and pressure values will be collected and stored at user define intervals. Figure 2.21 shows data graphical resultsd from a typical test.

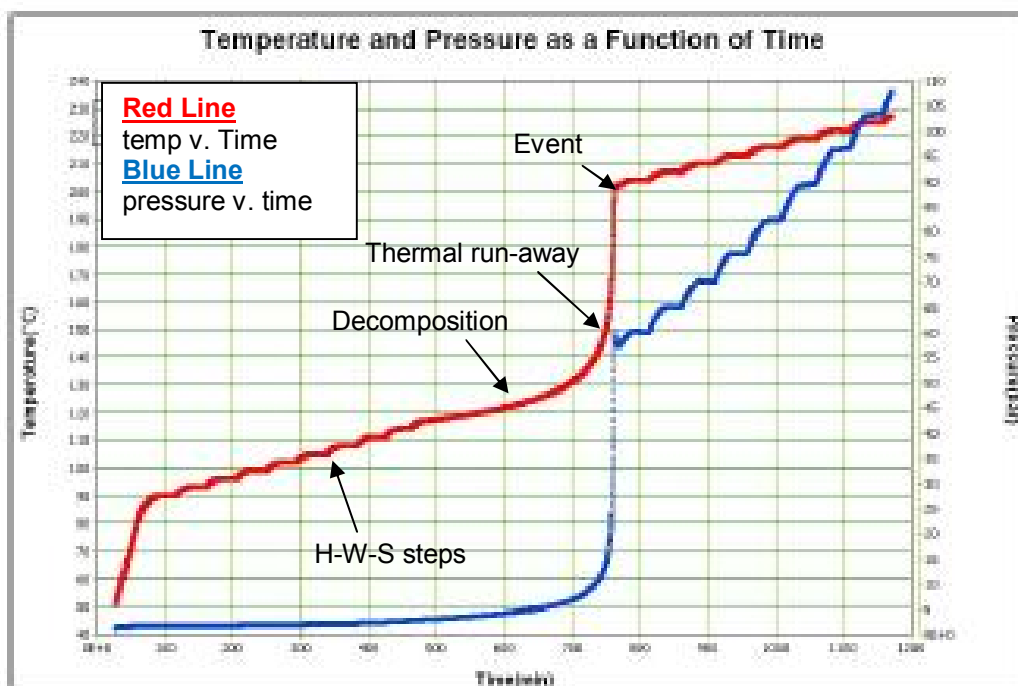


Figure 2.21: Example of data from a typical ARC test³.

³ In this example the gradual increase in heat element temperature maintains the heat of the sample until 120°C at 600min where self heating occurs until thermal run away and pressure increase at 130°C at 700min. The test is completed at circa 210°C at 760min where the sample is at the final stage of decomposition (or event).

The raw data plots are:

- Temperature against time.
- Pressure against time.
- Pressure against temperature
- Self heating against time.
- Pressure rate against temperature
- Self-heating rate against pressure rate.

Calculated plots (and tables of data) can be obtained for:

- Time to maximum rate against temperature.
- Activation energy determination
- Kinetic modelling.
- Thermodynamic heat of reaction determination.

2.6 Additional Thermal Testing Methods

Thermoanalytical data capture has been discussed previously but the application of these techniques to study explosives requires special considerations due to their mode of operation. With the previous 'fast/slow' heating testing methods, thermal run away on quantities under test may lead to instrumentation equipment and important data or wrongly interpreted as a result from a violent event. Therefore, heating rates may have to be carefully chosen to match a sample size in order to keep reactions under control while producing signals and measurable magnitude. STANAG 4515 details specific techniques and mechanisms and summarised as follows:

2.6.1 Differential Thermal Analysis (DTA)

This involves the heating (or cooling) of test samples and an inert reference sample under identical conditions and recording any temperature difference

which develops between them (the ΔT shown in figure 2.22). Any change (physical and/or chemical) between the samples by evolution of heat will cause the test sample to rise above the reference sample, thus giving the rise on to an exothermic peak on a DTA plot. Conversely, a process which is accompanied by the absorption of heat will cause the temperature of the test sample to lag behind that of the reference material, leading to an endothermic peak. DTA will also provide information on melting, boiling, crystalline transitions, dehydration, decomposition, oxidation and reduction reactions (Akhavan 2004, pp 114 - 115).

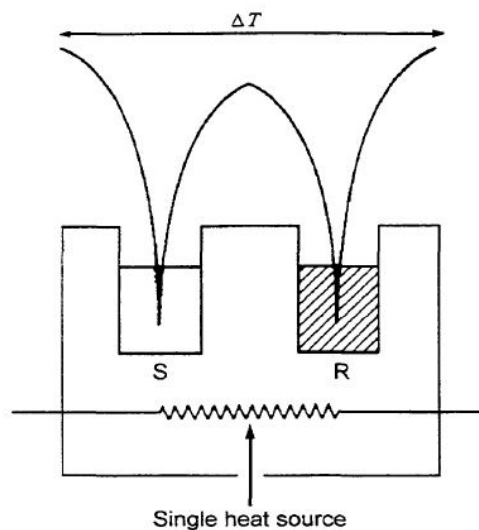


Figure 2.22: Schematic Diagram of DTA (STANAG 4515)

When this technique is used in conjunction with a photo detector system, strategically placed above the sample (figure 2.23) the ignition temperature can be assessed.

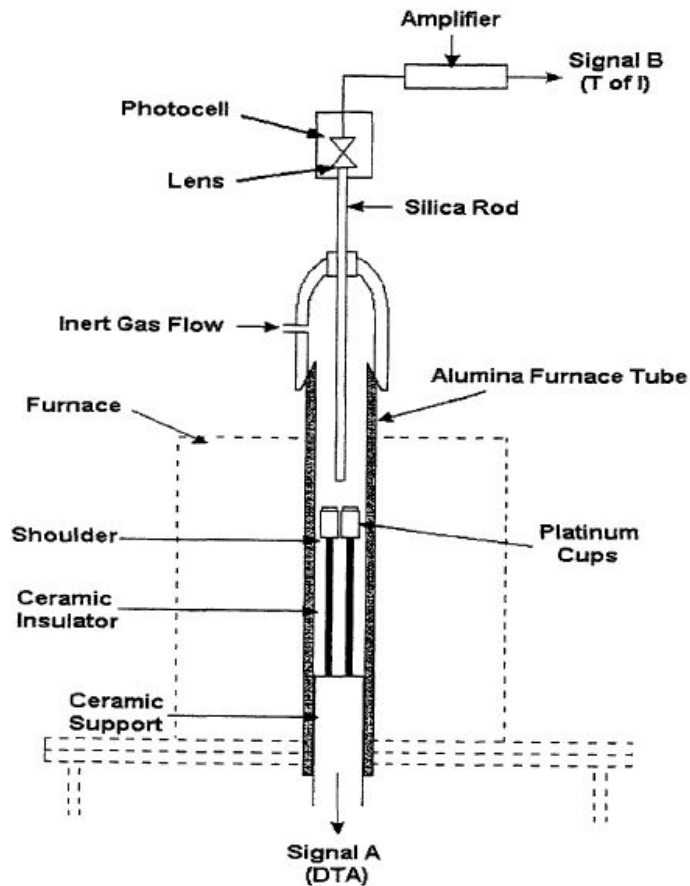


Figure 2.23: Schematic Diagram showing DTA Temperature of Ignition Test

2.6.2 Differential Scanning Calorimetry (DSC)

This technique is similar to DTA. The peaks in a DTA thermogram represent a difference in temperature between the sample and reference, whereas the peaks in a DSC thermogram (see figure 2.24) represent the amount of electrical energy supplied to the system to keep the sample and reference at the same temperature. The areas under the DSC peaks will be proportional to the enthalpy change of the reaction. DSC is generally used for the study of equilibria, heat capacities and kinetics of explosive reactions in the absence of phase changes (Akhavan 2004, pp 116 - 117).

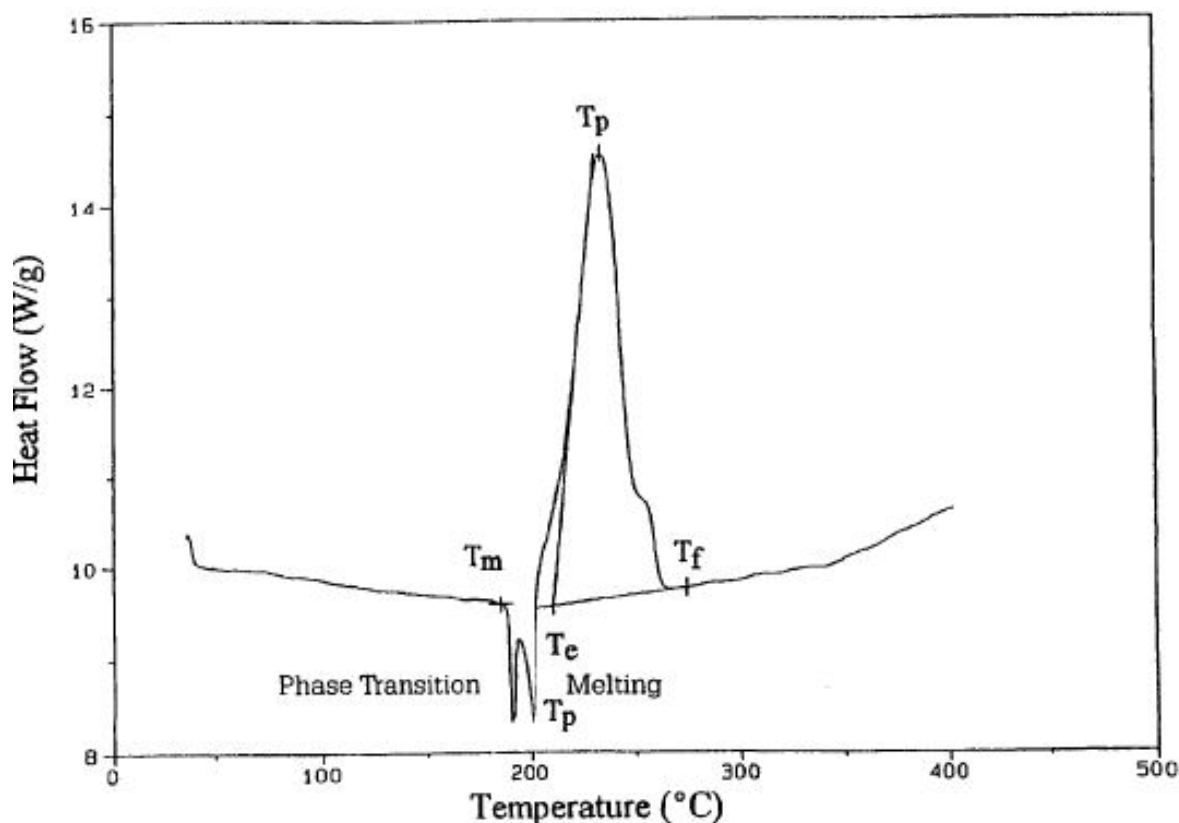


Figure 2.24: Graph showing typical DSC Thermogram (STANAG 4515)

2.6.3 Thermo Gravimetric Analysis (TGA)

This process is suitable for studying explosive reactions. The sample is placed on a balance inside an oven and heated at a desired rate and the loss in weight of the sample is recorded (due to evaporation of moisture, evolution of gases and/or chemical decomposition). TGA is normally combined with DTA, and a plot of the loss in weight together with DTA thermogram is recorded. The plots give information on the physical and chemical processes that are taking place. In an explosive reaction there is rapid weight loss after ignition due to the production of gaseous substances and at the same time heat is generated. Thermal pre-ignition and ignition temperatures of explosive substances can be obtained from DTA and TGA thermograms (Akhavan 2004, pp 116).

2.6.4 Consideration of Cook-Off Temperature Testing Equipment for Experiments

Options	Advantages	Disadvantages
SCB	Can replicate fast cook-off of propellants.	Complexity and damage to test vehicles
SSCB	Can replicate fast cook-off of propellants.	Limited positioning of thermocouples
Tube Test	Can replicate fast cook-off of propellants (inc. Thermal lag).	Damage to test rigs, visibility of Cook-Off and time consuming
Confined Slow Test	Cheap and easy test to conduct.	Not suitable to replicate gun cook-off scenarios.
VCCT	Reasonably easy test to conduct and measure results.	Damage to test components. More suited to primary explosives.
STEX	Rapid heating to simulate gun temperatures	More suited to quantify violence of thermal explosions
ODTX	Excellent method to measure time to explosion	Difficult to prepare loose propellant into test crucible.
ARC	Good identifying the onset of decomposition onset.	Expensive and time consuming tests.
DTA, DSC and TGA	Good to monitor comparison and control.	More suited for stability testing and weight loss during the heating of pyrotechnics and primary explosives

Table 2: Cook-Off methodology and testing comparison

3. CHAPTER 3. Test Equipment Methods

3.1 Review of Options

To achieve a partial aim of the assessment, the ODTX initially seemed the most suitable test in identifying initial decomposition to explosion. Although a possible candidate to conduct the cook-off tests, harnessing loose propellant into a capsule as test samples would have been a challenge compared to the more readily compact primary explosive samples used in the work of Graaw (1998) and Matei (2001). The loose propellant sample would have to be contained in a metallic sphere which would have to be filled (it splits in 2 halves; primary explosives easier to place as a solid compared to a loose grain/powder).

The ARC is another possible candidate in obtaining accurate decomposition rate, thermal run away and eventually cook-off data. However, this still did not replicate the cook-off within a 'hot' gun barrel scenario (fast cook-off rate). Tuisku (1996) completed a project on self-ignition (cook-off) of gun propellants and explosives and this test equipment was designed using small samples (20g – 30g). Also Cottle et al (1974) designed a disposable oven comprising of a heating element and aluminium body to evaluate the effects on 30mm aircraft cannon ammunition (including high explosive shell).

It was decided that although ARC data would be useful in determining the onset of self heating and decomposition, an additional heating rig that would be required to measure the cook-off of a gun propellant.

Pakula (1984) used a SCB that involved the explosive samples being contained in a 400mm steel vessel with walls 3mm thick and this vessel was heated with two 400w electric heaters. The SCB was instrumented with one or two plate thermocouples (these being the vessel wall and explosive under test). Data recording during the test were taken from strip-chart records, and an assessment of the severity of the reaction was made from the number and condition of the vessel fragments and condition of the witness plate. Whilst the

report conclusions supported the use of this SCB test fixture as an excellent technique to determine the thermal behaviour of explosives with regard to cook-off time it also involved high degree equipment destruction.

3.2 Experimental Methodology and Equipment Selection

The ARC was to be used for assessment of the onset to decomposition and locally designed test apparatus would be required for the recording cook-off temperatures using the ARC results as a base line. An interesting report by Bunyan et al (2002) recommended the use of ARC but recommended the ARC result(s) be validated by the use of heat conduction calorimetry experiments conducted at temperatures below the temperature where self heating hazards of a system of interest are being considered. To achieve the follow on experimental goals, the apparatus design would be along the lines of a SSCB heating chamber and be able to experiment individual quantities up to 5g. To add the reality of gun chamber cook-off, the apparatus would contain Small Arms Ammunition (SAA) and attempt to heat the SAA to a chamber temperature representing a gun heating (and cooling during) cyclic gun fire operation. Thermocouples would also be used to record outer wall cartridge case and cartridge case core (centre) propellant temperatures by the use of thermocouples (TC). The temperatures of these TCs would be recorded continuously as the temperature ramps up and until cook-off occurs or the experiment terminated 1800s from the start of test (which ever soonest). The SAA cartridges will be fired 7.62mm fired (post Shrivenham 25m range) inert cases (no primer or propellant). These cartridges will be separated into identical batches and filled with a propellant (Bulls Eye'⁴) held at the University

⁴ This is a double base (DB) pistol propellant and the MHDS is detailed at Appendix A. The ideal propellant would have been the single based (SB) NRN 41 FN/T, Nitro Chemie Wimmis 1500, N150 or NC 1331 but none were available apart from the option of ammunition dismantling containing this grade of propellant. The 'Bulls Eye' propellant was the only propellant to hold a MHDS to allow transport and external ARC testing outside the Shrivenham estate.

explosive storage area. The cartridges will be filled to 2g (+/- 0.01g)⁵ and a thermocouple held by a plug designed and built to sit where the bullet head would normally be located (see Appendix B). The primer will have also been expended and that area inert, and not required for the purpose of this assessment.

Stage 1 will involve one test by ARC testing to obtain onset to decomposition, thermal run-away and time to explosion data of Bulls Eye DB propellant. Stage 2 to will be a design and production of a locally manufactured heating rig. Stage 3 would be to use the locally designed cook-off apparatus and carry out a number of cook-off experiments on the 7.62mm cartridges containing Bulls Eye propellant.

3.3 Locally Manufactured Fast Cook-Off Test Rig

The experiments and equipment to be designed had to meet the following criteria:

- Be able to heat the cartridge case to 150°C under 300s
- Equipment and manufacturing procured within the project budget.
- Be re-usable for number of experiments planned and with a repeatable performance (limited scope for equipment damage).
- Be able to record outer cartridge temperature and propellant core temperature at the point of cook-off.
- A system to record and produce time/temperature data throughout each test procedure.

⁵ The UK 7.62mm cartridges are normally filled to 2.85 to 2.9g of its standard propellant. However, during preparation it became impracticable to achieve this. No doubt due to the differences in the SB and DB grain form and/or density.

- Be operated and controlled remotely (safety)

3.3.1 The Copper Heater

This is the main rig to hold the experimental SAA cartridge with a modified copper bar test component previously used on unconnected experiments by AWE⁶. This heater will contain 3 x electrical heater elements, 3 x thermocouples and the cartridge under experiment. Two other small holes, one in the top and one on the side, were required to be drilled to hold thermocouples (TC) in place. The centre hole in the copper bar to contain the cartridge case under experiment also required drilling to expand the diameter and lengthen the depth (details at figure 3.1).

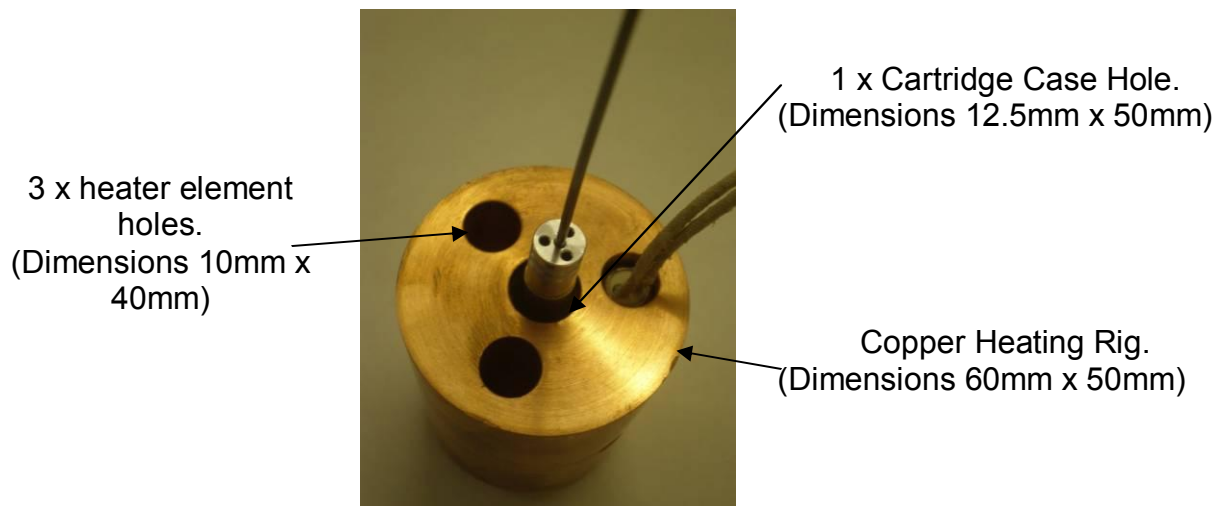


Figure 3.1 Copper heating Rig

Due to a number of necessary modifications to the copper heating rig throughout the experiments, the copper heaters had to undergo a number of iterations which is explained further into this thesis. Six heaters were

⁶ The AWE cook-off experiments apparatus was purely acting as a heater mass containing heating elements and a thermocouple. No EM or test samples were contained inside the copper heater. The base of the copper heater has a screw thread for the AWE experimental purposes only and performed no useful function in the Bulls Eye propellant experiments.

manufactured and were marked A and F for experimental recording purposes. The copper heater went through two design changes due to damage to the components. Essentially, three versions were developed during the experiments with the only difference being a slight reduction in weight of the copper heater due to the modifications required. The potential impact would be heater transfer rates on the experimental results and these weight changes are recorded in table 3. The three variants produced are identified as Versions 1, 2 and 3. In all Versions the cooked-off cartridge had to be removed from the copper heater by use of a hammer and drift.

3.3.1.1 Cooper Heater (Version 1)

This Version was essentially the 'Mk1' and has already been described in beginning of this chapter. During experimental use at least one or more TCs and one or more heater elements were damaged in each experiment (see figure 3.2).

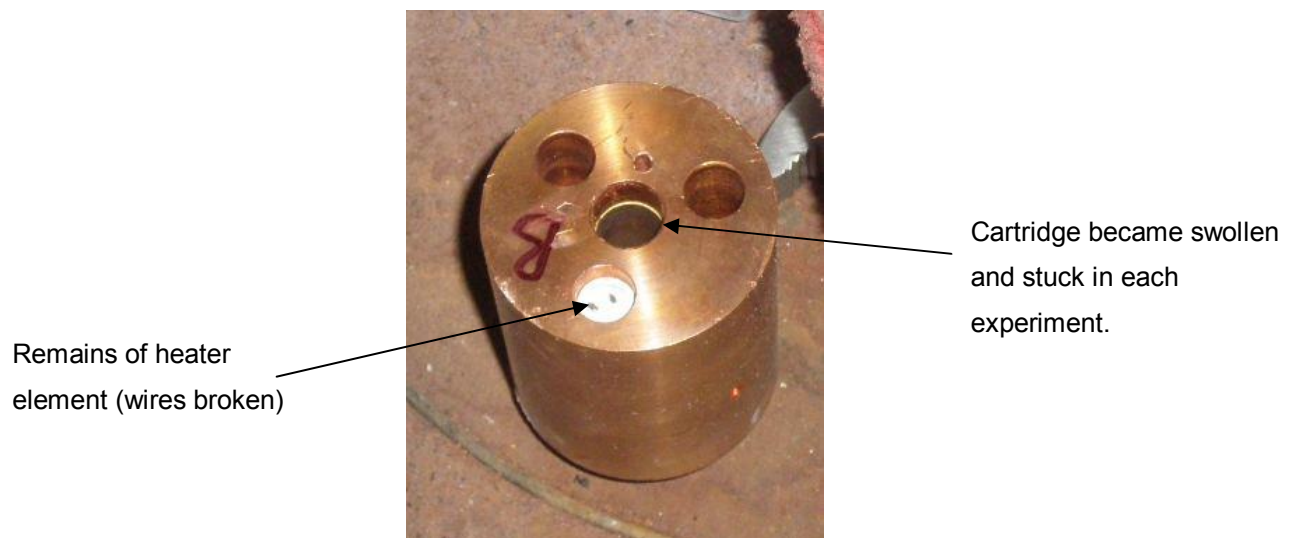


Figure 3.2 Damage to 'Serial B' Copper Heater

3.3.1.2 Copper Heater (Version 2)

The aim was to reduce the damage to the TC's and Heater Elements. An aluminium tube 250mm x 13mm was used to protect TC A3 and the heater element wires. A small recess was drilled at the copper heater cartridge entrance to hold the aluminium (Al) tube in place during the experiment (figure 3.3 and 3.4).

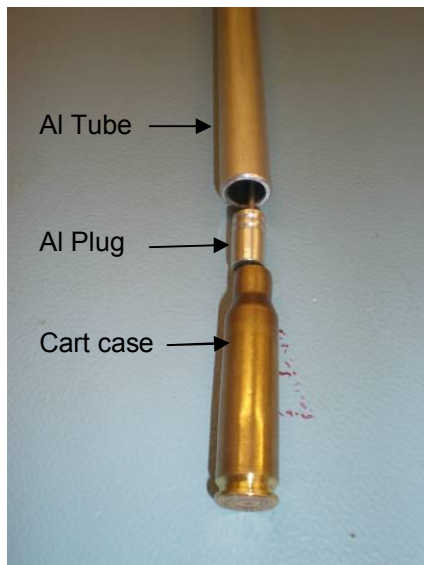


Figure 3.3 Al bar, cart and TC A3 combination (Version 2)

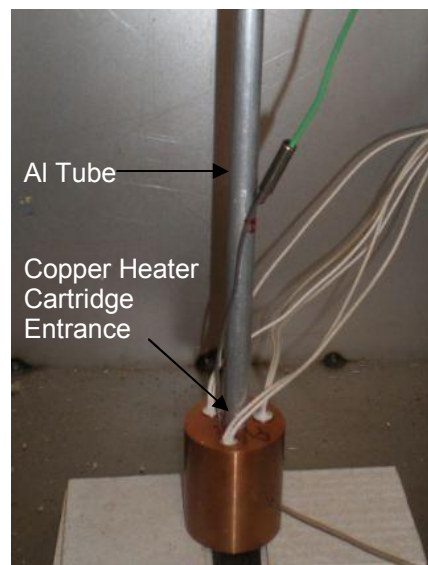


Figure 3.4 Version 2 combination fitted to Copper Heater

Version 2 (with AL tube) was only used for experiments 7, 34 and 35. The latter two experiments were used to demonstrate by video the violence what a small amount of DB propellant can cause as a result of cook-off (damage to Al tube shown at figure 3.6)



Figure 3.5 All 3 heater elements fitted to copper heater



Figure 3.6 20mm of Al tube destroyed with remaining fragments shown.

3.3.1.3 Copper Heater (Version 3)

With the failure of Version 2 causing excessive damage to heater element wires (can be seen at figure 3.5) a re-design was required. The copper heater (Serial 'A') used during the Version 2 was modified by drilling the heater elements holes completely through (previously blind holes) so the fitting of these elements could be achieved from the bottom (screw thread) end of copper heater. This would ensure less flame / cartridge fragmentation impact to cables. Also the cartridge case hole in the copper heater was deepened by a further 10mm to reduce the damage on TCs A1 and A3 (figure 3.7).

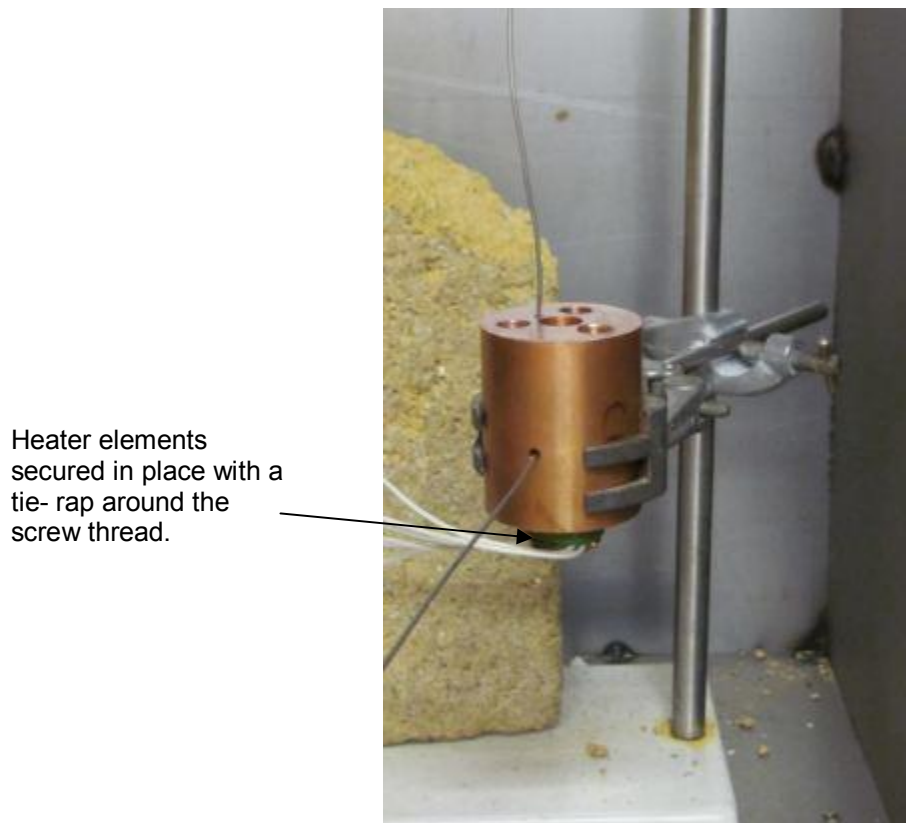


Figure 3.7 Version 3 with heater elements fitted from the bottom

Copper Heater – Recorded Weights (g)

Body Serial	Version 1	Version 2	Version 3
A	912	904	887
B	904	897	885
C	917	906	893
D	906	900	886
E	904	898	885
F	882	873	860

Table 3: Detailing the recorded weights of the manufactured copper heater rigs⁷

⁷ A total of 6 copper rigs were produced (Serials A to F). The different versions were due to modifications required during the cook-off tests which resulted in a weight reduction in each Version affecting the masses of Body Serials A to F.

3.3.2 Aluminium (Al) Vent and TC holder Plugs⁸

An Aluminium plug was used to hold a thermocouple (TC) in the centre of the propellant with three venting holes so any pressure build up during a heating stage would not dislodge the TC from its monitoring position.

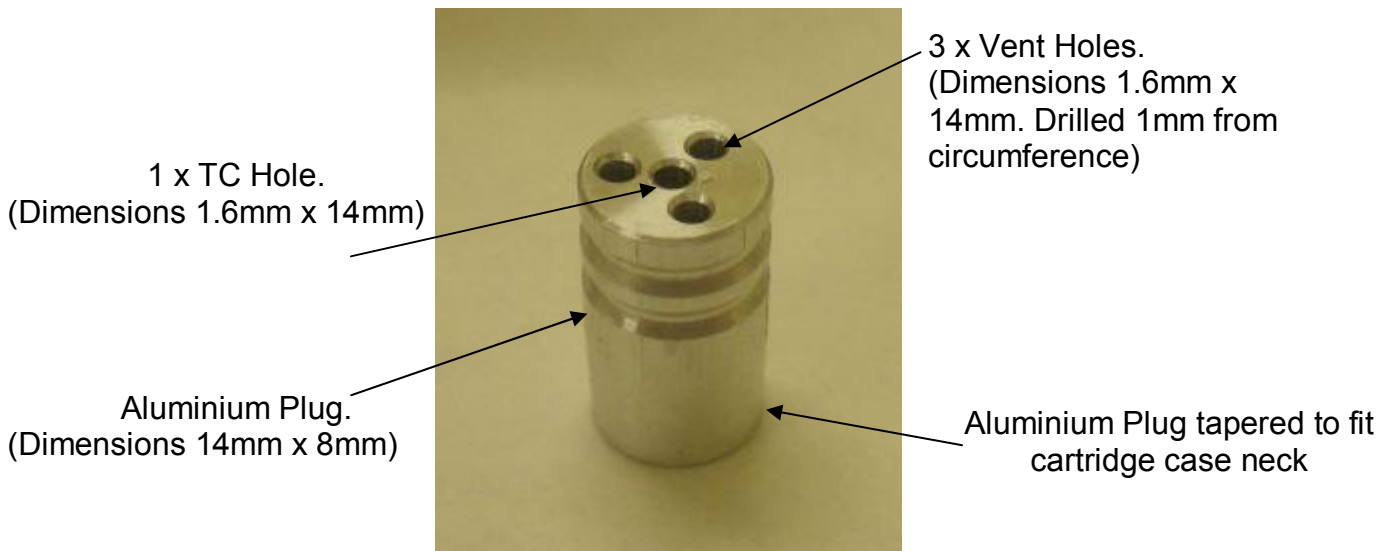


Figure 3.8 Details of aluminium plug construction

3.3.3 Cartridge Cases

The calibre of cartridge cases used was 7.62mm x 51mm and the manufacture and batch details varied. For the purpose of this project quantity 35 x 7.62mm cases were selected as being 'fit for purpose' (i.e. symmetrical in size and shape with no deformation) and the cartridge necks slightly reamed to accept the tapered aluminium plugs comfortably. Internally contaminants (burnt propellant residue) were removed by emersion in an acetone bath (figure 3.10). Prior to the test, 2g (+/- 0.01g) was added to each prepared cartridge case and recorded in table 4. A more detailed analysis of cartridge batch, weights and dimensions are detailed in Appendix C.

⁸ The design and manufacturing drawings detailed in Appendix B.

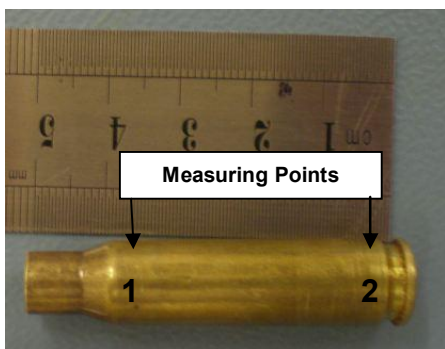


Figure 3.9 7.62 mm Cartridge case



Figure 3.10 Cartridge cases (post acetone clean)

Prior to the heating of the cartridges the diameters were measured with a vernier calibre at the points highlighted in figure 3.9 and recorded in the more detailed table in Appendix C.

Serial	Type (Base markings)	Qty	Average Weight (g)
0.308(1-19)	0.308 WIN Match FNM	19	11.67
FFV(1-9)	FFV 92	9	12.37
1a	1K07 7.62x52	1	11.55
2a	CG01	1	12.23
3a	L2A2 MG 99	1	11.39
4a-c	RG 90	3	10.71
5	RG 93	1	11.77

Table 4: Types, quantities and average weights of experimental cartridges

3.3.4 Heater Elements

Three elements (10mm x 40mm) with an electrical lead length of 250mm were used and for the majority of the experiments were rated at a 100w which required a 120v AC supply. The supplies of these were via COTS purchase from RC Components, a nationwide supplier of electrical/electronic items. For two of the later experiments 200w heater elements were used.

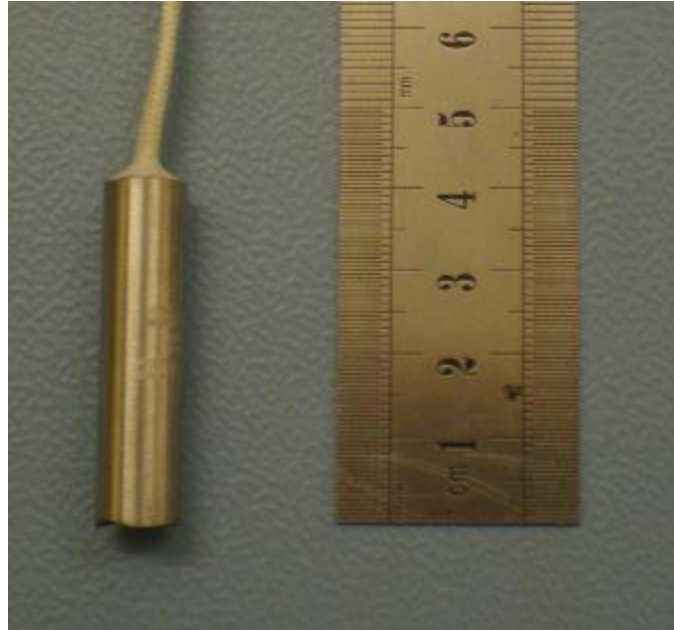


Figure 3.11 100w Heater element

3.3.5 Thermocouples (TC)

For each experiment a quantity of three TCs were required. These were again procured from RC Components and two varieties were used (RC identification No. 228-7445 and 397-1236). Both were K-Type TCs and identical in probe diameter (1.5mm) with a maximum / minimum operating temperatures of 1100°C / 0°C. The only difference being that of the electrical cable length, this being 150mm (figure 3.12) and 250mm (figure 3.13) respectively. For the first 25 experiments the 150mm cable length type was used and the data received during the experiments would be identical from both variants of TC.



Figure 3.12 150mm TC



Figure 3.13 250mm TC

The TC probes measured the following in each experiment:

- The Copper heater (feed back to temperature controller) – TC A1
- The surface of the cartridge case – TC A2
- The centre core of the Bulls Eye propellant – TC A3

These TCs were used in each experiment as follows:

3.3.5.1 TC A1

Used to monitor and regulate the temperature of the voltage regulator / controller box throughout each experiment. It ensures that the temperature of the core copper heater does not rise beyond the designated experimental temperature and/or can be held at a particular temperature (figure 3.14)

Position of TC A1
(Hole drilled 1.7mm x 5mm)

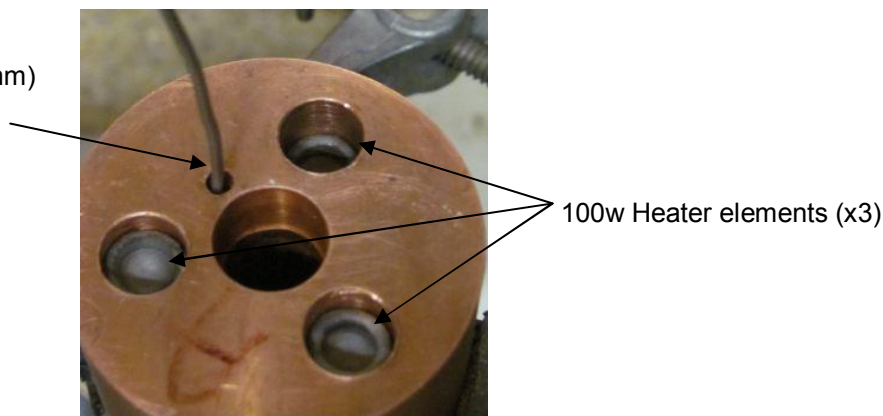


Figure 3.14 Position of TC A1 in Copper Heater

3.3.5.2 TC A2

TC used to monitor the external surface of the cartridge during each experiment. Drill 1.7mm dia. and 35mm from top of copper heater (figure 3.15)

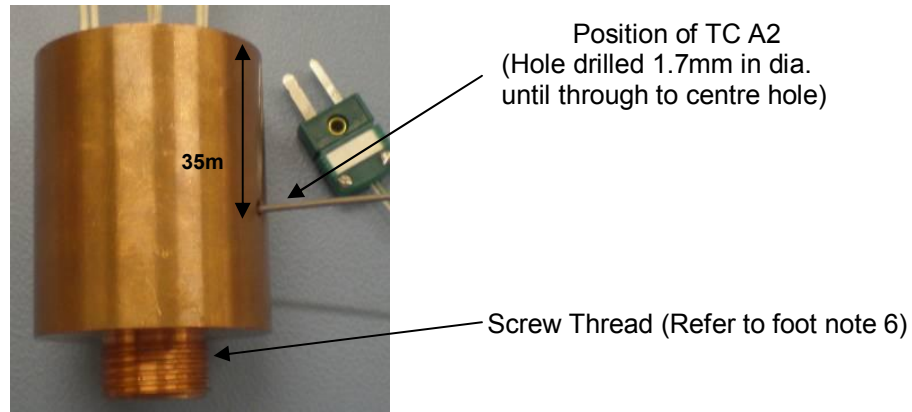


Figure 3.15 Position of TC A2 in Copper Heater

3.3.5.3 TC A3

This TC would sit in the centre of the propellant, the approximately 25mm from the base of the cartridge case (figure 3.16). This TC became a guaranteed 'sacrificial' item in each experiment (failed to survive beyond cook-off in every experiment).

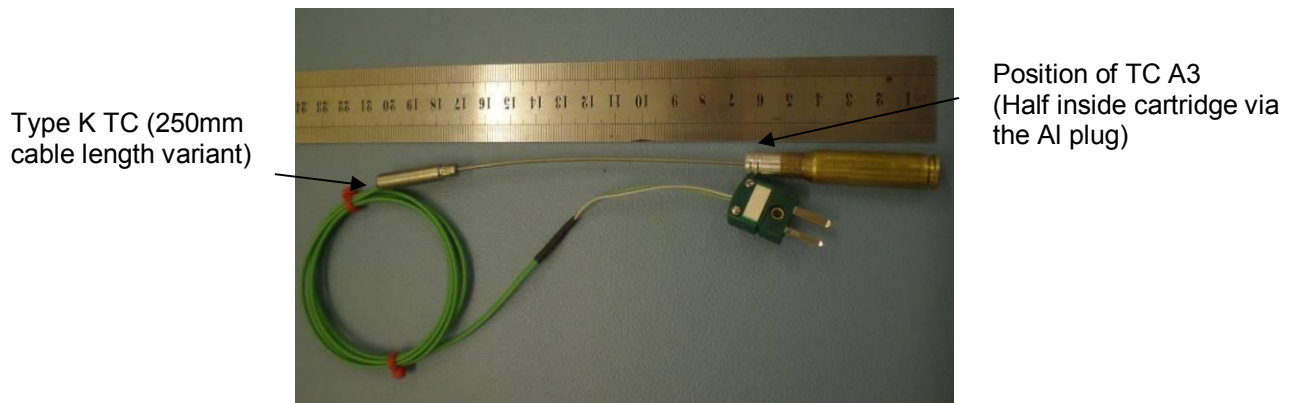


Figure 3.16 Position of TC A3 in cartridge case and AI plug combination

3.3.6 Assembled Heating Apparatus

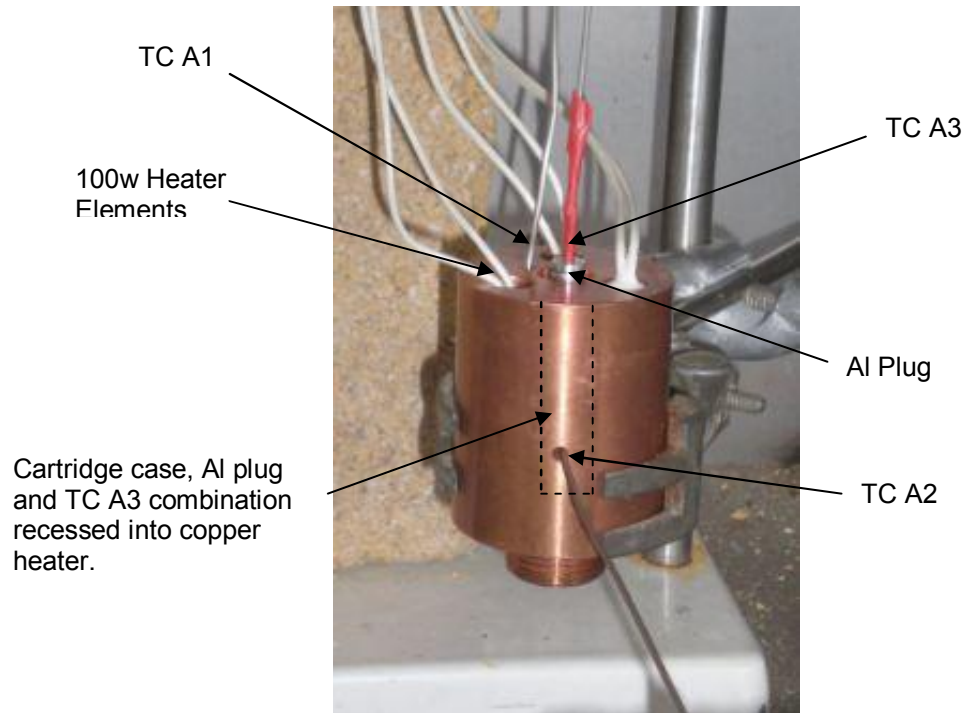


Figure 3.17 Position of all test rig components as completed item

3.3.7 Pre-Experimental Testing

The Copper heater, TC A1 and 1 x heater element were used to demonstrate the capability and the predicted heat rise per minute. The voltage converter and Nico software system (figures 3.18 and 3.19) were also proven⁹

⁹ Nico is the software that reads the TC data and converts into time vs. temperature via graphical display during the experiment. The information is available to the test operator at 'real time' and easily converted in MS excel for graphical records and analysis.



Figure 3.18 Copper Heater, TC A1 and Nico controller.

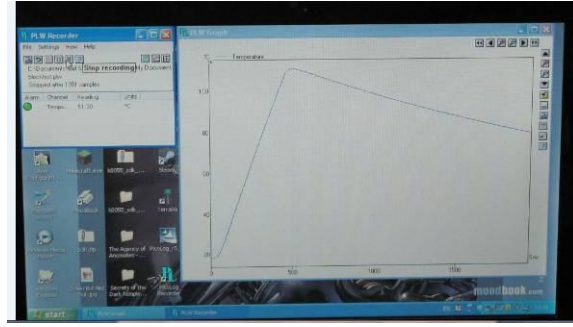


Figure 3.19 Laptop display with Nico software graph.

Prior to the first live experiment a 'dry run' was conducted to prove the heating rate when all three heater elements are fitted. Once the experimental runs commence, a 10s run time was applied to allow for thermocouple registration confirmation before any heating of the copper heater was started. All results in the experiments will include these 10s lead in times (real time results would normally be 10s short but for ease of standardising and recording they will be included throughout this thesis).

Although pressure readings were available in the ARC testing, for the purposes of the remaining copper heater experiments pressure readings were not recorded or considered due to the unpredictability of the propellant under heating (hence the vent holes in the Al plug to keep the position of TC A3 extant for each experiment).

4. CHAPTER 4 Testing Procedures

4.1 ARC Testing

These were conducted off site at a company called Thermal Hazards Technology (THT) Ltd. and are located at Bletchley, near Milton Keynes. Only one test was conducted due to financial restrictions and this was the 'H-W-S' procedure to obtain the point of decomposition, the thermal run away point/duration and the time to explosion¹⁰. The sample weight was restricted to 0.5g due to the unknown violence from a Bulls Eye propellant energetic reaction. The procedure and outputs are detailed in Appendix D and are recorded in the Results section and further analysed in the discussions section.

4.2 Locally Manufactured Test Rig

4.2.1 Test Procedure.

This test involved the gradual heating of Bulls Eye Small Arms propellant in a used 7.62mm Cartridge Case (CC). Up to 35 individual experiments took place and each CC contained no more than 2g of propellant^{11, 12}. The filled CC was placed in the robust metallic rig (copper) and heated incrementally until Cook-Off of the propellant occurs. Temperature readings were obtained via Thermocouples (TC) fitted internal and external to the CC; TC A3 fitted in to the locally manufactured Al plug which will be loosely fitted to the neck of the CC prior to the addition of the propellant. Cook-Off will be identified by vertical lifting

¹⁰ This information and ignition temperature was lacking in the MHDS.

¹¹ The UK 7.62mm cartridge actually contains between 2.85g and 3g of propellant (DMP 1400-1000-1 2012). This is a different propellant type (SB) to 'Bulls Eye' (DB) and only 2g of propellant could be filled into the cartridges to test comfortably without the risk of 'crimping' loose propellant with the Al plug.

¹² Test 34 had an additional 0.5g (2.5g total) of propellant added for video recording / change of NEQ comparison purposes.

of the plug / TC combination on propellant Cook-Off, noise of the event and / or by the visible temperature fall on the recording thermocouples.

4.2.1.1 Experiment Aims

- a. A series of 3 experiments, each comprising of 35 cook-off experiments. The temperature rise time with 1 heating element in the cooper vessel is approximately 50°C / minute.
- b. Initial first few experiemnts to establish a constant temperature rise and time (recorded) to establish 1st series temperature start point.
- c. The 1st series will vary the temperature raised 150°C - 180°C and the increase the cook-off time recorded (temperature held and switched off).
- d. The 2nd series will vary the temperature raised 165°C - 170°C, switch off and the cook-off time recorded.
- e. The 3rd series will have the temperature raised 161°C - 163°C, switched off and the cook-off time recorded.
- f. The wait time for cook-off to occur in each experiment will be 30 minutes. If no event occurs within this time frame, the temperature will be increased until cook-off occurs.
- g. Once the 3 series are completed, the remaining cartridges will have the vent holes in the plugs sealed and a temperature determined from the previous experiment used.
- h. Experiments scheduled to start 13th May 2012 and be completed by 20th June 2012

4.2.1.2 Individual Experiment Procedure

- a. Cartridges filled in an appropriated explosives laboratory (2g $\pm$ 0.01g). Aluminium cap fitted, marked with a serial number cartridges placed in ACA until use.
- b. Before preparing apparatus, ensure all electrical supplies are disconnected.
- c. The ACA with prepared cartridges not to be opened until required.
- d. Ensure Cooper jig at room temperature or $< 50^{\circ}\text{C}$.
- e. Insert 1st thermocouple (TC A3) into aluminium cap (pre-measured for length).
- f. Removed cartridge from ACA and close lid immediately.
- g. Place aluminium cap / TC assembly lightly into cartridge (visual look to ensure no loose propellant inside cartridge neck).
- h. Insert cartridge/ aluminium cap / TC assembly into copper jig. Fit TCs A1 and A2 into copper jig.
- i. Ensure apparatus contained & shielded.
- j. Ensure instrumentation is functioning.
- k. Connect power and set transformer to voltage required to achieve.
- l. Temperature required. Once achieve, disconnect power remotely and wait until cook-off or 30 minutes; whichever soonest.
- m. If cook-off does not occur within 30 minutes, increase temperature until propellant ignites.
- n. Repeat procedures a to m above until all experiments complete.

4.2.1.3 Complete Live Firing Experimentation Testing Set Up

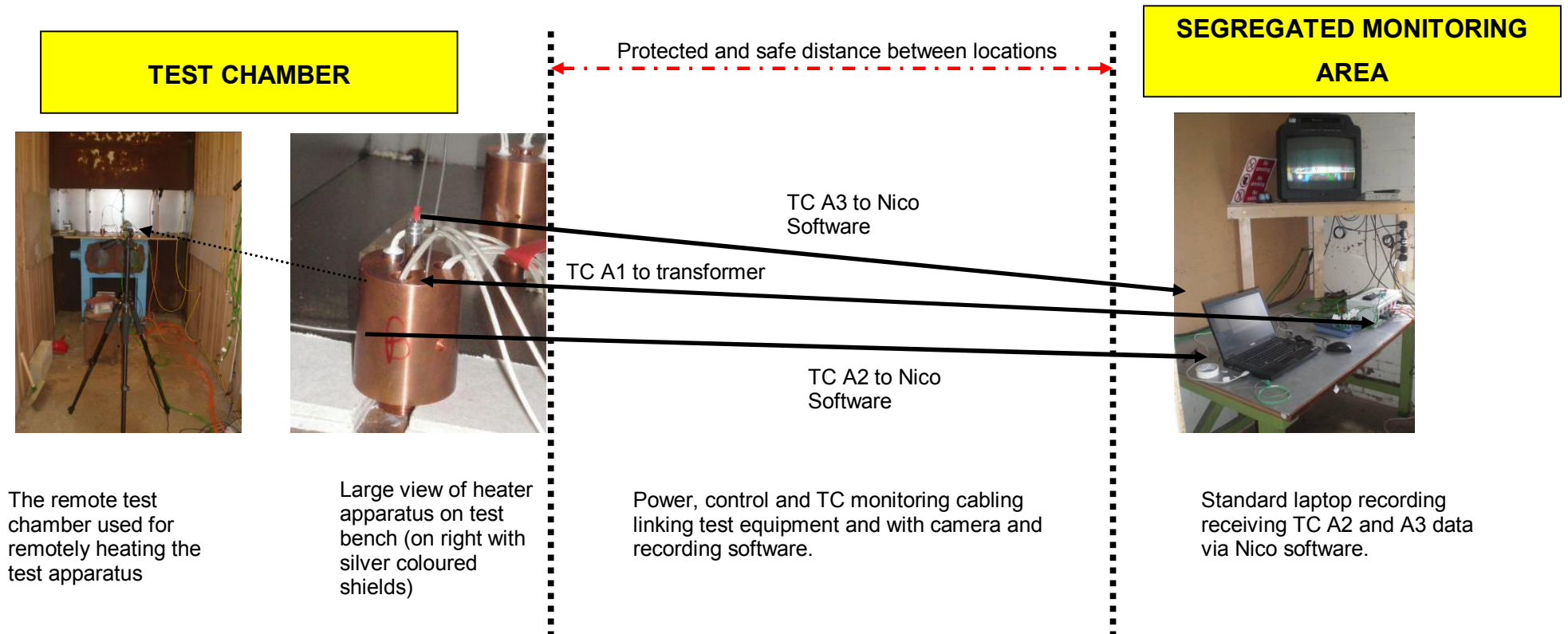


Figure 4.1 Diagram showing test house facilities and apparatus interfaces

5. CHAPTER 5. Results

5.1 ARC Results

Observed Onset Temperature	(T_o)	115.49 °C
Self Heat Rate at T_o	(M_o)	0.069 °C/min
Temp. at Maximum Rate	$(T_{mr})_{sys}$	176.51 °C
Max. Self Heat Rate	$(M_{mr})_{sys}$	2788.82 °C/min
Final Adiabatic Temp.	$(T_{ab})_{sys}$	184.09 °C
Adiabatic Temp. Rise	$(\Delta T_{ab})_{sys}$	68.60 °C

$$(where (\Delta T_{ab})_{sys} = (T_{ab})_{sys} - T_o)$$

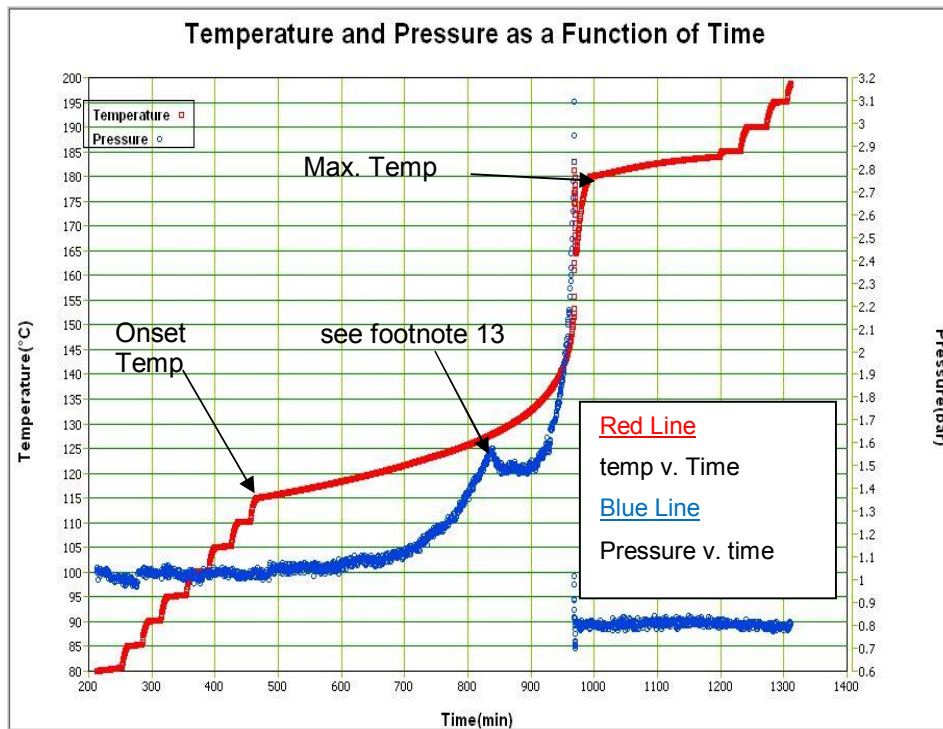


Figure 5.1 Graphical results showing time, temperature & pressure relationship during the ARC testing¹³.

¹³ Although pressure would be outside the scope of the copper heater experiments, it is interesting to note the pressure rise and drop at the 120°C / 825s point. This is probably due to the reactive flammable nature of the Rosin (extract from pine wood resin) at this point.

5.2 Copper Heater Results

The measurements were recorded via the Nico software (figure 5.2 is a typical output via PLW graph) and converted into excel spread sheet format and graphs to be found at Appendix E. Tables 5 - 12 have recorded the TCs A2 and A3 temperature and time points of interest throughout each experiment. Out of the 35 runs, 34 were seen to produce credible data for the purposes of these experiments. The temperature runs varied from 150°C to 180°C; some of these were held at these temperatures and others switched off once TC A1 had reached the required temperature. Temperature readings were recorded on TCs A2 and A3 whether the propellant cooked-off or not. In addition, two experiment Serials 34 and 35 used 200w heater elements in lieu of the 100w elements for comparison for heat rise rates and cook-off points. These last two firings were also video recorded using the Version 2 aluminium tube.

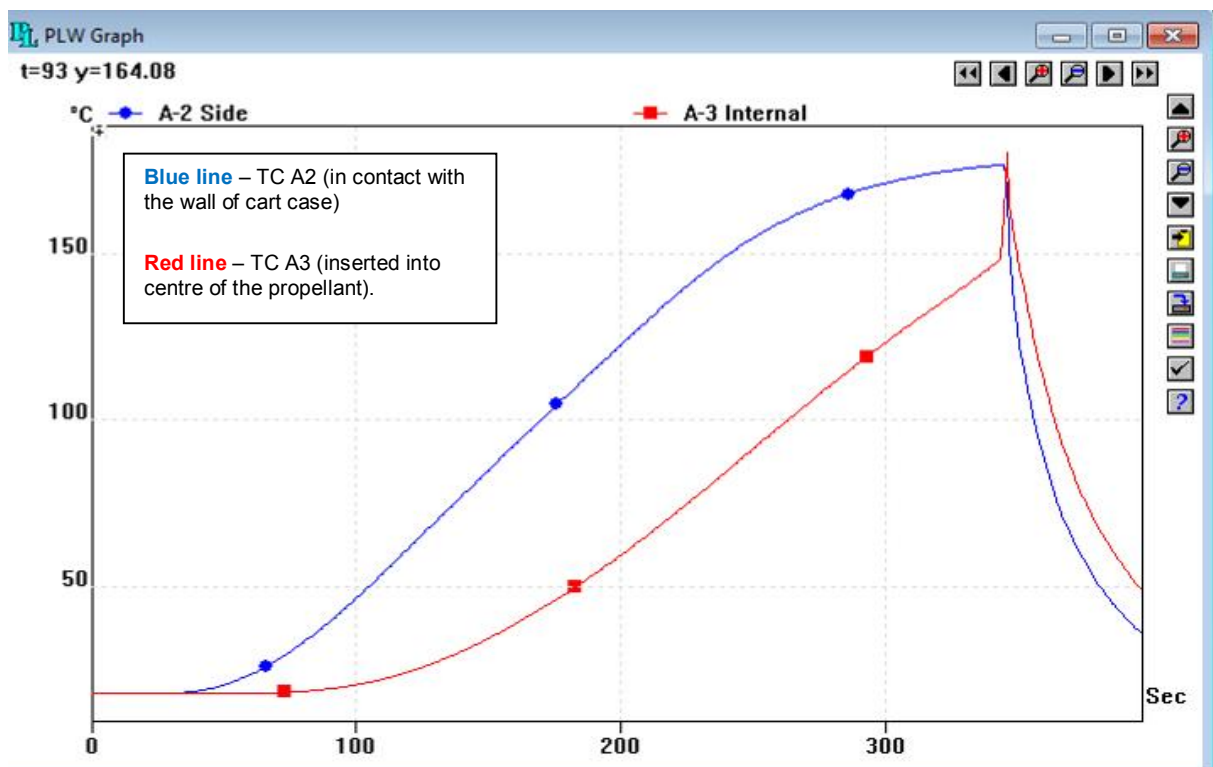


Figure 5.2: Example of a Nico result (the 1st experiment serial)

Copper Heater Results

Exp. Serial	Cartridge Details	T/C A1 Temp	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
1	RG 93	180°C	180°C at 336s	180°C at 336s	149°C at 336s	149°C at 336s	B (1)	Raise heater temperature to 180°C and hold at this temperature.
2	1K 07	150°C	145°C at 339s	145°C at 1861s	157°C at 1861s	157°C at 1861s	A (1)	Raise heater temperature to 150°C and hold at this temperature.
3	L2A2	165°C	165°C at 328s	165°C at 418s	171°C at 418s	171°C at 418s	A (1)	Raise heater temperature to 165°C and hold at this temperature.
4	CG 01	160°C	156°C at 320s	See Obs	165°C at 536s	See Obs	B (1)	Raise heater temperature to 160°C and hold at this temperature. Even at A3 peak temperature of 165°C no cook-off occurred. When A2 reached 100°C, A1 was raised to 170°C. Cook-Off at A3 eventually occurred at 170°C after 1843s (A2 temp was 166°C)

Table 5: Experiment (Exp) Serials 1 to 4

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
5	0.308(19)	See Notes	See Obs	140°C at 1189s	See Obs	174°C at 1189s	D (1)	Raise temperature of T/C A1 to 100°C then switch off for 60s then repeat at 120 / 140 / 160 180°C increments. Used to simulated gun firing / 60s pauses until cook-off.
6	0.308(1)	170°C	168°C at 338s	166°C at 384s	166°C at 384s	166°C at 384s	E (2)	Raise heater temperature to 170°C and switch off
7	FFV(1)	170°C	169°C at 334s	167°C at 376s	163°C at 376s	163°C at 376s	F (3)	Raise heater temperature to 170°C and switch off
8	0.308(3)	170°C	167°C at 334s	164°C at 393s	163°C at 393s	163°C at 393s	A (3)	Raise heater temperature to 170°C and switch off
9	FFV(2)	170°C	168°C at 305s	168°C at 352s	164°C at 352s	164°C at 352s	B (3)	Raise heater temperature to 170°C and switch off

Table 6: Experiment (Exp) Serials 5 to 9

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
10	FFV(3)	170°C	169°C at 319s	166°C at 374s	166°C at 374s	166°C at 374s	A (3)	Raise heater temperature to 170°C and switch off
11	0.308(4)	170°C	167°C at 311s	163°C at 364s	163°C at 366s	163°C at 366s	B (3)	Raise heater temperature to 170°C and switch off
12	0.308(5)	165°C	160°C at 314s	155°C at 452s	170°C at 452s	170°C at 452s	A (3)	Raise heater temperature to 165°C and switch off
13	FFV(3)	165°C	164°C at 315s	160°C at 394s	166°C at 394s	166°C at 394s	B (3)	Raise heater temperature to 165°C and switch off
14	FFV(5)	165°C	163°C at 299s	158°C at 396s	169°C at 396s	169°C at 396s	A (3)	Raise heater temperature to 165°C and switch off

Table 7: Experiment (Exp) Serials 10 to 14

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
15	0.308(6)	165°C	162°C at 295s	154°C at 428s	167°C at 428s	167°C at 428s	B (3)	Raise heater temperature to 165°C and switch off
16	0.308(7)	165°C	161°C at 297s	156°C at 391s	167°C at 391s	167°C at 391s	A (3)	Raise heater temperature to 165°C and switch off
17	FFV(6)	165°C	162°C at 300s	156°C at 391s	167°C at 391s	167°C at 391s	B (3)	Raise heater temperature to 165°C and switch off
18	FFV(7)	162°C	159°C at 327s	134°C at 514s	170°C at 514s	170°C at 514s	A (3)	Raise heater temperature to 162°C and switch off
19	0.308(8)	162°C	161°C at 386s	149°C at 550s	168°C at 550s	168°C at 550s	B (3)	Raise heater temperature to 162°C and switch off

Table 8: Experiment (Exp) Serials 15 to 19

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
20	0.308(9)	161°C	160°C at 335s	See Obs	164°C at 505s	See Obs	A (3)	Raise heater temperature to 161°C and switch off. No cook-off at initial peaks. A1 temp elevated to 161°C and held. Cook-Off: A2 = 161°C at 1287s and A3 = 165°C at 1287s
21	FFV(8)	161°C	160°C at 369s	146°C at 580s	167°C at 580s	167°C at 580s	B (3)	Raise heater temperature to 161°C and switch off
22	FFV(9)	161°C	159°C at 241s	See Obs	159°C at 438s	See Obs	A (3)	Raise heater temperature to 161°C and switch off. No cook-off at initial peaks. A1 temp elevated to 161°C and held. Cook-Off: A2 = 158°C at 925s and A3 = 170°C at 925s
23	0.308(10)	161°C	161°C at 350s	See Obs	167°C at 551s	See Obs	A (3)	Raise heater temperature to 161°C and switch off. . No cook-off at initial peaks. A1 temp elevated to 161°C and held. Cook-Off: A2 = 163°C at 1049s and A3 = 166°C at 1049s.

Table 9: Experiment (Exp) Serials 20 to 23

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
24	0.308(11)	161°C	160°C at 254s	See Obs	159°C at 432s	See Obs	E (3)	Raise heater temperature to 161°C and switch off. No cook-off at initial peaks. A1 temp elevated to 161°C and switch off. Cook-Off: A2 = 159°C at 922s and A3 = 172°C at 922s
25	0.308(12)	162°C	160°C at 343s	See Obs	162°C at 540s	See Obs	B (3)	Raise heater temperature to 162°C and switch off. . No cook-off at initial peaks. A1 temp elevated to 162°C and switch off. Cook-Off: A2 = 153°C at 920s and A3 = 170°C at 920s
26	0.308(13)	150°C	149°C at 159s	149°C at 1069s	164°C at 1069s	164°C at 1069s	E (3)	Raise heater temperature to 150°C and hold temperature.
27	0.308(14)	163°C	161°C at 347s	149°C at 516s	169°C at 516s	169°C at 516s	B (3)	Raise heater temperature to 163°C and switch off.

Table 10: Experiment (Exp) Serials 24 to 27

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
28	0.308(15)	162°C	161°C at 241s	See Obs	164°C at 444s	See Obs	E (3)	Raise heater temperature to 162°C and switch off. No cook-off at initial peaks. A1 temp elevated to 161°C and switch off. Cook-Off: A2 = 157°C at 706s and A3 = 175°C at 706s
29	RG 09(A)	162°C	189°C at 569s	See Obs	See Obs	See Obs	E (3)	Raise heater temperature to 162°C and switch off. Nil reading from A3. Temp elevated until propellant cooked off.
30	RG 09(B)	162°C	160°C at 332s	143°C at 566s	166°C at 566s	166°C at 566s	B (3)	Raise heater temperature to 162°C and switch off. .
31	RG 09(C)	161°C	161°C at 216s	See Obs	158°C at 404s	See Obs	E (3)	Raise heater temperature to 161°C and switch off. No cook-off at initial peaks. A1 temp elevated to 143 / 150°C and switch off. Cook-Off: A2 = 149°C at 1475s and A3 = 164°C at 1475s

Table 11: Experiment (Exp) Serials 28 to 31

Exp. Serial	Cartridge Details	T/C A1 Temp (°C)	T/C A2 Temp (°C) / Time (s)		T/C A3 Temp (°C) / Time (s)		Heater Body Serial	Additional Information on procedure and Observations (Obs)
			Peak	Cook-Off	Peak	Cook-Off		
32	0.308(16)	162°C	159°C at 210s	141°C at 478s	161°C at 378s	156°C at 478s	D (3)	Raise heater temperature to 162°C and switch off. 3 x 200w heaters used in copper heater jig – temp rise limited by controller.
33	0.308(17)	165°C	163°C at 349s	See Obs	153°C at 496s	See Obs	B (3)	Raise heater temperature to 165°C and switch off. No Cook-off. Heat increased to 165°C for video of cook-off (tube B used).
34	0.308(18)	170°C	168°C at 195s	167°C at 324s	153°C at 324s	153°C at 324s	D (3)	Raise heater temperature to 170°C and hold temperature. Video of cook-off (tube C used), a further 0.5g of propellant added and 200w heater elements
35	0.308(2)	180°C	179°C at 388s	179°C at 400s	162°C at 400s	162°C at 400s	B (3)	Raise heater temperature to 180°C and switch off. Test conducted as equipment confirmation test on 13 Jun 12.

Table 12: Experiment (Exp) Serials 32 to 35

CHAPTER 6 Observations and Analysis of Results.

6.1 ARC Results Analysis

The testing procedure has been previously described in Chapter 2 and further again along with the results from the THT report at Appendix D. The testing method used was H-W-S procedure and the propellant placed in a titanium spherical 'bomb' (figure 6.1). The volume of the bomb was 8.75ml and the 0.5g of Bulls Eye propellant tested equated to 4.5ml in volume. The thermal inertia calculation is detailed in paragraph 4 of Appendix D and the result being $\Phi = 9.78^{14}$. The key parameters were as follows:

Start Temperature – 50°C

End Temperature – 405°C

Heat Step Temperature – 5°C/min

Wait time – 15 min

The bomb ruptures at the final stage of the test, proving that the EM created a violent event (figure 6.2).



Figure 6.1 Titanium Bomb at Start



Figure 6.2 Titanium Bomb at Finish

¹⁴ THT Report Statement - Caution should be used when relating accelerated rate calorimetry data due to an actual plant process especially if significant extrapolations of the data or in the thermal inertia (Φ) are involved. Also the report highlights that many of the mathematical relationships used in the report are assumed valid only for non-autocatalytic, simple, single reactions.

The results have been recorded and a number of these have been graphically illustrated at paragraph 8 of appendix D detailing temperature, time, pressure and exotherm data combinations.

The temperature at maximum rate of pressure¹⁵ was around 150°C, this is probably due to the stabiliser DPA or EC being depleted. Figures 6.3 and 6.4 are examples of explosive ARC results previously conducted for other THT customers. Although not a useful for the Bulls Eye propellant ARC results comparison these can be used to compare against the results obtained for from the copper heater experiments.

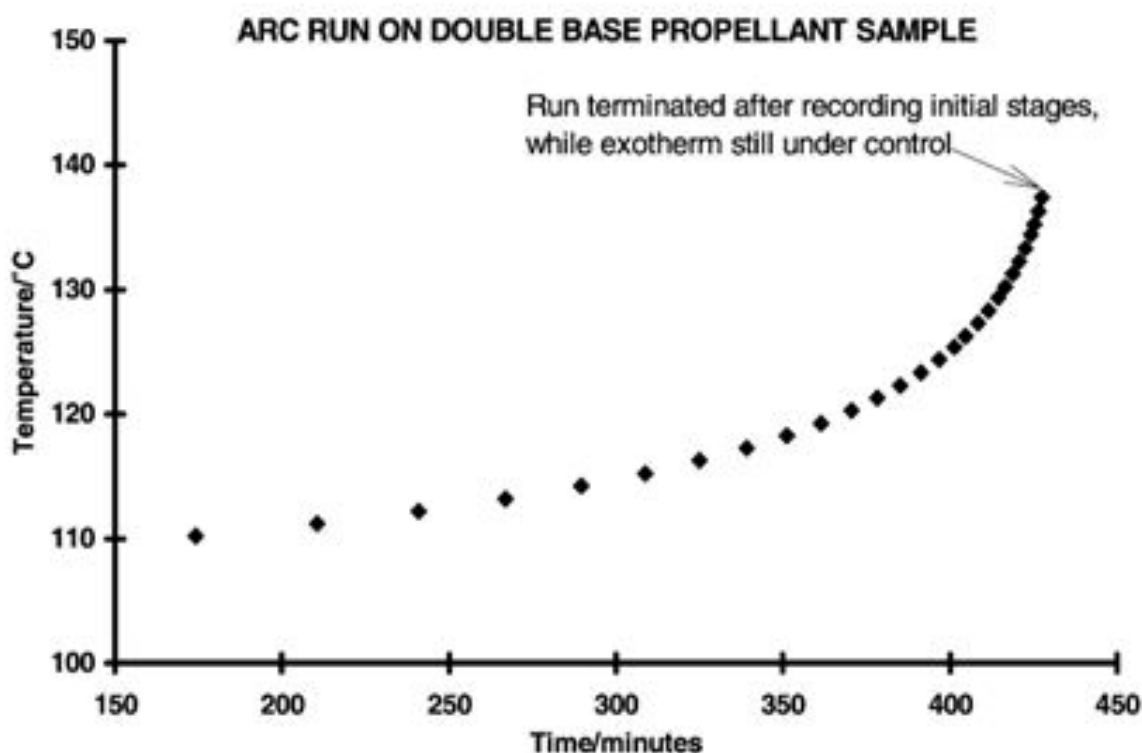


Figure 6.3 DB Propellant – Adiabatic temperature v. time curve¹⁶

¹⁵ The copper heater experiments have no correlation in respect to pressure. It was this data that underpinned the decision to produce vent holes in the aluminium plug of the locally designed apparatus.

¹⁶ These were obtained from a THT Ltd. power point presentation on 23rd April 2012.

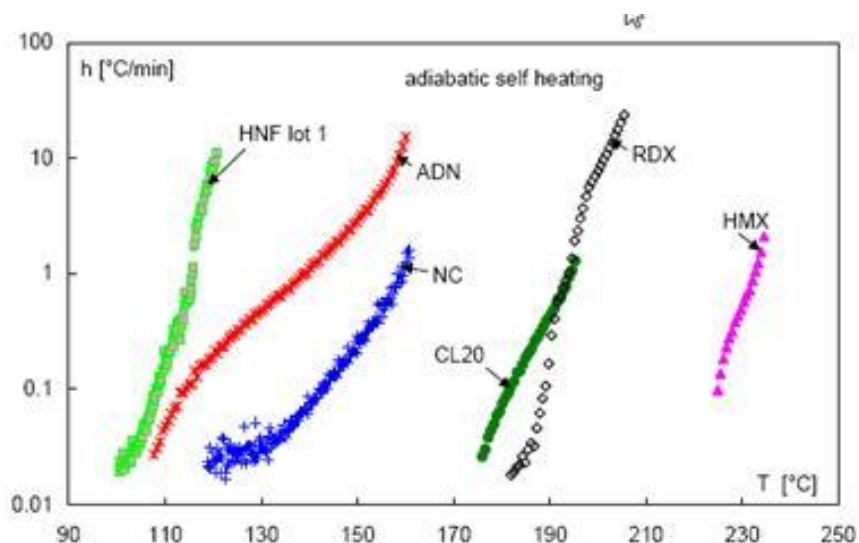


Figure 6.4 Adiabatic self-heating rates (h) as a function of the adiabatically reached temperature of other NC and other explosives¹⁶

6.2 Local Experimentation Results Analysis

The copper heater experiments highlight a number of successful results which was surprising considering this apparatus was development in process. Of the 35 experiments conducted 34 of these can be usefully analysed. The one remaining experimental result (test serial no. 29) was not evaluated because of a TC A3 failure (connecting plug). All graphical data corresponding to the results in tables 5 to 12 are found at Appendix E. The summary of the cook-off points are highlighted below:

6.2.1 TC A1 - 180°C

Two experiments were conducted at this ramp rise¹⁷. Both had different cartridge batches but the same Copper Heater serial¹⁸. Due to the slight difference in how the initial temperature was controlled for both experiments the

¹⁷ The first serial was taken to the experiment temperature and held. The second experiment was switched off on reaching the required temperature.

¹⁸ Experiment serial no.1 used Copper heater B(1) and experiment serial no.35 used B(3)

only data safe to compare is the initial ramp up temperature which took between 338s and 388s for TC A2 to reach its peak temperature.

6.2.2 TC A1 - 170°C

Six experiments were conducted at this ramp temperature. A seventh test was conducted at this temperature but additional propellant was added (0.5g) and the 100w heaters elements exchanged for more powerful 200w¹⁹. Cook-Off occurred on each of the experiments, and for the copper heaters with the 100w elements the cook-off range was:

- TC A2 'Peak' rise time variation – 167°C to 169°C @ 305s to 338.
- TC A2 Cook-Off temperature variation - 163°C to 168°C
- TC A3 Cook-Off time variation – 352s to 393s
- TC A3 Cook-Off temperature variation - 163°C to 166°C

The 200w element experiment (serial no.34) had a rapid TC A2 peak temperature of 168°C @ 195s. The TC A2 and A3 cook-off recordings were low at 153°C. This may have a similar correlation (excluding pressure) to the ARC results where the DPA and EC stabiliser decomposition may have occurred.

6.2.3 TC A1 - 163°C to 165°C

Between these temperature ranges there were 9 firings and all achieved 100% cook-off. The time vs. temperature ranges are detailed below:

- TC A2 'Peak' rise time variation – 160°C to 165°C @ 295s to 314s.
- TC A2 Cook-Off temperature variation - 149°C to 162°C
- TC A3 Cook-Off time variation – 394s to 516s

¹⁹ The temperature was held at 170°C for this experiment

- TC A3 Cook-Off temperature variation - 164°C to 171°C

6.2.4 TC A1 - 161°C to 162°C

Between these temperature ranges there were 5 out of 13 firings achieved cook-off rate (39%). The time vs. temperature ranges are detailed below:

- TC A2 'Peak' rise variation – 159°C to 161°C @ 327s to 386s.
- TC A2 Cook-Off temperature variation – 134°C to 149°C
- TC A3 Cook-Off time variation – 394s to 580s
- TC A3 Cook-Off temperature variation - 166°C to 170°C

6.2.4.1 Use 200w Heater Elements.

For experiment serials no. 32 and 34, the three 100w heaters were exchanged for three 200w heaters. The test was conducted by raising the TC A1 temperature to 162°C and 170°C for each firing. On both occasions TC A1 was switched off when the respective temperatures were reached. The only other difference was the mass NEQ of experiment serial no. 34 was increased by 0.5g (total NEQ in cartridge case 2.5g +/- 0.01g). Results as follows:

- Experiment Serial No.32 - cart serial no.0.308(16) and Copper Heater Serial D3
 - TC A2 'Peak' rise Temperature - 159°C @ 210s
 - TC A2 cook-off temperature - 141°C @ 478s
 - TC A3 'Peak' rise Temperature - 161°C @ 378s
 - TC A3 cook-off temperature - 156°C @ 478s
- Experiment Serial No.34 - cart serial no.0.308(18) and Copper Heater Serial D3
 - TC A2 'Peak' rise Temperature - 168°C @ 195s

- TC A2 cook-off temperature - 167°C @ 324s
- TC A3 'Peak' Temperature - 153°C @ 324s
- TC A3 cook-off temperature - 153°C @ 324s

A comparison with the use of 100w heaters in experiment serials 8 and 19 where TC A1 was raised to 170°C and 162°C respectively:

- Experiment Serial No.8 - cart serial no.0.308(3) and Copper Heater Serial A3²⁰
 - TC A2 'Peak' rise Temperature - 167°C @ 334s
 - TC A2 cook-off temperature - 164°C @ 393s
 - TC A3 'Peak' rise Temperature - 163°C @ 393s
 - TC A3 cook-off temperature - 163°C @ 393s
- Experiment Serial No.19 - cart serial no.0.308(3) and Copper Heater Serial B3²¹
 - TC A2 'Peak' rise Temperature - 161°C @ 386s
 - TC A2 cook-off temperature - 149°C @ 550s
 - TC A3 'Peak' rise Temperature - 168°C @ 550s
 - TC A3 cook-off temperature - 163°C @ 550s

Although these were statistically low numbers of experiments with use of 200w and 100w heaters for comparison of results, the limited experiments still identified a faster ramp up temperatures with the 200w heaters ranging from

²⁰ The weight of Copper Heater A3 is 887g.

²¹ The weight of Copper Heater B3 is 888g

162°C to 170°C. They also identified a much earlier and quicker cook-off points (and using the same 0.308 cartridge case batches as a standard).

6.2.4.2 Statistical Analysis of 162°C Temperature Zone

The table below extracts all relevant data of these experiments (but only where the temperature controller was switched off once TC A1 reached the peaks of 162°C and 161°C). With the exception of one cartridge, the 0.308 CCs did not cook-off. The 0.308 that did show an event, the TC A2 reading indicated a higher temperature compared to the FFV(7) and RG 09(B) cartridges under experiment (see table 13).

Cart Serial	Copper Heater Serial	A2 Peak Temp	A2 Cook-Off	A3 Cook-Off
FFV(7)	A(3)	159°C @ 327s	134°C @ 514s	170°C @ 514s
0.308(8)	B(3)	161°C @ 386s	149°C @ 550s	168°C @ 550s
0.308(12)	B(3)	160°C @ 343s	No Cook-Off	Peak temp of 162oC @ 540s but no Cook-Off
0.308(15)	E(3)	161°C @ 241s	No Cook-Off	Peak temp of 164oC @ 444s but no Cook-Off
RG 09(A)	E(3)	189°C ²²	Test Terminated	Test Terminated
RG 09 (B)	B(3)	160°C @ 332s	143°C @ 556s	166°C @ 556s

Table 13: Summary of Results from a 162°C Ramp Temperature

²² Experiment was terminated due to TC A3 connecting plug failure/damage from previous experiment.

6.2.4.3 Statistical Analysis of 161°C Temperature Zone

Six experiments with one cook-off (table 14). It has been observed that the heavier cartridge case (FFV variant) was the only one to cook-off in this series.

Cart Serial	Copper Heater Serial	A2 Peak Temp	A2 Cook-Off	A3 Cook-Off
0.308(9)	A(3)	160°C @ 335s	No Cook-Off	Peak temp of 164°C @ 505 s but no Cook-Off
FFV(8)	B(3)	160°C @ 369s	146°C @ 580s	167°C @ 580s
FFV(9)	A(3)	159°C @ 241s	No Cook-Off	Peak temp of 159°C @ 438s but no Cook-Off
0.308(10)	A(3)	161°C @ 350s	No Cook-Off	Peak temp of 167°C @ 551s but no Cook-Off
0.308(11)	E(3)	161°C @ 350s	No Cook-Off	Peak temp of 159°C @ 432s but no Cook-Off
RG 09(C)	E(3)	161°C @ 350s	No Cook-Off	Peak temp of 158°C @ 404s but no Cook-Off

Table 14: Summary of Results from a 161°C Ramp Temperature

6.2.5 TC A1 - 160°C

One experiment with 100w heater elements (experiment serial no. 4) and switched off on reaching the required temperature. TC A2 Peak temperature was 156°C at 320s and the TC A3 peak being 165°C at 536s but cook-off did not occur.

6.2.6 TC A1 - 150°C

One experiment with 100w heater elements (experiment serial no. 2) and the temperature held. TC A2 Peak temperature was 148°C at 339s and the TC A3 peak being 157°C at 1861s until cook-off (figure 6.5). From 1500s (approx.) there was evidence on the graph that the propellant (TC A3) is gradually increasing in temperature at 152°C whilst TC A2 is held at 148°C. This is a similar comparison to the ARC run-away point but within a shorter period.

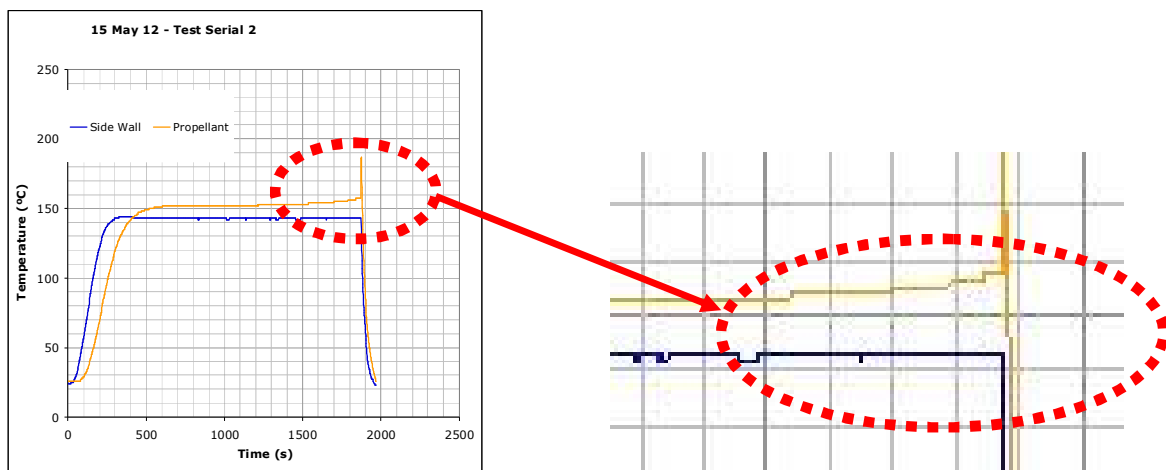


Figure 6.5: Serial No.2 TC A2 temperature constant at 148°C shown by blue line on graph. Decomposition to thermal run away identified.

6.3 Other Observations

Most of the experiments had a start temperature at room temperature which was circa 20°C. These were recorded by the Nico software via TCs A2/A3 and can be clearly seen on the spreadsheet graphs at Appendix E. However, due to experimental time constraints some start temperatures were above room

temperature – occasionally peaking 43°C prior to the commencement of an experimental run. These seemed to make very little difference in accelerating the ramp temperatures (15 seconds at best) and were insignificant on cook-off times and temperatures variations recorded from TC A3.

Experiment serial no.28 is where 100w heaters were used to raise TC A1 to 162°C and switched off. The TC A2 initial peak temperature maximum was reached in 241s; nearly 60s ahead of comparison experiments. In this experiment the TC A3 reached a peak 164°C in 444s but failed to cook-off. Whilst an earlier identical experiment (serial no.19) took 386s for TC A2 to reach peak temperature (with the same TC A1 ramp temperature) and TC A3 reached a peak temperature of 164°C at 550s (and cooked-off at this point). The only logical reason for this can only be that one or more 200w heater element were mistakenly used but recorded as a 100w heater element experiment. Also the rapid temperature rate rise and less time/heat exposure to the cartridge made a difference by not cooking off.

Experiment serial no. 32 is also worthy of note. The use of 200w heater elements brought the copper core temperature to 150°C in approx. at 120s. TC A2 then recorded a peak temperature of 160°C at 220s. The propellant core temperature peaked to 161°C at 378s and cooked off 156°C at 478s; the copper core / cartridge wall temperature had dropped to 141°C (20°C below the propellant core temperature). In essence although the core propellant was cooling, and the surrounding temperature was also cooling, the propellant still cooked-off.

7. CHAPTER 7 Discussions

7.1 Comparisons with Previous Experiments

Because of the harsh conditions encountered in actual gun firings, the acquisition of experimental data can be quite difficult to obtain. It has been calculated that cook-off occurs from a propellant charge of an APDS 120mm round in 120s if the combustible cartridge case is in contact with steel at 200°C whereas at 250s cook-off occurs in a few seconds (Lawton and Klinenberg 2009 p443). It is also stated in this reference that a suitably safe temperature is about 180°C when it takes about 1800s before cook-off may occur (however, as a barrel will cool significantly in this period it is unlikely that cook-off will ever really occur). In addition, it is also stated that until this 'limit' is reached the gun can be fired at any suitable rate, but once the cook-off temperature is reached the firing rate must be drastically reduced. High temperature rates must be considered along with rates of fire which can lead to early barrel wear rate.

Experiments on Cook-Off temperatures were carried on DB Hercules Green Spot propellant out by Tuisku (1996) who recorded cook-off results from 196°C @ 29s to 264°C @ 0.62s; this is in clear contrast on the ramp temperatures that could be achieved with the slower copper heater. Tuisku's test equipment involved the use of a heated container for the propellant sample that can be brought into contact with the heating surface under pressure by a weighted probe. There was no cartridge case but the thermal property of brass had been calculated at 0.3s to reach a steady state temperature and was stated not to give much thermal protection. This once again is in contrast to the copper heater results where the FFV variant cartridges cooked-off earlier than the lighter mass 0.308 WIN cartridges. However, analysis of Tuisku's results graph identified a characteristic 'hump' on the plotted results in the case of a cylindrical grain lying on its side on the heating surface. It was suggested that the total heat capacity before initiation of the grain varies non-linearly as the initiation point moves deeper into the grain (in response to a decrease in the heating element temperature). Of interest, Tuisku has also highlighted from the

experiments where propellant was forced onto the heated surface (by application of mechanical load of the apparatus) could greatly decrease cook-off time. In SAA, loading propellant charges to full capacity may lead to situations where forcing the bullet down onto the propellant could have this unwarranted situation. Unfortunately, this particular apparatus was not pursued for conducting this thesis experiments because of the longer cook-off times could not be replicated to that of the copper heater.

7.2 Comparisons with Previous Gun Trials

STANAG 4432 sets out the S³ requirements for Cannon ammunition (12.7 to 40mm). Within this document 'Hot Gun Cook-Off' testing requirement is included but test procedures are referenced to national procedures and could not be expanded due to the classified nature of the material available (unlike other munition test requirements which are covered in further detail as unclassified STANAGs). However, the following is an unclassified military approach to trials meeting the cook-off points and an critical firing limitation instruction:

7.2.1 27mm Aircraft Cannon

This cook-off testing was based on a trial conducted by Gunther and Amrhein (2008). The ammunition is FAP (no high explosive, just propellant and primer EMs) and has a baseline qualification test that it shall withstand +120°C for hours without pyrotechnic function or explosive event. The test ammunition was conditioned at a temperature of +70°C²³ for more than 4 hours but no more than 10 hours with three test temperatures defined as +124°C, +126°C and +128°C. In addition to the cook-off test, the rounds are required to be fired if surviving the cook-off tests to ensure chamber pressures are comparable to former pre-heated ammunition.

²³ The reason for initial high conditioning temperatures are calculated on the thermal effects that ammunition and armament stores experience from fast jet aero-kinetic heating effects

7.3 Results Comparison against Previous HT Tool Modelling

A previous MSc Explosive Ordnance Engineering module experiment conducted by Pitcher (2010) can be compared using the results on the Shrivenham 25m range using a 5.56mm Minimi Light Machine Gun (MLMG). The purpose of this experiment was to compare the HT Tool model to compare and simulate that with the live firing of this LMG. In this experiment only the outer heat of the barrels were recorded by use of TCs and software to give time vs. temperature results.

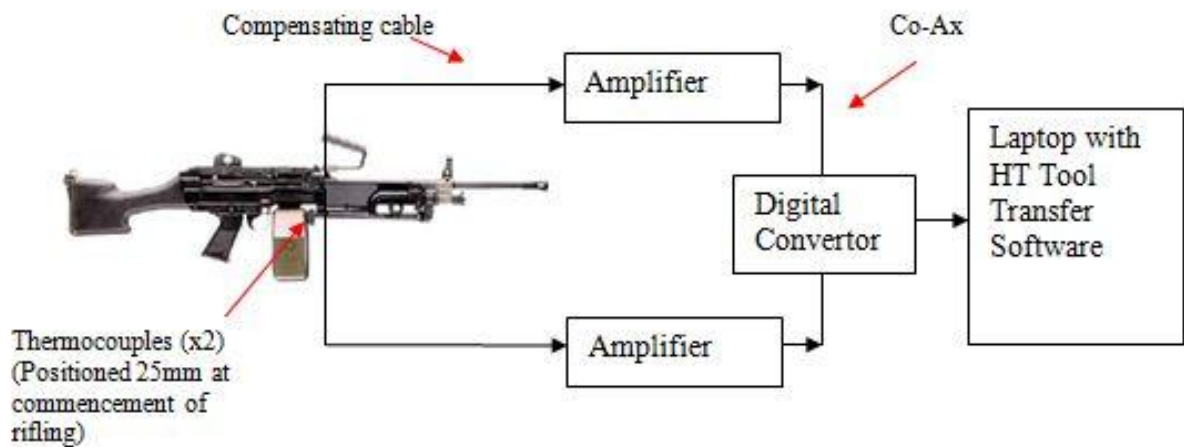


Figure 7.1 MLMG Experiment Set Up for measurement of barrel temperature

Ambient temperatures were recorded (approx. 0 – 5°C) prior to firing and a burst of 5 rounds fired at 20s intervals by manual reloading of 10 belts of ammunition (i.e. 10 bursts). The cycle equated to 0.5s of firing and 19.5s of non-firing for 10 cycles. The use of HT (Heat Transfer)²⁴ Tool during the experiment vs. modelling results did not differ considerably.

²⁴ The HT Tool is software that was used during the Propellants, Internal ballistics and Weapon thermodynamics phase of the EOE MSc for the calculation of heat transfer through a gun barrel.

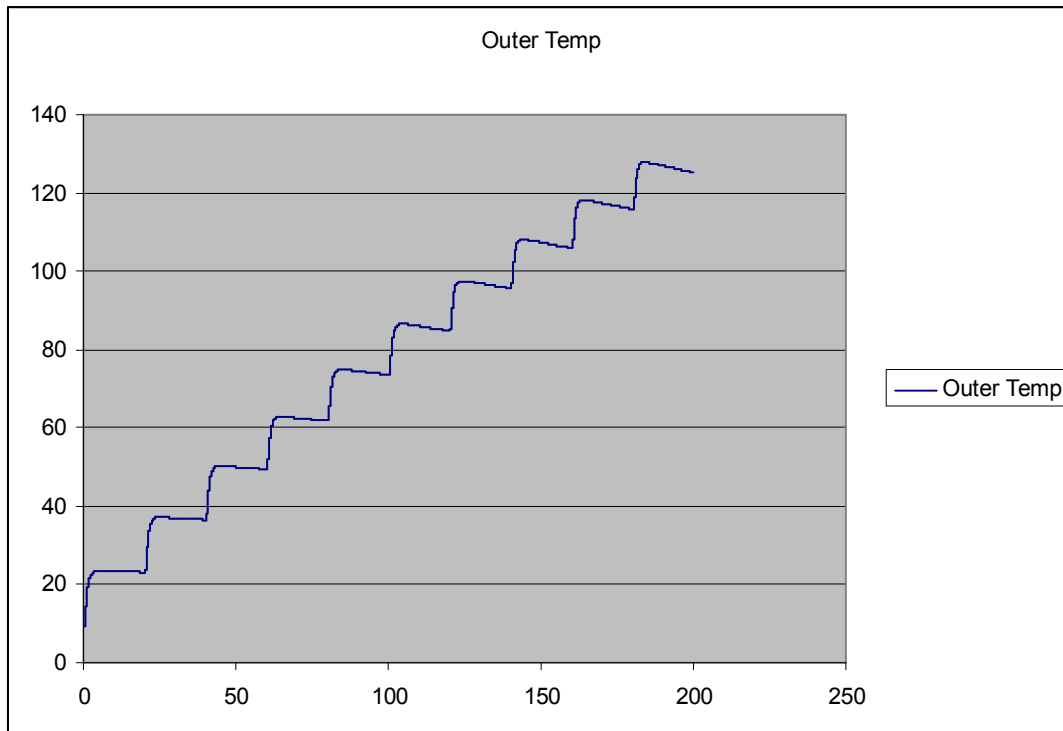


Figure 7.2 HT Tool chart showing the experimental firing run (200s)

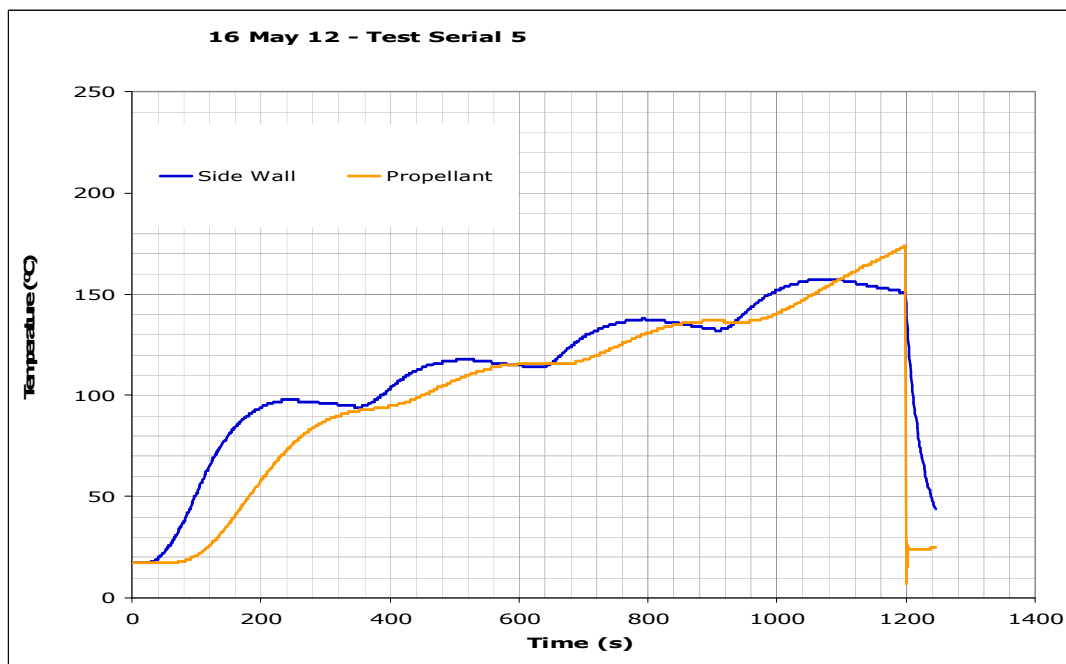


Figure 7.3 Copper Heater experiment No.5 simulating gun fire (heat, wait and increase)

From the HT Tool graph in figure 7.2 it can be seen that the outer barrel temperature is approx. 130°C at 200s (although bore temperature peaks to nearly 180°C for less than 1s). As a comparison, the copper heater result shown in graph of figure 7.3 is nearly 100°C within the same time period (but this temperature rise to the propellant is uninterrupted during this time period – one constant rise during this initial ramp temperature). Although specific heat capacities of the copper and steel material properties differ (0.38 vs. 0.44 kJ/kg K) and there is certainly scope to use the copper heater experiment outputs into HT tool to predict cook-off values. Although the study on the cool down rates could be challenging with the copper heater mass as it currently stands.

7.4 Experimentation vs. Modelling

Other modelling attempts have been attempted and matched to experimental data. Stroud (1976) produced a report supported by modelling vs. experimentation on comparison for cook-off of 30mm practice ammunition (steel case with only primer and propellant EMs researched). The aim was to predict the critical temperatures for both these EMs and it is clearly evident that the critical EM is the AR5401 propellant (primer has a high cook-off at circa. 250°C). In comparing the experimental and modelling data for the propellant, Stroud found the cook-off results differed by 3°C (173°C in favour of the experimental measurement). The theoretical measurements used were the Frank-Kamenetski theory and HF modelling code. The practical experiment was the test of 0.2g in a TC / cylinder combination heated at 5°C/min. Although this heat rate rise is considerably lower than the Copper Heater experiments, Stroud rightly initially questioned his inherited data of 130°C propellant cook-off critical temperature as this would take approximately 4 hours to achieve this event. He argued quite sensibly that from his experimental results that this critical temperature should be at least 150°C for operational gun firing purposes.

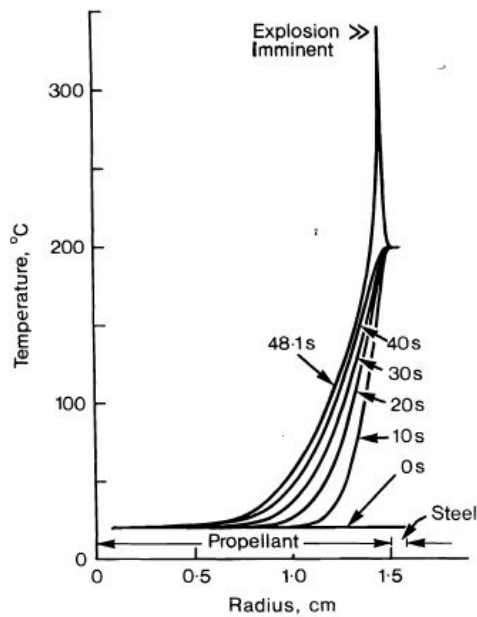


Figure 7.4 HF Modelling at 200°C

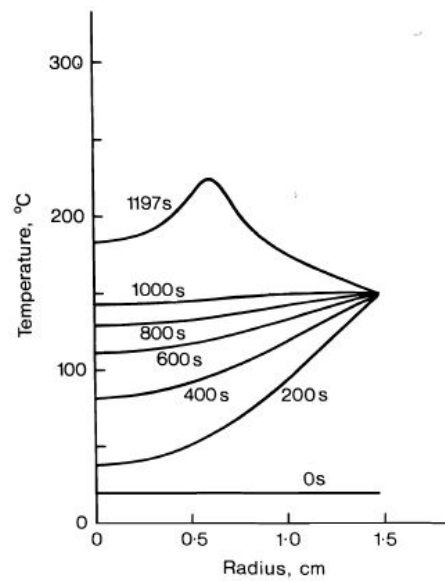


Figure 7.5 HF Modelling at 150°C

Stroud's modelling attempts at Figure 7.4 profiles the cylindrical section (case and propellant) with the round placed in the breech cylinder at 200°C. The radius is 15mm (side wall of cartridge to centre of propellant) with the temperature rise in °C, the calculation performed in time steps of 5×10^{-4} seconds and a total of 40 cells used (4 being in the steel case). Within the graph the times are clearly shown when a temperature is reached within a certain zone of the cartridge. Cook-Off is predicted when the surface of the case is $>300^{\circ}\text{C}$. The modelling at figure 7.5 is when the breech cylinder is at 150°C and the time within the graph highlights the temperatures throughout the propellant (steel case omitted from calculation to conserve computation time, only calculated with 30 nodes and 1s time steps). The core temperature obviously rises with exposed time to the elevated temperature but the spike indicates that a cook-off is imminent.

Of interest from the results table within the Stroud report is the cook-off temperature at 180°C is 250s, considerably less than the 1800s Lawton and Klingenberg (1996) prediction as safe limit at this temperature. The conclusions

still left the findings robust enough to support the 150°C limit but a recommended 134°C critical rise temperature should be the safety limit until further evidence is available. In Stroud's analysis of the round, it is maintained that the round is in perfect thermal contact with the breech cylinder and this acts as a constant temperature heat source and/or sink. However, unreferenced evidence is quoted to suggest that the round is in poor thermal contact and the flow of heat would be impeded, and aid a slight decrease in critical temperature. I would question this evidence based on results from the copper heater experiments where the slight differences in case diameters made little difference in time to cook-off but lighter mass cartridges extended time to cook-off. I would agree with Stroud's analysis that an experimental temperature rise of 5°C/min is significantly less than could be expected in an operational 30mm cannon and the rationale of maintaining the low critical temperature due to the limited evidence at the time of this report²⁵.

7.5 Propellant Cook-Off Temperature Prediction

In addition to Cook-Off issues to gun systems, this is also seen as the critical ignition temperature and important parameter required insuring safe storage and process operations related to propellants. Pickard (2000) covers a practical approach for prediction and validation of Initial temperature (T_i) data. This area was briefly covered in Chapters 1 and 2 of this thesis and the following equations define the net heat flow for systems governed by convective and conductive heat flow:

²⁵ This experiment and modelling report was conducted in 1976. Computers have become considerably more powerful, so computation time for the same experiment now would certainly be worth re-visiting.

$$\rho C_p \frac{dT}{dt} = \rho Q A e^{-E/RT} - \frac{hS}{V} (T - T_0)$$

$$\rho C_p \frac{dT}{dt} = \rho Q A e^{-E/RT} + \lambda \nabla^2 T$$

(E = activation energy, A = pre-exponential factor, Q = the total heat, ρ = density, C_p = heat capacity, T = reactant temperature, t = time, R = the radial gas law, h = the heat transfer coefficient, λ = the thermal conductivity, S = the surface area and V = the volume)

Equation 6: Defining the net heat flow (convective and conductive)

Zinn and Mader (1960) obtained a numerical solution for the case of conductive heat transfer. The above steady state equations above were derived by Semenov and Frank-Kamenetskii and are given by the following equations (Pickard 2000).

$$\frac{\rho r^2 Q A E e^{-E/RT}}{\lambda R T^2} = \delta$$

Equation 7: Showing shape parameters with various values

$$\frac{\rho V Q A E e^{-E/RT}}{h S R T^2} = e^{-1}$$

Equation 8: For use when the potential for thermal run-away exists

In equation 7, δ is a shape parameter with values of 0.88, 2.0 and 3.32 for an infinite slab, cylinder and sphere respectively. Equations (Eq.) 7 and 8 are critical conditions; if the left hand side of Eq. 7 exceeds $1/e$ or Eq. 8 exceeds δ the potential for thermal runaway exists. These equations assume zero-order kinetics. On validating predictions for T_i reliable data for A, E and Q are required and Pickard maintains that single set of kinetic parameters do not

necessary exist for explosives. The reason being that the variations in impurities or traces of residual solvents can influence the apparent kinetic parameters (small perturbations may have a significant influence upon T_i). Pickard describes a conductive heat flow for PETN experimental; very similar to the copper heat experiments but with the exclusion of Al plug vent holes and suggest its use for the measurements of small charges such as found in air bags. Figure 7.6 shows a cook-off experiment on an unspecified smokeless propellant.

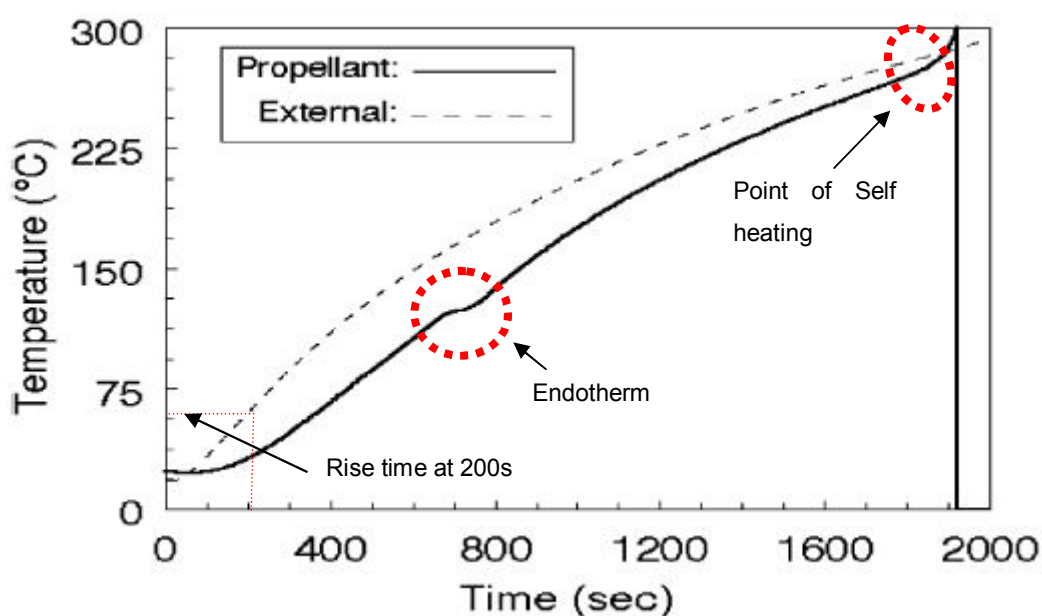


Figure 7.6 Graph showing a cook-off of a smokeless propellant (Pickard 2001)

The type of propellant in figure 7.6 is not referenced but being smokeless it is assumed to be DB. The heating chamber under experiment was 80mm x 5mm (assumed thin walls at these dimensions), of metallic construction and containing 0.6g of commercial propellant. The run time of the experiment was approximately 1800s where the cook-off occurred at approximately 300°C. The self heating commenced at approximately 255°C and a small endotherm at approximately 150°C. The result is a significantly higher cook-off temperature then compared to Test no 35 shown in Appendix E where cook off occurred at

163°C in approximately 400s. However, it must be noted that the cartridge wall rise temperature was significantly higher than that of Pickard's experiment (at 200s – approximately 60°C v. 110°C copper heater serial no.35). Once again this underpins that predicting cook-off in gun chambers requires realistic parameters from experimentation before calculation and modelling methods are attempted.

7.6 Real Time Prediction Assumptions and Cook-Off Reduction

As past research has shown, and this thesis generally supports, is that the likelihood of cook-off occurring is when left in a barrel at elevated temperatures for an extended period of time (i.e. from a misfire). A thermal analysis conducted by Witherill and Pflegl (2001) made a valid assumption that the heat required for initiating propellant cook-off must be conducted rearward from the hotter barrel material located forward of the case mouth. Rear ward conduction of heat will cause the peak temperature on the inside surface of the case to decrease with the axial distance from the case mouth. Since the case shoulder region is the most forward position where propellant can reside, and therefore the most likely region where propellant cook-off will be initiated during a misfire event. The copper heater experiment was unable to apply the ideal thermal transfer in this location and may have to be assumed as an error in representing a true gun chamber scenario in the experiments.

Witherill's testing used steel and aluminium casings with the steel cased rounds cooking off later than the aluminium (the cook-off was circa 120°C in 900s period). Although with the use of chromium plated barrels a minimum cook-off time of 1780s was reported (however a nitrated barrel showed that time reduced to 640s). In attempts to increase the rate of fire before cook-off occurring, Cohn (1961) describes approaches that have been used to prevent cook-off by preventing temperature reaching this threshold. Methods like

blowing air over the gun²⁶ or refrigerating the system have been known to work well but there is the penalty in weight, space and cost. Cook-Off in cannons fitted to aircraft can be catastrophic with possible loss of aircrew and an expensive platform, but space for a large coolant system will be limited. Cohn reported that a simpler solution was used on the M39A2 gun, which is an automatic 'revolver' type firing about 25 rounds per second from a single barrel. To achieve a reduced heating transfer, seals are required in the 'revolver' close to the clearance space between the revolving drum and the rear barrel face.

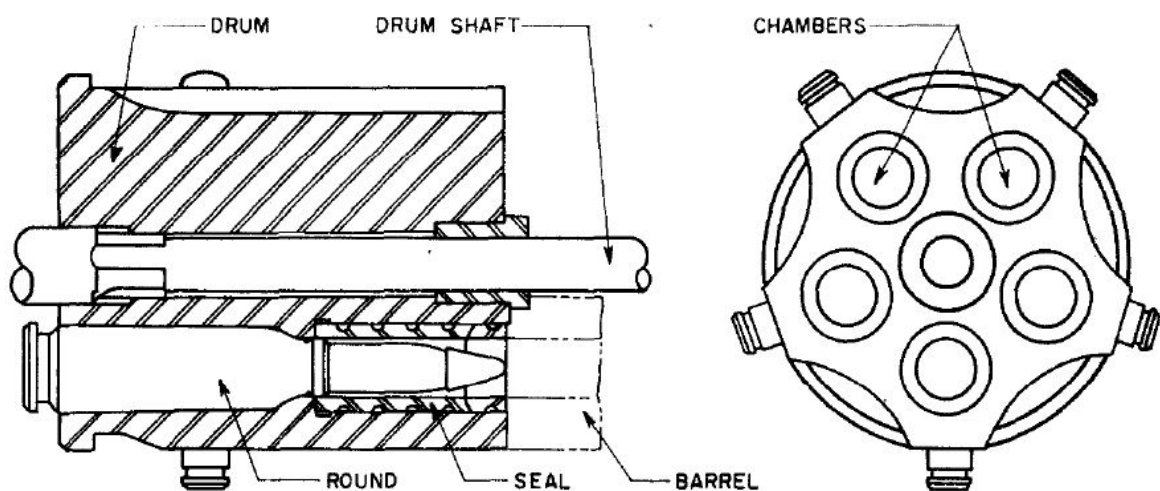


Figure 7.7 Drum of M39A2 show seal and other drum components

The seals (one per chamber) are housed in the cavity in the front of the drum (figure 7.7). This is the region where burning gases leave the cartridge case, gets hottest. The air space and drum retards the transfer of heat to the cooler portions of the drum. The seals are skirted and have a smaller mass and, therefore, less heat capacity. The report highlighted the simple seal innovation has not only extended the time to cook-off (increased firing rate) but also eliminated drum erosion and the seals can survive >10,000 rounds fired. From

²⁶ This has been tried on modern guns fitted to fast jet platforms by the use of taking in slip stream air during flight. However, consideration must be taken into account for aero-dynamic kinetic heating caused by turbulent air flow at high Mach speed at low altitude levels. In essence, this could increase the heating rate and induce earlier cook-off times.

this idea, the change to material/weights supports the mass reduction or reduced thermal capacity around a propellant in line with the findings of the copper heater experiments with cartridge case mass reduction.

CHAPTER 8 Conclusions

This project researched the phenomena of cook-off temperature of gun propellants in 'hot' gun chamber situations. It also researched methods and equipment available to obtain useful propellant cook-off data in research of predicting a safe operating temperature at which the propellant would be at an unacceptable limit.

A very useful and acceptable tool was modified from an existing experimental apparatus to produce a number of credible measurements in simulating a 7.62mm cartridge containing 2g of DB Bulls Eye smokeless propellant. It has been compared to previous experimentation methods and attempted modelling techniques to produce a credible range of results to aid propellant cook-off points.

From the results, the critical temperature on the wall of the cartridge was between 161°C and 162°C. Below the 161°C limit, the propellant was unlikely to cook-off and above 163°C cook-off was assured. However, this did depend on the rate of heat rise where in one experiment 200w heater elements were used the propellant core temperature was 156°C at cook-off (compared with the mean average of 162 - 168°C using the 100w heater elements).

Of interest was the effect of cartridge mass difference (0.308 WIN vs. FFV variants). These were the largest in number within the batches experimented and the FFV variant had a 6% weight advantage and slightly larger in diameter on average by 0.19mm. The air space difference between the cartridge wall and copper heater is assumed to be initially insignificant for heat transfer losses, and so was the cartridge mass difference (average 0.74g). However, there was evidence that although cook-off temperature did not significantly increase in identical tests the time to explosion did for the lighter 0.308 WIN cartridge. It can be reasonably assumed that once the copper heater had reached the required test temperature and switched off, the heavier mass of the cartridge retained

heat longer to accelerate the decomposition of the propellant leading to earlier cook-off.

It can also be seen that at a certain heating rate the Bull Eyes core propellant can be liable to cook-off even if the wall temperature is significantly lower (between 15 and 20°C). These cartridges can also be prone to cook-off within 1800s at cartridge wall constant temperature of approximately 145°C; the core propellant started thermal run away at approximately 1600s until the ignition temperature of 148°C.

The use of ARC certainly aided in the unknown thermal properties of this propellant type and the 'H-W-S' method was the correct one to use at the early stage of the critical temperature analysis. It would have been of interest to compare the copper heater heating rates with the ARC apparatus in lieu of the 'H-W-S' method.

The copper heater experimental apparatus performed well and Version 3 survived each experiment with only the loss of only 1 x TC and the occasional heater element (the cost of each <£20).

As with all new experiments there are number errors which should be considered if this approach to cook-off in gun chambers is pursued further.

- Statistical Numbers were low and not fully validating the cook-off results.
- The Nico software for measuring the cartridge wall and core propellant is accurate to 0.01°C. These experiments results were rounded up to the nearest 1°C.
- The temperature controller had a built in system which slowed down the heat rise as the required TC A1 temperature was reached. This made the heat rise non-linear as this limit was approached (and slowing the true heat rise, possibly delaying and increasing the cook-off points)
- It was assumed that all cartridges were all of a brass grade 70/30 (copper/zinc) and no additional material properties

- From research, it was established most heat transfer in a barrel/chamber of a gun will be from the cartridge case shoulder that will first influence propellant cook-off. The copper heater could not achieve this.
- Not all the T_i was identical but the differing temperatures may be useful for future follow on experimentation and analysis.
- The experiments were vented and not really comparable to other researched cook-off data which contained a pressure (this venting was a safety and a TC integration issue. The ARC results identified an early onset temperature when the pressure starts to increase).
- The amount of propellant and type did not replicate that of the UK 7.62mm round. This experiment will lack read across to assess the realistic cook-off for this round.
- The experiment did not truly replicate the temperature rise of a firing gun and introduction of a fresh round into a 'hot' chamber.

With a few iterations to the design of the copper heater apparatus and temperature inputs, this methodology and equipment has the potential to be an excellent, cost effective foundation stone in the measurement of cook-off temperatures of propellants in SAA < 12.7mm in calibre. There may also be potential for gun ammunition > 12.7mm with larger copper heater and additional heater elements. These results certainly (and other referenced documents) cautions the use of the 'generic' the propellant cook-off safe limit of 1800s at temperature not exceeding 180°C in gun barrels.

CHAPTER 9 Recommendations for Further work.

The following recommendations are from the lessons and experiences undertaking this project:

- Modify the current copper heaters to allow a method of inserting cartridge / plug / TC combination remotely into the copper heater at elevated temperatures (simulating fresh round loaded into a hot breach).
- Based on Witherill's experimentation and analysis, consider modification of the copper heater by fitting the TC A3 via the primer / base end of cartridge case. The copper heater will require a redesign where the internal cartridge case space replicates the chamber and barrel space.
- Experiment with SB and other DB propellants.
- Experiment with 200w and 300w heater elements until the ramp temperatures match that of fast operational firing guns.
- If repeating these experiments, manually control the temperature controller for a more linear temperature approach to the required temperature.
- More statistical firings conducted in larger batch numbers.
- Investigate the copper heater outputs / methodology for modelling (possibly the HT Tool).
- Consider use of HPLC to determine stabiliser content of Bulls Eye propellant pre and post heating to 150°C.
- A creation of a separate STANAG to assist the S³ test requirements for cook-off testing (this can include the addition of aero-kinetic effects on aircraft cannon systems).
- Use apparatus with different cartridge materials and/or liners.

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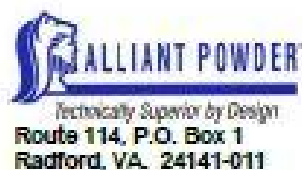
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Appendix A MHDS



Material Safety Data Sheet

Section 1. Product Information

Product Name:	Bullseye® Smokeless Powder
MSDS Number:	N/A
Product ID#:	N/A
Preparation Date:	Revised 05/11/2010
Business Phone/Hours:	540-639-8143
Emergency Telephone #:	540-639-7323
24 Hr. Spill (Chemtrec):	1-800-424-9300

Section 2. Composition/Information On Ingredients

Component Name	Common Name	CAS Number
Nitroglycerin	NG	55-63-0
Ethyl Centralite	EC	85-98-3
Diphenylamine	DPA	122-39-4
Nitrocellulose	NC	9004-70-0
Rosin		8050-09-7

Section 3. Hazard Identification

Emergency Overview:

DANGER! EXTREMELY FLAMMABLE - EXPLOSIVE.

ACCIDENTAL FIRE OR EXPLOSION IS LIKELY TO CAUSE SEVERE INJURY OR DEATH. DUST MAY CAUSE MODERATE TEMPORARY EYE, SKIN, RESPIRATORY, OR THROAT IRRITATION

Eye:	May cause eye irritation
Skin:	Absorption through the skin can cause severe headache. May cause allergic skin reaction (sensitization) in susceptible individuals.
Ingestion:	Ingestion may cause severe headache, nausea, vomiting, abdominal pain, fatigue, diarrhea, trembling, ringing in the ears.
Inhalation:	Inhalation of dust or vapor can cause severe headache. Avoid breathing dust, vapor, or mist.
Exposure Symptoms:	Irritation

Cancer Information:	NTP:	NA	OSHA:	NA	IARC:	NA
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Section 4. First Aid Measures

Eyes:	Remove contact lenses. Hold eyelids apart. Immediately flush eyes with plenty of low-pressure water for at least 15 minutes. Get immediate medical attention.
Skin:	Wash thoroughly with soap and water. Remove contaminated clothing. Thoroughly wash clothing before reuse. Render unusable and discard contaminated shoes and leather articles.
Ingestion:	Do Not Induce vomiting. Call a physician or poison control center immediately. NEVER give anything by mouth to an unconscious person. NEVER induce vomiting in an unconscious person.
Inhalation:	Remove to fresh air. If breathing is difficult, give oxygen. Get immediate medical attention.
Doctor Notes:	NA

Section 5. Fire Fighting Measures

Flammable Properties:	Flammable EVACUATE AREA IMMEDIATELY. DO NOT fight fire.
Hazardous Decomposition Products:	Oxides of carbon and nitrogen, trace metal oxides
Extinguishing Media:	Deluge with large quantities of water as quickly as possible by automatic sprinklers or fire hose from a protected location.
Protective Equipment:	Wear positive-pressure self-contained breathing apparatus (SCBA) and protective fire clothing (includes fire fighting helmet, coat, pants, boots, and gloves).
Unusual Fire and Explosion Hazards:	Explosive hazard, may detonate under the right conditions.

Section 6. Accidental Release Measures

Spills and Leaks:	Cleanup spills with a soft bristle brush and conductive rubber pan or rubber shovel. Use conductive containers. Avoid pinching material or metal to metal contact. Avoid sharp objects, sand, glass, grit or other material that sensitizes explosives.
Waste Disposal:	Disposal (if explosive) should be carried out under the direct supervision of a qualified person. For industrial disposal, federal hazardous waste regulation allows open burning of explosive waste in permitted facilities. Check with Local authorities before disposal.

Section 7. Handling Information

DO NOT PRESSURIZE OR EXPOSE CONTAINERS TO HEAT, FLAME, SPARKS, STATIC ELECTRICITY, OR OTHER SOURCES OF IGNITION

Follow appropriate D.O.D., N.F.P.A. and B.A.T.F. explosive safety measures. Local ordinances may apply.

For handling and storage requirement see 29 CFR 1910.109.

Avoid exposure to sunlight or artificial ultraviolet light.

Do not keep deteriorated or salvaged powders. Check old powders for deterioration regularly.

Destroy deteriorated powders immediately.

Section 8. Exposure Control Measures / Personal Protection

Component Name	%	CAS #	Applicable Exposure Limits
Nitroglycerin	4.0 - 40%	55-63-0	OSHA - 0.2 ppm (Skin*) ACGIH - 0.05 ppm TLV (Skin*)
Nitrocellulose	Remainder	9004-70-0	Not Established
Diphenylamine	0 - 1%	122-39-4	ACGIH Threshold Limit Value (TLV): 10 mg/m ³ OSHA (TWA) 10 mg/m ³
Ethyl Centralite	0 - 1%	85-98-3	Not Established
Rosin	0 - 4%	8050-09-7	Not Established

Engineering Controls:	Prevent build-up of static electric charges. Use process enclosures, local exhaust ventilation, or other engineering controls to control airborne levels below recommended exposures limits. Discharge from the ventilation system should comply with applicable air pollution control regulations.
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Respiratory Protection:	Appropriate respiratory protection is required to reduce airborne contaminants, which may exceed acceptable limits. Respirators should be selected and used in accordance with OSHA, Subpart I (29 CFR 1910.134) and manufacturer's recommendations.
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Hand Protection:	Impervious gloves
Eye Protection:	Safety glasses
Hygiene Controls:	Avoid contact with eyes, skin and clothing. Avoid breathing dust, vapor, or mist. Handle in areas with adequate ventilation. Wash thoroughly after handling, and before eating, drinking, or smoking. Avoid contamination of food, beverages, or smoking materials. Remove contaminated clothing promptly and clean thoroughly before reuse.

Section 9. Physical And Chemical Properties

Boiling Point:	NA	Solubility:	Negligible at 20°C
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Melting Point:	NA	Specific Gravity:	1.5 (approximate)
Vapor Pressure:	Negligible at 20°C	pH:	NA
Vapor Density:	NA	Odor:	NA
Flash Point:	NA	Appearance:	Small black/gray flakes
Ignition Temp.:	NA		
UEL:	NA		
LEL:	NA		

Section 10. Stability and Reactivity

Stability:	Stable under recommended handling and storage conditions. Material is sensitive to friction, shock, impact, and electrostatic discharge.
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Incompatibilities:	Acids, oxidizing agents, alkalis and amines and strong sunlight or ultraviolet light.
Conditions to Avoid:	Avoid storing product near incompatible materials. Do not store near flammable materials. Do not keep deteriorated or salvaged product. Keep away from heat, flame sparks and other ignition sources. Do not store in direct sunlight or expose to UV radiation

Hazardous Decomposition Products:	None anticipated under normal or recommended handling and storage conditions.
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Section 11. Toxicological Information

Component - Nitroglycerin: WARNING! Inhalation may cause severe headache. Ingestion may cause severe headache and blood pressure lowering. Absorption through skin may cause severe headache. Repeated skin contact may cause allergic skin reaction (skin sensitization). <u>Medical conditions generally recognized as being aggravated by exposure:</u> Severe untreated anemia and glaucoma may be aggravated by extreme overexposure to nitroglycerin in the workplace. Aggravation of these conditions has never been reported as a consequence of workplace exposure but has been reported following protracted medicinal over dosage of nitroglycerin. Alcohol has been reported to intensify any adverse reaction to nitroglycerin. Not listed as a carcinogen by National Toxicity Program (NTP); not regulated as a carcinogen by Occupational Safety and Health Administration (OSHA); not evaluated by International Agency for Research on Cancer (IARC). Rats dosed for a lifetime showed a <u>decrease</u> in the number of mammary and pituitary tumors that usually occur. CARCINOGENICITY/TERATOGENICITY INFORMATION <u>Reported Human Effects:</u> The effects reported above are only ones that have been reported following workplace exposure. The following additional effects have been reported following medicinal usage or over usage: faintness, skin flush, palpitation, rapid

heartbeat, dizziness, abdominal pain, retrosternal discomfort, muscle twitches, Heinz bodies (black spheres in red blood cells), methemoglobinemia, depression, confusion, skin irritation, and allergic skin reaction.

Not teratogenic in rats or rabbits. Inactive in dominant lethal mutagenicity test. No effect on fertility in reproduction test. At many times the dose that would be received following exposure at the TLV, methemoglobinemia and fibrous tissue formation in the bile ducts were seen. Testicular effects were seen in rats dosed for 3 months at over 20,000 times the dose a worker would receive working in the TLV. In lethal dose determination in rats, rapid breathing, poor muscle tone, raised hair and excessive activity were seen.

Section 12. Ecological Data

No ecological studies were found on this product.

Section 13. Disposal Considerations

Disposal of material should be in accordance with Federal, State and local regulations.

Section 14. Transportation Information

U.S. DOT Proper Shipping Name:	Smokeless powder for small arms(100 pounds or less)
UN ID No.:	NA3178
Class & Division:	4.1
Packing Group.:	I

Section 15. Regulatory Information

OSHA:			
TSCA:	Section 12(b) Listed: X	Unlisted:	Exempt:
CERCLA:	Reportable Quantity: 25 pounds (based on nitroglycerin content)		
SARA Title III:			
Section 302:	Chemical Name: NA		
	Threshold Planning Quantity: NA		
	Reportable Quantity: 25 pounds (CERCLA RQ)		
Section 311/312:	Hazard Class:		
	☒ Acute ☒ Chronic ☒ Fire ☒ Reactive ☒ Sudden Release of Pressure		
Section 313:	Chemical Name:	CAS Number/Category Code:	Weight %:
	Nitroglycerin	55-63-0	4.0-40
	Diphenylamine	122-39-4	0-1

Other:	Wastes associated with the propellant portion of this product may be considered RCRA Hazardous Wastes (40 CFR Part 261) (a) because it is listed (P081) due to Nitroglycerin content
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Section 16. Information Sources

Label Information:

DANGER !

FLAMMABLE/EXPLOSIVE

KEEP AWAY FROM HEAT, SPARKS OR OPEN FLAME

MAY CAUSE SKIN IRRITATION

Residual dust may ignite.

Ingestion may cause nausea, vomiting and possible loss of consciousness.

FIRST AID: In case of contact, immediately flush skin with water. Remove contaminated clothing and shoes. Call physician if irritation develops and persists.

In case of spill: Use soft natural bristle brush and conductive rubber or conductive plastic shovel and dispose accordingly.

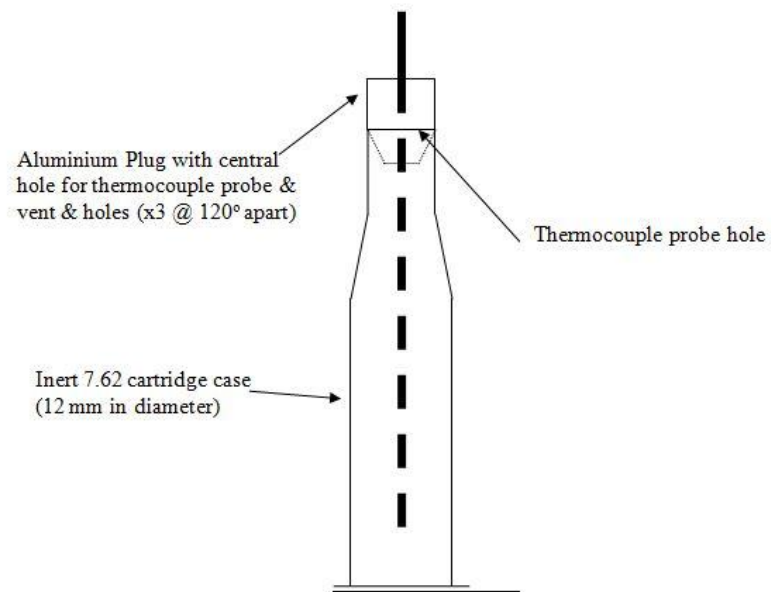
Read product-specific MSDS for more details

Abbreviation Key:

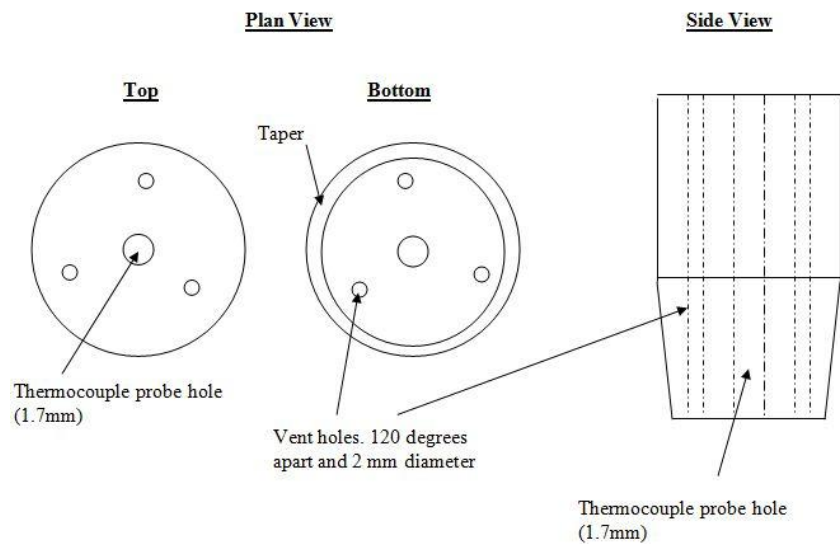
ACHIH:	American Conference of Governmental Industrial Hygienist
AICS:	Australian Inventory of Chemical Substances
AIHA WEEL:	American Industrial Hygienists Association - Workplace Environmental Exposure Level
ANSI:	American National Safety Institute
C:	Ceiling
CASRN:	Chemical Abstracts Service Registry Number
CERCLA:	Comprehensive Emergency Response, Compensation and Liability Act
DSL:	Domestic Substances List (Canadian)
EIECCS:	European Inventory of Existing Commercial Chemical Substances
HMIS:	Hazardous Materials Identification System
IARC:	International Agency for Research on Cancer
MITI:	Ministry of International Trade and Industry (Japanese)
N/A:	Not Applicable
NDSL:	Non-domestic Substances List (Canadian)
NFPA:	National Fire Prevention Association
NOR:	Not Otherwise Regulated
NTP:	National Toxicology Program
OSHA:	Occupational Safety and Health Administration
PEL:	Permissible Exposure Limit
RCRA:	Resource Conservation and Recovery Act
RQ:	Superfund Amendment Reauthorization Act
STEL:	Short Term Exposure Limit
TLV:	Threshold Limit Value (ACGIH)
TPQ:	Threshold Planning Quantity
TSCA:	Toxic Substances Control Act
TWA:	Time Weighted Average

Appendix B – Cartridge Case and Aluminium Plug Drawings

Inert 7.62mm with Aluminium Thermocouple Plug Fitted



Aluminium Thermocouple Cartridge Plug



Appendix C – Cartridge Weight and dimensions

Serial	Type (Base markings)	Weight (g)	Cart Diameter
0.308(1)	0.303 WIN Match FNM	11.64	(1) 11.61 (2) 11.85
0.308(2)	0.303 WIN Match FNM	11.67	(1) 11.62 (2) 11.83
0.308(3)	0.303 WIN Match FNM	11.62	(1) 11.62 (2) 11.82
0.308(4)	0.303 WIN Match FNM	11.66	(1) 11.61 (2) 11.82
0.308(5)	0.303 WIN Match FNM	11.72	(1) 11.64 (2) 11.84
0.308(6)	0.303 WIN Match FNM	11.73	(1) 11.61 (2) 11.91
0.308(7)	0.303 WIN Match FNM	11.61	(1) 11.61 (2) 11.91
0.308(8)	0.303 WIN Match FNM	11.56	(1) 11.69 (2) 11.96
0.308(9)	0.303 WIN Match FNM	11.61	(1) 11.66 (2) 11.84
0.308(10)	0.303 WIN Match FNM	11.75	(1) 11.62 (2) 11.88
0.308(11)	0.303 WIN Match FNM	11.62	(1) 11.62 (2) 11.85
0.308(12)	0.303 WIN Match FNM	11.79	(1) 11.61 (2) 11.86
0.308(13)	0.303 WIN Match FNM	11.69	(1) 11.61 (2) 11.88
0.308(14)	0.303 WIN Match FNM	11.62	(1) 11.64 (2) 11.86
0.308(15)	0.303 WIN Match FNM	11.73	(1) 11.63 (2) 11.88
0.308(16)	0.303 WIN Match FNM	11.63	(1) 11.68 (2) 11.85
0.308(17)	0.303 WIN Match FNM	11.64	(1) 11.66 (2) 11.88

Serial	Type (Base markings)	Weight (g)	Cart Diameter (mm)
0.308(18)	0.303 WIN Match FNM	11.53	(1) 11.62 (2) 11.86
0.308(19)	0.303 WIN Match FNM	11.74	Not Recorded
FFV(1)	FFV 92	12.33	(1) 11.70 (2) 12.02
FFV(2)	FFV 92	12.32	(1) 11.68 (2) 11.99
FFV(3)	FFV 92	12.33	(1) 11.70 (2) 11.98
FFV(4)	FFV 92	12.42	(1) 11.69 (2) 12.05
FFV(5)	FFV 92	12.39	(1) 11.65 (2) 12.06
FFV(6)	FFV 92	12.36	(1) 11.68 (2) 12.02
FFV(7)	FFV 92	12.32	(1) 11.68 (2) 12.09
FFV(8)	FFV 92	12.38	(1) 11.66 (2) 12.05
FFV(9)	FFV 92	12.36	(1) 11.63 (2) 12.02
1a	1K07 7.62 x 51	11.55	Not Recorded
2a	CG01	12.23	Not Recorded
3a	L2A2 MG 99	11.39	Not Recorded
4a	RG 90(A)	10.91	(1) 11.69 (2) 11.94
4b	RG 90(B)	11.63	(1) 11.66 (2) 11.98
4c	RG 90(C)	11.66	(1) 11.67 (2) 11.98
5	RG 93	11.77	Not Recorded

Appendix D – THT ARC Bulls Eye Test Results

thermal hazard technology 

Thermal Hazard Technology,
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Phone: +44 1908 646800 Fax: +44 1908 645209

Thermal Hazard Technology,
Suite 1203, 255 Old Newbrunswick Road, Piscataway, NJ 08854, USA.
Phone: (732) 562 1123 1-888-688 7487 Fax: (732) 462 0778

E-mail: info@thermalhazardtechnology.com Web: www.thermalhazardtechnology.com

ACCELERATING RATE CALORIMETER

EXPERIMENTAL REPORT

REPORT NO: tht12088

REPORT DATE: 30/04/12

PREPARED FOR: Mr Phil Pitcher
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OPERATOR: Stephen Ottaway

POSITION: International Product Specialist

SIGNATURE: _____

DATE: _____

EXPERIMENTAL PROCEDURE

Accelerating rate calorimetry aids in the evaluation of hazard prediction for reactive chemicals. The Accelerating Rate Calorimeter permits a small sample of a chemical to undergo thermal decomposition due to self-heating. The primary data given in this report are the temperature and pressure variations as a function of time during such a 'thermal runaway' reaction. Secondly, certain modelling functions based upon the experimental values are presented.

The specified amount of sample is introduced into the sample container (or 'bomb'), which is in turn mounted in the calorimeter, and the temperature and pressure sensors are attached.

The calorimeter is then rapidly heated to the 'Start Temperature' after which either an Isothermal Age mode or a Heat-Wait-Search sequence is entered, to determine onset of sample self-heating. After each 'Heat' step there is a 'Wait' period intended to allow the sample-bomb system to reach thermal equilibrium. This in turn is followed by a self-heating 'Search' period.

When less than the present self-heat rate threshold ('Slope Sensitivity') is observed during the Search mode, or at the end of the Isothermal Age time, a Heat step is applied to the sample and bomb. The Heat-Wait-Search sequence is repeated until the 'End Temperature' is reached or until self-heating is detected above the 'Slope Sensitivity' threshold. At this point the reaction is allowed to proceed under very closely controlled adiabatic conditions and time, temperature and pressure data are collected and stored. Detailed procedures and information can be found in the Accelerating Rate Calorimeter Instruction Manual.

GENERAL PRECAUTIONS

Accelerating rate calorimetry is useful for evaluating exothermic runaway processes. However, caution should be used when relating accelerating rate calorimetry data to an actual plant process especially if significant extrapolations of the data or in the thermal inertia, ϕ , are involved. Also, many of the mathematical relationships used herein are assumed valid only for non-autocatalytic, simple, single reactions. It is strongly recommended that predictions for full scale chemical processing, storage or handling procedures should not be based solely upon the results of this report.

DISCLAIMER

The work carried out and reported herein has been obtained with all due care and competence by the signatory or other professionally qualified and fully trained person. The Accelerating Rate Calorimeter used in this work has been utilised in as expert a way as possible. The method conforms to the method described in ASTM E27 procedure. The results submitted on the basis that they constitute and are considered to be a reliable representation of the received material and its behaviour in the test. Further to this no warranty can be given to the data and its application. Acceptance of the data implies agreement with this. Whilst every effort will be given to aid the Customer in understanding and interpreting the data, help will not be given on the specific application of the data.

1. GENERAL

1.1	Run Number	tht12088
1.2	File Names	arc12088.dat & arc12088.exe

2. SAMPLE DATA

2.1	Designation (Code)	Bullseye Smokeless Pistol Powder
2.2	Chemical	---
2.3	Formula	---
2.4	Molecular Weight	---
2.5	Density	---
2.6	Assay	---
2.7	State	---
2.8	Diluent/Solvent	---
2.9	Batch Number	---
2.10	Comments	---

3. BOMB DATA

3.1	Type	Spherical
3.2	Material	Titanium
3.3	Bomb Volume (approx.)	8.75ml
3.4	Total System Volume (approx.)	10ml
3.5	Sample Volume	4.5ml
3.6	Head Space Volume (approx.)	5.5ml
3.7	Head Space Atmosphere	Air
3.8	Bomb Loading Comments:	Funnel used.

4 THERMAL INERTIA

$$\Phi = 1 + \frac{(M_b) \cdot (C_b) + (M_{cl}) \cdot (C_{cl})}{(M_s) \cdot (C_s)}$$

where:

Φ	=	Thermal Inertia
M	=	Mass (g)
C	=	Specific Heat (J/g °C)

and subscripts:

b	=	bomb
cl	=	clip
s	=	sample

M _b	=	8.49g
M _{cl}	=	— g
M _s	=	0.508 g
C _b	=	0.53 J/g °C
C _{cl}	=	— J/g °C
C _s	=	1.0 J/g °C

Φ	=	9.78
--------	---	------

The value of Φ was calculated using the data shown above.

5 CALIBRATION DATA

Latest calibration:	Run Number	arc12083
	File Name	arc12083.cal
	Run Date	26 th April 2012

6. EXPERIMENTAL CONDITIONS

PARAMETER	VALUE
Test Type	Heat-Wait-Seek
Start Temperature	50 °C
End Temperature	405 °C
Slope Sensitivity	0.02 °C/min
Heat Step Temperature	5 °C
Wait Time	15 min
Data Step Temperature	1 °C
Data Step Time	0.5 min
Calculation Step Temperature	0.2 °C

The data above together with other instrument parameters and the calibration parameters are obtainable on request.

7. EXPERIMENTAL RESULTS

The values below are for Bomb/Sample system, i.e. not phi - corrected.

7.1	Observed Onset Temperature	(T_o)	115.49 °C
7.2	Self Heat Rate at T_o	(M_o)	0.069 °C/min
7.3	Temp. at Maximum Rate	$(T_{mr})_{sys}$	176.51 °C
7.4	Max. Self Heat Rate	$(M_{mr})_{sys}$	2788.82 °C/min ##
7.5	Final Adiabatic Temp.	$(T_{ab})_{sys}$	184.09 °C
7.6	Adiabatic Temp. Rise	$(\Delta T_{ab})_{sys}$	68.60 °C

where $(\Delta T_{ab})_{sys} = (T_{ab})_{sys} - T_o$

7.7 COMMENTS:

Max Rate determined from...	Temperature
Bomb Rupture...	Yes.
Burst Disc Rupture...	Used.
Nature of Sample Residue:	—

- Based on highest rate recorded.

8. RESULTS CORRECTED FOR THERMAL INERTIA (Φ)

WARNING! MANY OF THESE CALCULATIONS ARE VALID ONLY FOR NON-AUTOCATALYTIC SIMPLE SINGLE REACTIONS. CAUTION IS ADVISED!

8.1 Adiabatic Temp. Rise (δT_{ab}) 670.91 °C **

$$\delta T_{ab} = \Phi \cdot (\delta T_{ab})_{sys}$$

8.2 Final Adiabatic Temp. (T_{ab}) 786.40 °C **

$$T_{ab} = \delta T_{ab} + T_o$$

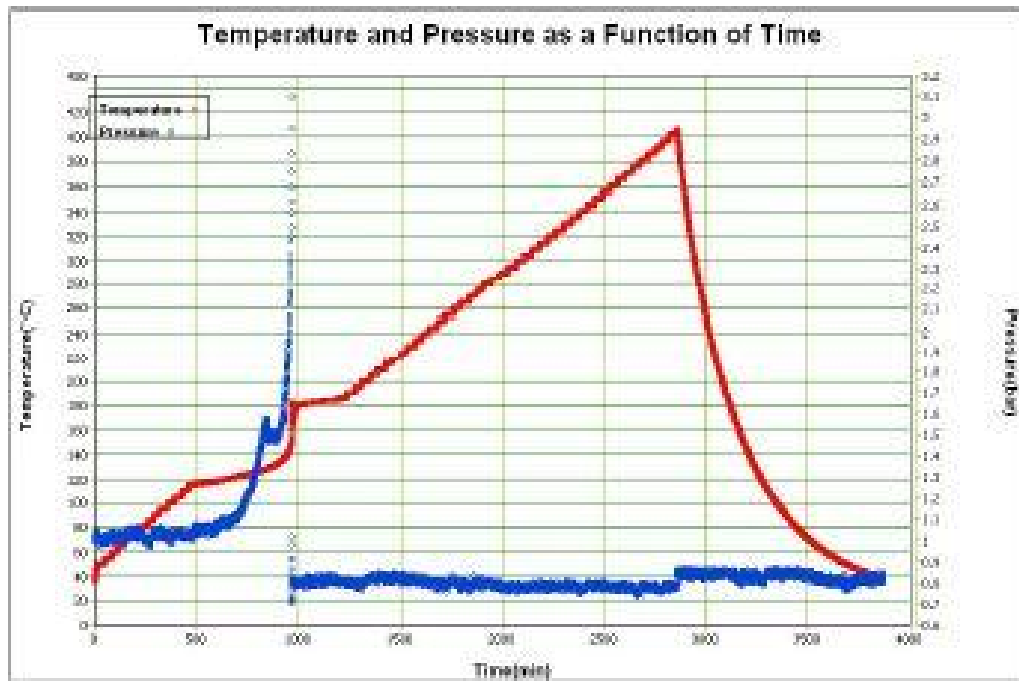
8.3 Heat of Reaction (δH) Not Calculated

$$\delta H = (Cs) \cdot (\delta T_{ab}) \frac{\text{mol. weight}}{\text{purity}}$$

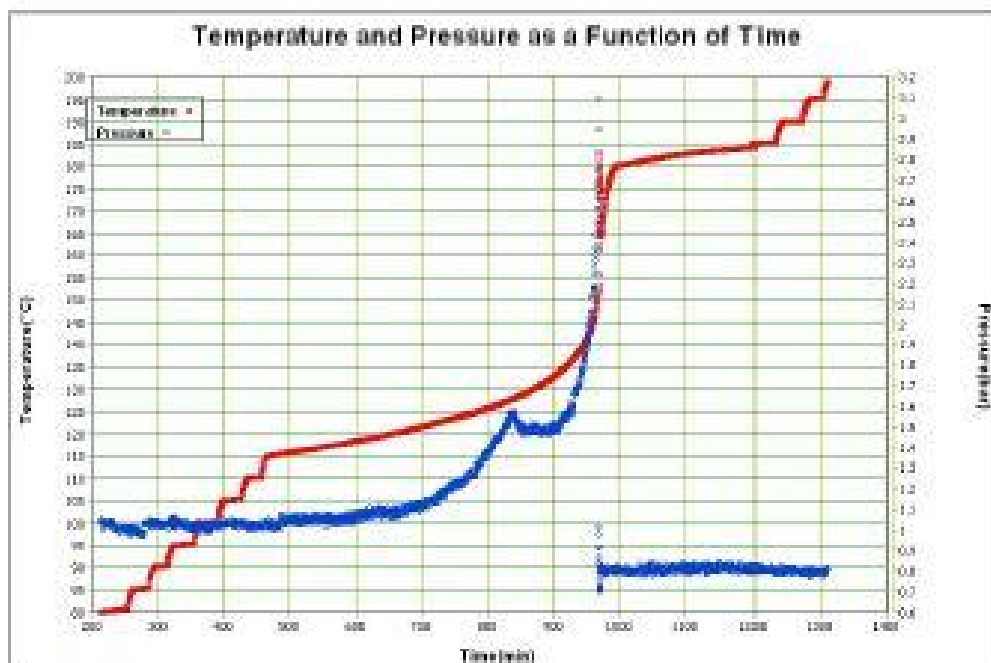
** Based on final data point measured

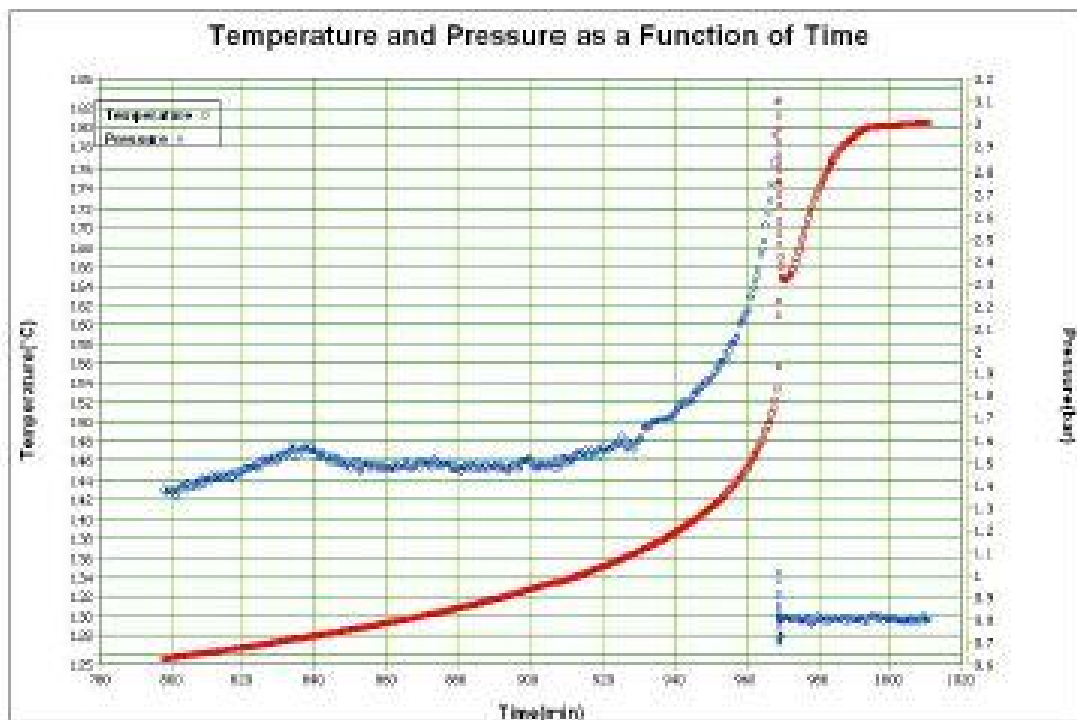
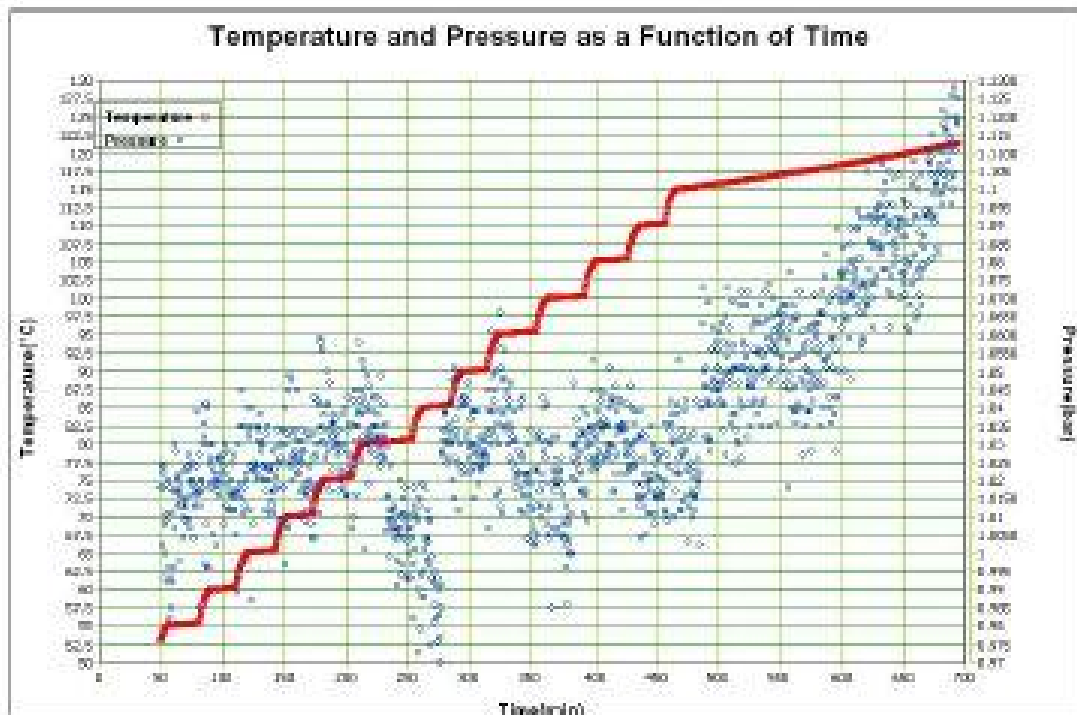
APPENDICES

Real Time Data



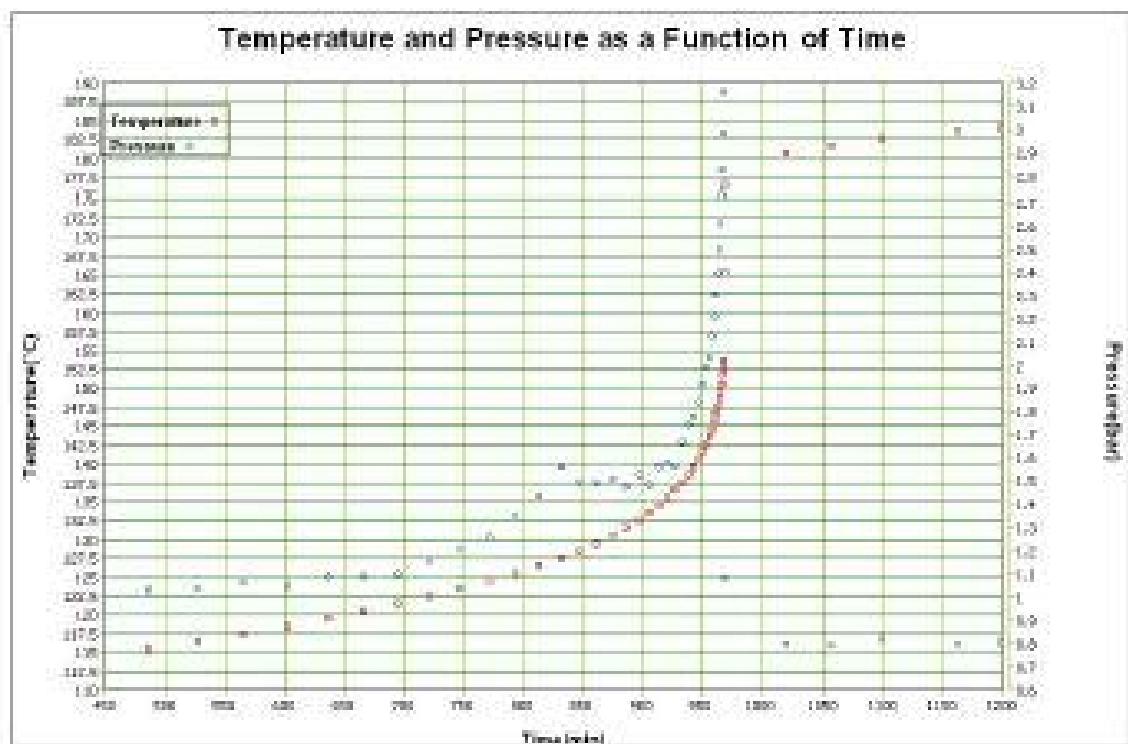
Overall profile of the test. Cell rupture occurs.



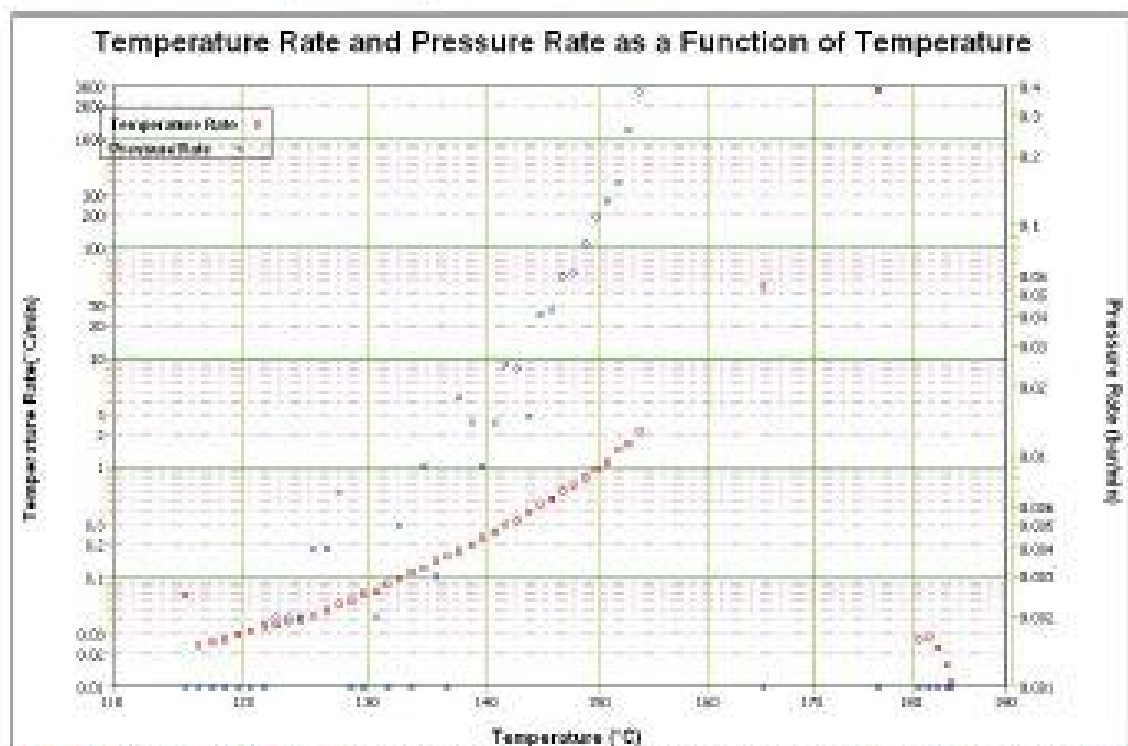


Exotherm Data

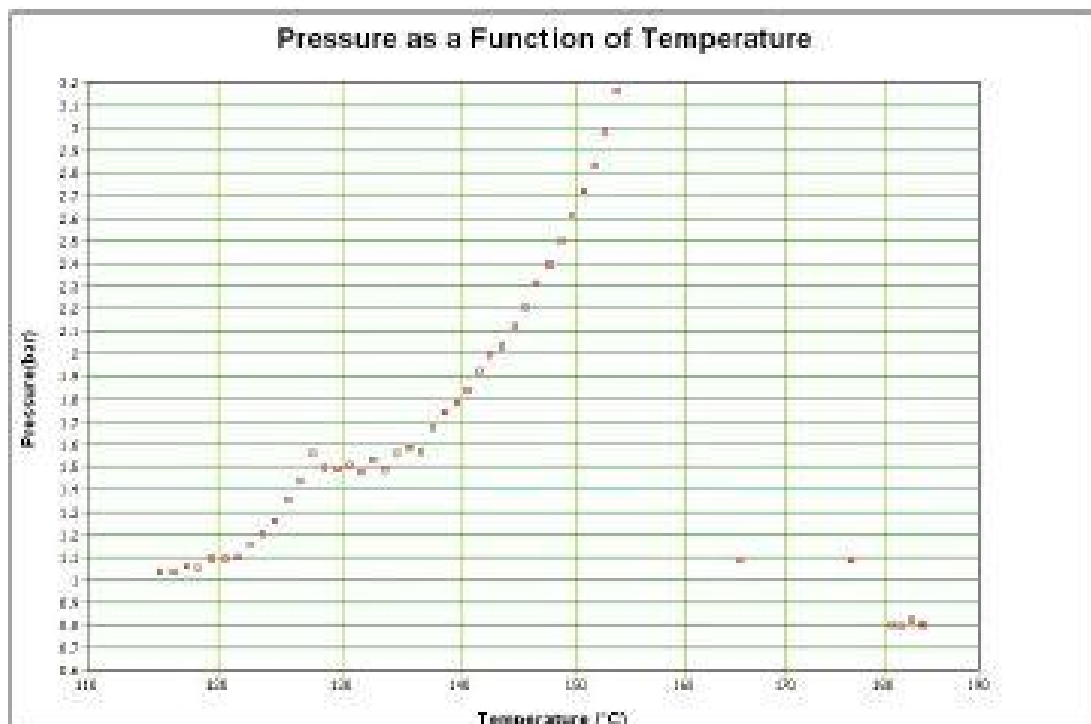
Time	Temperature, T	Rate	Pressure, P	Rate		
485.949	115.490	0.069	1.035	-0.011	0.000	
527.310	116.494	0.024	1.036	0.000	0.000	
565.740	117.506	0.026	1.064	0.001	0.000	
602.466	118.506	0.027	1.050	-0.000	0.000	
636.112	119.508	0.030	1.092	0.001	0.000	
666.666	120.508	0.033	1.092	-0.000	0.000	
694.733	121.523	0.036	1.100	0.000	0.000	
721.771	122.523	0.037	1.158	0.002	0.000	
747.326	123.525	0.039	1.204	0.002	0.000	
771.972	124.527	0.041	1.261	0.002	0.000	
794.275	125.528	0.045	1.352	0.004	0.000	
814.154	126.529	0.050	1.438	0.004	0.000	
831.753	127.531	0.057	1.563	0.007	0.000	
847.872	128.533	0.062	1.495	-0.004	0.000	
862.195	129.534	0.070	1.489	-0.000	0.000	
875.490	130.536	0.075	1.509	0.002	0.000	
887.174	131.555	0.087	1.474	-0.003	0.000	
897.649	132.556	0.096	1.528	0.005	0.000	
906.665	133.563	0.112	1.487	-0.004	0.000	
914.972	134.570	0.121	1.560	0.009	0.000	
922.120	135.578	0.141	1.582	0.003	0.000	
928.459	136.584	0.159	1.567	-0.002	0.000	
934.391	137.591	0.172	1.673	0.018	0.000	
939.375	138.602	0.200	1.745	0.014	0.000	
943.699	139.607	0.232	1.783	0.009	0.000	
947.598	140.609	0.257	1.837	0.014	0.000	
950.882	141.613	0.306	1.920	0.025	0.000	
953.894	142.620	0.334	1.994	0.024	0.000	
956.442	143.621	0.393	2.033	0.015	0.000	
958.634	144.628	0.459	2.124	0.041	0.000	
960.610	145.631	0.508	2.208	0.043	0.000	
962.237	146.632	0.616	2.306	0.060	0.000	
963.677	147.636	0.697	2.395	0.062	0.000	
964.901	148.642	0.821	2.498	0.064	0.000	
965.929	149.643	0.974	2.611	0.110	0.000	
966.817	150.646	1.130	2.725	0.129	0.000	
967.501	151.652	1.470	2.829	0.152	0.000	
968.093	152.654	1.692	2.984	0.262	0.000	
968.557	153.667	2.185	3.161	0.383	0.000	
968.813	155.352	45.644	1.086	-8.108	0.000	
968.817	176.507	2788.822	1.086	0.000	0.000	
968.853	185.307	-74.585	0.910	-4.429	0.000	
968.869	184.079	-76.700	0.873	-2.301	0.000	
968.885	182.975	-69.035	0.868	-0.288	0.000	
968.905	181.797	-58.915	0.847	-1.074	0.000	
968.929	180.646	-47.948	0.835	-0.511	0.000	
968.953	179.570	-44.836	0.839	0.179	0.000	
968.977	178.538	-42.982	0.833	-0.243	0.000	
969.001	177.449	-45.396	0.829	-0.160	0.000	
969.029	176.288	-41.645	0.813	-0.575	0.000	
969.057	175.148	-40.738	0.804	-0.329	0.000	
969.069	174.720	-35.594	0.804	0.000	0.000	
1020.506	180.696	0.027	0.803	0.001	0.000	
1055.556	181.702	0.029	0.795	-0.000	0.000	
1099.350	182.710	0.023	0.824	0.001	0.000	
1162.926	183.712	0.016	0.803	-0.000	0.000	
1198.936	184.091	0.011	0.804	0.000	0.000	



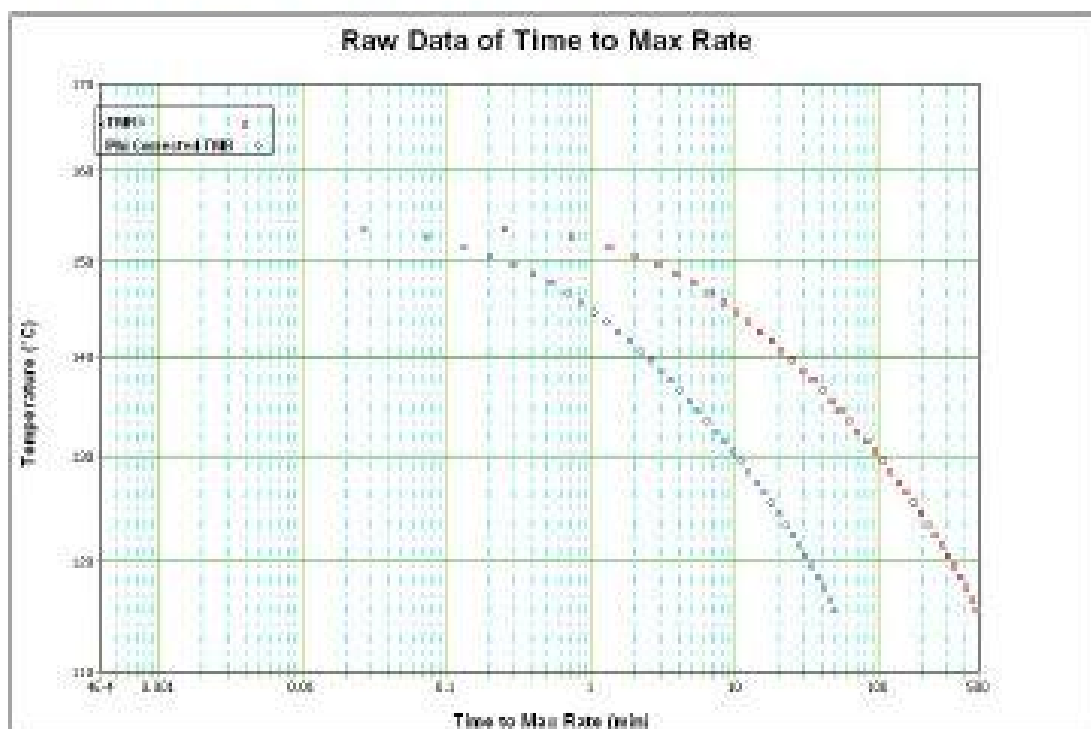
The rate accelerates until explosive decomposition occurs at around 153°C.

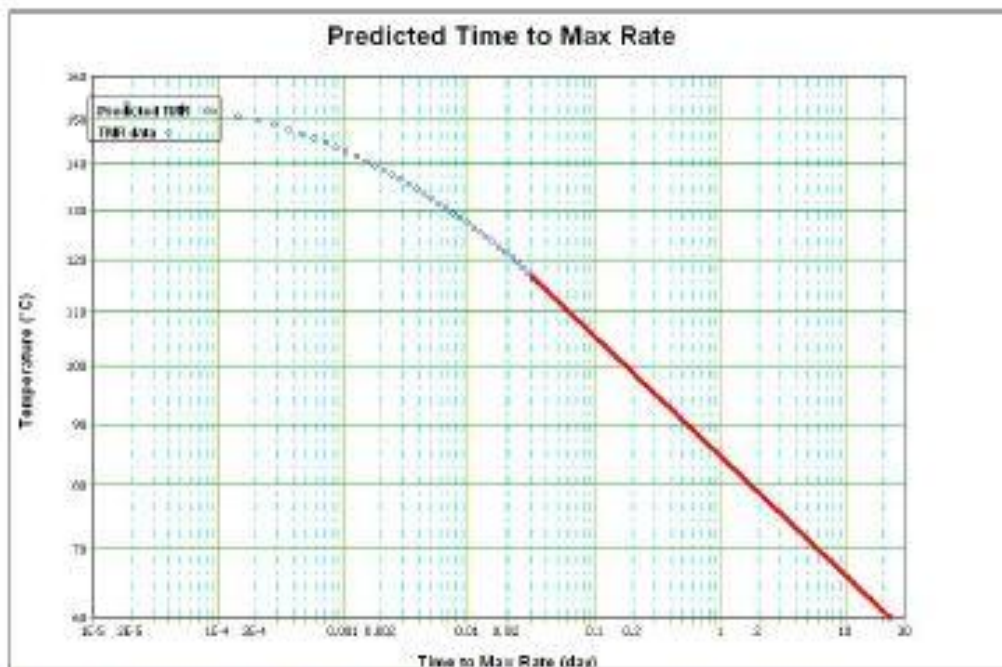


From the onset temperature the rate increases steadily, reaching 0.03°C/min at 120°C, 0.08°C/min at 130°C, 0.25°C/min at 140°C and 1°C/min at 150°C.



Upon explosive decomposition cell rupture occurs, destroying the cell.





Phi corrected time to maximum rate data. Note the time is in hours.

Images



Cell post test

Appendix E

Experiment Serials 1 and 2

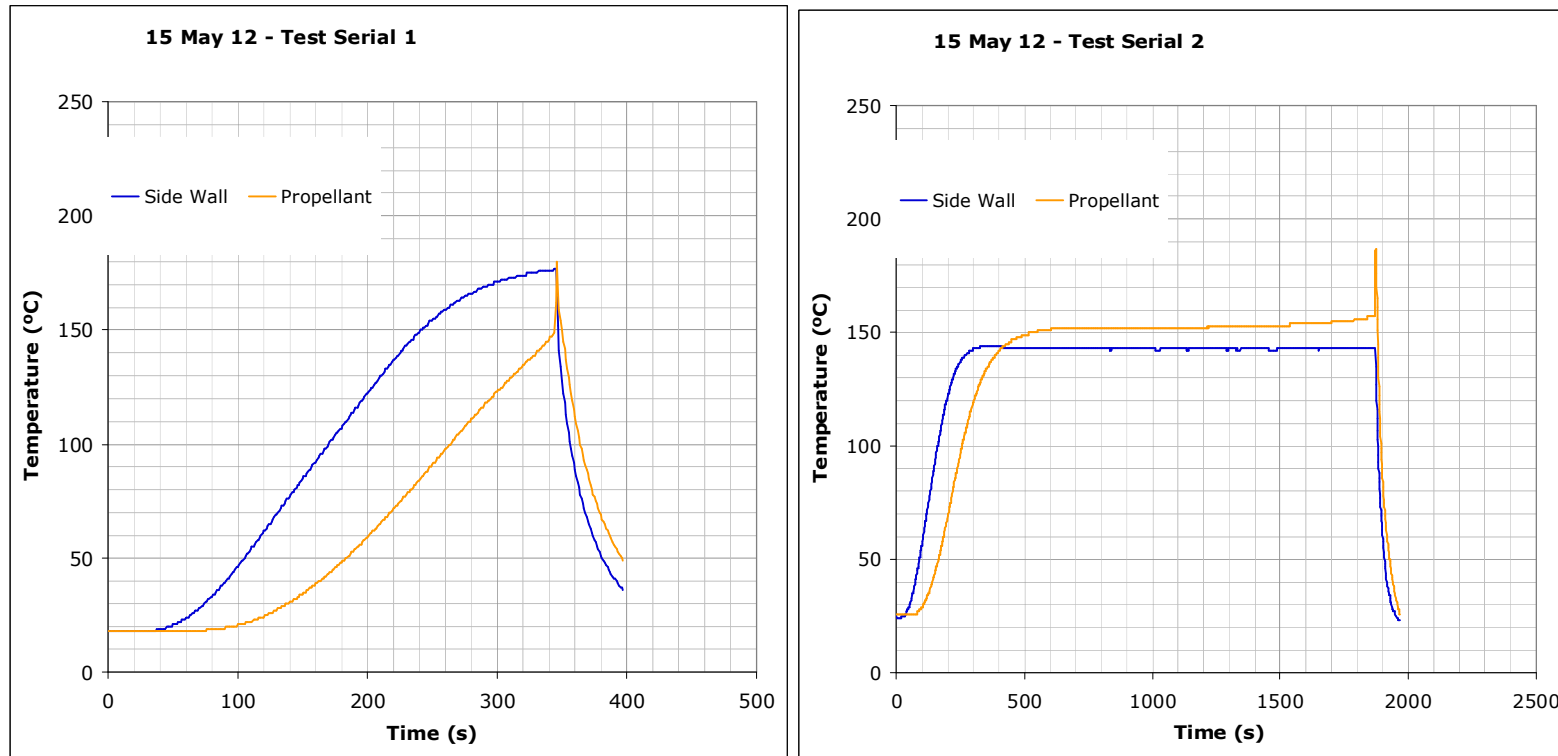


Figure E1.1: Raise temperature of T/C A1 to 180°C and held. Figure E1.2: Raise temperature of T/C A1 to 150°C and held.

Experiment Serials 3 and 4

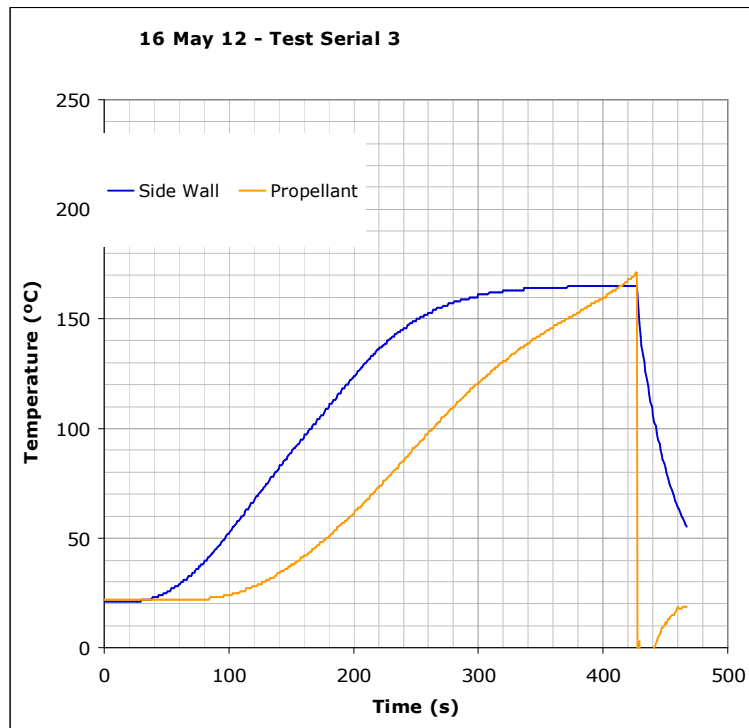


Figure E1.3: Raise temperature of T/C A1 to 165°C and held.

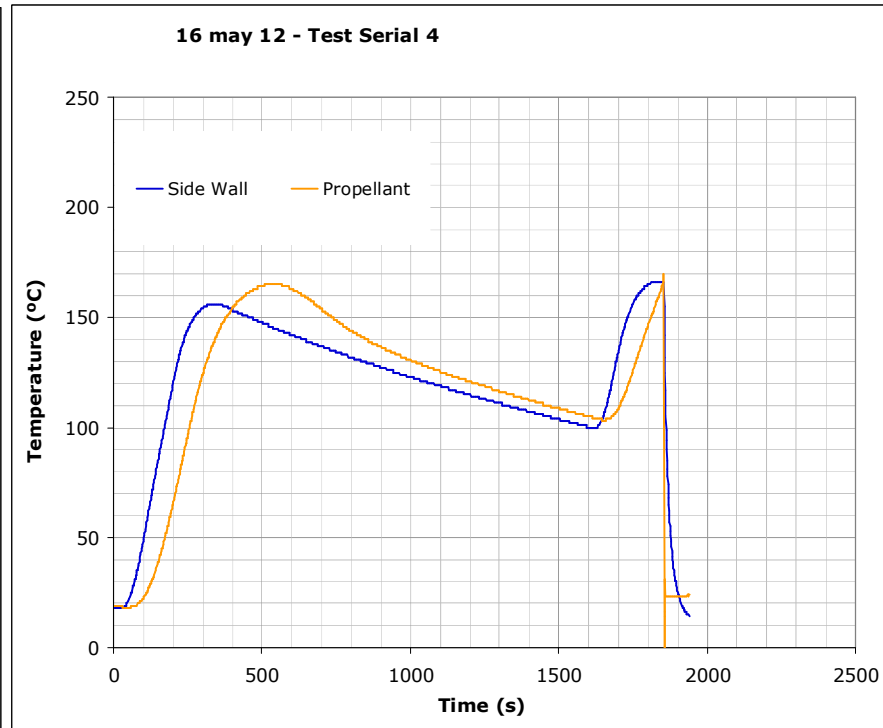


Figure E1.4: Raise temperature of T/C A1 to 160°C and then Switch Off

Experiment Serials 5 and 6

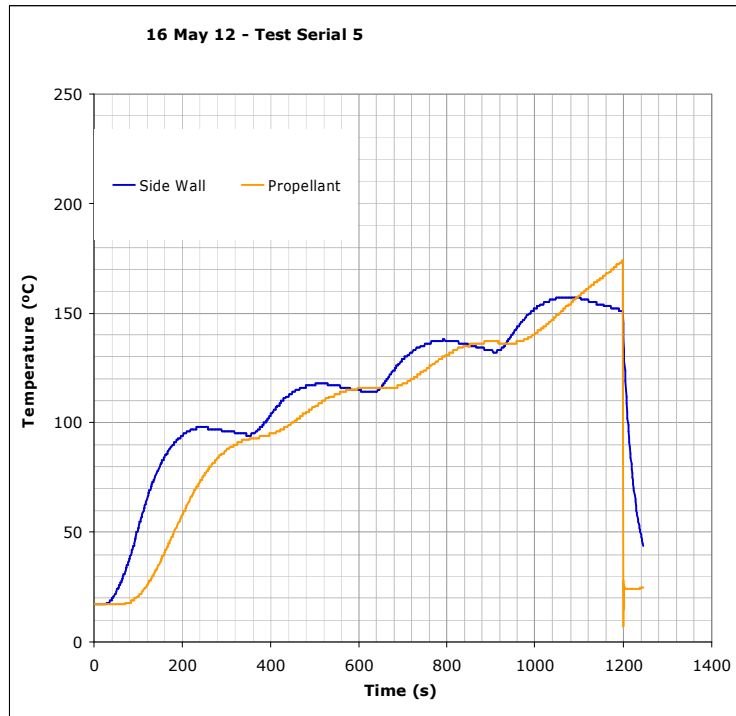


Figure E1.5: Raise temperature of T/C A1 to 100°C then switch off for 60s then repeat at 120 / 140 / 160 180°C increments

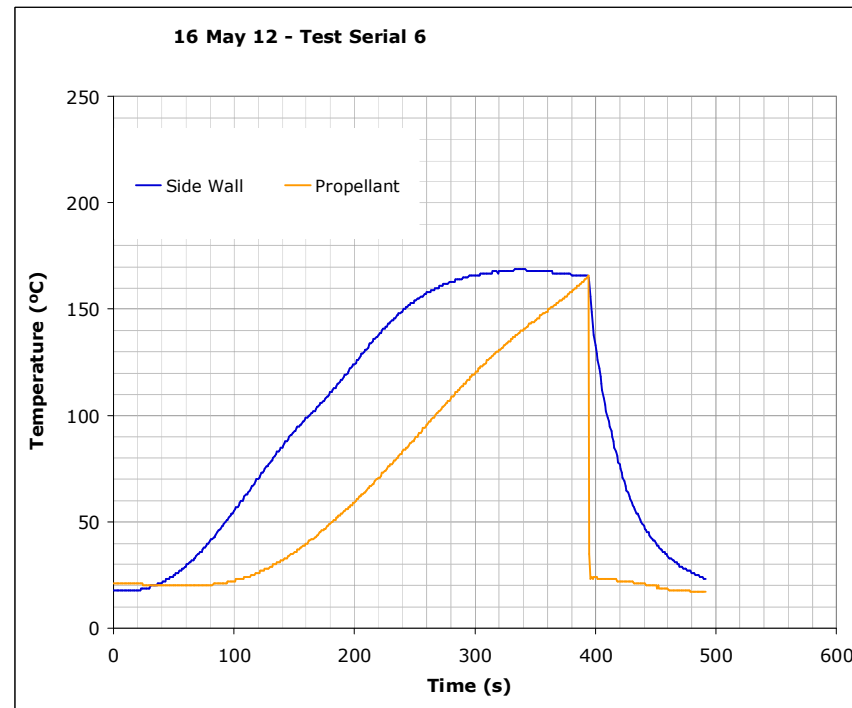


Figure E1.6: Raise temperature of T/C A1 to 170°C and Switched Off

Experiment Serials 7 and 8

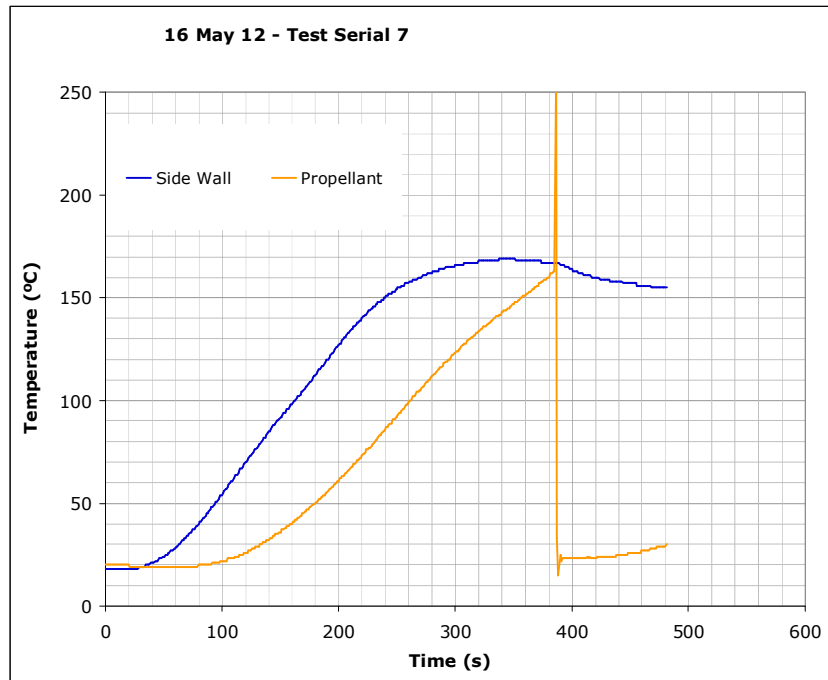


Figure E1.7: Raise temperature of T/C A1 to 170°C and Switched Off

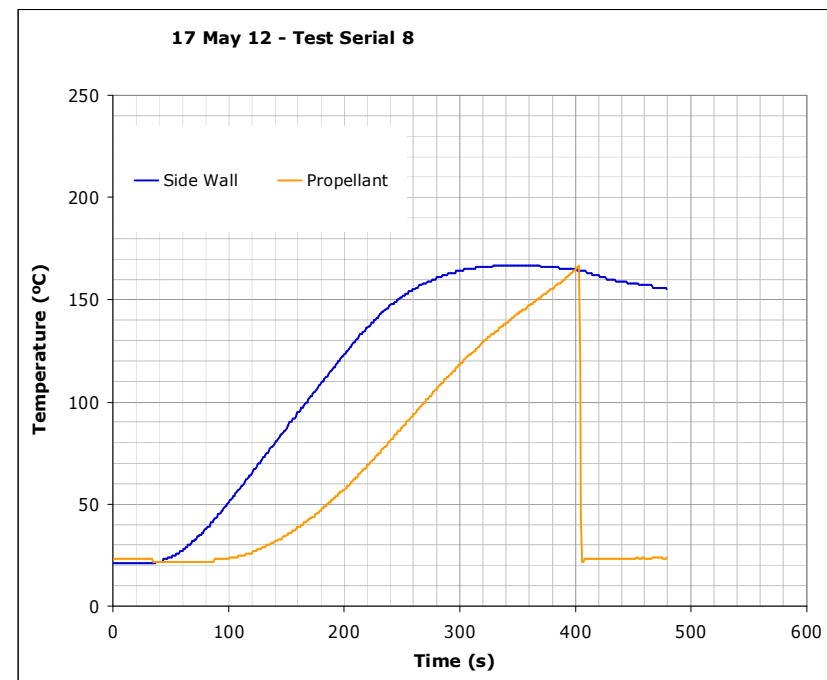


Figure E1.8: Raise temperature of T/C A1 to 170°C and Switched Off

Experiment Serials 9 and 10

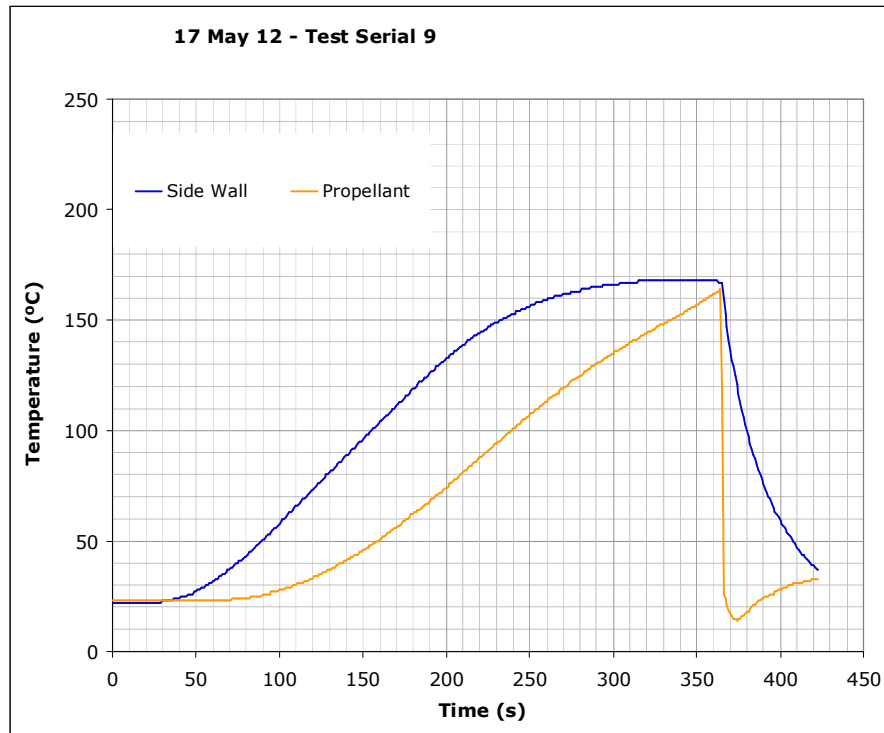


Figure E1.9: Raise temperature of T/C A1 to 170°C and Hold

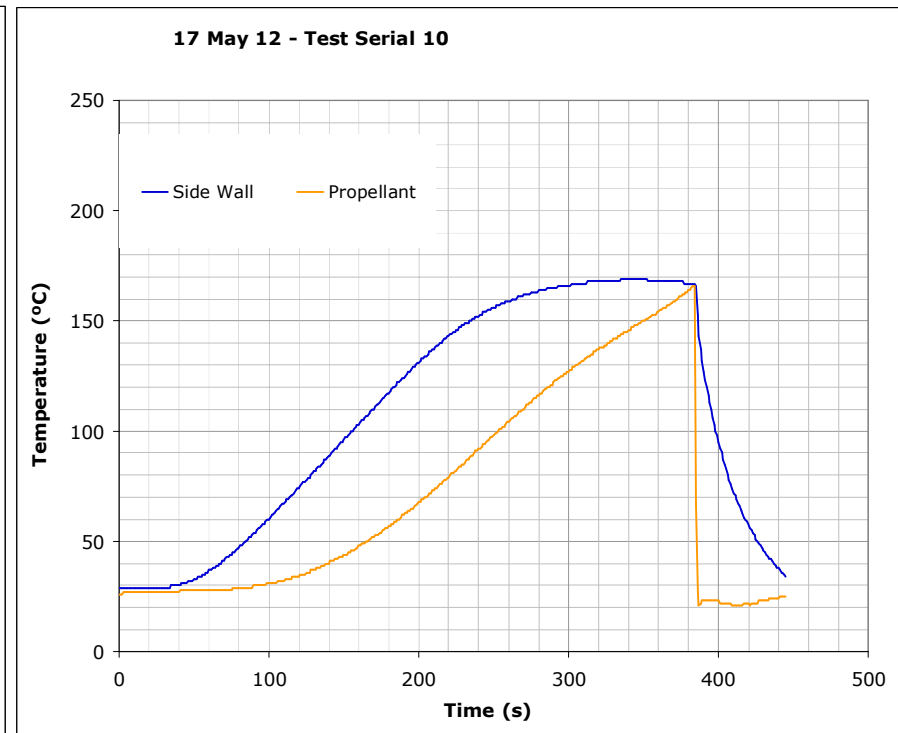


Figure E1.10: Raise temperature of T/C A1 to 170°C and Switch Off

Experiment Serials 11 and 12

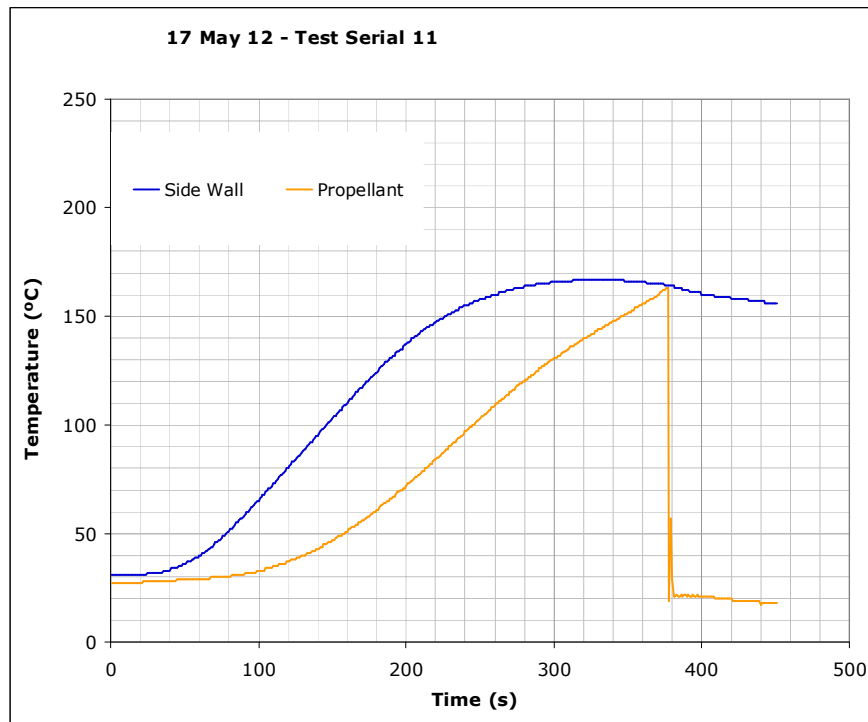


Figure E1.11: Raise temperature of T/C A1 to 170°C and Switch Off

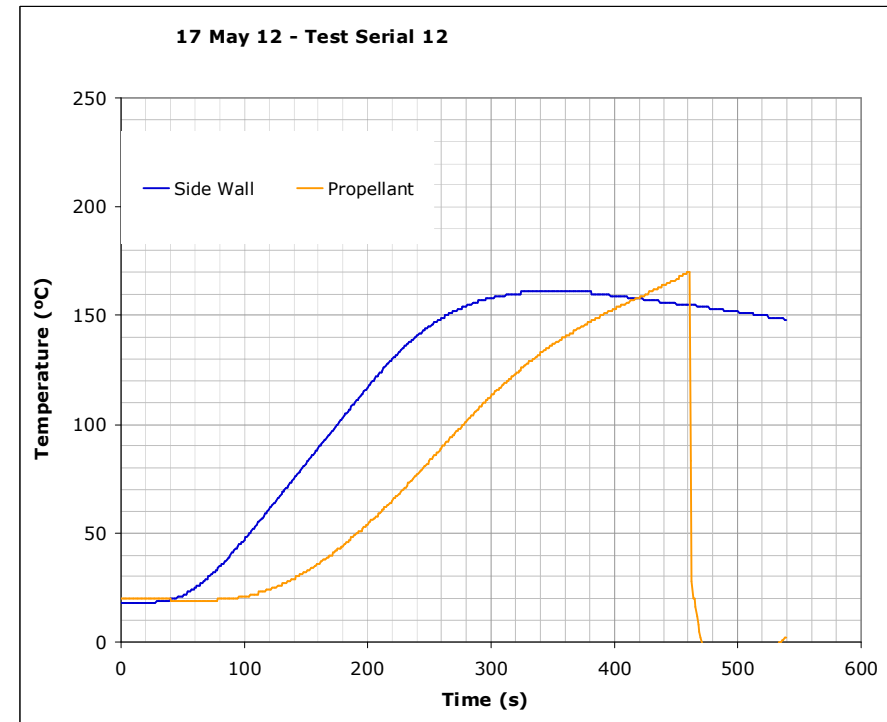


Figure E1.12: Raise temperature of T/C A1 to 165°C and Switch Off

Experiment Serials 13 and 14

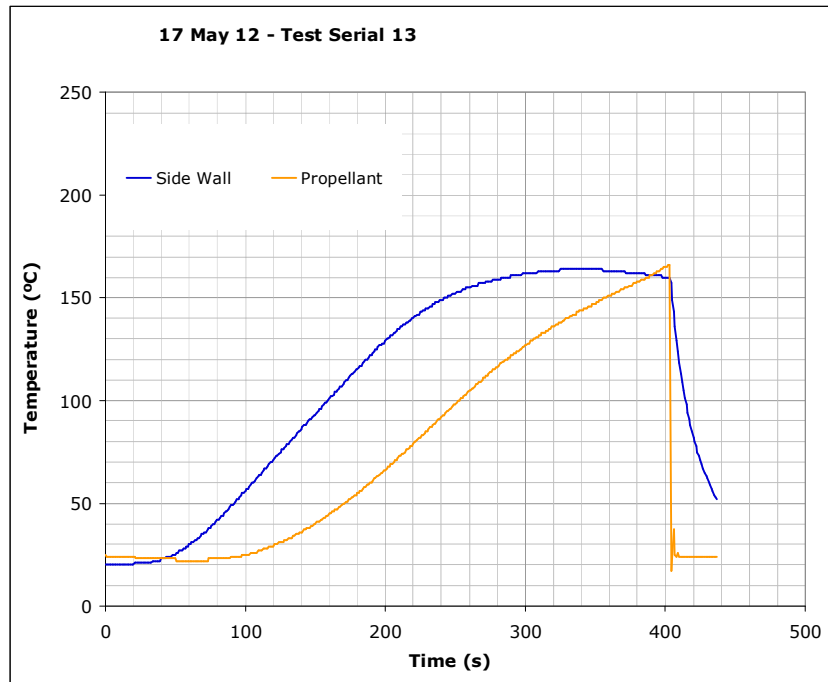


Figure E1.13: Raise temperature of T/C A1 to 165°C and Switch Off

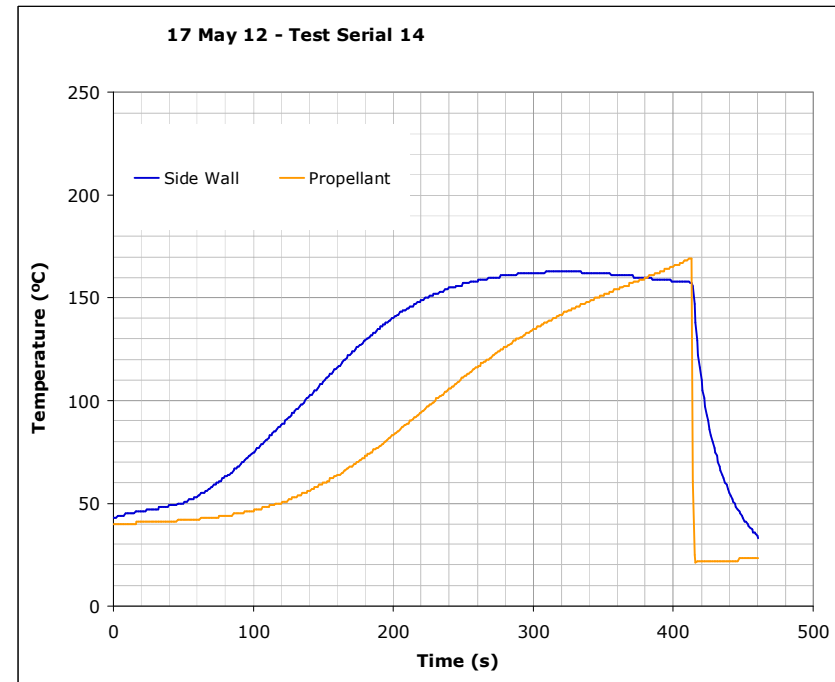


Figure E1.14: Raise temperature of T/C A1 to 165°C and Switch Off

Experiment Serials 15 and 16

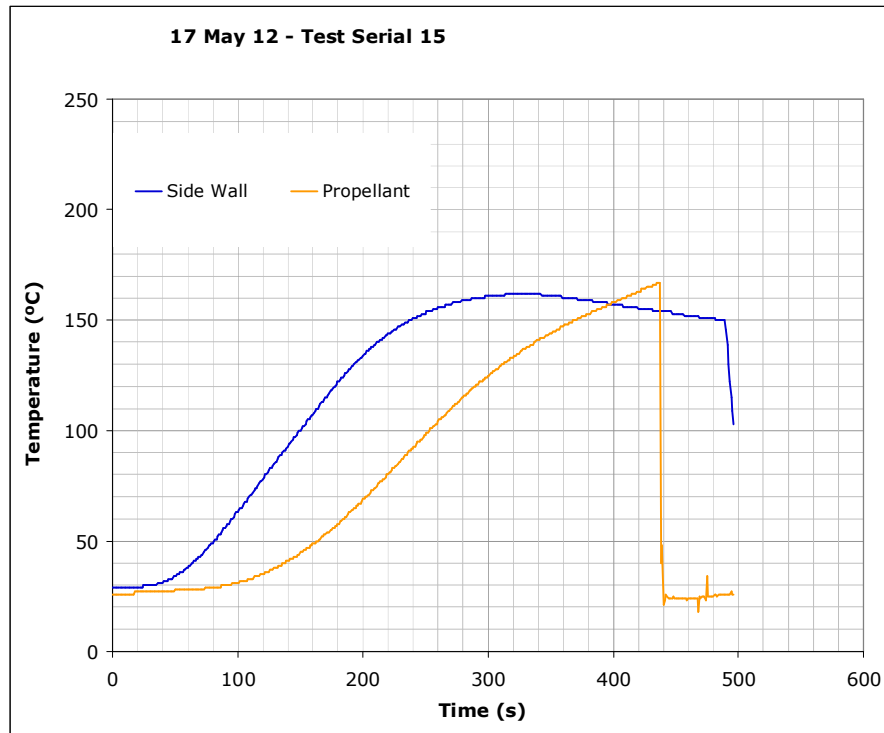


Figure E1.15: Raise temperature of T/C A1 to 165°C and Switch Off

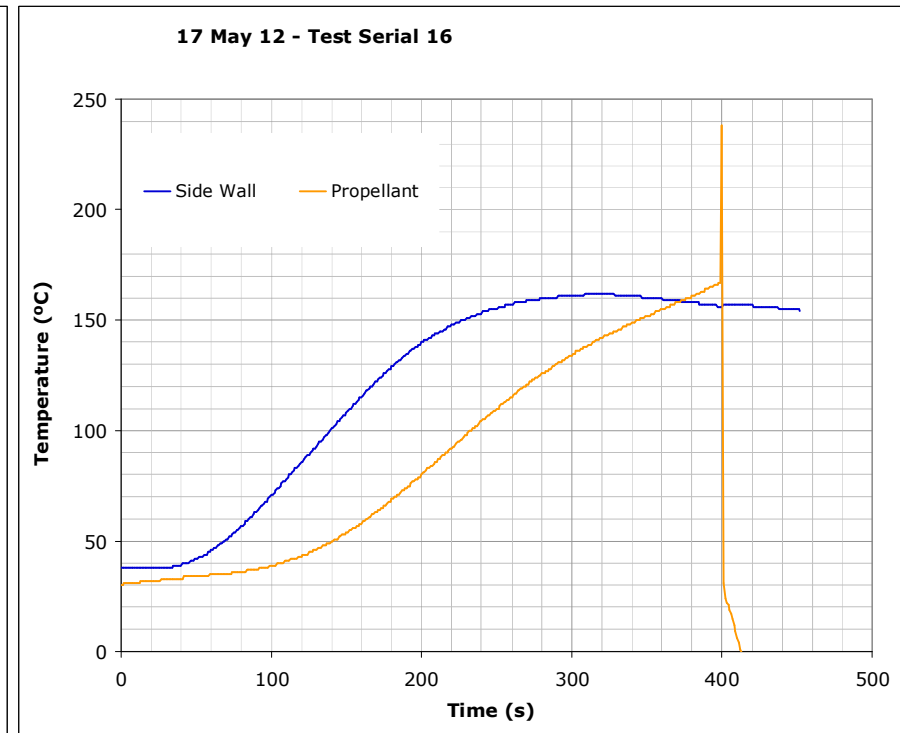


Figure E1.16: Raise temperature of T/C A1 to 165°C and Switch Off

Experiment Serials 17 and 18

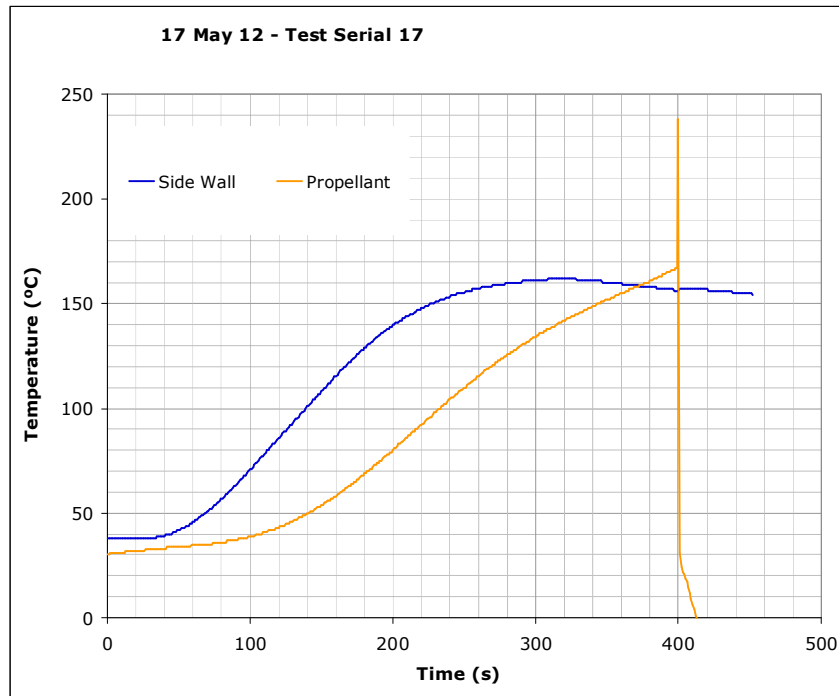


Figure E1.17: - Raise temperature of T/C A1 to 165°C and Switch Off

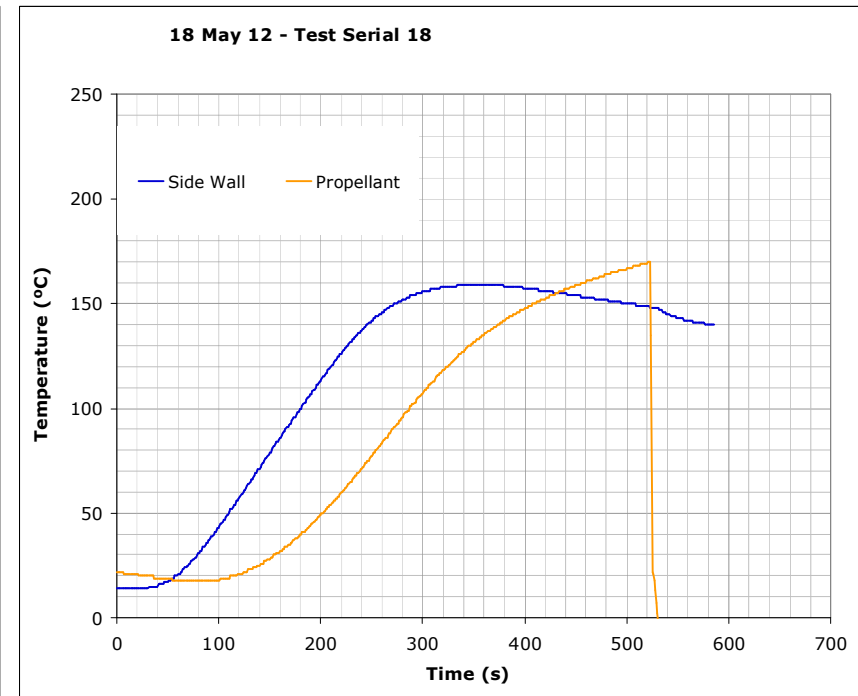


Figure E1.18: Raise temperature of T/C A1 to 162°C and Switch Off

Experiment Serials 19 and 20

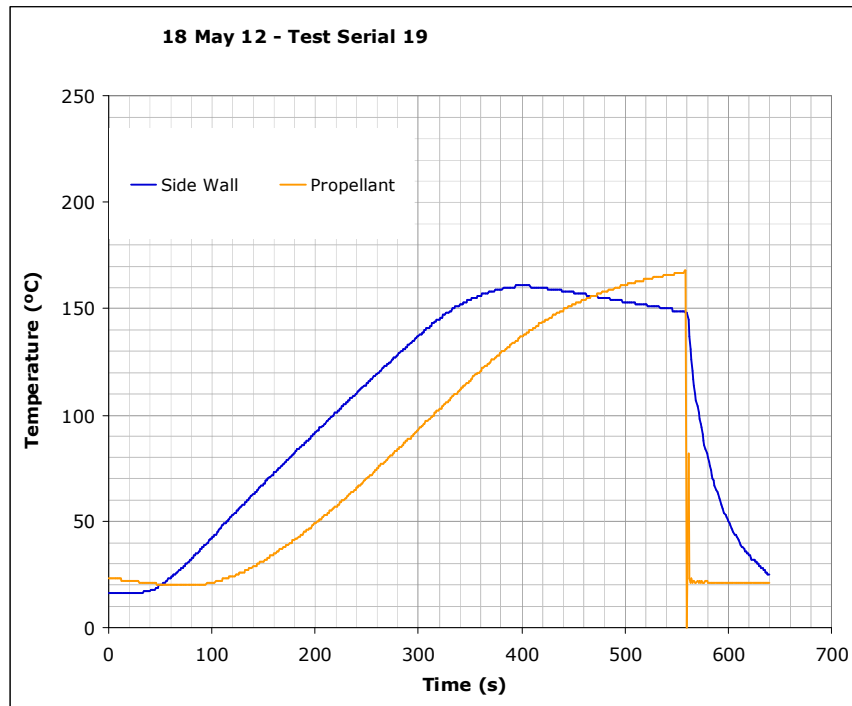


Figure E1.19: Raise temperature of T/C A1 to 162°C and Switch Off

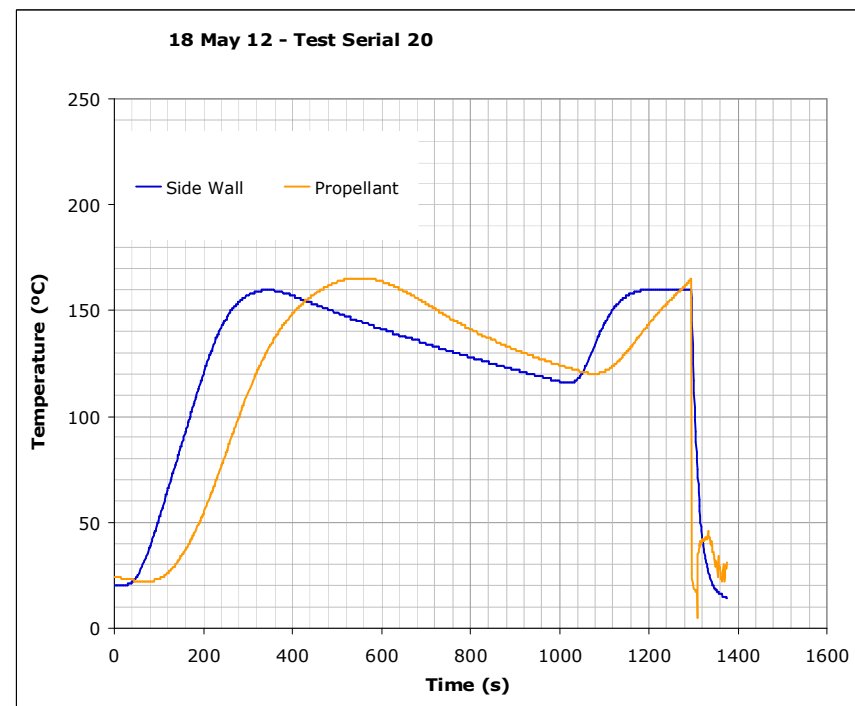


Figure E1.20: Raise temperature of T/C A1 to 161°C and Switch Off

Experiment Serials 21 and 22

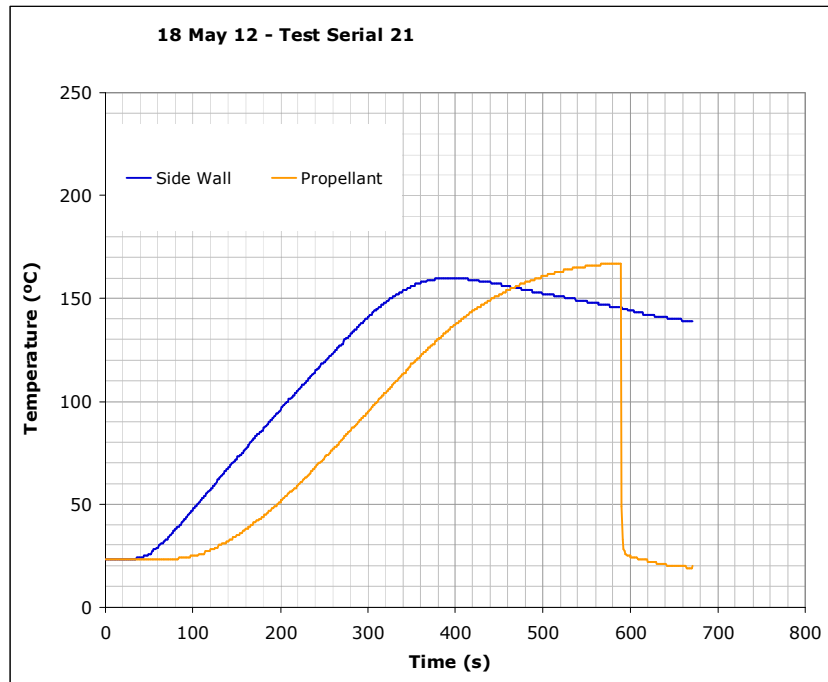


Figure E1.21: Raise temperature of T/C A1 to 161°C and Switch Off

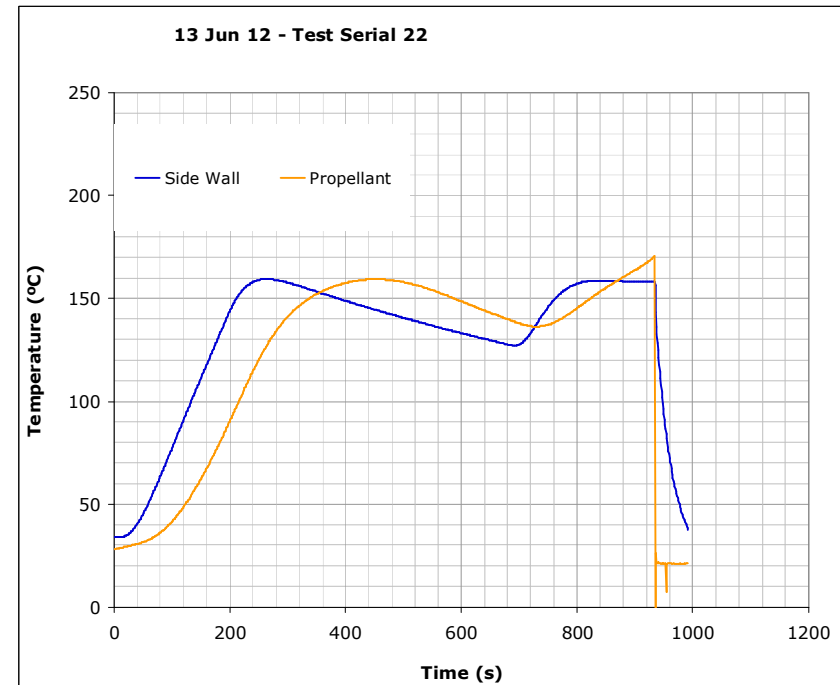


Figure E1.22: Raise temperature of T/C A1 to 161°C and Switch Off

Experiment Serials 23 and 24

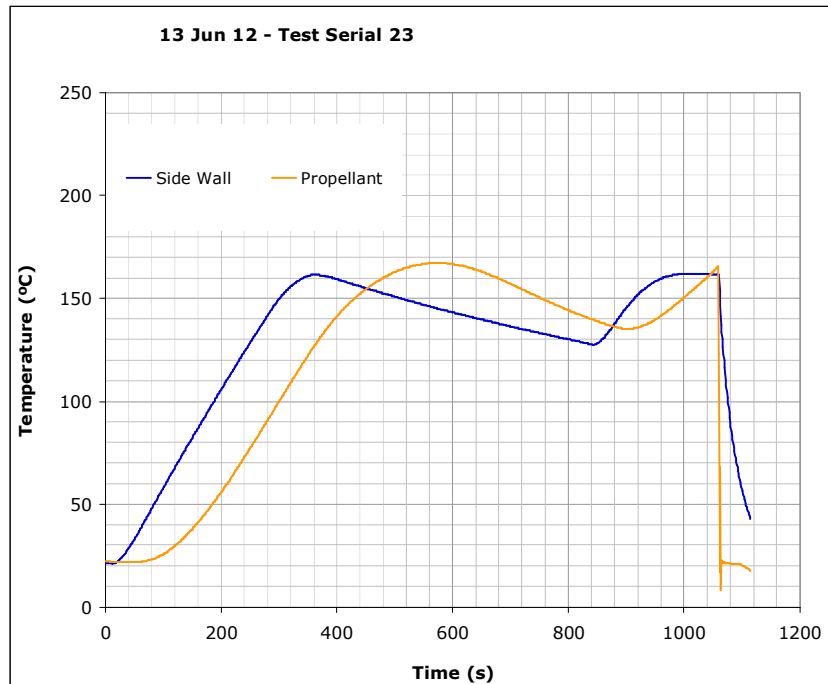


Figure E1.23: Raise temperature of T/C A1 to 161°C and Switch Off

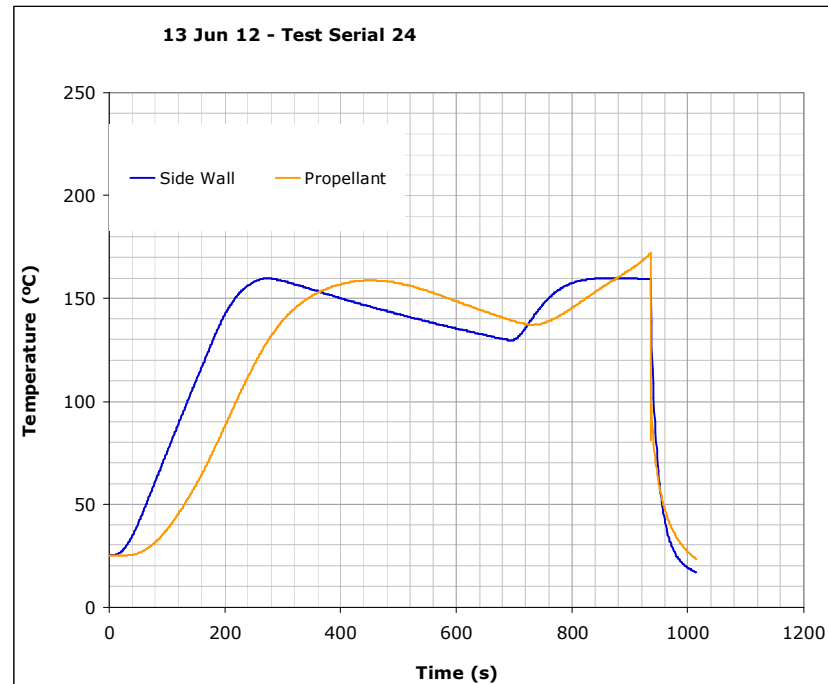


Figure E1.24: Raise temperature of T/C A1 to 161°C and Switch Off

Experiment Serials 25 and 26

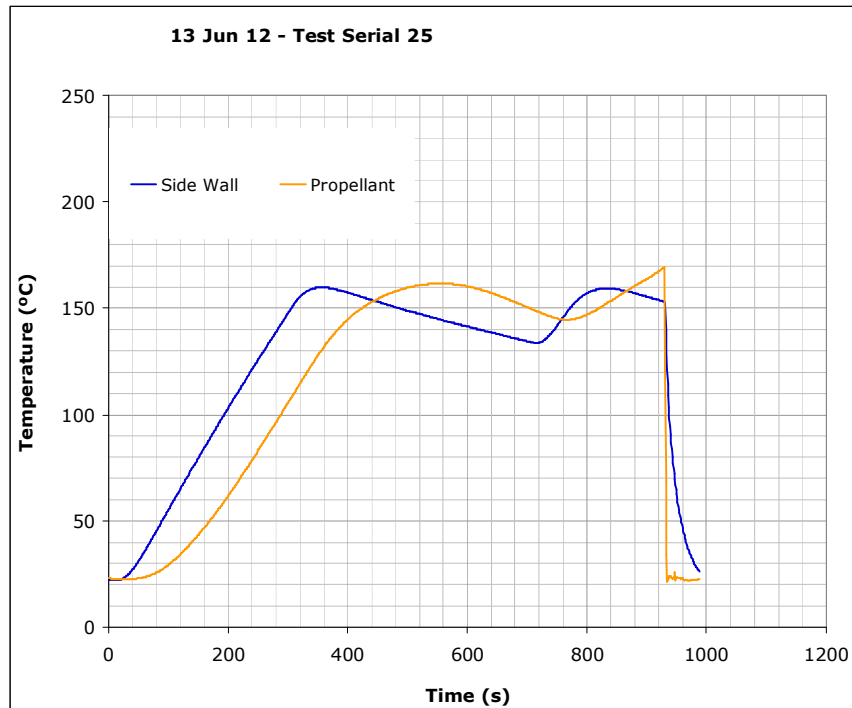


Figure E1.25: Raise temperature of T/C A1 to 162°C and Switch Off

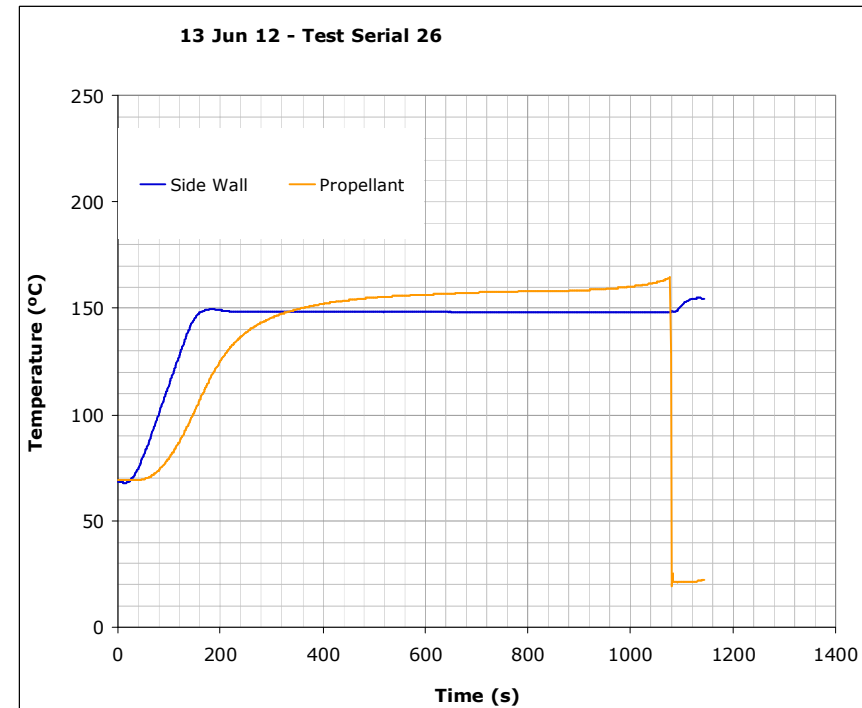


Figure E1.26: Raise temperature of T/C A1 to 150°C and Switch Off

Experiment Serials 27 and 28

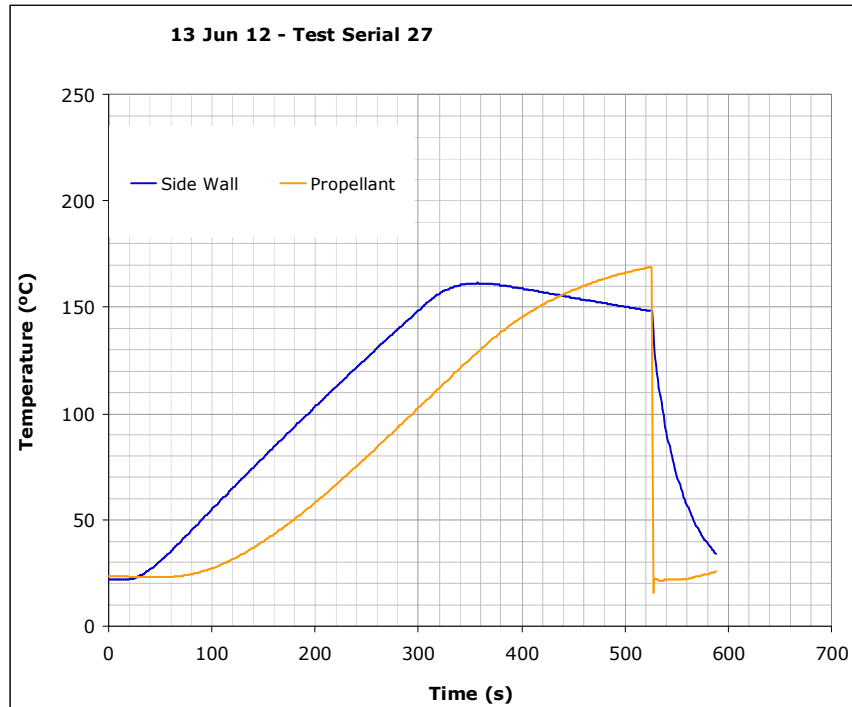


Figure E1.27: Raise temperature of T/C A1 to 163°C and Switch Off

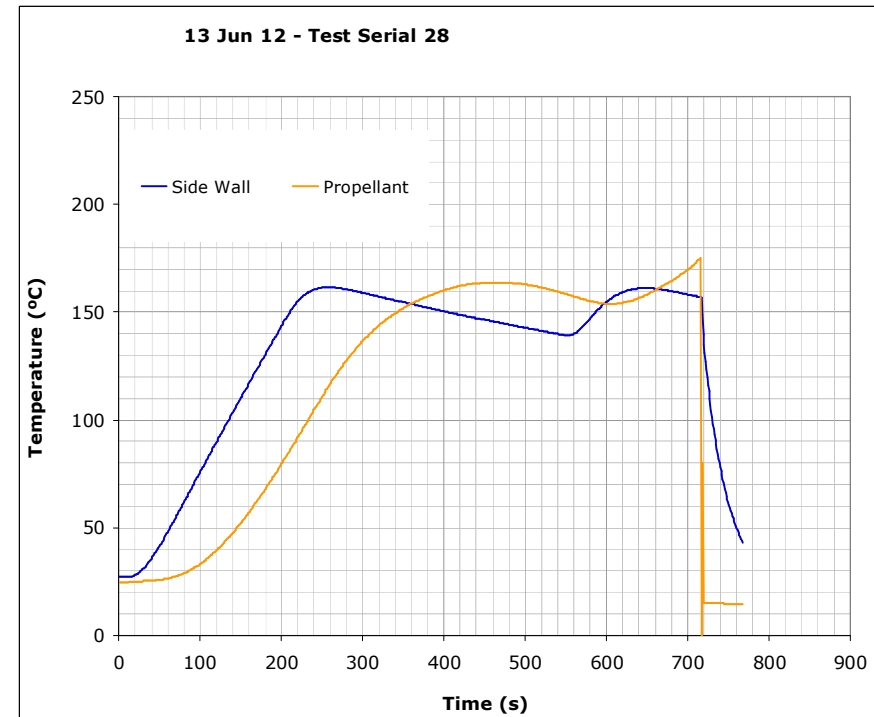


Figure E1.28: Raise temperature of T/C A1 to 162°C and Switch Off

Experiment Serials 29 and 30

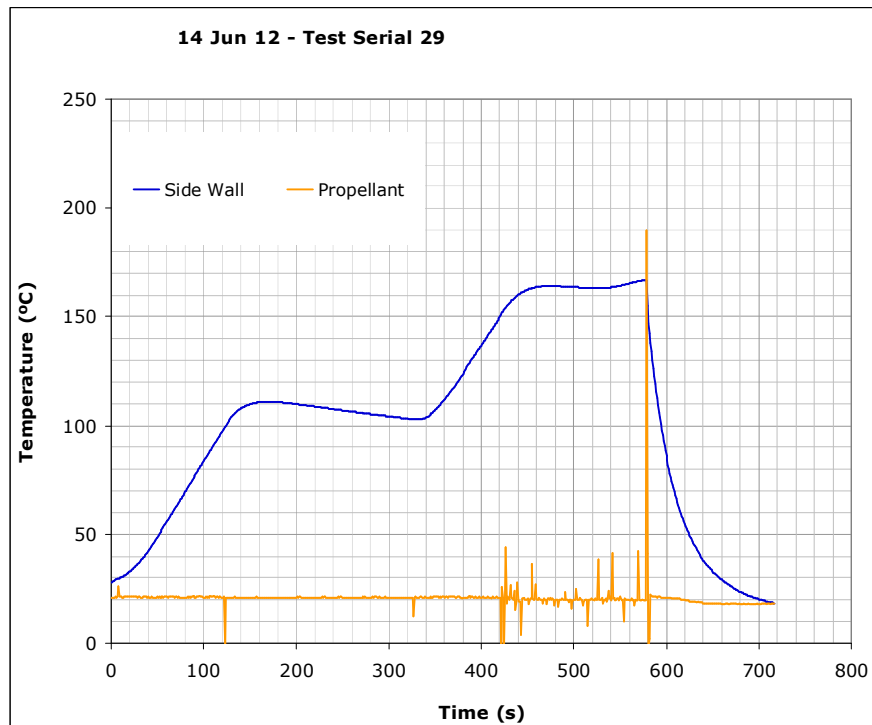


Figure E1.29: Raise temperature of T/C A1 to 162°C and Switch Off (TC A3 Failure – experiment terminated)

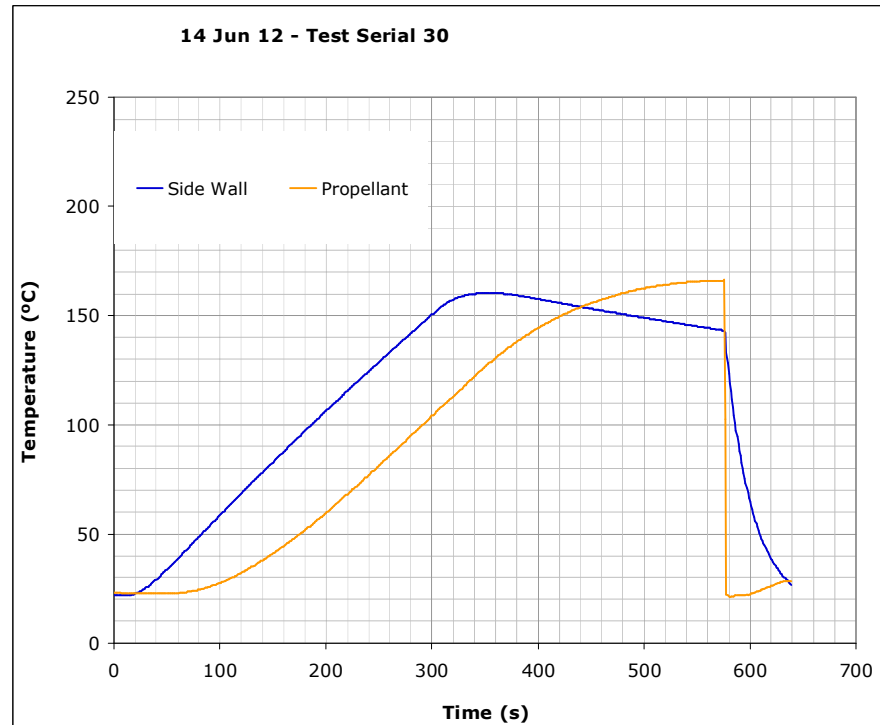


Figure E1.30: Raise temperature of T/C A1 to 162°C and Switch Off

Experiment Serials 31 and 32

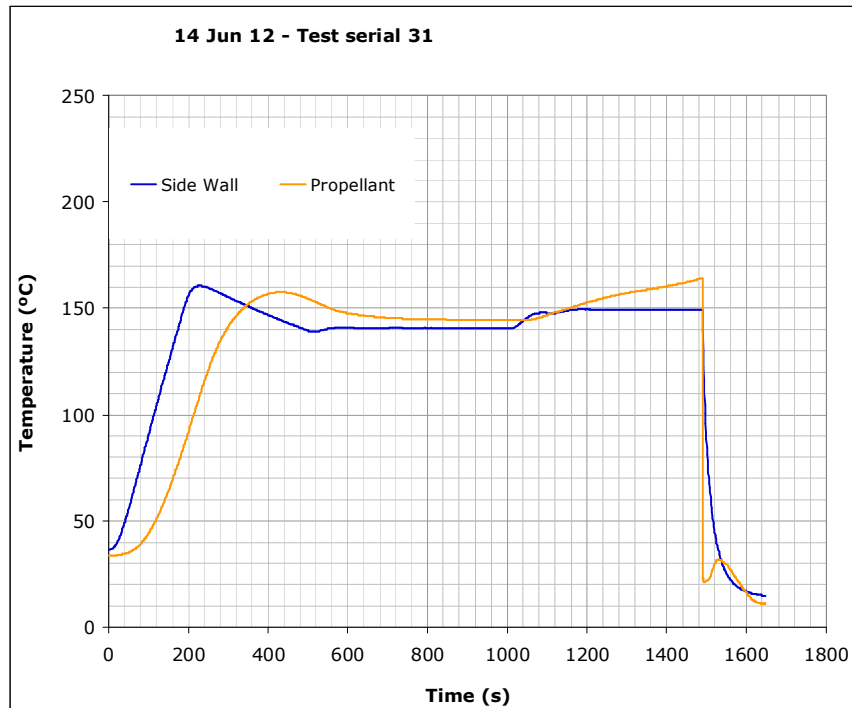


Figure E1.31: Raise temperature of T/C A1 to 161°C and Switch Off

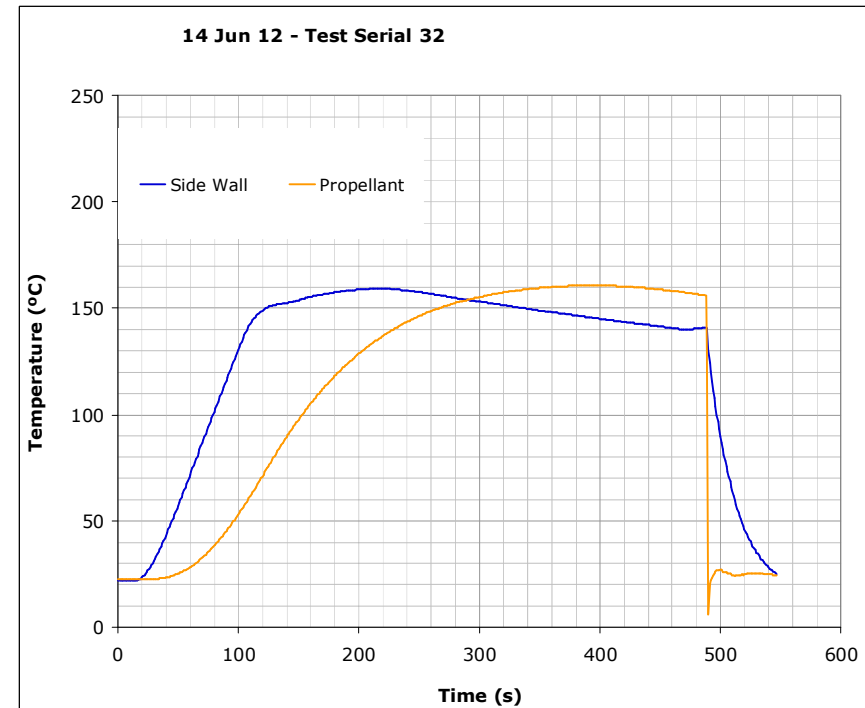


Figure E1.32: Raise temperature of T/C A1 to 162°C and Switch Off

Experiment Serials 33 and 34

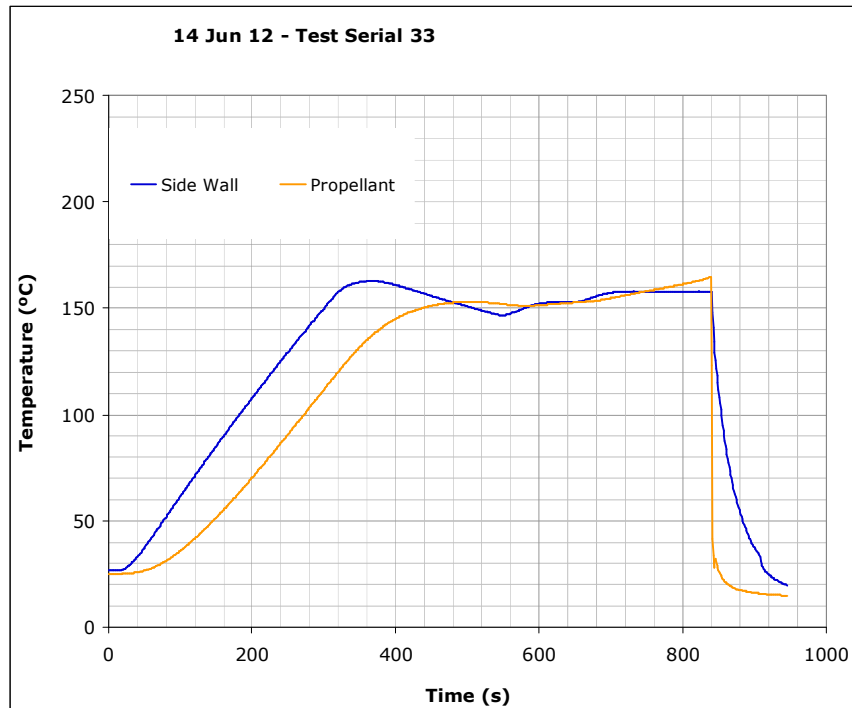


Figure E1.33: Raise temperature of T/C A1 to 162°C and Switch Off

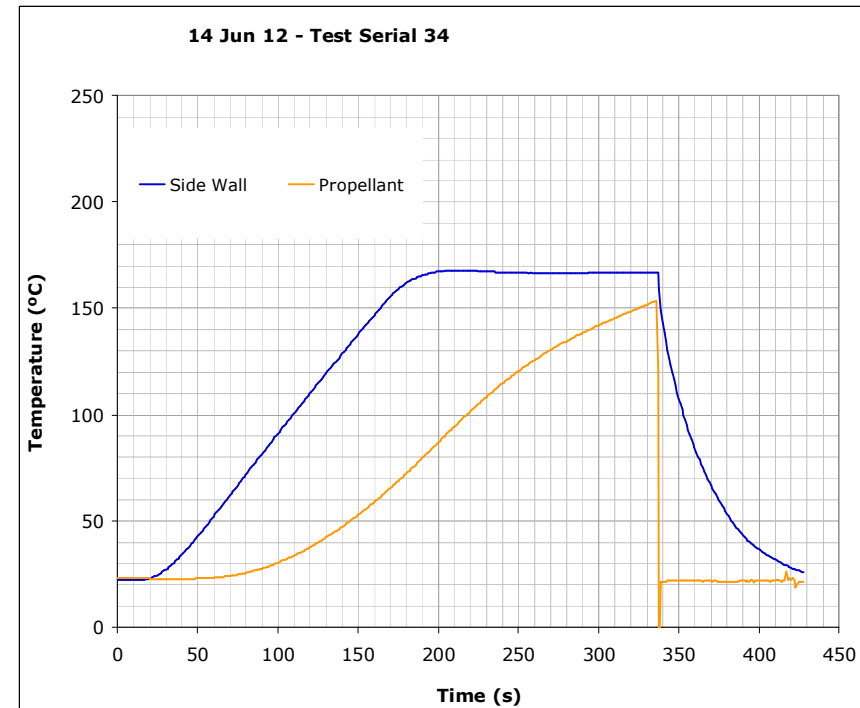


Figure E1.34: Raise temperature of T/C A1 to 162°C and Switch Off

Experiment Serial 35

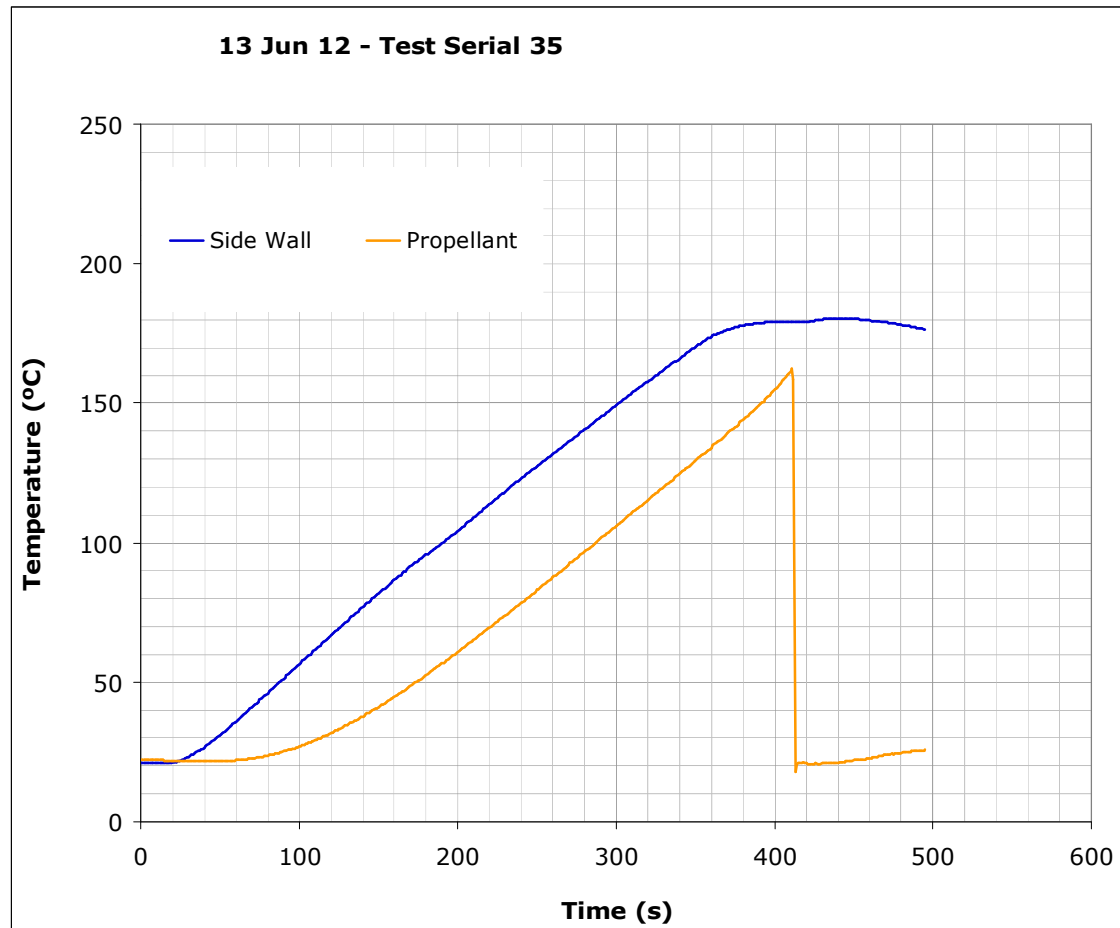


Figure E1.35: Raise temperature of T/C A1 to 180°C and Switch Off