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NATO STANDARD

AOP-4491

ENERGETIC MATERIALS, THERMAL SENSITIVENESS AND EXPLOSIVENESS TESTS

Edition A, Version 1

MARCH 2022



NORTH ATLANTIC TREATY ORGANIZATION

ALLIED ORDNANCE PUBLICATION

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NATO LETTER OF PROMULGATION

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Major General, GRC (A)
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INTRODUCTION

Participating countries who have ratified this standard have decided to accept these tests as standard procedures for determining the thermal sensitiveness and explosiveness of energetic materials and to use the data exchange formats as indicated in the annexes. One small scale test should be performed from each of Annex A and two larger scale tests from Annex B, one of which is a fast cook-off test (FCO) and one a slow cook-off test (SCO). The small-scale tests are performed in order to improve the characterization of the test energetic materials and to provide safety information relevant to the handling of small quantities of it and the larger scale tests are performed in order to assist in building a predictive capability in relation to potential systems hazards.

This standard calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way, absolves the user from statutory obligations relating to health and safety at any stage during use.

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Annex A to
AOP 4491

ANNEX A: TEMPERATURE OF IGNITION (Small-scale, low confinement)

A1. TEMPERATURE OF IGNITION

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the ignition temperature of solid energetic materials.

2. PRINCIPLE

Small weighed quantities of the explosive under test are heated in borosilicate glass test tubes placed in a steel block or other suitable heat sink, the temperature of which is controlled to rise at a steady rate of 5°C/minute, until an event occurs.

3. APPARATUS

The test tubes should comply with the following dimensions: length 114 ± 15 mm, external diameter 12-16 mm, wall thickness (light wall) 0.5-1.25 mm with rimmed end. A typical example of the apparatus is shown in Figure 1. Alternative designs may also be used. A hollow mild steel block suitable as a heat sink is wound with a layer of mica insulation and a nickel-chromium heating tape of suitable resistance to provide a sample temperature rise rate of up to 10°C per minute. The block is contained within a metal enclosure packed with thermal insulation. The hollow centre is also packed with thermal insulation. A programmer monitors the block temperature via a Pt/Pt-Rh thermocouple and controls the temperature rise rate. The standard rate is 5°C/minute. A 4.8 mm diameter platinum resistance thermometer is located in a drilling at the same pitch circle diameter in the block as that of the samples. It monitors the temperature at this point and displays it on a digital meter.

4. SAMPLE PREPARATION.

Solid powders should all pass through a 3-mm standard sieve. Rubbery or dough-like samples are either cut into a cube shape of the correct mass or chopped to pass through the sieve.

5. TYPES OF REACTION.

Reaction can take the form of burning with the development of a flame, rapid decomposition (puff of smoke) or explosion.

6. TEST PROCEDURE.

200 ± 2 mg samples of the test material are loaded into glass tubes and inserted to a depth of at least 20 mm into the heat sink. For unknown samples or those thought to be primary explosives a smaller quantity (usually 50 mg) is loaded. Any unused drillings in the block are filled with empty glass test tubes. The temperature controlling programmer is switched on and the sample tubes are observed through the laminated plastic safety screen for any sign of reaction. At least two samples are tested.

7. REPORTING OF RESULTS / DATA-SHEET

The result of the test is reported as a "Temperature of Ignition" and is the lowest temperature at which reaction is observed. If the duplicated samples produce results differing by 3°C or more the test is repeated. Samples which give no sign of reaction up to the usual maximum temperature of the test (400°C) are allowed to cool and then examined. If upon examination no decomposition is apparent the result is recorded as "Not less than 400°C". When examination does reveal decomposition to have occurred the result is recorded as "Decomposes slowly".

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8. REFERENCE DATA

Heating rate 5°C/min

Test explosive	Ignition temperature °C	Reaction type
Lead styphnate	269	Explodes
Service lead azide	319	Explodes
Double base propellants	167	Ignition
AP/HTPB propellant	271	Ignition
TNT	288	Ignition
Tetryl	183	Ignition
RDX	219	Ignition
HMX	273	Ignition
PETN	186	Ignition

9. REACTION DESCRIPTORS

No Reaction

Decomposition – decomposes slowly

Ignition

Explosion

Detonation

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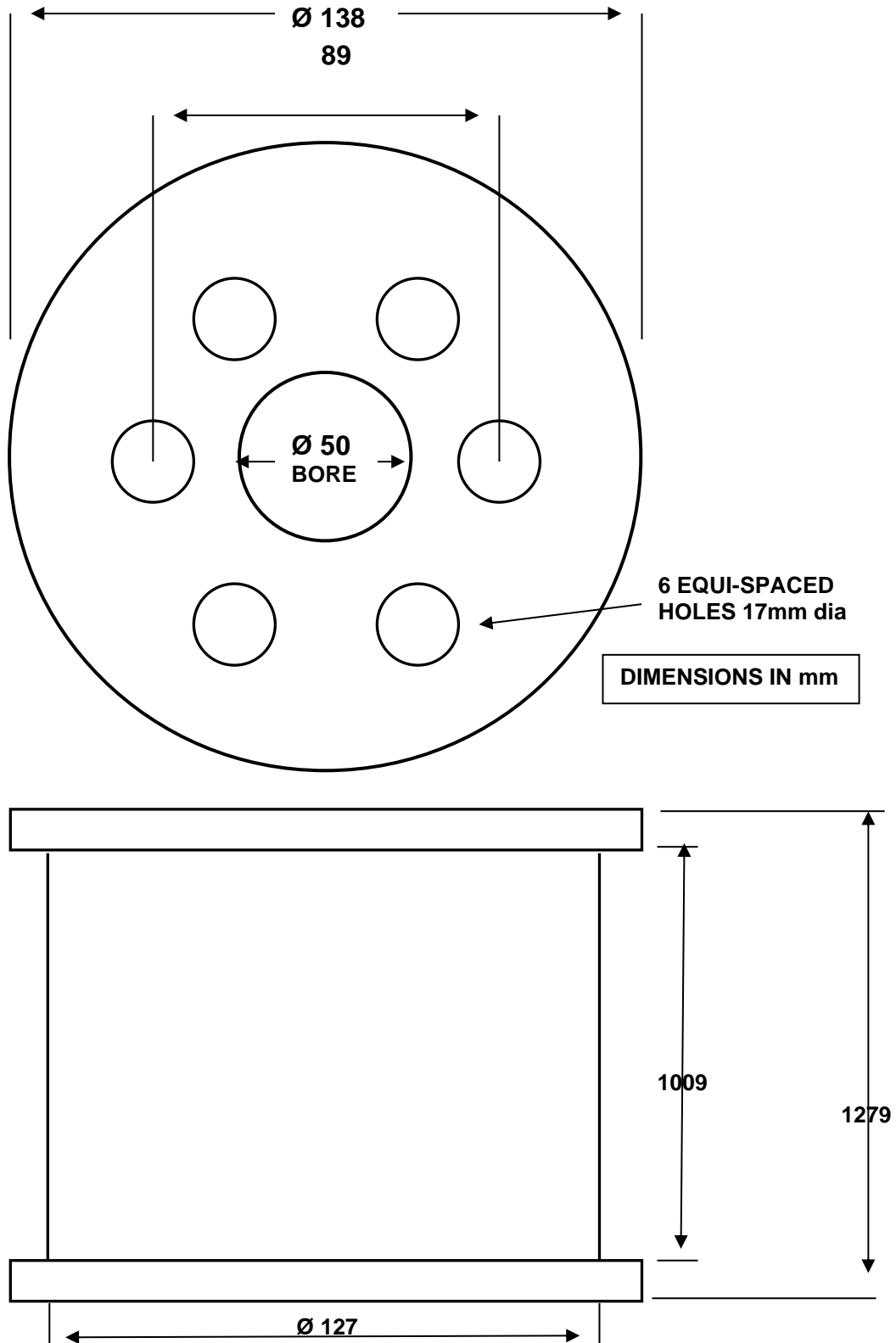


FIGURE 1 – EXAMPLE OF HEATING BLOCK

A-3

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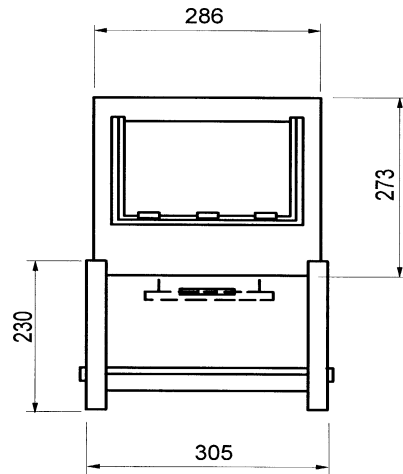
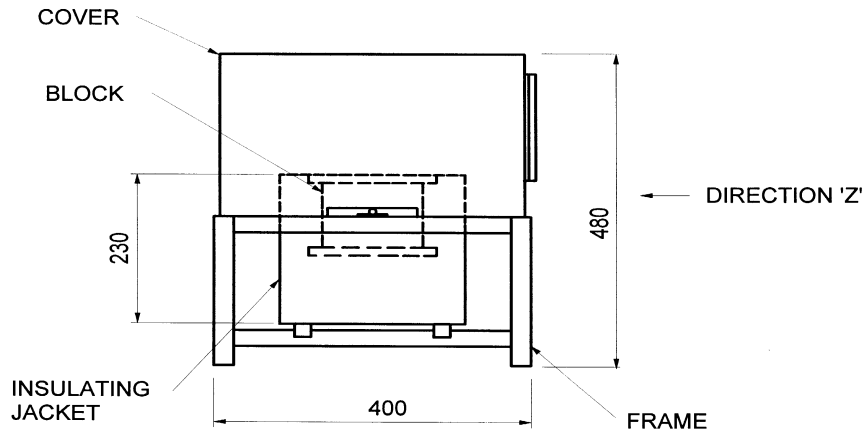
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VIEW IN
DIRECTION 'Z'

DIMENSIONS IN mm

FIGURE 2 - TYPICAL ENCLOSURE

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**Annex B to
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ANNEX B: COOK-OFF (Larger scale variable or higher confinement)

B1. KOENEN TEST (FCO only)

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the small scale thermal sensitivity of solid energetic materials.

2. PRINCIPLE

The thermal sensitivity of explosive substances to a strong and sustained thermal stimulus is determined in the steel tube test or Koenen test. Samples of the test material are confined in a steel tube fitted with an orifice plate at the top end. The tube is heated by four propane burners at a pre-calibrated rate of $3.3 \pm 0.3 \text{Ks}^{-1}$ and both the time to and nature of any ensuing reaction is noted. Most explosive substances will demonstrate a change from a burning response to an explosive response at a limiting orifice plate diameter and explodes at all smaller orifice diameters. Thermal sensitivity increases with increasing limiting orifice diameter and decreasing time to event and time to explosion.

3. APPARATUS

The apparatus is shown in Figures 1 and 2. It consists of a protective steel box fitted with the four propane gas burners, three in the walls and one in the base, each set up so that the tip of the inner blue flame just touches the tube and each heating a different part of the tube. Also, there are two 4 mm support rods, the steel tube and the orifice plate. The 24-mm internal diameter, 75 mm long steel tube is drawn from sheet steel and a flange at the top seals against a selected chrome steel orifice plate. These components are held together by a threaded collar and box nut. The threaded collar and box nut are made from manganese chrome steel. Plates with an orifice diameter of 1, 1.5, 2, 2.5, 3, 4, 5, 6, 8, 10, 12, 14, 16, 18 and 20 mm can be used. The protective box is located in a test chamber which can be viewed through an armoured glass window in a strong wall.

4. SAMPLE PREPARATION

Solid explosive substances are tested in the dry state. Powdered substances are sieved and the fraction with a particle size of 0.5 to 1.0 mm is used for testing. Pressed, cast, or otherwise compacted substances are crushed and then sieved. Small grain gun propellants are tested in the original size. Liquid explosive substances are tested without preparation. Composite rocket propellants are cast directly into the steel tube.

5. TYPES OF REACTION

These range from an undamaged tube to the whole tube assembly being damaged or fragmented. It is possible to sub-divide the types of reaction into nine categories as follows:

- : Tube undamaged
- A: Tube bottom bulged
- B: Tube bottom and wall bulged
- C: Tube bottom severed
- D: Tube torn open
- E: Tube split into two fragments
- F: Tube split into three or more mainly large fragments
- G: Tube split into many mainly small fragments
- H: as G; threaded collar, box nut and/or orifice plate damaged or fragmented

For the purposes of this test only F, G and H are positive reactions.

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6. TEST PROCEDURE.

Solid, liquid or gelatinous substances can be tested. Each trial involves the filling of a tube to a depth of 60 mm, i.e.. 15 mm from the top of the tube. The empty tube is weighed. Powdered substances are lightly tamped using a wooden rod and by tapping on the outside of the tube. Gelatinous substances are incrementally loaded into the tube using a spatula and light tamping with the wooden rod. Explosive substances can be tested in a compacted state by either casting or pressing directly into the tube or by machining to the tube dimensions. The tube is re-weighed, then fitted with an orifice plate and the nut assembly fitted and tightened. It is then suspended between the two supporting rods in the protective box, the pilot flame is ignited and the door to the test chamber is closed. The burners are ignited and the time (t_1) between ignition of the burners and the first reaction of the sample is noted. If the first reaction is not an explosion, the further time elapsed between t_1 and explosion (t_2) is noted. Should an explosion not occur, t_2 is the time from the first reaction of the test substance to its total consumption. After each test the gas is turned off and the chamber is ventilated. Then the steel tube or its fragments are collected for inspection. All subsequent tests are performed at the same sample density as was involved with the first test.

7. REPORTING OF RESULTS / DATA-SHEET

The thermal sensitivity of an explosive substance is expressed as the orifice plate limiting diameter; that is the largest diameter giving at least one explosion in three trials. Explosion is defined as those events causing the steel tube to fragment into three or more pieces.

8. REFERENCE DATA

Formal pass / fail criteria are not applied to the test results. Measurements of t_1 and t_2 are only used to differentiate between two explosives if they have the same limiting orifice diameter.

Test explosive	Limiting orifice diameter, mm	Reaction category
TNT	4	F
PETN	5	G
Tetryl	6	G
RDX	6	G
HMX	8	F

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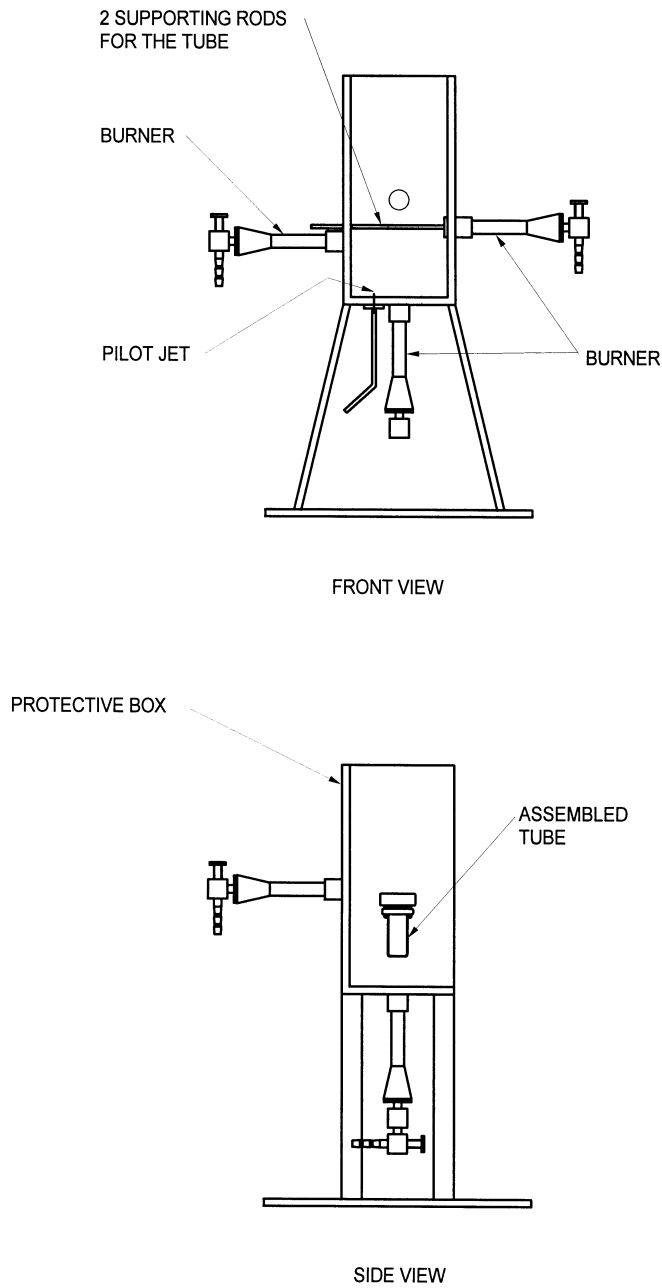


FIGURE 1 - TYPICAL HEATER ENCLOSURE

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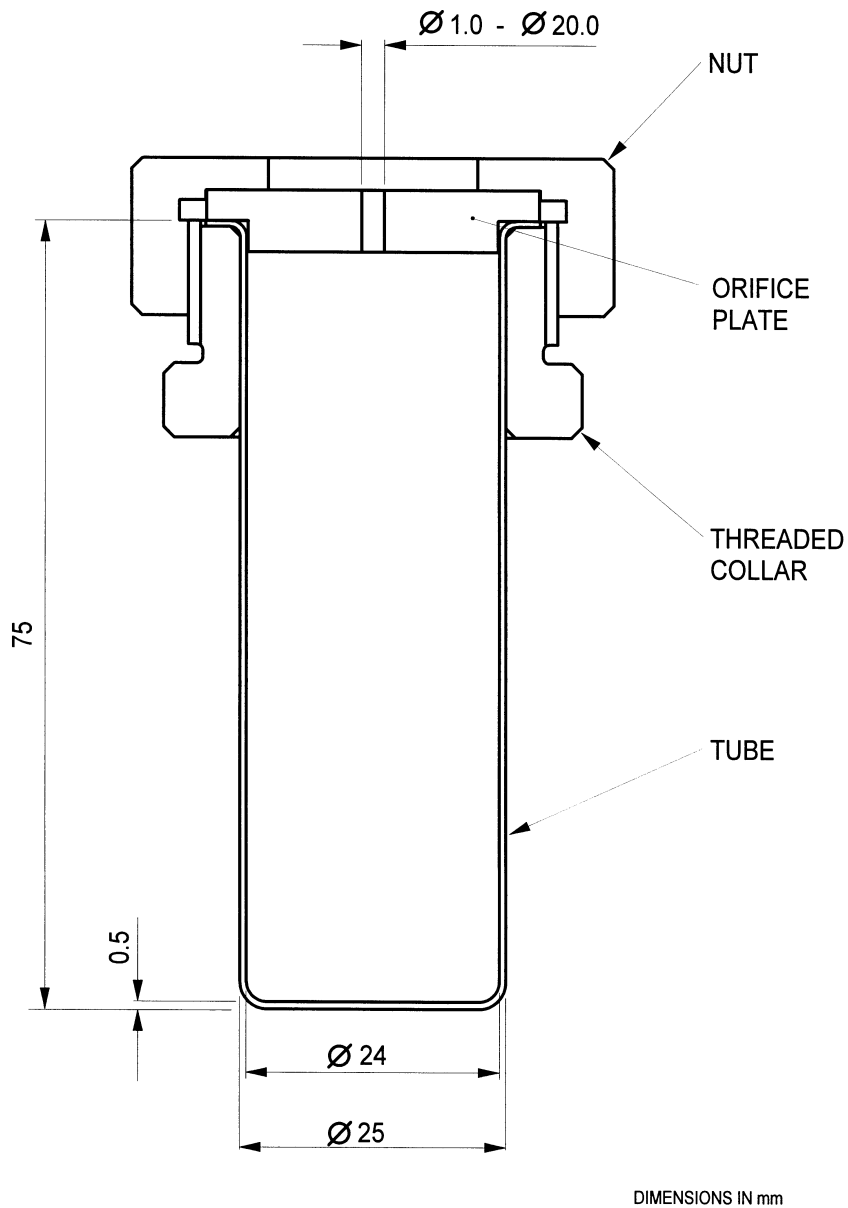


FIGURE 2 - TUBE AND ACCESSORIES

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B-2. VARIABLE CONFINEMENT (FCO AND SCO) TEST

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the explosiveness of solid and bulk energetic materials.

2. PRINCIPLE

A sample of explosive, propellant, or pyrotechnic (bulk or solid), confined in an aluminium liner within a steel tube, is heated by means of electrical windings. A typical fast cook-off heating rate is shown in Figure 1. Slow cook-off rate is 3.3°C/h. The degree of tube fragmentation is used to determine the reaction category and the objective of the test is to determine the median tube thickness just resulting in a deflagration reaction rather than burning.

3. APPARATUS

This is shown in Figures 2 to 7 and consists of an aluminium liner, one from a series of increasing wall thickness steel tubes, heating bands, thermocouples, steel spacer washers (aluminium foil covered), steel end plates and retaining bolts. The thickness of the aluminium sleeve is 2.5 mm and the thickness of the steel tube can be 0.375 to 3 mm in 0.375 mm increments. Two thermocouples are fitted, one in each of two diametrically opposing grooves in the aluminium sleeve. Either two mica-insulated band heaters or an insulated nichrome wire winding are located on the steel tube, spacer washers are added to each end and the assembly is located in recesses between steel witness plates. The retaining bolts are evenly tightened to a torque of 3.39 +/- 0.4 N-m.

N.B: For the bottom steel washers place one piece of aluminium foil on top. For the top washer's place one aluminium foil piece below the sample to sandwich the test materials with the aluminium foil when the material is a powder or granular form.

Witness plate and bolts sizing:

9.53mm (0.375") witness plates and 6.35mm (0.250") bolts for T-15 and T-30 confinement sleeves. 11.11mm (0.4375") witness plates and 6.35mm (0.250") bolts for T-45 and T-60 confinement sleeves. 12.7mm (0.500") witness plates and 9.53mm (0.375") bolts for T-75, T-90, T-105, and T-120 confinement sleeves.

4. SAMPLE PREPARATION

Each energetic material sample weighs approximately 50 g and can consist of a single piece or three pellets as solid and varies for bulk materials based on bulk density.

5. TYPES OF REACTION

- (1) Burning: The steel sleeve is recovered in one piece. The aluminium sleeve is usually recovered in one or two pieces. Witness plates exhibit no deformation. Retaining bolts usually remain intact, although in some cases they may be bowed.
- (2) Pressure Rupture: The steel sleeve is recovered in no more than three large fragments. No more than two bolts sheared. Witness plates exhibit no deformation.
- (3) Deflagration: The steel sleeve is recovered in three or more large fragments. The aluminium sleeve usually fragments into large pieces. Witness plates exhibit slight deformation. Two or more retaining bolts fail in shear.
- (4) Explosion: Both steel and aluminium sleeves fragment into several small pieces. Witness plates exhibit some deformation. All bolts sheared.
- (5) Partial detonation: Steel and aluminium sleeves fragment into both large and small pieces. Witness plates exhibit severe deformation. All bolts sheared.

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(6) Detonation: Steel and aluminium sleeves fragment into very small pieces. Witness plates exhibit severe damage with a clean hole punched through one plate. All bolts sheared.

Cautionary Note: In the French language, the terms 'deflagration' and explosion' are used in the opposite sense to the definitions given above. In the French version of this STANAG the English word 'deflagration' is translated as 'explosion' and the English word 'explosion' by the French word 'déflagration'.

6. TEST PROCEDURE

A starting confinement thickness of steel tubing is chosen and a test vehicle is prepared. The temperature of the curved surface of the test explosive is raised at the prescribed rate as judged by the thermocouple outputs until reaction occurs. Following reaction, all debris is collected and assessed. Testing continues by altering the steel confinement thickness, until the transition region from a burning to a deflagration reaction has been first bracketed and then confirmed by a number of results on each side of the transition. If it is not possible to determine the burning to deflagration transition because, for example, the response is more violent even when under the lowest confinement level, then the trials results are recorded and reported as they stand.

7. REPORTING OF RESULTS / DATA-SHEET

The reaction categories of all of the trials conducted are reported and the steel confinement thickness to cause a transition from burning to deflagration is calculated.

8. REFERENCE DATA

These are shown in Table 1.

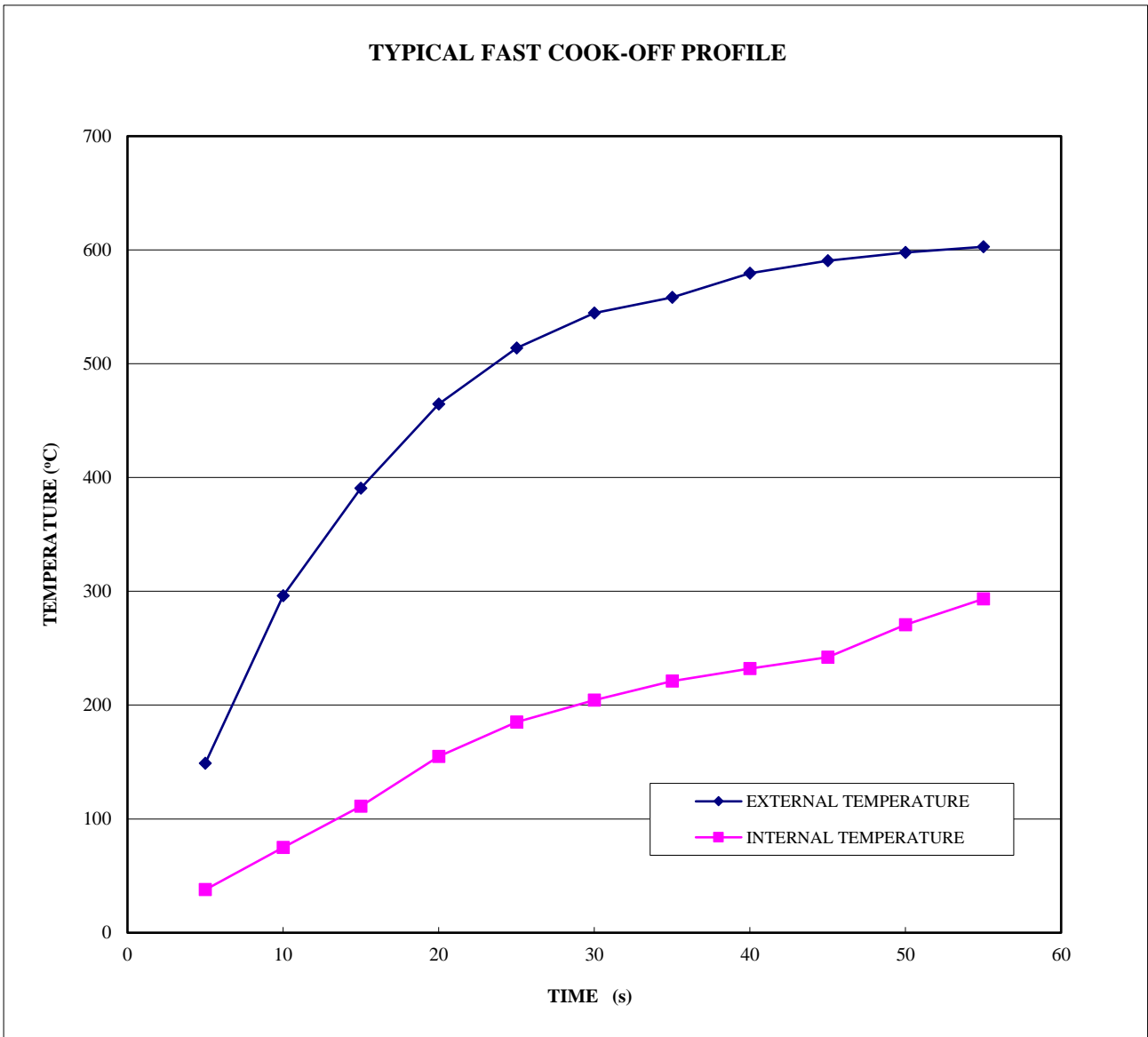


FIGURE 1

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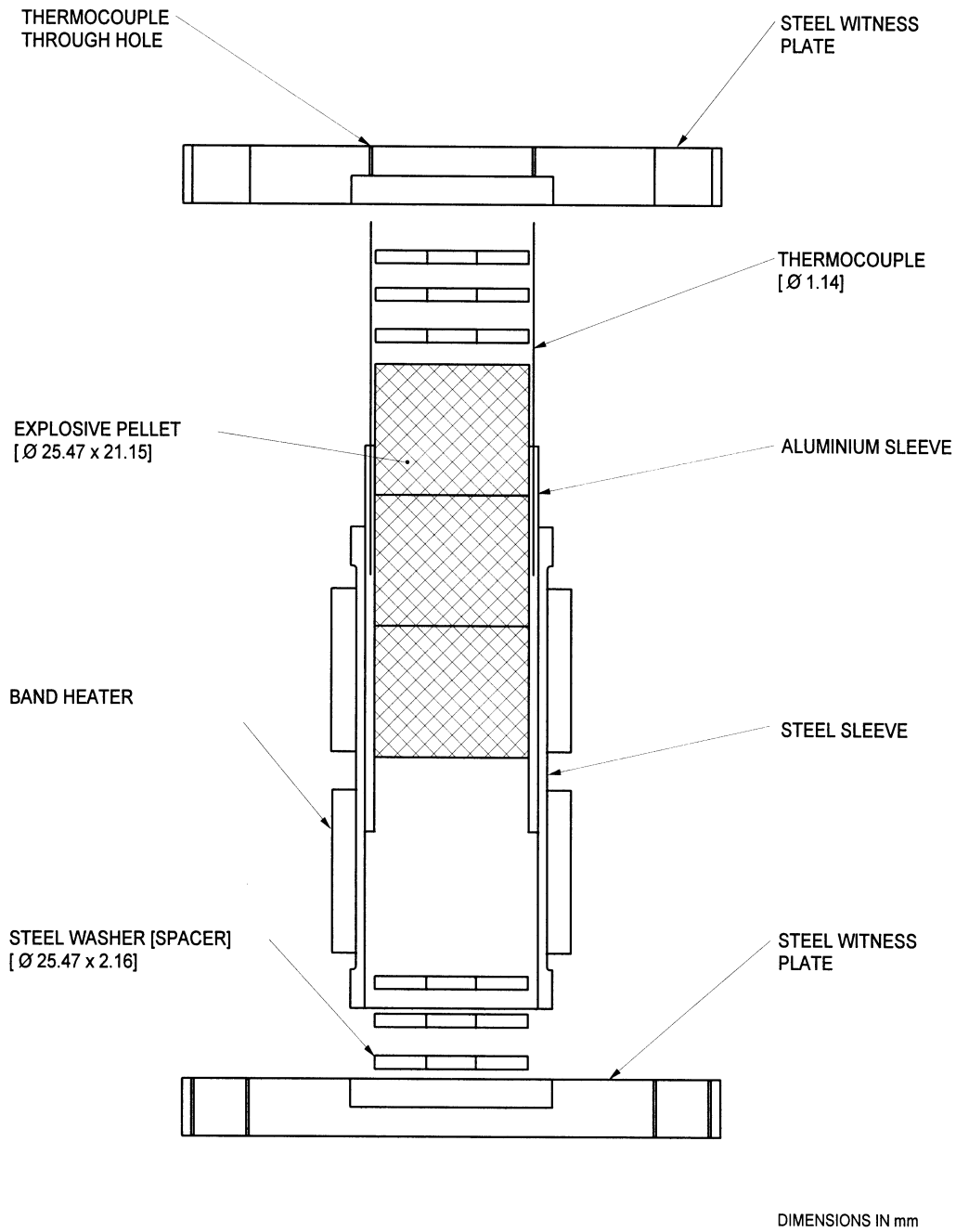


FIGURE 2 - ASSEMBLY, EXPLODED VIEW

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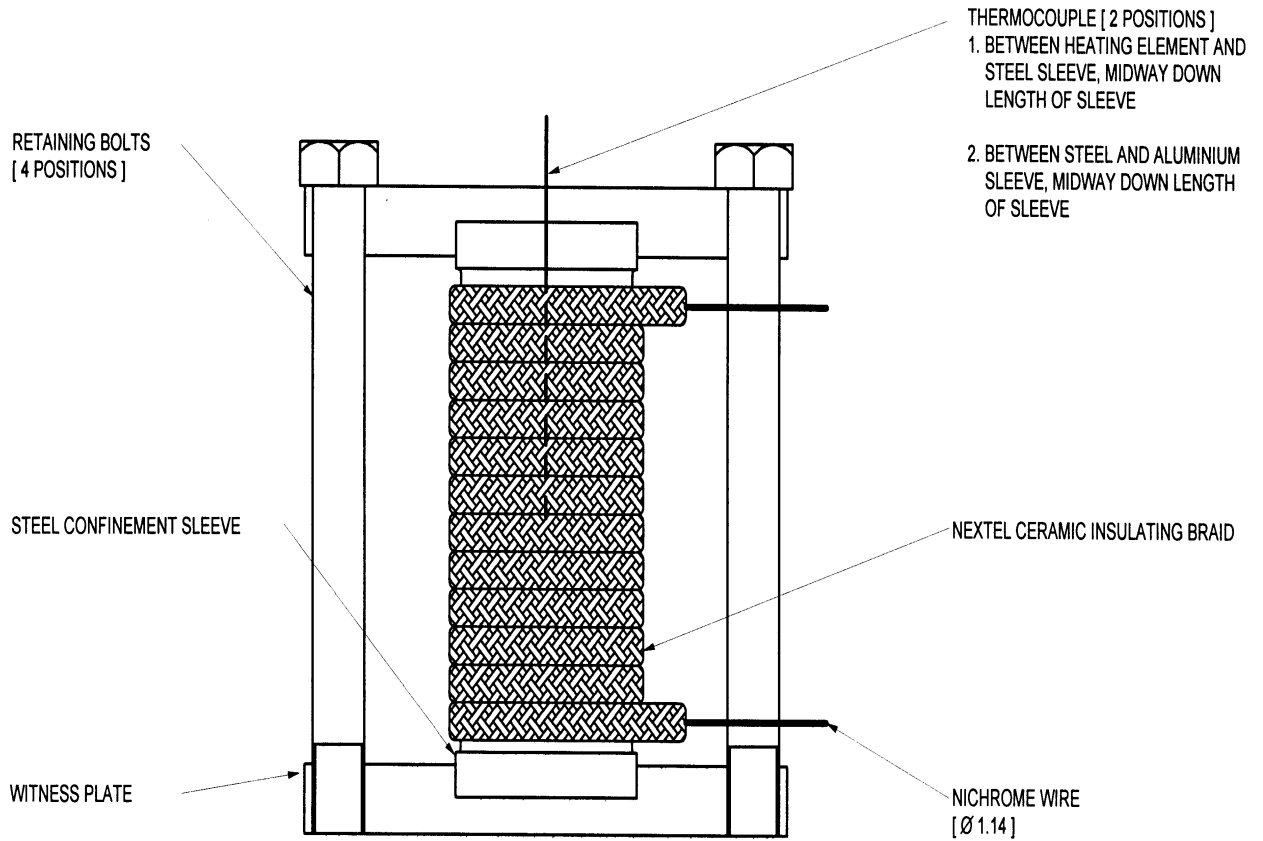


FIGURE 3 - ASSEMBLY, SHOWING HEATER

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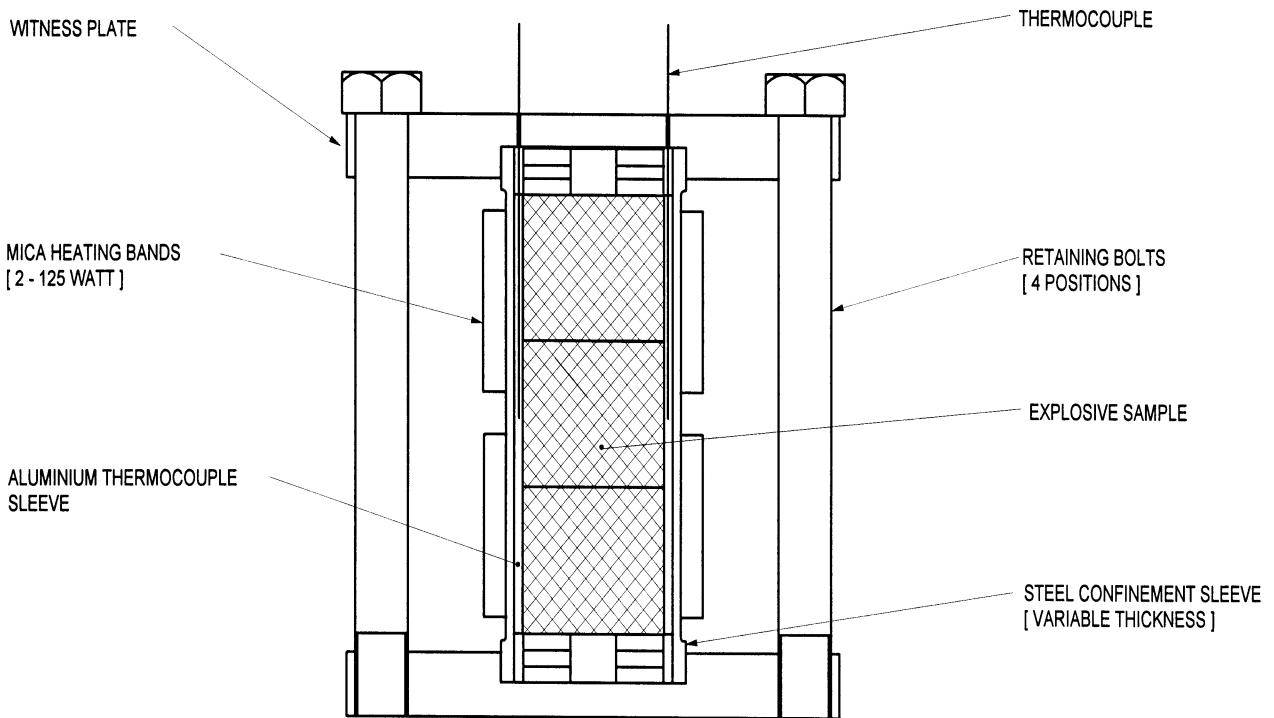


FIGURE 4 - ASSEMBLY SECTIONED VIEW

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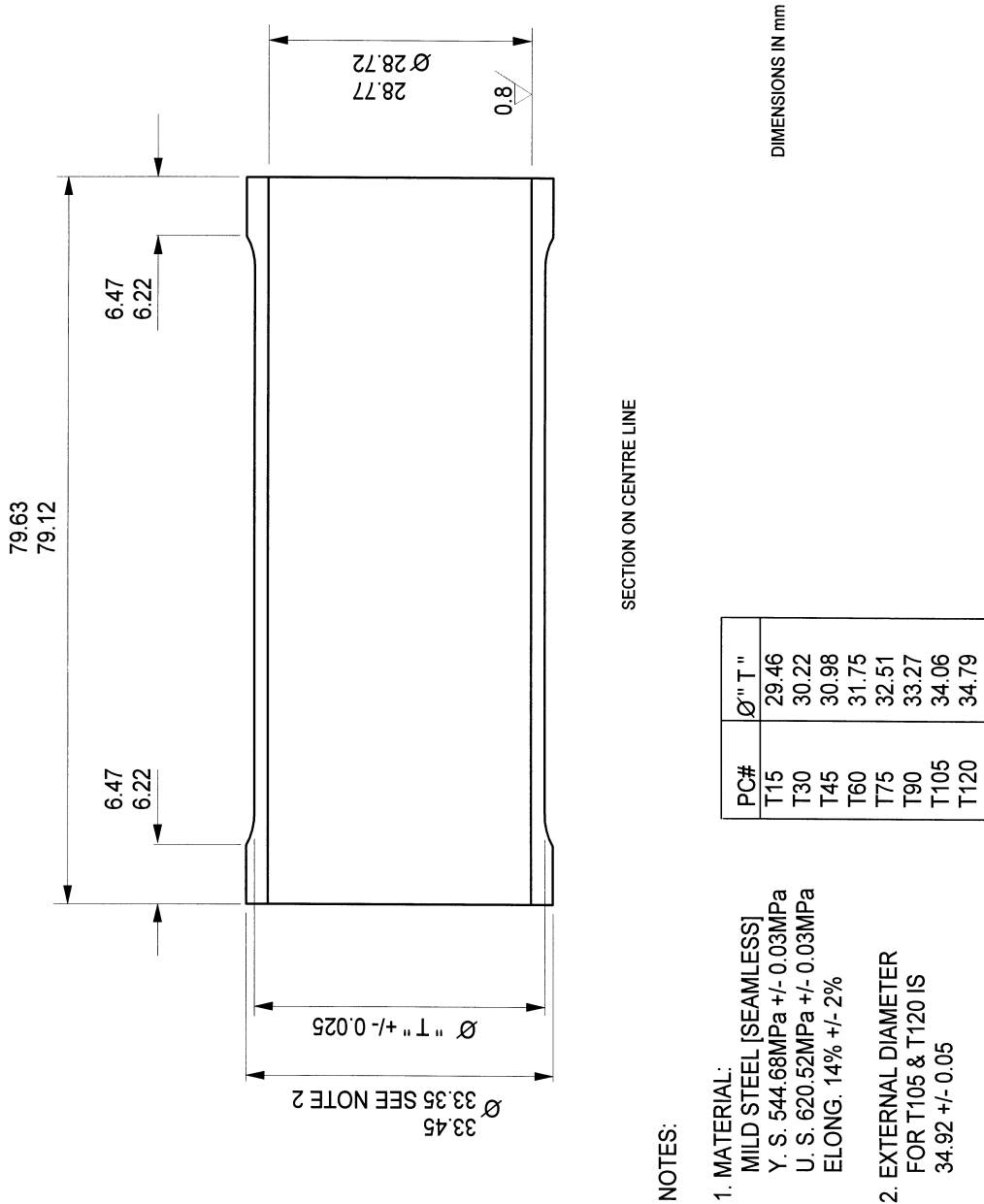


FIGURE 5 - STEEL SLEEVE

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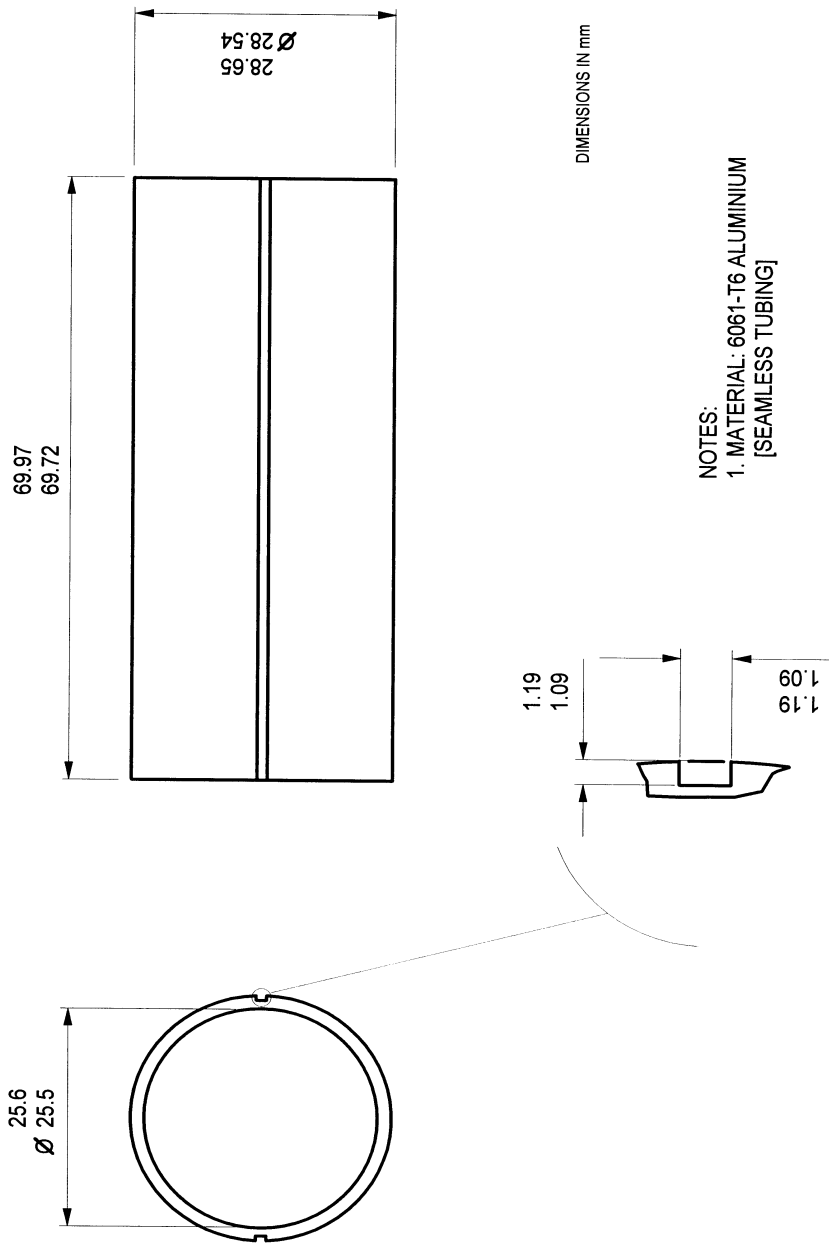


FIGURE 6 - ALUMINIUM SLEEVE

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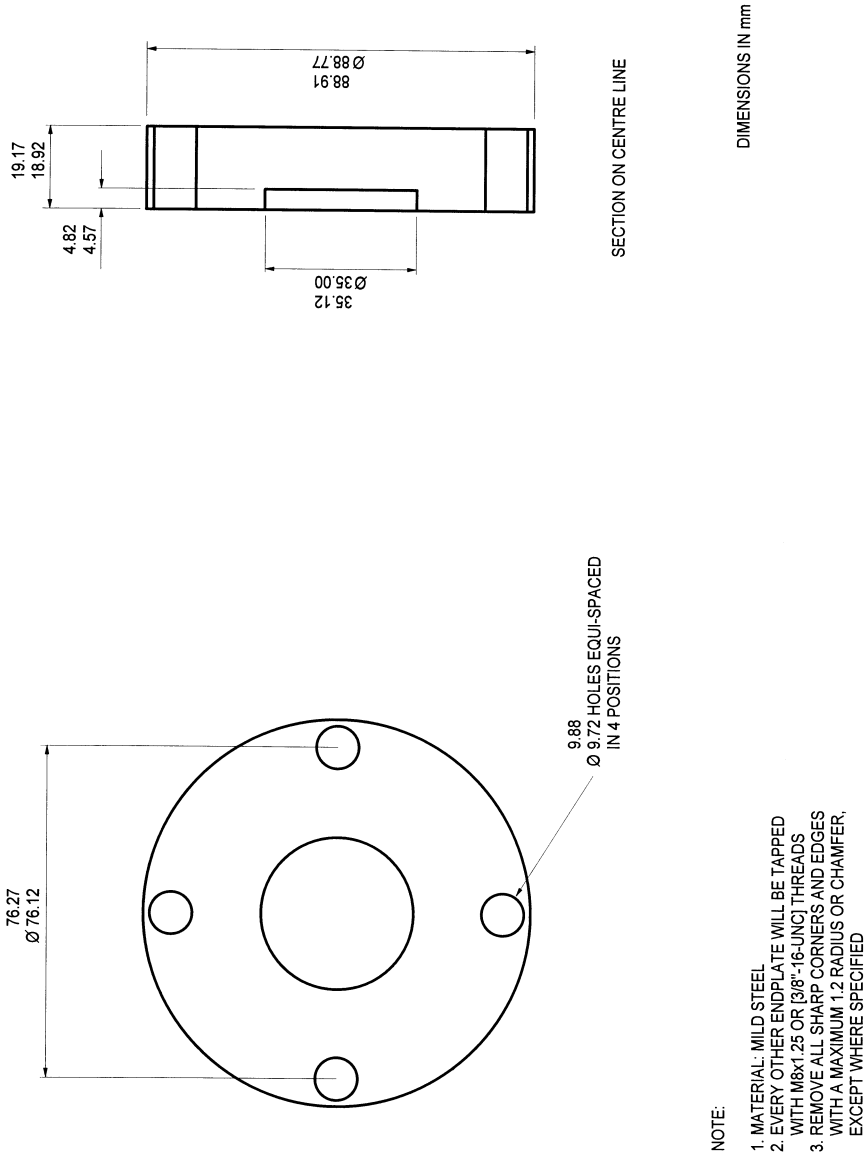


FIGURE 7 - WITNESS PLATE

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Test Explosive	Average Density (kg/m ³)	Steel tubes wall (mm)	External cook – off temperature (°C)	Internal cook – off temperature (°C)	Reaction Category
CH - 6	1.65	0.375	510	354	Explosion
CH - 6	1.65	0.750	621	338	Detonation
PBXN - 5	1.79	1.125	627	366	Burn
PBXN - 5	1.79	1.500	518	354	Deflagration
PBXN - 5	1.79	1.500	568	318	Burn
PBXN - 5	1.79	1.875	510	343	Explosion
PBXW - 11	1.79	2.250	538	366	Detonation
PBXW - 11	1.79	1.875	554	316	Burn
PBXN - 5	1.79	2.625	685	307	Deflagration
PBXN - 5	1.77	2.625	649	379	Burn
PBXN - 5	1.77	3.000	602	352	Deflagration
Comp A – 5	1.65	0.375	643	402	Detonation
Comp A – 5	1.65	0.750	579	318	Detonation

TABLE 1 – VCCT (FCO) RESULTS

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**Annex B to
AOP 4491**

B-3. TUBE TEST (FCO AND SCO)

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the explosiveness of solid energetic materials.

2. PRINCIPLE

A sample of explosive, confined in a steel tube, is heated by means of an external fire for FCO or by an electrical winding for SCO. The degree of tube fragmentation is used to assess the relative explosiveness of the composition under test.

3. APPARATUS

The assembled test vehicle is shown schematically as Figure 1. It consists of a solid cold drawn mild steel tube, of internal diameter 30 to 50mm, length 200 to 254mm and wall thickness 4 to 6mm, sealed by threaded end caps. The end caps must be as strong or stronger than the tube so that in low explosiveness events the tube wall fails before the end caps fail.

4. SAMPLE PREPARATION

The tube and end caps are weighed and then whenever possible, the explosive charge is filled directly into the test vehicle. Pressed or machined pellets can be loaded separately but they must be a good fit in the tube. The filling weight is recorded.

5. TYPES OF REACTION

Results are tabulated as shown in Table 1. The number of body fragments provides the most useful indication of the explosiveness of the test material and this number defines the reaction category. End cap fragments do not count as body fragments.

6. TEST PROCEDURE

Firings should be carried out in a facility capable of retaining at least a representative sample of the fragments produced. For FCO the filled test vehicle is supported over either a tray containing 2 litres of petrol or a wood fire. A proven method to produce a wood fire which has a balanced fuel / air ratio and therefore produces less smoke is to construct a lattice from air dried pieces of wood each not more than 50 mm thick and spaced at about 100 mm intervals. The fuel is remotely ignited and the time from ignition to event is recorded. When all signs of fire have disappeared and a suitable cooling off period has elapsed, the remains of the vehicle are collected along with any unreacted explosive. For SCO, the electrical heater winding is used.

The number of steel body fragments and the total number of fragments are recorded. A minimum of three trials are undertaken for each explosive under test.

The use of propane burners has been proposed by some member nations. This method can be used to carry out the FCO test, however, the reaction to this method has not been favourable as the heating rate is significantly higher than that of the 'traditional' method, thereby leading to spurious data.

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**Annex B to
AOP 4491**

7. REPORTING OF RESULTS / DATA-SHEET

Results are tabulated as shown in Table 2. The number of body fragments provides the most useful indication of the explosiveness of the test material.

8. REFERENCE DATA

These are shown in Table 3.

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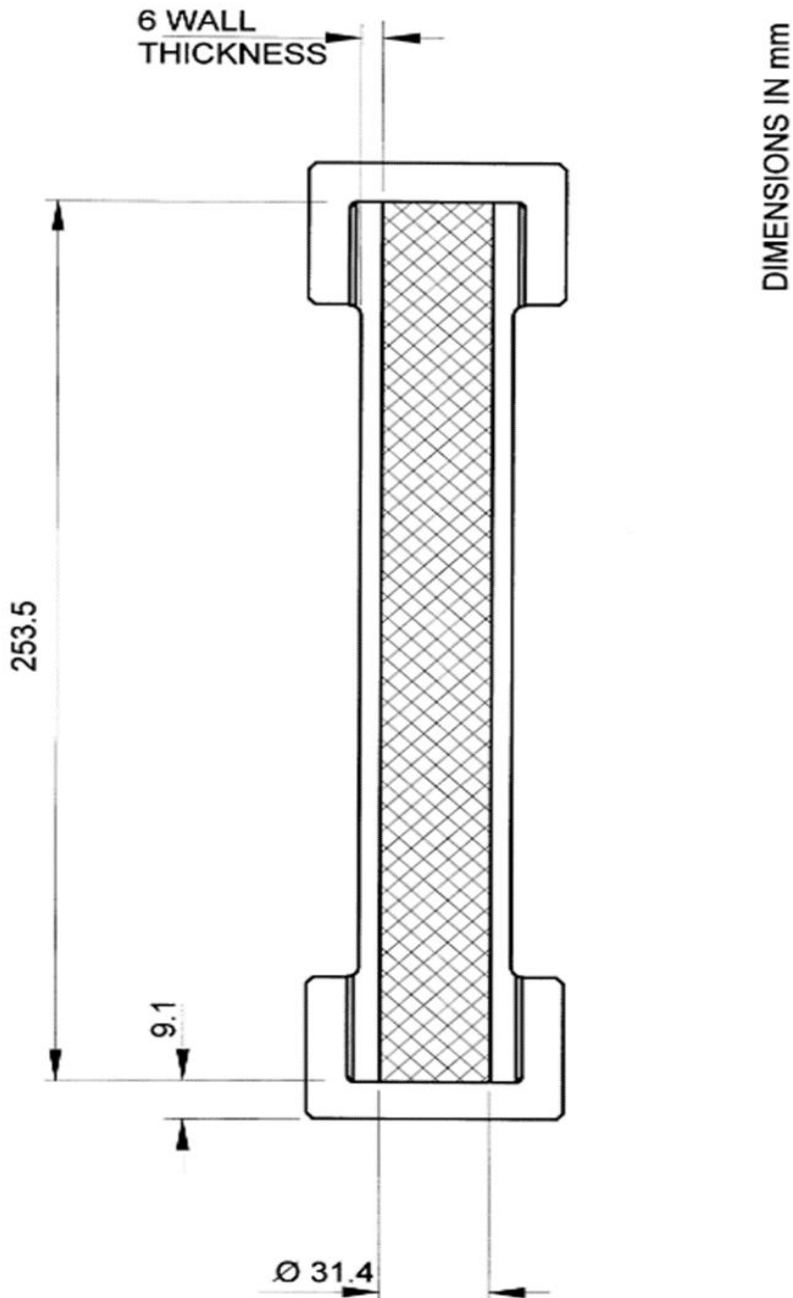


Figure 1:
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Category	Reaction Description	Observation
0	No reaction	Internal inspection.
0/1	Burning / decomposition	No disruption of test vehicle.
1	Deflagration	Test vehicle ruptured but one fragment approximates to initial weight.
2	Explosion	2 to 9 test vehicle body fragments.
3	Detonation	10 to 100 test vehicle body fragments showing evidence of detonation.
4	Detonation	>100 test vehicle body fragments showing evidence of detonation.

TABLE 1: DESCRIPTION OF REACTION CATEGORIES

NB: The categories shown are NOT IM reaction categories.

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**Annex B to
AOP 4491**

Explosive Formulation: TNT/HNS 99.5 / 0.5

Filling Method: Open cast.

Filled Density: 1590 kg/m³

Round No	Trial Date	Recovered Fragments			%Filling Recovered	Reaction category	Time to Event (s)	Comments
		Total No	Body no	% Weight				
1	12/7/89	111	98	80	0	4	408	Venting prior to event
2	ditto	53	46	80	0	3	367	
3	ditto	124	108	55	0	4	447	
4	ditto	52	43	85	0	3	301	
5	ditto	74	64	95	0	3	315	
6	ditto	83	77	60	0	4	255	
7	ditto	58	51	95	0	3	236	
8	ditto	158	148	75	0	4	357	
9	ditto	142	136	85	0	4	304	
10	13/7/89	95	44	95	0	3	302	

TABLE 2: TUBE TEST (FCO) RESULTS FORMAT

NB: The reaction categories shown are NOT IM reaction categories.

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**Annex B to
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Composition	Reaction Category, (No. of Events)	Average No. of Body fragments	Average Time to Event (s)
HMX/TNT 85/15	3(4), 4(6)	> 100	174
HMX/PU 85/15	1(10)	1	171
RDX/Wax/Al/PIB 71/7/20/2 +0.5% Carbon black	1(4), 2 (6)	1	163
TNT/HNS 99.5/0.5	3(5), 4(5)	55 / > 100	329

TABLE 3: TYPICAL TUBE TEST (FCO) RESULTS

NB: The reaction categories shown are NOT IM reaction categories.

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**Annex B to
AOP 4491**

B4-UK Test 35: TUBE TEST – INTERNAL IGNITION

Appreciation

1. The internal ignition version of the Tube Test can be used to evaluate the explosiveness of solid energetic materials and propellants. It assesses the behaviour of explosive charges when ignited under confinement. The level of violence of this reaction, and its potential to cause damage to the surroundings, is referred to as "explosiveness".
2. Explosiveness is influenced by several variables including the nature of the stimulus, the temperature and physical form of the explosive and its confinement. The Internal Ignition Tube Test has been developed to assess explosiveness under one set of well-defined conditions. These conditions are more representative of a moderately confined explosive in munitions such as shells but the data generated form a basis for comparing the relative explosiveness of energetic materials. Because the test is relatively cheap and simple to perform it is used routinely in assessing the potential hazards of new explosive formulations.
3. (See also Tube Test – Fast Heating, EMTAP Test No 41, and Tube Test – Electrically Heated, EMTAP Test No 42).

Test

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

Apparatus and Materials

5. The assembled test vehicle is shown schematically, with dimensions, in Figure 1. It consists of a cold drawn seamless mild steel tube of wall thickness 6.0 mm, which is sealed by two mild steel end-caps. One cap has a 2.4 mm diameter hole to accommodate the igniter leads.
6. The explosive is filled at its usual density directly into the test vehicle if possible. The nominal explosive mass is 335 g at a density of 1.80 g/cm³. A 21-mm deep cavity is left at one end of the filling to accommodate the igniter. The exposed surface of the explosive should have a composition representative of the bulk of the filling and be relatively smooth. It may not be possible to press-fill explosives directly into the tube, in which case a single pre-formed pellet, or 5 pellets, each approximately 46.5 mm in length, which are made to adhere to each other by a thin film of compatible cyano-acrylate adhesive, should be used.

Procedure

7. 0.75 g of Bullseye™ or M9 propellant, is weighed out and placed in the centre of a 4 ± 1cm square piece of tissue paper. A single E-Type fuzehead to specification Spec Air 1447 is placed in the centre of the propellant and the tissue drawn around the fuzehead to form a containing bag. The bag is closed around the leads of the fuzehead by means of thin wire. The end-cap with the hole is detached from the filled Tube and the leads to the fuzehead fed through the hole.
8. A further 0.75 g of the propellant is poured into the cavity in the Tube and the end cap, containing the igniter, is re-screwed into position and the complete assembly is weighed.
9. The igniter is fired by means of a low voltage firing pack, with the tube laid horizontally on wooden blocks.

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Annex B to AOP 4491

10. The remains of the assembly are collected, together with any unconsumed explosive. Both items are weighed and recorded as a percentage of their original weights. The total number of steel fragments recovered is recorded and the number of fragments produced by the steel tube representing the body of the assembly.
11. All fragments are photographed before being disposed of. Each photograph should include a legend and a scale for reference purposes.
12. Normally, 10 firings are undertaken for each explosive under test.

Reporting of Results

13. Results are tabulated in the format shown in Table 1. The number of fragments recovered provides the most useful indication of the relative explosiveness of the material under test. In those experiments where less than 100% of the test vehicle is recovered, the number of body fragments recorded in Table 1 may be divided by their fraction of the intact vehicle to give a "normalised number of body fragments". This number is then used to assign a reaction category to the event using the descriptions listed in Table 2. For clarity, the reaction description should be included as well as the category when reporting the results.
14. Typical results for a range of common explosives are summarised in Table 3. A category 0 reaction does not necessarily imply low explosiveness unless adequately supported by other low category results.

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Table 1 – Explosive Composition: Torpex 2 (2.1) ex MK9 Mine No 1765

Filling Method: Open Cast (Re-Cast)

N.B: 1 fragment = item intact, Igniter = 1.5g Ballistite

Round No	Assembly No	Trial date	HE Density (g/cm ³)	Recovered Fragments			% HE Recovered	Degree of Reaction	Comments
				Total No	Body No	% Wt.			
11		09.83	1.72	20	15	90	0	3	
12		"	"	19	14	90	0	3	
13		"	"	15	7	90	0	2	
14		"	"	16	9	75	0	3	
15		"	"	15	7	90	0	2	
16		"	"	14	10	80	0	3	
17		"	"	17	12	90	0	3	
18		"	"	23	14	100	0	3	
19		"	"	18	7	90	0	2	
20		"	"	16	10	90	0	3	

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AOP 4491**

Table 2 – Description of Reaction Categories

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

Table 3 – Typical Internal Ignition Tube Test Results, Achieved with Tube to Drawing EMI/5110/2

Explosive	Degree of Reactions (Number of Tests)	Average Number of Body Fragments (Normalised)
Composition A5		100
Debrix 18AS		23
EDC 1(G)		25
PE4		1
PROX-4		1
TNT		3
Torpex 2		11
Torpex 2B		7
Rowanex 4100		8 from 10 failed to ignite others rapidly quenched, 90% HE unburnt.
Tetryl (NATO standard)		> 200, 11 (two distributions)

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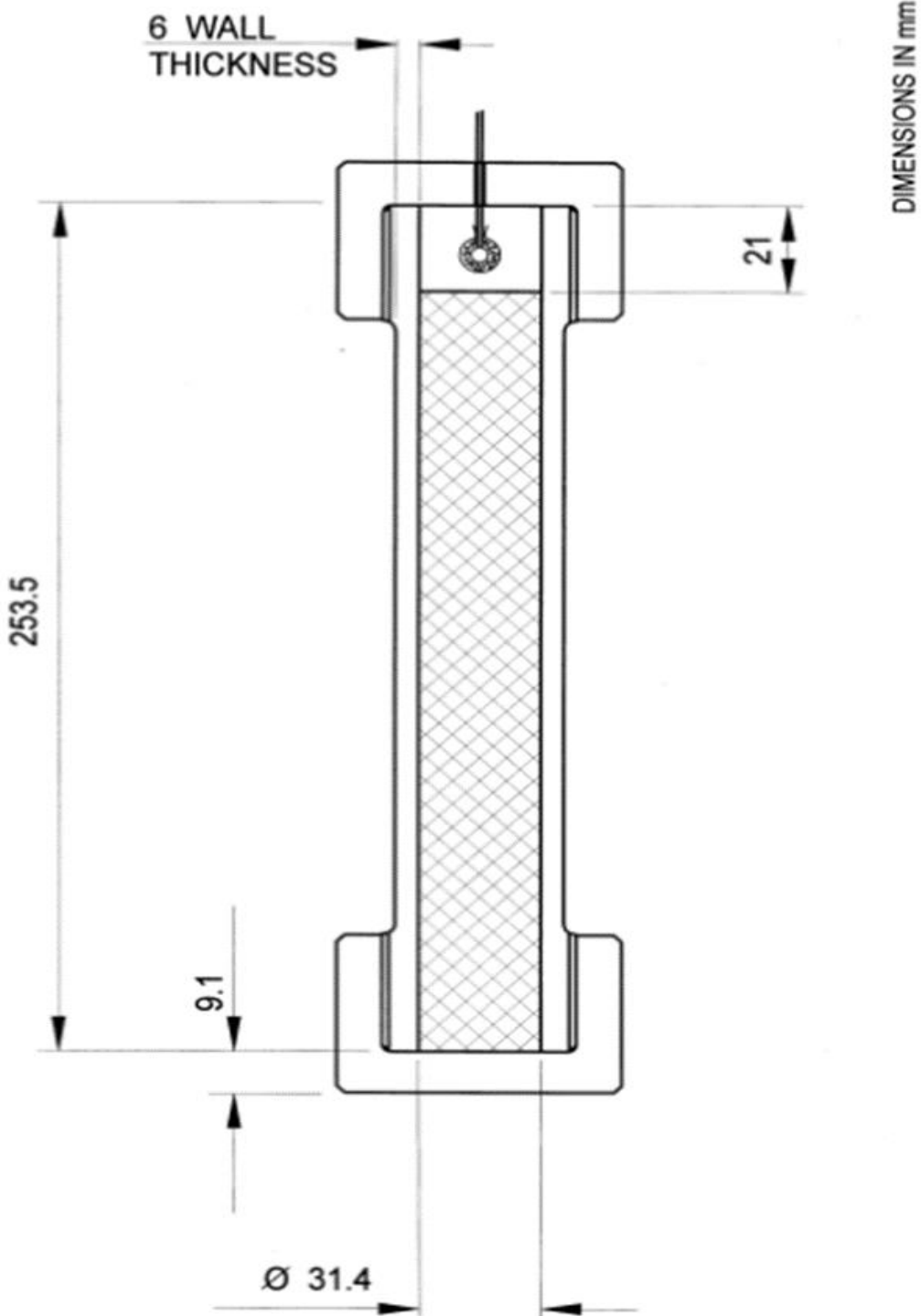


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**Annex B to
AOP 4491**

B5-UK Test 41: TUBE TEST – FAST HEATING

Appreciation

1. The Fast Heating version of the Tube Test assesses the behaviour of energetic material or propellant charges when subjected to rapid external heating. The level of violence of this reaction and its potential to cause damage to the surroundings, is referred to as "explosiveness".
2. Explosiveness is influenced by several variables including the nature of the stimulus, the temperature and physical form of the explosive and its confinement. The Fast Heating Tube Test has been developed to assess explosiveness under one set of well-defined conditions. These conditions are more representative of a moderately confined munition, such as a shell, but the data generated form a basis for comparing relative explosiveness of energetic materials. The test uses the same test vehicle as the Tube Test – Internal Ignition (EMTAP Test No 35), and is often used in assessing new formulations. Results from the Fast Heating Tube Test generally show explosiveness at least as high as those of EMTAP Test No 35.

Test

3. A sample of energetic material, confined in a steel tube, is heated by means of a petrol fire. The degree of fragmentation of the tube is used to assess the relative explosiveness of the material under test.

Apparatus and Materials

4. The assembled test vehicle is shown schematically, with dimensions, in Figure 1. It consists of a cold drawn seamless mild steel tube, of wall thickness 6.0 mm, which is sealed by threaded mild steel end caps.
5. The explosive is filled directly into the test vehicle if possible. The charge weight and filling density should be recorded. The nominal explosive mass is 335 g at a density of 1.80 g/cm³.
6. It may not be possible to press-fill explosives directly into the tube in which case a single preformed pellet, or 6 pellets each approximately 42.00 mm long which are made to adhere to each other by a thin film of compatible adhesive, should be used.
7. It is considered advisable that a means of remote viewing is used while conducting this test.

Procedure

8. The test vehicle is placed over a tray containing 2 litres of petrol.
9. The petrol is ignited by a remote method and the time from ignition to any event(s) is recorded.
10. When all signs of fire have disappeared once it is safe to do so, the remains of the vehicle are collected along with any unreacted explosive. The number of steel body fragments and the total number of fragments are recorded. The body fragments and the unreacted explosive are weighed and recorded as a percentage of their original weights.
11. All fragments are photographed before being disposed of. Each photograph should include a legend and a scale for reference purposes.
12. Normally 10 firings are undertaken for each explosive under test.

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Reporting of Results

13. Results are tabulated in the form shown in Table 1. The number of body fragments provides the most useful indication of the explosiveness of the material under test. In those experiments where less than a 100% of the body is recovered, the body fragment count may be "normalised" by dividing the number of body fragments by the proportion recovered. This number is then used to assign a reaction category to the event using the descriptions listed in Table 2.
14. Typical results for a range of common explosives are summarised in Table 3.

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Table 1: Tube Test – Fast Heating

Explosive Composition: TNT/HNS 95.5/0.5

Filling Method: Open Cast

Round No	Vehicle No	Trial Date	Filling Density (g/cm ³)	Recovered fragment			% Filling Recovered	Reaction category	Time to Event (s)	Comments
				Total No	Body No	% Wt.				
1	1	12.07.89	1.59	111	98	80	0	4	408	Venting prior to event
2	2	"		53	46	80	0	3	367	Venting prior to event
3	3	"		124	108	55	0	4	447	Venting prior to event
4	4	"		52	43	85	0	3	301	Venting prior to event
5	5	"		74	64	95	0	3	315	Venting prior to event
6	6	"		83	77	60	0	4	255	Venting prior to event
7	7	"		58	51	95	0	3	236	Venting prior to event
8	8	"		158	148	75	0	4	357	Venting prior to event
9	9	"		142	136	85	0	4	304	Venting prior to event
10	10	13.07.89		54	44	95	0	3	302	Venting prior to event

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Table 2 – Reaction Categories

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

Table 3 – Typical Results

Composition	Reaction Category (Number of events)	Average Number of Body Fragments	Average Time to Event (s)
HMX/TNT 85/15	3(4) 4(6)	>100	174
HMX/PU 85/15	1(10)	1	171
RDX/WAX8/Al/PIB 71/7/20/2 + 0.5% Carbon Black	1(4) 2(6)	1	163
TNT/HNS 99.5/0.5	3(5) 4(5)	55/>100	329

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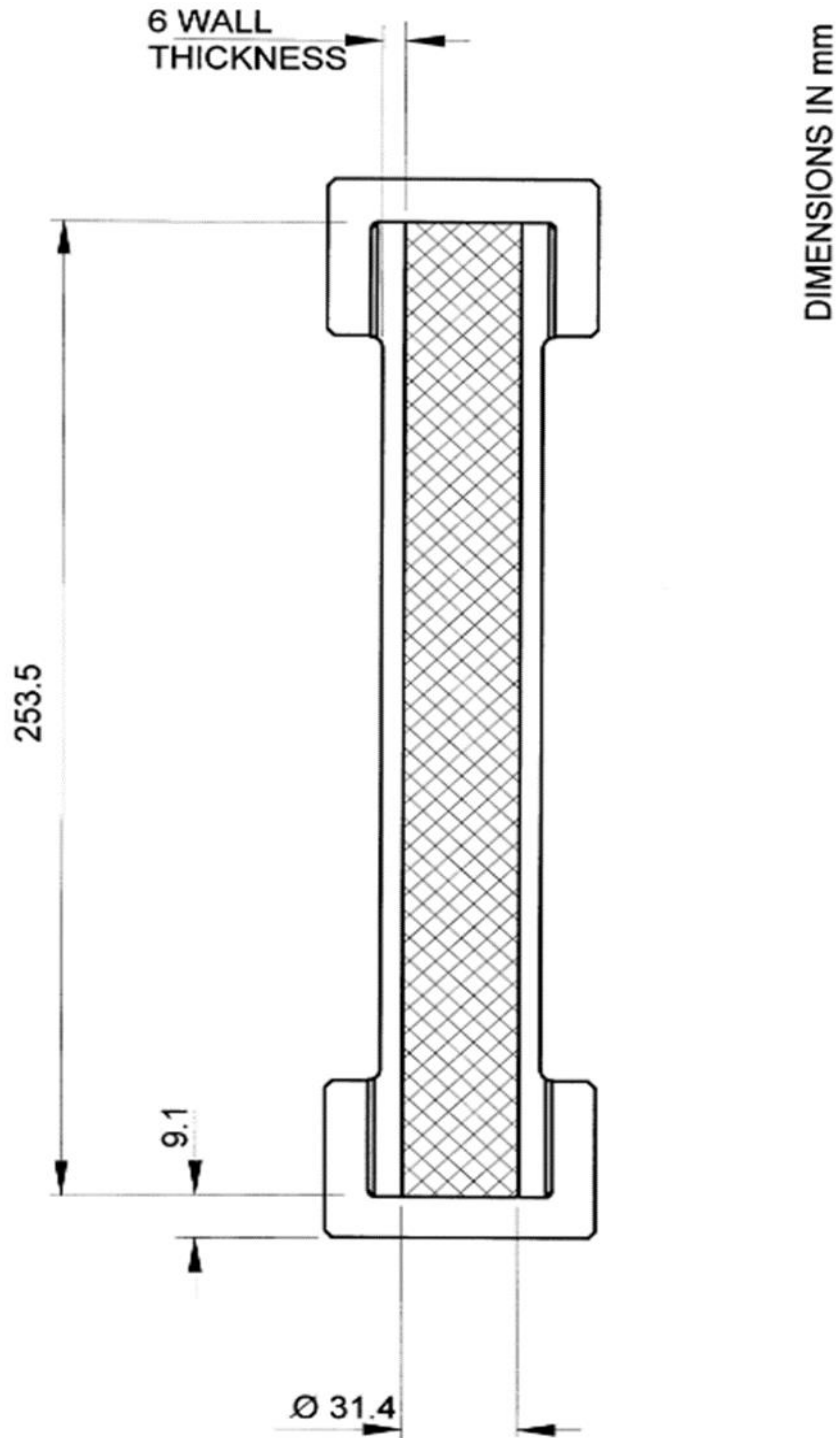


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B6-UK Test 42: TUBE TEST – ELECTRICALLY HEATED

Appreciation

1. The electrically heated version of the tube test assesses the behaviour of energetic materials or propellant charges when confined and subjected to various heating rates that can be as low as 3.3°C per hour.
2. The tube and its contents are heated by means of an electrical heating tape. The heating rate can be varied from one trial to the next by altering the power supplied to the heating jacket. Thermocouples are mounted at the centre of the wall of the tube and monitored to ensure that the heating rate in each trial is constant. The power can be controlled manually, or by the use of an automatic PID controller.
3. One trial is performed with a thermocouple mounted internally in the centre of the charge so that information on the thermal profile within the charge can be obtained throughout the test.
4. This test uses the same tube as is used for EMTAP tests 35 and 41.

Test

5. A sample of energetic material, confined in a steel tube, is heated at controlled rates. The relative explosiveness of the material under these test conditions is assessed from the degree of fragmentation of the tube body.

Apparatus and Materials

6. The test vehicle is shown schematically in Figure 1. It consists of a cold drawn seamless mild steel tube of wall thickness 6.0 mm which is fitted with mild steel end-caps. The nominal explosive mass is 335 g at a density of 1.80 g/m³.

Procedure

7. Each tube has a K-type thermocouple in intimate contact half way along the tube. Two tubes are fitted with a second K-type thermocouple located in one of the end caps and positioned centrally on the long axis of the tube. This is fitted into position before a cast explosive is filled into the tube. Pressed pellets need to be machined to permit subsequent assembly of this more fully instrumented test vehicle.
8. Where possible, cast explosives are filled directly into the test vehicle. It is not possible to press-fill explosives directly into the tube, so 6 pellets, each approximately 42 mm in length, made to adhere to each other by a thin film of compatible adhesive, should be used. The exposed surface of the explosive should have a composition representative of the bulk of the filling and be relatively smooth.
9. The end caps are screwed into position on the loaded tube.
10. A suitable heating tape is wound uniformly around the tube.
11. Five test vehicles are prepared in this manner, two of which are fitted with the additional thermocouple.
12. A suitable container is half-filled with vermiculite insulation and the vehicle is laid horizontally atop the vermiculite and clear of the sides. The vehicle is completely covered with vermiculite. The thermocouple(s) are connected to the data acquisition system. The heater leads are then connected to the power leads.
13. The heater tape is attached to a suitable power supply through a temperature control device e.g. a Proportional, Integration and Differentiation (PID) controller.

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14. Power is applied to the heater tape with the time to explosion being taken from the first application of power to the tape. The test terminates when an explosive event occurs or when the temperature reaches 400 °C, whichever occurs first.
15. Once the test is completed, the power is switched off and the fragments are allowed to cool. Once it is safe to do so, the remains of the assembly are collected, together with any unconsumed explosive. Both are weighed and recorded as a percentage of their original weights. The total number of steel fragments recovered is recorded as is the number of fragments produced by the steel tube representing the body of the assembly.
16. The fragments are photographed before being disposed of. Photographs should include a legend and a scale for reference purposes.
17. Trials should be performed to investigate the range of explosiveness responses exhibited over a wide spectrum of heating rates. Suitable rates could be 10 °C/hour and 1, 5, 10 and 100 °C/minute. One of the trials will involve the use of two thermocouples, usually that involving the slowest heating rate.
18. Once the explosiveness assessment trials have been completed, a further trial is carried out at 5 °C/minute using the second test vehicle fitted with an internal thermocouple.

Reporting of Results

19. The number of fragments recovered provides the most useful indication of the relative explosiveness of the material under test. Every effort should be made to recover as many as possible, but in those experiments where less than 100% of the test vehicle is recovered, the number of body fragments may be divided by their fraction of the intact vehicle to give a "normalised number of body fragments". This number is then used to assign a reaction category to the event using the descriptions listed in Table 1.

Table 1 – Description of Reaction Categories

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

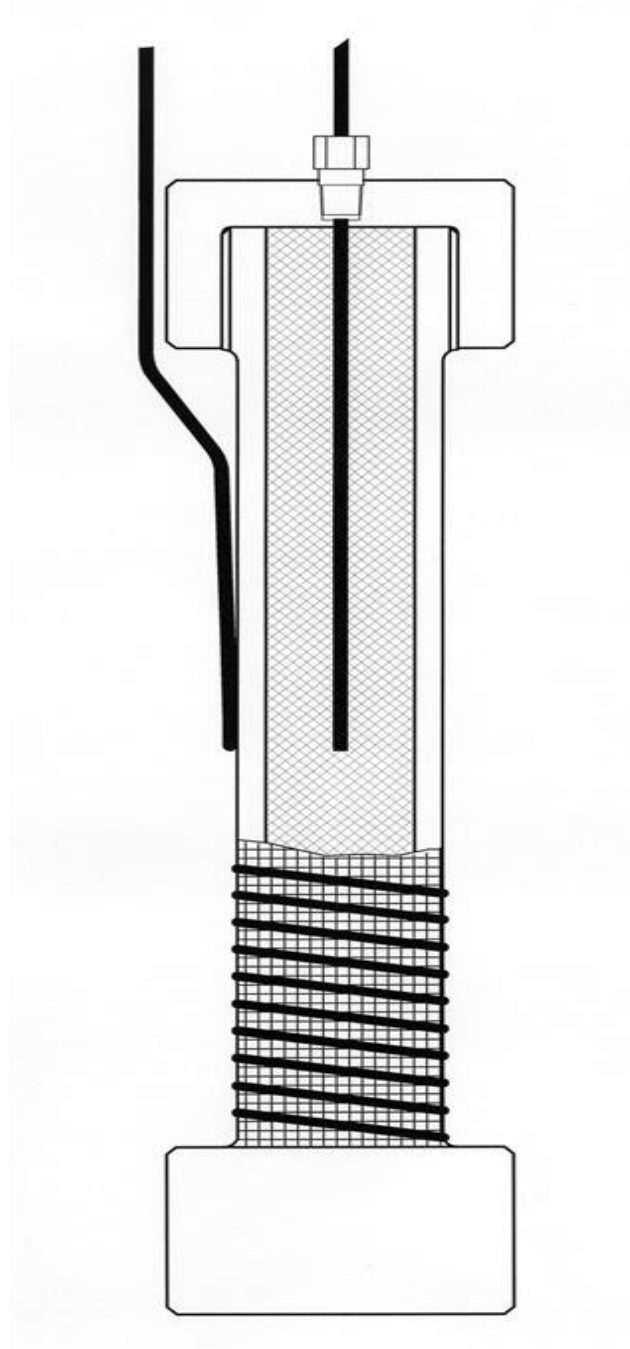
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Electrically heated tube test: Diagrammatic position of thermocouples

Figure 1:
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