Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals

10 April 2014

Sub-Committee of Experts on the Transport of Dangerous Goods

Forty-fifth session
Geneva, 23 June – 2 July 2014
Item 11(i) of the provisional agenda
Issues relating to the Globally Harmonized System of Classification and Labelling of Chemicals: miscellaneous

Sub-Committee of Experts on the Globally Harmonized System of Classification and Labelling of Chemicals

Twenty–seventh session
Geneva, 2–4 July 2014
Item 3 (a) (ii) of the provisional agenda
Classification criteria and related hazard communication:
Work of the Sub-Committee of Experts on the Transport of Dangerous Goods (other relevant issues)

Alignment of the Manual of Tests and Criteria with the GHS: Part I (Sections 16 to 18) of the Manual

Note by the secretariat

1. Reference is made to document ST/SG/AC.10/C.3/2014/61–ST/SG/AC.10/C.4/2014/8.

2. This document contains the suggested amendments to Part I (Sections 16 to 18) of the Manual.

Annex

"~~SECTION~~ CHAPTER 16

TEST SERIES 6

16.1 Introduction

16.1.1 The results from four types of series 6 tests are used to determine which division, amongst Divisions 1.1, 1.2, 1.3 and 1.4, corresponds most closely to the behaviour of a product if a load is involved in a fire resulting from internal or external sources, or an explosion from internal sources (boxes 26, 28, 30, 32 and 33 of Figure 10.3). The results are also necessary to assess whether a product can be assigned to Compatibility Group S of Division 1.4 and whether or not it should be excluded from the class of explosives (transport Class 1) (boxes 35 and 36 of Figure 10.3). The four types of test are:

Type 6 (a): a test on a single package to determine if there is mass explosion of the contents;

Type 6 (b): a test on packages of an explosive substance or mixture or explosive articles, or non-packaged explosive articles, to determine whether an explosion is propagated from one package to another or from a non-packaged article to another;

Type 6 (c): a test on packages of an explosive substance or mixture or explosive articles, or non-packaged explosive articles, to determine whether there is a mass explosion or a hazard from dangerous projections, radiant heat and/or violent burning or any other dangerous effect when involved in a fire; and

Type 6 (d): a test on an unconfined package of explosive articles to which special provision 347 of Chapter 3.3 of the Model Regulations applies, to determine if there are hazardous effects outside the package arising from accidental ignition or initiation of the contents.

16.2 Test methods

16.2.1 The test methods currently used are listed in Table 16.1.

Table 16.1: Test methods for Test Series 6

Test code	Name of Test	Section
6 (a)	Single package test[a]	16.4.1
6 (b)	Stack test[a]	16.5.1
6 (c)	External fire (bonfire) test[a]	16.6.1
6 (d)	Unconfined package test[a]	16.7.1

[a] *Recommended test.*

16.2.2 Test types 6 (a), 6 (b), 6 (c) and 6 (d) are performed in alphabetical order. However, it is not always necessary to conduct tests of all types. Test type 6 (a) may be waived if explosive articles are carried without packaging or when the package contains only one article. Test type 6 (b) may be waived if in each type 6 (a) test:

(a) The exterior of the package is undamaged by internal detonation and/or ignition; or

(b) The contents of the package fail to explode, or explode so feebly as would exclude propagation of the explosive effect from one package to another in test type 6(b).

Test type 6 (c) may be waived if, in a type 6 (b) test, there is practically instantaneous explosion of virtually the total contents of the stack. In such cases the product is assigned to Division 1.1.

Test type 6(d) is a test used to determine whether a 1.4S classification is appropriate and is only used if special provision 347 on Chapter 3.3 of the Model Regulations applies.

The results of test series 6 (c) and 6 (d) indicate if 1.4S is appropriate, otherwise the classification is 1.4 other than S.

16.2.3 If a substance or mixture gives a "—" result (no propagation of detonation) in the Series 1 type (a) test, the 6(a) test with a detonator may be waived. If a substance or mixture gives a "—" result (no or slow deflagration) in a Series 2 type (c) test, the 6 (a) test with an igniter may be waived.

16.2.4 Explanations of certain terms used in the assignment of divisions and compatibility groups are given in the Glossary in Appendix B of the Model Regulations (e.g. mass explosion, pyrotechnic substance, entire load, total contents, explode, explosion of the total contents).

16.3 Test conditions

16.3.1 Tests from series 6 should be applied to packages of explosive substances or mixtures and articles in the condition and form in which they are offered for transport[RG1]. The geometrical arrangement of the products should be realistic in regard to the packing method and the conditions of transport [RG2]and should be such as to produce the most disadvantageous test results. Where explosive articles are to be carried without packaging, the tests should be applied to the non-packaged articles. All types of packaging containing substances, mixtures –or articles should be subjected to the tests unless:

(a) The product, including any packaging, can be unambiguously assigned to a division by a competent authority on the basis of results from other tests or of available information; or

(b) The product, including any packaging, is assigned to Division 1.1.

16.4 Series 6 type (a) test prescription

16.4.1 *Test 6 (a): Single package test*

16.4.1.1 *Introduction*

This is a test on a single package to determine if there is mass explosion of the contents.

16.4.1.2 *Apparatus and materials*

The following items are required:

(a) A detonator to initiate the substance, mixture or article;

(b) An igniter just sufficient to ensure ignition of the substance, mixture or article;

(c) Suitable confining materials; and

(d) A sheet of 3.0 mm thick mild steel to act as a witness plate.

Blast measuring equipment may be used.

16.4.1.3 *Procedure*

16.4.1.3.1 The test is applied to packages of explosive substances, mixtures and articles in the condition and form in which they are offered for transport[RG3]. Where explosive articles are to be carried without packaging, the tests should be applied to the non-packaged articles. The decision to use either an initiating stimulus or an igniting stimulus is based on the following considerations.

16.4.1.3.2 For packaged substances~~:~~ or mixtures

(a) If the substance or mixture is intended to function by detonation, it should be tested with a standard detonator (Appendix 1);

(b) If the substance or mixture is intended to function by deflagration, it should be tested with an igniter just sufficient (but not more than 30 g of black powder) to ensure ignition of the substance within the package. The igniter should be located in the center of the substance or mixture in the package;

(c) Substances and mixtures not intended for use as explosive, but provisionally accepted into the class of explosives (transport Class 1), should be tested first with a standard detonator (Appendix 1) and, if no explosion occurs, with an igniter as in (b) above. If a substance or mixture gives a "—" result (no propagation of detonation) in the Series 1 type (a) test, the test with a detonator may be waived and if a substance or mixture gives a "—" result (no or slow deflagration) in a Series 2 type (c) test, the test with an igniter may be waived.

16.4.1.3.3 For packaged articles[1]:

(a) Articles provided with their own means of initiation or ignition:

The functioning of an article near the center of the package is stimulated by the article's own means of initiation or ignition. Where this is impracticable, the article's own means of initiation or ignition is replaced by another form of stimulus having the required effect;

(b) Articles not provided with their own means of initiation or ignition:

(i) An article near the centre of the package is caused to function in the designed mode; or

(ii) An article near the centre of the package is replaced by another article which can be caused to function with the same effect.

16.4.1.3.4 The package is placed on a steel witness plate on the ground. The preferred method of confinement consists of containers, similar in shape and size to the test package, completely filled with earth or sand and placed as closely as possible around the test package to a minimum thickness of confinement, in every direction of 0.5 m for a package not exceeding 0.15 m^3 and 1.0 m for a package greater than 0.15 m^3. Alternative methods of confinement are to use boxes or bags filled with earth or sand placed around and on top of the package or to use loose sand.

16.4.1.3.5 The substance, mixture or article should be initiated and observations made on the following: evidence of thermal effects, projection effects, detonation, deflagration or explosion of the total contents of the package. ***A safe waiting period, prescribed by the test agency, should be observed after initiation.*** The test should be performed three times unless a decisive result occurs earlier (e.g. explosion of the total contents). If the results of the recommended number of tests do not enable unambiguous interpretation of the results, the number of tests should be increased.

16.4.1.4 *Test criteria and method of assessing results*

Mass explosion (see definition in Chapter 2.1, paragraph 2.1.1.4 (a) of the Model Regulations and Chapter 2.1, paragraph 2.1.2.1 (a) of the GHS) indicates a candidate for Division 1.1. Evidence of such an indication includes:

(a) A crater at the test site;

(b) Damage to the witness plate beneath the package;

(c) Measurement of a blast; and

(d) Disruption and scattering of the confining material.

[1] *Subject to the proviso that in the case of articles containing a very small quantity of substance(s) of compatibility group A only, a sufficient number of such items should be initiated simultaneously to cause not less than 0.2 g of primary explosive to explode.*

If the product is accepted into Division 1.1, further testing is not necessary; otherwise proceed to a test of type 6 (b).

16.4.1.5 *Examples of results*

Substance	Packaging	Initiation system	Events	Result
Ammonium perchlorate (12 µm)	10 kg fibreboard drum	Detonator	Detonation	Candidate Division 1.1
Musk xylene	50 kg fibreboard drum	Detonator	Localised decomposition	Not Division 1.1
Musk xylene	50 kg fibreboard drum	Igniter	Localised decomposition	Not Division 1.1
Single base propellant (non-porous)	60 litre fibreboard drum	Igniter	No explosion	Not Division 1.1
Single base propellant (porous)	60 litre fibreboard drum	Igniter	Explosion	Candidate Division 1.1

16.5 Series 6 type (b) test prescription

16.5.1 *Test 6 (b): Stack test*

16.5.1.1 *Introduction*

This is a test on packages of an explosive substance or mixture or explosive articles, or unpackaged explosive articles, to determine whether an explosion is propagated from one package to another or from a unpackaged article to another.

16.5.1.2 *Apparatus and materials*

The following items are required:

(a) A detonator to initiate the substance, mixture or article;

(b) An igniter just sufficient to ensure ignition of the substance, mixture or article;

(c) Suitable confining materials; and

(d) A sheet of 3.0 mm thick mild steel to act as a witness plate.

Blast measuring equipment may be used.

16.5.1.3 *Procedure*

The test is applied to a stack of packages of an explosive product or a stack of unpackaged articles, in each case, in the condition and form in which they are offered for transport[RG4]. Where explosive articles are to be carried without packaging, the tests should be applied to the unpackaged articles. Sufficient packages or articles to give a total volume of 0.15 m^3 are stacked on a steel witness plate on the ground. If the volume of an individual package (or unpackaged article) exceeds 0.15 m^3, then the test is performed with at least one acceptor placed in the position most likely to result in communication between the individual products (see 16.3.1). If this position is not known, several acceptors are used. The preferred method of confinement consists of containers, similar in shape and size to the test packages completely filled with earth or sand and placed as closely as possible around the test package to create a minimum thickness of confinement, in every direction, of 1 m. Alternative methods of confinement are to use boxes or bags filled with earth or sand placed around and on top of the stack or to use loose sand. If loose sand is used for confinement, the stack should be covered or protected to ensure that no sand falls into the interstices between adjacent packages or non-packaged articles. Articles carried without packaging are confined in a manner analogous to that used for packaged articles. The decision to use either an initiating stimulus or an igniting stimulus is based on the following considerations.

16.5.1.4 For packaged substances and mixtures:

(a) If the substance or mixture is intended to function by detonation, it should be tested with a standard detonator (Appendix 1);

(b) If the substance or mixture is intended to function by deflagration, it should be tested with an igniter just sufficient (but not more than 30 g of black powder) to ensure ignition of the substance or mixture within one individual package. The igniter should be located in the centre of the substance or mixture in the package;

(c) Substances or mixtures not intended for use as explosives, but provisionally accepted into the class of explosives (transport Class 1), should be tested using whichever initiation system gave a "+" result in a type 6 (a) test.

16.5.1.5 For packaged articles and unpackaged articles[2]:

(a) Articles provided with their own means of initiation or ignition.

The functioning of an article at the centre of the package near the centre of the stack should be stimulated by the article's own means of initiation or ignition. Where this is impracticable, the article's own means of initiation or ignition may be replaced by another form of stimulus having the required effect;

(b) Articles not provided with their own means of initiation or ignition:

(i) An article at the centre of the package near the centre of the stack is caused to function in the designed mode; or

(ii) An article at the centre of the package near the centre of the stack is replaced by another article which can be caused to function with the same effect.

16.5.1.6 The point of ignition or initiation should be situated in a package near the centre of the stack. Articles carried without packaging are tested in a manner analogous to that used for packaged articles.

16.5.1.7 The substance, mixture or article should be initiated and observations made on the following: evidence of thermal effects, projection effects, detonation, deflagration or explosion of the total contents of the package. ***A safe waiting period, prescribed by the test agency, should be observed after initiation.*** The test should be performed three times unless a decisive result occurs earlier (i.e. explosion of the total contents). If the results of the recommended number of tests do not enable unambiguous interpretation of the results, the number of tests should be increased.

16.5.1.8 *Test criteria and method of assessing results*

If in test 6 (b) explosion of the contents of more than one package or unpackaged article occurs practically instantaneously, then the product is assigned to Division 1.1. Evidence of such an occurrence includes:

(a) A crater at the test site appreciably larger than that given by a single package or unpackaged article;

(b) Damage to the witness plate beneath the stack which is appreciably greater than that from a single package or unpackaged article;

[2] *Subject to the proviso that in the case of articles containing a very small quantity of substance(s) of compatibility group A only, a sufficient number of such items are initiated simultaneously to cause not less than 0.2 g of primary explosive to explode.*

(c) Measurement of blast which significantly exceeds that from a single package or unpackaged article; and

(d) Violent disruption and scattering of most of the confining material.

Otherwise proceed to a test of type 6 (c).

16.5.1.9 *Examples of results*

No examples of results are given as these are too specific to the packaging or article tested.

16.6 Series 6 type (c) test prescription

16.6.1 *Test 6 (c): External fire (bonfire) test*

16.6.1.1 *Introduction*

This is a test performed on packages of an explosive substance or mixture or explosive articles, or unpackaged explosive articles, to determine whether there is a mass explosion or a hazard from dangerous projections, radiant heat and/or violent burning or any other dangerous effect when involved in a fire.

16.6.1.2 *Apparatus and materials*

The following items are needed:

(a) If the volume of the package of substance, mixture or articles, or unpackaged article, is less than 0.05 m^3, sufficient packages or unpackaged articles to give a total volume of not less than 0.15 m^3;

(b) If the volume of the package of substance, mixture or articles, or unpackaged article, is equal to or greater than 0.05 m^3, three packages or unpackaged articles. If the volume of one package or unpackaged article is greater than 0.15 m^3, the competent authority may waive the requirement for three packages or unpackaged articles to be tested;

(c) A metal grid to support the products above the fuel and allow adequate heating. If a wooden crib fire is used, the grid should be 1.0 m above the ground and if a liquid hydrocarbon pool fire is used then the grid should be 0.5 m above the ground;

(d) Strapping or wire, if necessary, to hold the packages or unpackaged articles together on the support grid;

(e) Enough fuel to keep a fire burning for at least 30 minutes or, if necessary, until the substance, mixture or article has clearly had sufficient time to react to the fire (see 16.6.1.3.8);

(f) Suitable means of ignition to ignite the fuel on at least two sides e.g. for a wood fire, kerosene to soak the wood and pyrotechnic igniters with wood wool;

(g) Three 2 000 mm × 2 000 mm × 2 mm 1100-0 aluminium sheets (Brinell Hardness 23, tensile strength 90 MPa), or equivalent, to act as witness screens together with suitable supports to hold them vertically. The witness screens shall be mounted rigidly to their frames. When more than one panel is used to make a witness screen, each panel shall be supported at all joints;

(h) Cine or video cameras, preferably high speed and normal speed, to record events in colour.

Blast gauges, radiometers and associated recording equipment may also be used.

16.6.1.3 *Procedure*

16.6.1.3.1 The required number of packages or unpackaged articles, in the condition and form in which they are offered for transport[RG5], are arranged as close as possible to one another on the metal grid. Packages should be oriented in such a way that a maximum probability for projections hitting the witness screens is obtained. If necessary, the packages or unpackaged articles may be encircled with a steel strip to support them during the test. Fuel is placed beneath the grid so that the fire will engulf the packages or unpackaged articles. Precautions against side winds may be required to avoid dissipation of the heat. Suitable methods of heating include a wood fire using a lattice of wooden laths, a liquid or gas fuel fire, that produces a flame temperature of at least 800 °C.

16.6.1.3.2 One method is to use a wood fire which has a balanced air/fuel ratio, thereby avoiding too much smoke which would obscure the events, and which burns with sufficient intensity and duration to bring many kinds of packaged explosives to reaction in 10 to 30 minutes. A suitable method involves using air-dried pieces of wood (approximately 50 mm square section), stacked to form a lattice beneath the grid (1 m off the ground), and up to the base of the grid supporting the packages or unpackaged articles. The wooden laths should extend beyond the packages or unpackaged articles to a distance of at least 1.0 m in every horizontal direction and the lateral distance between the laths should be about 100 mm.

16.6.1.3.3 A receptacle filled with suitable liquid fuel, a combination of both wood and liquid fuel fire may be used as an alternative to the wood fire providing it is as severe. If a liquid pool fire is used, the receptacle should extend beyond the packages or unpackaged articles to a distance of at least 1.0 m in every direction. The distance between the grid platform and the receptacle should be approximately 0.5 m. Before using this method, consideration should be given to whether any quenching action or adverse interaction between explosives and liquid fuel can occur such as might bring the results into question.

16.6.1.3.4 If gas is to be used as a fuel, the burning area must extend beyond the packages or unpackaged articles to a distance of at least 1.0 m in every direction. The gas must be supplied in such a manner to ensure that the fire is evenly distributed around the packages. The gas reservoir should be large enough to keep the fire burning for at least 30 minutes. Ignition of the gas may be accomplished either by remotely ignited pyrotechnics or by remote release of the gas adjacent to a pre-existing source of ignition.

16.6.1.3.5 The vertical witness screens are erected in each of three quadrants at a distance of 4 m from the edge of the packages or unpackaged articles. The downwind quadrant is not used for screens because prolonged exposure to flames may change the resistance of the aluminium sheets to projections. The sheets should be placed so that the centres are level with the centre of the packages or unpackaged articles or, if this is less than 1.0 m above the ground, in contact with the ground. If there are any perforations or indentations in the witness screens before the test, they should be marked so that they can be clearly distinguished from those created during the test.

16.6.1.3.6 The ignition system should be put into place and the fuel ignited on two sides, one the upwind side, simultaneously. The test should not be performed under conditions where the wind speed exceeds 6 m/s. ***A safe waiting period, prescribed by the test agency, should be observed after the fire has extinguished.***

16.6.1.3.7 Observations are made on the following:

(a) Evidence of explosion;

(b) Potentially hazardous projections; and

(c) Thermal effects.

16.6.1.3.8 The test is normally performed once only but if the wood or other fuel used for the fire is all consumed leaving a significant quantity of unconsumed explosive substance or mixture in the remains or in the vicinity of the fire, then the test should be performed again using more fuel, or a different method, to increase the intensity and/or duration of the fire. If the result of the test does not enable the hazard division to be determined, a further test should be performed.

16.6.1.4 *Test criteria and method of assessing results*

16.6.1.4.1 The following criteria are used to answer the questions in Figure 10.3 (boxes 26, 28, 30, 32, 33, 35 and 36) in order to assess the results and classify the product.

16.6.1.4.2 If mass explosion occurs then the product is assigned to Division 1.1. A mass explosion is considered to occur if a substantial proportion explodes so that the practical hazard should be assessed by assuming simultaneous explosion of the whole of the explosive content of the packages or unpackaged articles.

16.6.1.4.3 If a mass explosion does not occur but any one of the following occurs:

(a) A perforation of any of the witness screens (see 16.6.1.3.5);

(b) A metallic projection with a kinetic energy exceeding 20 J as assessed by the distance - mass relation given in Figure 16.6.1.1;

then the product is assigned to Division 1.2.

16.6.1.4.4 If none of the events occur which would require the product to be assigned to Division 1.1 or 1.2 but any one of the following events occurs:

(a) a fireball or jet of flame which extends beyond any of the witness screens;

(b) a fiery projection emanating from the product is thrown more than 15 m from the edge of the packages or unpackaged articles;

(c) a burning time of the product measured to be less than 35 seconds for 100 kg net explosive mass (see 16.6.1.4.8 Notes for scaling time measurements in evaluating thermal flux effects). Alternatively, in the case of articles and low energy substances, the irradiance of the burning product exceeds that of the fire by more than 4 kW/m^2 at a distance of 15 m from the edge of the packages or unpackaged articles. The irradiance is measured over 5 seconds, during the period of maximum output;

then the product is assigned to Division 1.3.

16.6.1.4.5 If none of the events occur which would require the product to be assigned to Division 1.1, 1.2 or 1.3, but any one of the following events occurs:

(a) a fireball or jet of flame which extends more than 1 m from the flames of the fire;

(b) a fiery projection emanating from the product is thrown more than 5 m from the edge of the packages or unpackaged articles;

(c) an indentation in any of the witness screens of more than 4 mm;

(d) a metallic projection with a kinetic energy exceeding 8 J as assessed by the distance - mass relation given in Figure 16.6.1.1;

(e) a burning time of the product measured to be less than 330 seconds for 100 kg net explosive mass (see 16.6.1.4.8: Notes for scaling time measurements in evaluating thermal flux effects);

then the product is assigned to Division 1.4 and to a compatibility group other than Compatibility Group S.

16.6.1.4.6 If none of the events occur which would require the product to be assigned to Division 1.1, 1.2, 1.3 or 1.4 other than Compatibility Group S, the thermal, blast, or projection effects would not significantly hinder fire-fighting or other emergency response efforts in the immediate vicinity, and if hazardous effects are confined within the package, then the product is assigned to Division 1.4 Compatibility Group S.

16.6.1.4.7 If there are no hazardous effects at all, the product is considered for exclusion from the class of explosives (transport Class 1). The possibilities, as shown by boxes 35 and 36 in Figure 10.3, are:

(a) If the product is an article manufactured with a view to producing a practical explosive or pyrotechnic effect, then:

(i) If there is some effect (projection, fire, smoke, heat or loud noise) external to the device itself, the device is not excluded from the class of explosives (transport Class 1) and the product, as packaged, is assigned to Division 1.4 and to Compatibility Group S. Paragraphs 2.1.1.1 (b) of the Model Regulations and 2.1.1.2 (b) of the GHS refers explicitly to the device, rather than the package, so it is usually necessary to make this assessment on the basis of a test involving functioning of the device without packaging or confinement. Sometimes the stated effects are observed in test 6 (c), in which case the product is classified 1.4 S without further tests;

(ii) If there is no effect (projection, fire, smoke, heat or loud noise) external to the device itself, the unpackaged device is excluded from the class of explosives (transport Class 1) in accordance with paragraph 2.1.1.1 (b) of the Model Regulations and 2.1.1.2 (b) of the GHS. ~~Paragraph 2.1.1.1 (b) of the Model Regulations~~These paragraphs refers explicitly to the device, rather than the package, so it is usually necessary to make this assessment on the basis of a test involving functioning of the device without packaging or confinement;

(b) If the product is not manufactured with a view to producing a practical explosive or pyrotechnic effect, it is excluded from the class of explosives (transport Class 1) in accordance with paragraph 2.1.1.1 of the Model Regulations and 2.1.1.2 of the GHS.

16.6.1.4.8 Notes for scaling time measurements in evaluating thermal flux effects

Notes:

(1) The value of 35 seconds/100 kg (see 16.6.1.4.4 (c)) correlates to an average thermal flux of 4 kW/m^2 at 15 m and is based on an assumed heat of combustion of 12 500 J/g. If the true heat of combustion is significantly different, the burning time of 35 s may be corrected; for example, a true heat of combustion of 8 372 J/g burning for (8 372/12 500) × 35 s = 23.4 s would produce the same flux level. Corrections for masses other than 100 kg are made according to the scaling relations and examples in Table 16.2.

(2) The value of 330 seconds/100 kg (see 16.6.1.4.5(e)) correlates to an average thermal flux of 4 kW/m^2 at 5 m and is based on an assumed heat of combustion of 12 500 J/g. If the true heat of combustion is significantly different, the burning time of 330 s may be corrected; for example, a true heat of combustion of 8 372 J/g burning for (8 372/12 500) × 330 s = 221 s would produce the same flux level. Corrections for masses other than 100 kg are made according to the scaling relations and examples in Table 16.2.

(3) In some burn time trials, individual packages or articles will be observed to burn in separate, identifiable events; in such cases, the burning times and masses of each separate event should be used.

Table 16.2: Comparative thermal flux values for varying masses

	1.3/1.4		1.4/1.4S	
Mass (kg)	**Flux (15 m)**	**Burn time (s)**	**Flux (5 m)**	**Burn time (s)**
20	1.36 kW/m^2	21.7	1.36 kW/m^2	195
50	2.5	29.6	2.5	266
100	4	35	4	330
200	6.3	46.3	6.3	419
500	11.7	63.3	11.7	569

NOTE: Thermal flux is scaled on basis of $(m/m_0)^{2/3}$.
Times are scaled on basis of $(m/m_0)^{1/3}$.

The values of thermal flux can be calculated from the equation:

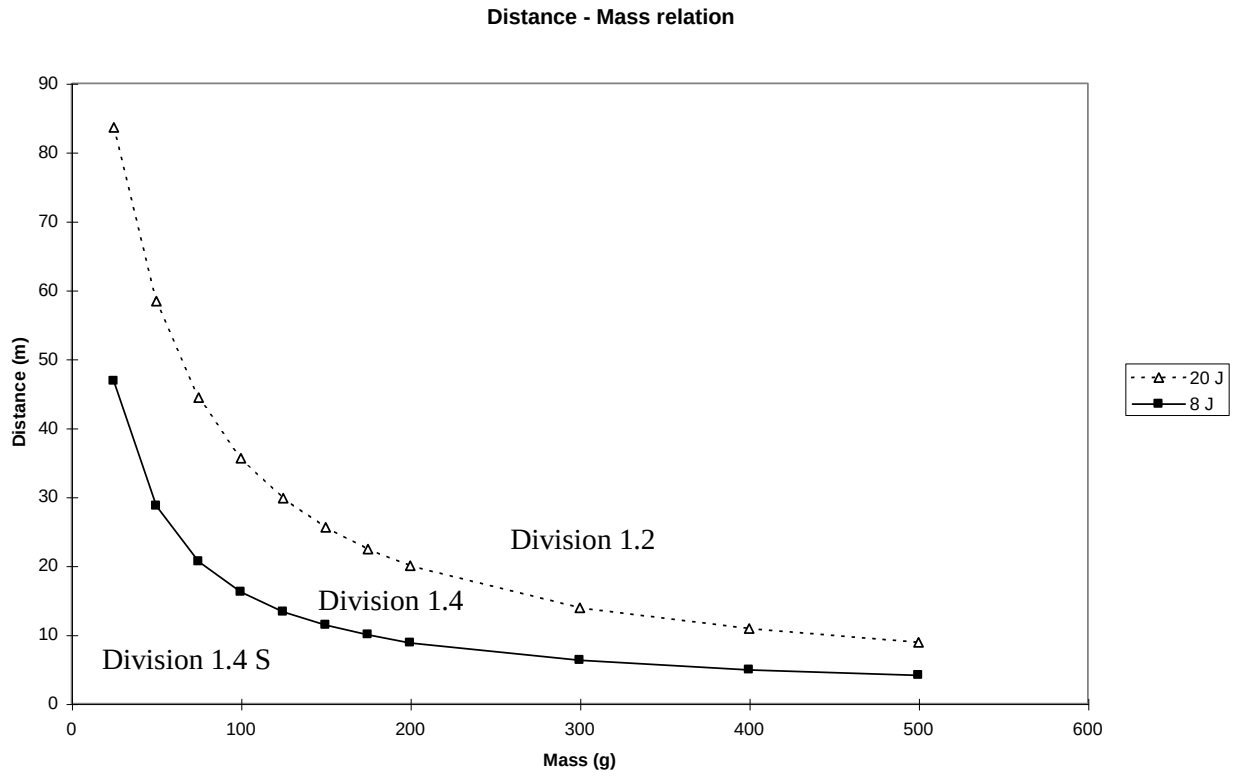
$$F = \frac{C \times E}{4\pi R^2 t}$$

where:

F = thermal flux in kW/m^2;
C = constant = 0.33;
E = total energy content in joules;
R = distance from fire to exposed position in meters;
T = observed burn time in seconds.

16.6.1.5 *Examples of results*

Substance	Packaging	Events	Result
Musk xylene	3 × 50 kg fibreboard drums	Slow burning only	Not an explosive (Not transport Class 1)



Mass	Projection distance (m)	
(g)	20 J	8 J
25	83.6	46.8
50	58.4	28.7
75	44.4	20.6
100	35.6	16.2
125	29.8	13.3
150	25.6	11.4
175	22.43	10
200	20	8.8
300	13.9	6.3
400	10.9	4.9
500	8.9	4.1

Example data for metallic projections with a kinetic energy of 20 J and 8 J

FIGURE 16.6.1.1: Distance-mass relation for metallic projections with a kinetic energy of 20 J and 8 J [3]

[3] *The data presented in Figure 16.6.1.1 is based upon metallic projections. Non-metallic projections will produce different results and may be hazardous. Hazards from non-metallic projections should also be considered.*

16.7 Series 6 type (d) test prescription

16.7.1 *Test 6 (d): Unconfined package test*

16.7.1.1 *Introduction*

This is a test on a single package to determine if there are hazardous effects outside the package arising from accidental ignition or initiation of the contents.

16.7.1.2 *Apparatus and materials*

The following items are required:

(a) A detonator to initiate the article; or

(b) An igniter just sufficient to ensure ignition of the article; and

(c) A sheet of 3.0 mm thick mild steel to act as a witness plate.

Video equipment may be used.

16.7.1.3 *Procedure*

16.7.1.3.1 The test is applied to packages of explosive articles in the condition and form in which they are offered for transport[RG6]. Where explosive articles are to be carried without packaging, the tests should be applied to the non-packaged articles. The decision to use either an initiating stimulus or an igniting stimulus is based on the following considerations.

16.7.1.3.2 For packaged articles:

(a) Articles provided with their own means of initiation or ignition:

The functioning of an article near the centre of the package is stimulated by the article's own means of initiation of ignition. Where this is impracticable, the article's own means of initiation or ignition is replaced by another form of stimulus having the required effect;

(b) Articles not provided with their own means of initiation or ignition:

(i) an article near the centre of the package is caused to function in the designed mode; or

(ii) an article near the centre of the package is replaced by another article which can be caused to function with the same effect.

16.7.1.3.3 The package is placed on a steel witness plate on the ground without confinement.

16.7.1.3.4 The donor article should be initiated and observations made on the following: denting or perforation of the witness plate beneath the package, a flash or flame capable of igniting an adjacent material, disruption of the package causing projection of the explosives contents; or full perforation of the packaging by a projection. **A safe waiting period, prescribed by the test agency, should be observed after initiation.** The test should be performed three times, in different orientations, unless a decisive result is observed earlier. If the results of the recommended number of tests do not enable unambiguous interpretation of the results, the number of tests should be increased.

16.7.1.4 *Test criteria and method of assessing the results*

Inclusion in Compatibility Group S requires that any hazardous effects arising from functioning of the articles in this test are confined within the package. Evidence of a hazardous effect outside the package includes:

(a) Denting or perforation of the witness plate beneath the package;

(b) A flash or flame capable of igniting an adjacent material such as a sheet of 80 ± 3 g/m² paper at a distance of 25 cm from the package;

(c) Disruption of the package causing projection of the explosives contents; or

(d) A projection which passes completely through the packaging (a projection or fragment retained or stuck in the wall of the packaging is considered as non hazardous).

The competent authority may wish to take into account the expected effect of the initiator when assessing the results of the test, if these are expected to be significant when compared to the articles being tested. If there are hazardous effects outside the package, then the product is excluded from Compatibility Group S.

16.7.1.5 *Examples of results*

Article	**Packaging**	**Initiation system**	**Events**	**Result**
Cartridges, power device	Fibreboard box containing 20 articles (300 g of propellant each) each in a plastic bag	One of the articles	Articles ignited one by one, producing flames up to 2 m high outside package	Not consistent with Compatibility Group S
Detonator assemblies, non-electric	Fibreboard box containing 60 assemblies each in a plastic bag with its shock tube coiled in a figure 8, with attenuators on the detonators	One of the articles	One out of 60 detonators fired and no visible effects outside the box.	Consistent with Compatibility Group S
Detonators, electric	Fibreboard box containing 84 assemblies, each bundled with its wire so that the blast from a firing detonator would be attenuated	One of the articles	One out of 84 detonators fired. The reaction caused the box to break open and released some of the assemblies but it was judged that there were no hazardous effects outside the package.	Consistent with Compatibility Group S
Charges, shaped (open-face 19 g perforators)	Fibreboard box containing 50 charges in two layers so that pairs of charges were focused toward each other	Detonator with approxim-ately 60 mm of detonating cord	Three trials were conducted. In each of the trials, the witness plate was perforated with three to four charges reacting. The packages were blown apart scattering the remaining charges over a wide area.	Not consistent with Compatibility Group S
Detonators, electric	Fibreboard box containing 50 detonators each with a 450 mm lead wire. Each assembly was contained in its own fibreboard inner box. The boxes were separated by fibreboard panels	One of the articles	One out of 50 detonators fired causing one of the box flaps to open. There were no hazardous effects outside of the package.	Consistent with Compatibility Group S

~~SECTION~~ CHAPTER 17

TEST SERIES 7

17.1 Introduction

The question "Is it an extremely insensitive explosive article?" (box 40 of Figure 10.3) is answered by series 7 tests and any candidate for Division 1.6 should pass one of each of the ~~ten~~ eleven types of test comprising the series. The first six types of test (7(a) to 7(f)) are used to establish if a substance is an Extremely Insensitive ~~Detonating~~ Substance (EI~~D~~S) and the remaining ~~four~~ five types of test (7 (g), 7 (h), 7 (j), ~~and~~ 7(k) and 7(l)) are used to determine if an article containing an EI~~D~~S(s) may be assigned to Division 1.6. The ~~ten~~ eleven test types are:

Type 7 (a): a shock test to determine sensitivity to intense mechanical stimulus;
Type 7 (b): a shock test with a defined booster and confinement to determine sensitivity to shock;
Type 7 (c): a test to determine the sensitivity of the explosive substance or mixture to deterioration under the effect of an impact;
Type 7 (d): a test to determine the degree of reaction of the explosive substance or mixture to impact or penetration resulting from a given energy source;
Type 7 (e): a test to determine the reaction of the explosive substance or mixture to an external fire when the material is confined;
Type 7 (f): a test to determine the reaction of the explosive substance or mixture in an environment in which the temperature is gradually increased to 365 °C;
Type 7 (g): a test to determine the reaction to an external fire of an article which is in the condition as presented for transport[RG7];
Type 7 (h): a test to determine the reaction of an article in an environment in which the temperature is gradually increased to 365 °C;
Type 7 (j): a test to determine the reaction of an article to impact or penetration resulting from a given energy source; ~~and~~
Type 7 (k): a test to determine whether a detonation of an article will initiate a detonation in an adjacent, like article~~;~~. And
Type 7 (l): a test to determine the sensitivity of the article to shock directed at vulnerable components.

The question in box 40 is answered "no" if a "+" result is obtained in any series 7 test.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.2 Test methods

The test methods currently used are listed in Table 17.1.

Table 17.1: Test methods for Test Series 7

Test code	Name of Test	Section
Tests on substances		
7 (a)	EI~~D~~S cap test[a]	17.4.1
7 (b)	EI~~D~~S cap test[a]	17.5.1
7 (c) (i)	Susan test	17.6.1
7 (c) (ii)	Friability test[a]	17.6.2
7 (d) (i)	EI~~D~~S bullet impact test[a]	17.7.1
7 (d) (ii)	Friability test	17.7.2
7 (e)	EI~~D~~S external fire test[a]	17.8.1
7 (f)	EI~~D~~S slow cook-off test[a]	17.9.1
Tests on articles		
7 (g)	1.6 article external fire test[a]	17.10.1

Test code	Name of Test	Section
7 (h)	1.6 article slow cook-off test[a]	17.11.1
7 (j)	1.6 article bullet impact test[a]	17.12.1
7 (k)	1.6 article stack test[a]	17.13.2
7 (l)	1.6 article fragment impact test	17.14.1

[a] *Recommended test.*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.3 Test conditions

17.3.1 All explosive components must always be present in articles during Series 7 testing of types 7 (g) to 7 (l). Smaller explosive components containing substances not subjected to tests of type 7 (a) to 7 (f) shall be specifically targeted in tests 7 (j) and 7 (l) when it is assessed that they will cause the most severe reaction from the test article, to ensure the probability of accidental initiation or propagation of a Division 1.6 article remains negligible.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.3.~~1~~2 A substance or mixture intended for use as ~~the~~ a main explosive load in an article of Division 1.6 should be tested in accordance with Test Series 3 and 7. A substance or mixture intended for use as a larger (dimensionally) boostering component in an article of Division 1.6, where the volumetric size limit relative to the main explosive load it is boostering is met, should be tested in accordance with Test Series 3 and tests of type 7 (c) (ii) and 7 (e). Test Series 7 should be conducted on the substance or mixture in the form (i.e. composition, granulation, density etc.) in which it is to be used in the article.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.3.~~2~~3 An article being considered for inclusion in Division 1.6 should not undergo Series 7 testing until after ~~its~~ main explosive load and certain boostering component substances ~~has~~ have undergone appropriate tests of type 7 (a) to 7 (f) to determine whether they meet the substance or mixture requirements for Division 1.6. Guidance on substance or mixture testing determination process is given under section 10.4.3.6.~~it is an EIDS~~.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.3.~~3~~4 Tests of types 7 (g), 7 (h), 7 (j), ~~and~~ 7 (k) and 7 (l) should be performed to determine if an article with an EI~~D~~S main load(s) and appropriately insensitive boostering component may be assigned to Division 1.6. These tests are applied to articles in the condition and form in which they are offered for transport[RG8], except that non-explosive components may be omitted or simulated if the competent authority is satisfied that this does not invalidate the results of the tests.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.3.5 Response levels referred to within the following individual Test Series 7 test prescriptions are provided at Appendix 8 (Response descriptors), to aid in the assessment of the results of tests of types 7 (g), 7 (h), 7 (j), 7 (k) and 7 (l) and should be reported to the competent authority to support assignment to Division 1.6.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.4 Series 7 type (a) test prescription

17.4.1 *Test 7 (a): EI~~D~~S cap test*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.4.1.1 *Introduction*

This shock test is designed to determine the sensitivity of an EI~~D~~S candidate to intense mechanical stimulus.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.4.1.2 *Apparatus and materials*

The experimental set-up for this test is the same as for test 5 (a) (see 15.4.1).

17.4.1.3 *Procedure*

The experimental procedure is the same as for test 5 (a) (see 15.4.1).

17.4.1.4 *Test criteria and method of assessing results*

The result is considered "+" and the substance or mixture should not be classified as an EI~~D~~S if in any trial:

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

(a) The witness plate is torn or otherwise penetrated (i.e. light is visible through the plate) - bulges, cracks or folds in the witness plate do not indicate cap sensitivity; or

(b) The centre of the lead cylinder is compressed from its initial length by an amount of 3.2 mm or more.

Otherwise, the result is considered "-".

17.4.1.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	–
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.5 Series 7 type (b) test prescription

17.5.1 *Test 7 (b): EIDS gap test*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.5.1.1 *Introduction*

This test is used to measure the sensitivity of an EIDS candidate to a specified shock level, i.e. a specified donor charge and gap.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.5.1.2 *Apparatus and materials*

The set-up for this test consists of an explosive charge (donor), a barrier (gap), a container holding the test charge (acceptor), and a steel witness plate (target).

The following materials are to be used:

(a) United Nations Standard detonator or equivalent;

(b) 95 mm diameter by 95 mm long pressed 50/50 pentolite or 95/5 RDX/WAX pellet with a density of 1 600 kg/m^3 ± 50 kg/m^3 of either 50/50 pentolite or 95/5 RDX/WAX;

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(c) Tubing, steel, cold drawn seamless, with an 95 mm outer diameter of 95 mm ± 7.0 mm, 11.1 mma wall thickness of 9.75 mm ± 2.75 mm and an inner diameter of 73.0 mm ± 7.0 mm 10% variations, by 280 mm long having the following mechanical propertiesand with a length of 280 mm;:

- tensile strength = 420 MPa (± 20% variation)
- elongation (per cent) = 22 (± 20% variation)
- Brinell hardness = 125 (± 20% variation);

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(d) Sample substances or mixtures, machined to a diameter which is just under the diameter of the steel tubing. The air gap between the sample and tubing wall should be as small as possible;

(e) Cast pPolymethyl methacrylate (PMMA) rod, of 95 mm diameter by 70 mm long;

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(f) Mild steel plate, 200 mm ×by 200 mm ×x 20 mm;, having the following mechanical properties:

- tensile strength = 580 MPa (± 20% variation)
- elongation (per cent) = 21 (± 20% variation)
- Brinell hardness = 160 (± 20% variation);

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(g) Cardboard tubing, 97 mm inner diameter by 443 mm long;

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(hg) Wood block, 95 mm diameter and 25 mm thick, with a hole drilled through the centre to hold the detonator.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

17.5.1.3 *Procedure*

17.5.1.3.1 As shown in Figure 17.5.1.1, the detonator, donor, gap and acceptor charge are coaxially aligned above the centre of the witness plate. A 1.6 mm air gap is maintained between the free end of the acceptor charge and the witness plate with suitable spacers which do not overlap the acceptor charge. Care should be taken to ensure good contact between the detonator and donor, donor and gap and gap and acceptor charge. The test sample and booster should be at ambient temperature for the test.

17.5.1.3.2 To assist in collecting the remains of the witness plate, the whole assembly may be mounted over a container of water with at least a 10 cm air gap between the surface of the water and the bottom surface of the witness plate which should be supported along two edges only.

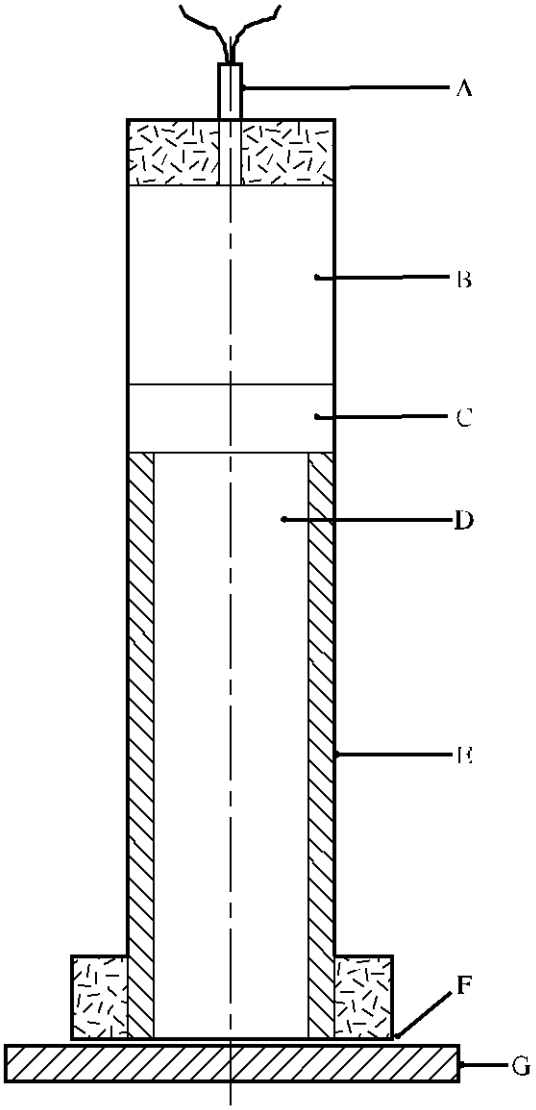
17.5.1.3.3 Alternative collection methods may be used but it is important to allow sufficient free space below the witness plate so as not to impede plate puncture. The test is performed three times unless a positive result is observed earlier.

17.5.1.4 *Test criteria and method of assessing results*

A clean hole punched through the plate indicates that a detonation was initiated in the sample. A substance or mixture which detonates in any trial is not an EI~~D~~S and the result is noted as "+".

17.5.1.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	+
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	+
RDX/inert binder (85/15), cast	+
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–
TNT, cast	+



(A)	Detonator	(B)	Booster charge
(C)	PMMA gap	(D)	Substance under test
(E)	Steel tube	(F)	Air gap
(G)	Witness plate		

Figure 17.5.1.1: EIDS Gap test

17.6 Series 7 type (c) test prescriptions

17.6.1 *Test 7 (c) (i): Susan impact test*

17.6.1.1 *Introduction*

The Susan Impact test is used to assess the degree of explosive reaction under conditions of high velocity impact. The test is conducted by loading the explosives into standardised projectiles and firing the projectiles against a target at a specified velocity.

17.6.1.2 *Apparatus and materials*

17.6.1.2.1 51 mm diameter, 102 mm long explosives billets, fabricated by normal techniques, are employed.

17.6.1.2.2 The Susan test employs the test vehicle shown in Figure 17.6.1.1. The projectile has an assembled weight of 5.4 kg and contains slightly less than 0.45 kg of explosive. The overall dimensions are 81.3 mm in diameter by 220 mm long.

17.6.1.2.3 The projectiles are fired from a 81.3 mm smooth-bore gun. The gun muzzle is positioned about 4.65 m from the 64 mm thick, smooth-surface, armour steel target plate. Projectile impact velocity is obtained by adjusting the propellant charges in the gun.

17.6.1.2.4 A schematic drawing of the firing range showing the target-gun layout and the relative positions of the diagnostic equipment is shown in Figure 17.6.1.2. The flight path is about 1.2 m above ground level.

17.6.1.2.5 The test site is equipped with calibrated blast gauges and recording equipment. The air blast recording system should have a system frequency response of at least 20 kHz. Measurements are made of impact velocities and air shock blast over-pressure. Air blast is measured at a distance of 3.05 m from the impact point (gauges (C) in Figure 17.6.1.2).

17.6.1.3 *Procedure*

17.6.1.3.1 The propellant charge in the gun should be adjusted to produce a projectile velocity of 333 m/s. The projectile is fired and the impact velocity and air blast, produced as a result of its reaction on impact, are recorded. If a velocity of 333 m/s (+ 10%, - 0%) is not obtained, the amount of propellant is adjusted and the test repeated.

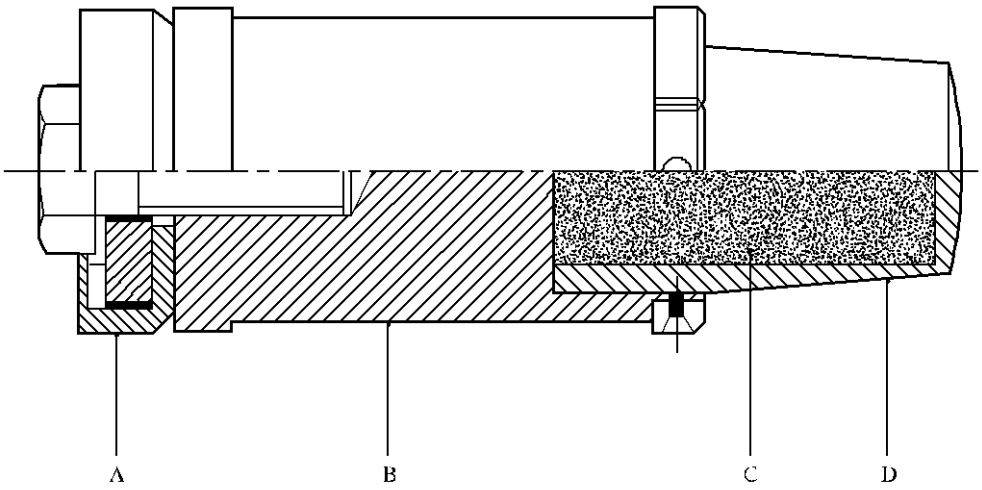
17.6.1.3.2 Once an impact velocity of 333 m/s is obtained, the test is repeated until accurate pressure-time records are obtained from at least five separate shots. On each of these accurate shots, the impact velocity should be 333 m/s (+ 10%, - 0%).

17.6.1.4 *Test criteria and method of assessing results*

The maximum air blast overpressure that is determined from each air blast is recorded. The average of the maximum pressures obtained from the five accurate shots is determined. If the average pressure obtained by such a procedure is greater than or equal to 27 kPa, then the substance or mixture is not an EI~~D~~S explosive and the result is noted as "+".

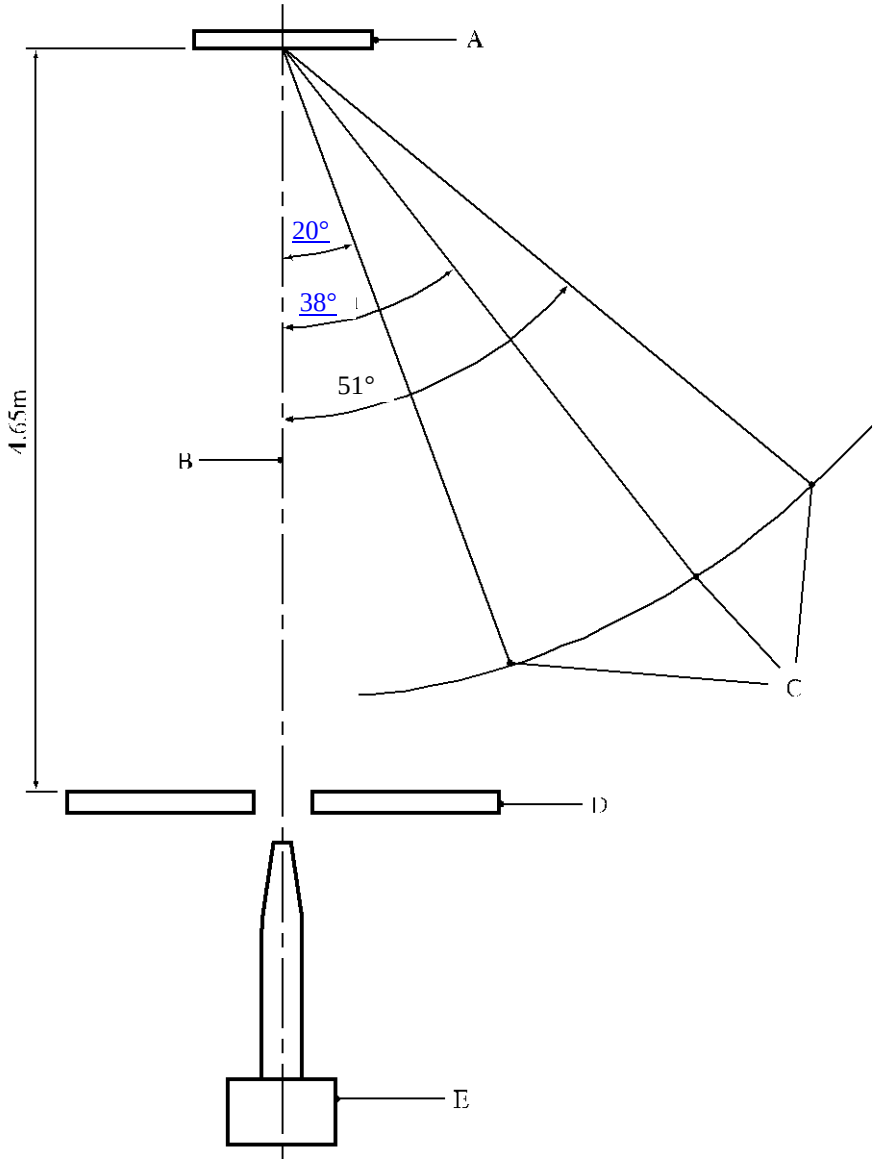
17.6.1.5 *Examples of results*

Substance/Mixture	Result
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	+
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–



(A)	Leather cup seal	(B)	Steel body
(C)	Explosive under test	(D)	Aluminium cup

Figure 17.6.1.1: SUSAN projectile



(A)	Target plate (6.4 cm thick)	(B)	Flight path
(C)	Air blast transducers (3.05 m from target point)	(D)	Smoke barrier
(E)	81.3 mm gun		

Figure 17.6.1.2: Schematic layout of SUSAN test (top view)

17.6.2 ***Test 7 (c) (ii): Friability test***

17.6.2.1 *Introduction*

The friability test is used to establish the tendency of a compact EI~~D~~S candidate to deteriorate dangerously under the effect of an impact.

17.6.2.2 *Apparatus and materials*

The following apparatus is required:

(a) A weapon designed to shoot 18 mm diameter cylindrical test pieces at a velocity of 150 m/s;

(b) A Z30C 13 stainless steel plate, 20 mm thick with a front face roughness of 3.2 microns (AFNOR NF E 05-015 and NF E 05-016 standards);

(c) A 108 ± 0.5 cm^3 manometric bomb at 20 °C;

(d) A firing capsule containing a heating wire on 0.5 g of black powder with a mean particle size of 0.75 mm. The composition of the black powder is 74% potassium nitrate, 10.5% sulphur and 15.5% carbon. The moisture content should be less than 1%;

(e) A cylindrical sample of compact substance of diameter 18 ± 0.1 mm. The length is adjusted so as to obtain a mass of 9.0 ± 0.1 g. The sample is brought to and maintained at a temperature of 20 °C;

(f) A fragment recovery box.

17.6.2.3 *Procedure*

17.6.2.3.1 The sample is projected against the steel plate at an initial velocity sufficient to give an impact velocity as close as possible to 150 m/s. The mass of fragments collected after the impact should be at least 8.8 g. These fragments are fired in a manometric bomb. Three tests are carried out.

17.6.2.3.2 The curve of pressure against time p = f (t) is recorded; this enables the curve (dp/dt) = f' (t) to be constructed. From this curve the maximum value $(dp/dt)_{max}$ is read off. This enables the value $(dp/dt)_{max}$, corresponding to an impact speed of 150 m/s, to be estimated.

17.6.2.4 *Test criteria and method of assessing results*

If the average maximum $(dp/dt)_{max}$ value obtained at a speed of 150 m/s is greater than 15 MPa/ms, the substance or mixture tested is not an EI~~D~~S and the result is noted as "+".

17.6.2.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	–
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.7 Series 7 type (d) test prescriptions

17.7.1 *Test 7 (d) (i): EIDS bullet impact test*

17.7.1.1 *Introduction*

The bullet impact test is used to evaluate the response of an EIDS candidate to the kinetic energy transfer associated with impact and penetration of a given energy source, i.e. a 12.7 mm projectile, travelling at a specified velocity.

17.7.1.2 *Apparatus and materials*

17.7.1.2.1 Explosive test samples fabricated by normal techniques are employed. The samples should have a length of 20 cm and a diameter to allow a close fit into a seamless steel pipe having an inside diameter of 45 mm (± 10% variation), a wall thickness of 4 mm (± 10% variation) and a length of 200 mm. The pipes are closed with steel or cast iron end caps, at least as strong as the tube, torqued to 204 Nm.

17.7.1.2.2 The bullet is a standard 12.7 mm armour-piercing bullet with a projectile mass of 0.046 kg, and is fired at the service velocity of about 840 ± 40 m per second from a 12.7 mm gun.

17.7.1.3 *Procedure*

17.7.1.3.1 A minimum of six test articles (explosive substance or mixture in a capped steel pipe) should be fabricated for the tests.

17.7.1.3.2 Each test article is positioned on a suitable pedestal at a convenient distance from the muzzle of the gun. Each test article must be secured in a holding device upon its pedestal. This device must be capable of restraining the item against dislodgement by the bullet.

17.7.1.3.3 A test consists of the firing of one projectile into each test item. There should be at least three tests with the test article oriented such that its long axis is perpendicular to the line of flight (i.e. impact through the side of the pipe). There should also be at least three tests with the test article oriented such that its long axis is parallel to the line of flight (i.e. impact through the end cap).

17.7.1.3.4 The remains of the test container are collected. Complete fragmentation of the container is indicative of explosion or detonation.

17.7.1.4 *Test criteria and method of assessing results*

A substance or mixture which explodes or detonates in any trial is not an EIDS and the result is noted as "+".

17.7.1.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	–
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.7.2 ***Test 7 (d) (ii): Friability test***

17.7.2.1 *Introduction*

The friability test is used to evaluate the response of an EI~~D~~S candidate to the kinetic energy transfer associated with impact and penetration of a given energy source travelling at a specified velocity.

17.7.2.2 *Apparatus and materials*

The following apparatus is required:

(a) A weapon designed to shoot 18 mm diameter cylindrical test pieces at a velocity of 150 m/s;

(b) A Z30C 13 stainless steel plate, 20 mm thick with a front face roughness of 3.2 microns (AFNOR NF E 05-015 and NF E 05-016 standards);

(c) A 108 ± 0.5 cm^3 manometric bomb at 20 °C;

(d) A firing capsule containing a heating wire on 0.5 g of black powder with a mean particle size of 0.75 mm. The composition of the black powder is 74% potassium nitrate, 10.5% sulphur and 15.5% carbon. The moisture content should be less than 1%;

(e) A cylindrical sample of compact substance of diameter 18 ± 0.1 mm. The length is adjusted so as to obtain a mass of 9.0 ± 0.1 g. The sample is brought to and maintained at a temperature of 20 °C;

(f) A fragment recovery box.

17.7.2.3 *Procedure*

17.7.2.3.1 The sample is projected against the steel plate at an initial velocity sufficient to give an impact velocity as close as possible to 150 m/s. The mass of fragments collected after the impact should be at least 8.8 g. These fragments are fired in a manometric bomb. Three tests are carried out.

17.7.2.3.2 The curve of pressure against time p = f (t) is recorded; this enables the curve (dp/dt) = f' (t) to be constructed. From this curve the maximum value $(dp/dt)_{max}$ is read off. This enables the value $(dp/dt)_{max}$, corresponding to an impact speed of 150 m/s, to be estimated.

17.7.2.4 *Test criteria and method of assessing results*

If the average maximum $(dp/dt)_{max}$ value obtained at a speed of 150 m/s is greater than 15 MPa/ms, the substance or mixture tested is not an EI~~D~~S and the result is noted as "+".

17.7.2.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	–
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.8 Series 7 type (e) test prescription

17.8.1 *Test 7 (e): EI~~D~~S external fire test*

17.8.1.1 *Introduction*

The external fire test is used to determine the reaction of an EI~~D~~S candidate to external fire when it is confined.

17.8.1.2 *Apparatus and materials*

Explosive test samples fabricated by normal techniques are employed. The samples should have a length of 20 cm and a diameter to allow a close fit into a seamless steel pipe having an inside diameter of 45 mm (± 10% variation), a wall thickness of 4 mm (± 10% variation) and a length of 200 mm. The pipes are closed with steel or cast iron end caps, at least as strong as the tube, torqued to 204 Nm.

17.8.1.3 *Procedure*

17.8.1.3.1 The experimental procedure is the same as for test 6 (c) (see 16.6.1.3) except as noted in paragraph 17.8.1.3.2 below.

17.8.1.3.2 The test is conducted as:

(a) One fire engulfing fifteen confined samples, stacked in three adjacent piles of two samples banded on top of three samples; or

(b) Three fires in which five samples are laid out horizontally and banded together.

Colour photographs are taken to document the condition of the samples after each test. Cratering and the size and location of confining pipe fragments are documented as an indication of the degree of reaction.

17.8.1.4 *Test criteria and method of assessing results*

An explosive substance or mixture which detonates or reacts violently with fragments thrown more than 15 m is not an EI~~D~~S and the result is noted as "+".

17.8.1.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/inert binder (85/15), cast	–
HMX/energetic binder (80/20), cast	+
HMX/aluminium/energetic binder (51/19/14), cast	–
RDX/inert binder (85/15), cast	+
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.9 Series 7 type (f) test prescription

17.9.1 *Test 7 (f): EI~~D~~S slow cook-off test*

17.9.1.1 *Introduction*

This test is used to determine the reaction of an EI~~D~~S candidate to a gradually increasing thermal environment and to find the temperature at which reaction occurs.

17.9.1.2 *Apparatus and materials*

17.9.1.2.1 Explosive test samples fabricated by normal techniques are employed. The samples should have a length of 200 mm and a diameter to allow a close fit into a seamless steel pipe having an inside diameter of 45 mm (± 10% variation), a wall thickness of 4 mm (± 10% variation) and a length of 200 mm. The pipes are closed with steel or cast iron end caps, at least as strong as the tube, torqued to 204 Nm.

17.9.1.2.2 The sample assembly is placed in an oven which provides a controlled thermal environment over a 40 °C to 365 °C temperature range and can increase the temperature of the surrounding oven atmosphere at the rate of 3.3 °C per hour throughout the temperature operating range and ensure, by circulation or other means, a uniform thermal environment to the item under test.

17.9.1.2.3 Temperature recording devices are used to monitor temperature at 10 minute or less intervals; continuous monitoring is preferred. Instrumentation with an accuracy of ± 2 per cent over the test temperature range is used to measure the temperature of:

(a) The air within the oven; and

(b) The exterior surface of the steel pipe.

17.9.1.3 *Procedure*

17.9.1.3.1 The test item is subjected to a gradually increasing air temperature at a rate of 3.3 °C per hour until reaction occurs. The test may begin with the test item pre-conditioned to 55 °C below the anticipated reaction temperature. The onset temperature at which the sample temperature exceeds the oven temperature should be recorded.

17.9.1.3.2 After the completion of each test, the pipe or any fragments of pipe are recovered in the test area and examined for evidence of violent explosive reaction. Colour photographs may be taken to document the condition of the unit and the test equipment before and after the test. Cratering, and the size and location of any fragments, may also be documented as indications of the degree of reaction.

17.9.1.3.3 Three tests are conducted for each candidate substance or mixture unless a positive result is observed earlier.

17.9.1.4 *Test criteria and method of assessing results*

A substance or mixture which detonates or reacts violently (fragmentation of one or two end caps and fragmentation of the tube into more than three pieces) is not considered an EI~~D~~S and the result is noted as "+".

17.9.1.5 *Examples of results*

Substance/Mixture	**Result**
HMX/inert binder (86/14), cast	–
HMX/energetic binder (80/20), cast	+
RDX/TNT (60/40), cast	+
TATB/Kel-F (95/5), pressed	–

17.10 Series 7 type (g) test prescription

17.10.1 *Test 7 (g): 1.6 ~~Article~~ article (or component level) external fire test*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.10.1.1 *Introduction*

The external fire test is used to determine the reaction of a possible Division 1.6 article to external fire as presented for transport[RG9].

17.10.1.2 *Apparatus and materials*

The experimental set-up for this test is the same as for test 6 (c) (see 16.6.1.2).

17.10.1.3 *Procedure*

17.10.1.3.1 The experimental procedure for this test is the same as for test 6 (c) (see 16.6.1.3), except that, if the volume of single item exceeds 0.15 m^3, only one item is required.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.10.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance[RG10] remains, fragmentation, blast, projections, cratering, witness screen damage, and thrust are documented as an indication of the article's response level.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.10.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.10.1.3.4 To classify complex articles containing multiple EIS main explosive loads, external fire testing at the individual main load component level should be conducted to fully characterise the article's response level.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.10.1.4 *Test criteria and method of assessing results*

If there is a ~~reaction~~ response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.11 Series 7 type (h) test prescription

17.11.1 *Test 7 (h): 1.6 article or component level slow cook-off test*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.11.1.1 *Introduction*

This test is used to determine the reaction of a candidate Division 1.6 article to a gradually increasing thermal environment and to find the temperature at which reaction occurs.

17.11.1.2 *Apparatus and materials*

17.11.1.2.1 The test equipment consists of an oven which provides a controlled thermal environment over a 40 °C to 365 °C temperature range and can increase the temperature of the surrounding oven atmosphere at the rate of 3.3 °C per hour throughout the temperature operating range, minimize hot spots, and ensure (by circulation or other means) a uniform thermal environment to the item under test. Secondary reactions (such as those caused by exudate and explosive gases contacting the heating devices) invalidate the test, but these can be avoided by providing a sealed inner container to surround articles transported bare. A means of relief should be provided for the increased air pressure generated during the test due to heating.

17.11.1.2.2 Temperature recording devices (permanent record types) are used to monitor temperature continuously or, at least, every 10 minutes. Instrumentation with an accuracy of ± 2% over the test temperature range is used to measure the temperature at:

(a) The atmosphere air gap adjacent to the unit under test; and

(b) The exterior surface of the unit.

17.11.1.3 *Procedure*

17.11.1.3.1 The test item is subjected to a gradually increasing, at a rate of 3.3 °C per hour, air temperature until unit reaction occurs. The test may begin with the test item pre-conditioned to 55 °C below the predicted reaction temperature. Temperatures and elapsed test time are measured and recorded.

17.11.1.3.2 Colour still photographs are taken to document the condition of the ~~unit~~ test item and the test equipment before and after the test. ~~Cratering and fragment size are documented as an indication of the degree of reaction. The energetic material may ignite and burn and the case may melt or weaken sufficiently to allow mild release of the combustion gases. Burning should be such that case debris and package elements stay in the area of test except for case closures which may be dislodged by the internal pressure and thrown up to about 15 metres.~~ Explosive substance[RG11] remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level. Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.11.1.3.3 The test is conducted twice unless a positive result is obtained earlier. To classify complex articles containing multiple EIS main explosive loads, slow cook-off testing at the individual main load component level should be conducted to fully characterise the article's response level.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.11.1.4 *Test criteria and method of assessing results*

If there is a ~~reaction~~ response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.

17.12 Series 7 type (j) test prescription

17.12.1 *Test 7 (j): 1.6 article or component bullet impact test*

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.1 *Introduction*

The bullet impact test is used to evaluate the response of a candidate Division 1.6 article to the kinetic energy transfer associated with the impact and penetration by a given energy source.

17.12.1.2 *Apparatus and materials*

A Three 12.7 mm gun is are used to fire service 12.7 mm armour-piercing (AP) ammunition with a projectile mass of 0.046 kg. and with sStandard propellant loads may require adjustment to achieve projectile velocities within tolerance. The guns is are fired by remote control and is protected from fragment damage by firing through a hole in a heavy steel plate. The firing gun muzzle should be at a maximum range of at least 10 m from the test item to assure stabilization prior to impact, and at a maximum range of 30 3 to 20 m from the test item depending upon the explosive weight of the test item. The test item should be secured in a holding device capable of restraining the item against dislodgement by the projectiles. The test is recorded visually by photographic or other means.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.3 *Procedure*

17.12.3.1 The candidate Division 1.6 article is subjected The test consists of subjecting a complete EIDS loaded item to a three-round burst fired at 840 ± 40 m/s velocity and 600 rounds/minute rate of fire. The test is repeated in three different orientations. In the appropriate orientation(s), the striking point on the test item in the most vulnerable areas as assessed by the competent authority. for the multiple impact is selected so that the impacting rounds penetrate the most sensitive material(s) that is not separated from the main explosive charge by barriers or other safety devices. The degree of reaction is determined by post-test inspection of test film and hardware. Fragmentation of the article into small pieces is indicative of detonation. These are areas for which an assessment of the explosive sensitivity (explosiveness and sensitiveness) combined with knowledge of the article design indicate the potential producing the most violent response level.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance [RG12]remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.3.4 To classify complex articles containing multiple EIS main explosive loads, bullet impact testing at the individual main load component level should be conducted to fully characterise the article's response level.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.12.1.4 *Test criteria and method of assessing results*

If there is a response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles. If a detonation results from any test, the item cannot be considered as a Division 1.6 article and the result is noted as "+". No reaction, burning or deflagration is considered a negative result and is noted as " ".
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.13 Series 7 type (k) test prescription

17.13.1 *Test 7 (k): 1.6 article stack test*

17.13.1.1 *Introduction*

This test is used to determine whether a detonation of a candidate Division 1.6 article, as offered for transport[RG13], will initiate a detonation in an adjacent, like article.

17.13.1.2 *Apparatus and materials*

The experimental set-up is the same as for test 6 (b) (see 16.5.1.2), with one trial conducted confined, and another unconfined. The test should only be conducted on detonable candidate Division 1.6 articles; the test 7 (k) article stack test is waived for non-detonable candidates for Division 1.6 (evidence is available to demonstrate that the article cannot support a detonation). Where the article is designed to provide a detonation output, the article's own means of initiation or a stimulus of similar power shall be used to initiate the donor. If the article is not designed to detonate but is capable of supporting a detonation, the donor shall be detonated using an initiation system selected to minimise the influence of its explosive effects on the acceptor article(s). ~~but without confinement. The donor article should be provided with its own means of initiation or a stimulus of similar power.~~
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.13.1.3 *Procedure*

The experimental set-up is the same as for test 6 (b) (see 16.5.1.3). Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance [RG14]remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented and used to assess whether or not any acceptor has detonated (including partially). Blast data may be used to supplement this decision. Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information. Comparing data from the two stack test trials to data from a single donor calibration shot, or to a calculated donor detonation pressure, can be useful in assessing the response level of acceptors.~~The test is performed three times unless detonation of an acceptor is observed earlier. Fragmentation data (size and number of acceptor article fragments), damage to the witness plate and crater dimensions are used to determine whether or not any acceptor has detonated. Blast data may be used to supplement this decision.~~

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.13.1.4 *Test criteria and method of assessing results*

If detonation in the stack is propagated from the donor to an acceptor, the test result is noted as "+" and the article cannot be assigned to Division 1.6. Acceptor article response~~s~~ level ~~identified~~ assessed as no reaction, burning, ~~or~~ deflagration or explosion as outlined in Appendix 8 are considered as negative results and noted as "—".

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

17.14 Series 7 type (l) test prescription

17.14.1 *Test 7 (l): 1.6 article (or component level) fragment impact test*

17.14.1.1 *Introduction*

This test is used to determine the response of an article in its transport configuration to a localised shock input representative of a fragment strike typical of that produced from a nearby detonating article.

17.14.1.2 Apparatus and materials

To reduce variability due to yaw, a gun system is recommended for firing a standard 18.6 g steel fragment in the shape of a right-circular cylinder with a conical nose, as detailed in Figure 17.14.1, at a candidate Division 1.6 article. The distance between the firing device and the test item should ensure that the fragment is ballistically stable at impact. Barricades should protect the remote control gun system from the potential damaging effects of the test item's reaction.

17.14.1.3 Procedure

17.14.1.3.1 The test is repeated in two different orientations, striking the test item in the most vulnerable areas as assessed by the competent authority. These are areas for which an assessment of the explosive sensitivity (explosiveness and sensitiveness) combined with knowledge of the article design indicate the potential for producing the most violent response level. Typically, one test would be conducted targeting a non-EIS boostering component and the second test would target the centre of the main explosive load. The orientation of impact should generally be normal to the outer surface of the article. The fragment impact velocity should be 2530 ± 90 m/s.

17.14.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance [RG15]remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level.

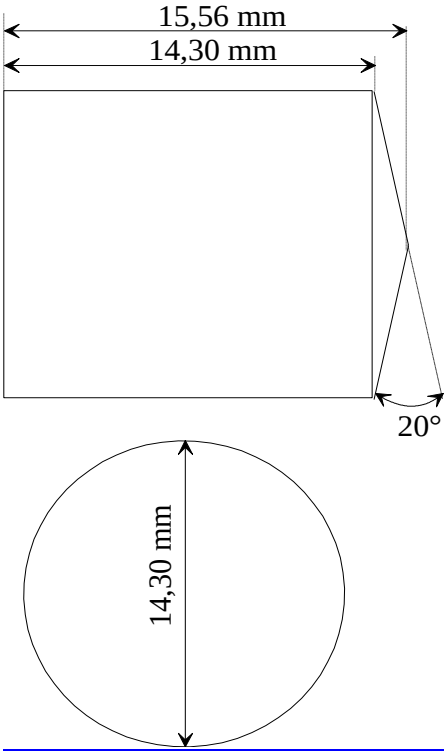
17.14.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

17.14.1.3.4 To classify complex articles containing multiple EIS main explosive loads, fragment impact testing at the individual main load component level should be conducted to fully characterise the article's response level.

17.14.1.4 Test criteria and method of assessing results

If there is a response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)



Notes:

Shape: a conical ended cylinder with the ratio $\frac{\text{L (length)}}{\text{D (diameter)}} > 1$ *for stability;*

Tolerances: *± 0.05 mm and ± 0°30';*
Fragment mass: *18.6 g;*
Fragment material: *a mild carbon steel with a Brinell Hardness (HB) less than 270.*

Figure 17.14.1: Standard fragment for 1.6 article fragment impact test

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.1)

~~SECTION~~ CHAPTER 18

TEST SERIES 8

18.1 Introduction

The assessment whether a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives (ANE)" is insensitive enough ~~for inclusion~~to be classified as an oxidizing liquid or solid (~~in~~ Division 5.1 for transport) is answered by series 8 tests and any such candidate for inclusion in this hazard class ~~Division 5.1~~ should pass each of the three types of tests comprising the series. The three test types are:

Type 8 (a): a test to determine the thermal stability;
Type 8 (b): a shock test to determine sensitivity to intense shock;
Type 8 (c): a test to determine the effect of heating under confinement;

Test series 8 (d) has been included in this section as one method to evaluate the suitability for the transport in tanks.

18.2 Test methods

The test methods currently used are listed in Table 18.1.

Table 18.1: Test methods for Test Series 8

Test code	Name of Test	Section
8 (a)	Thermal Stability Test for ANE[a]	18.4
8 (b)	ANE Gap Test[a]	18.5
8 (c)	Koenen test[a]	18.6
8 (d)	Vented pipe tests[b]	18.7

[a] *This test is intended for classification.*
[b] *These tests are intended for evaluating the suitability for transport in tanks.*

18.3 Test conditions

18.3.1 The substance or mixture should be tested as offered for transport, at the highest transport temperature (see 1.5.4 of this Manual)[RG16].

18.4 Series 8 Type (a) test prescription

18.4.1 *Test 8 (a): Thermal stability test for ammonium nitrate emulsions, suspension or gels*

18.4.1.1 *Introduction*

18.4.1.1.1 This test is used to measure the stability of a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" when subjected to elevated thermal conditions to determine if the emulsion is too dangerous to transport[RG17].

18.4.1.1.2 This test is used to determine whether the emulsion, suspension or gel is stable at temperatures encountered during transport[RG18]. In the way this type of test is normally carried out (see 28.4.4), the 0.5 litre Dewar vessel is only representative for packagings, IBC's and small tanks. For the transport of ammonium nitrate emulsions, suspensions or gels the test can be used to measure its stability during tank transport if the test is carried out at a temperature 20 °C higher than the maximum temperature which may occur during transport, including the temperature at the time of loading.

18.4.1.2 *Apparatus and materials*

18.4.1.2.1 The experimental equipment consists of a suitable test chamber, appropriate Dewar vessels with closures, temperature probes and measuring equipment.

18.4.1.2.2 ***The test should be performed in a test cell capable of withstanding fire and overpressure and, preferably, should be fitted with a pressure relief system e.g. a blow out panel.*** The recording system should be housed in a separate observation area.

18.4.1.2.3 A thermostatically controlled drying oven (which may be fan-assisted) large enough to allow air circulation on all sides of the Dewar vessel may be used. The air temperature in the oven should be controlled so that the desired temperature for a liquid inert sample in the Dewar vessel can be maintained with a deviation of not more than ± 1 °C for up to 10 days. The air temperature in the oven should be measured and recorded. It is recommended that the door of the oven be fitted with a magnetic catch or replaced by a loosely fitting insulated cover. The oven may be protected by an appropriate steel liner and the Dewar vessel housed in a wire mesh cage.

18.4.1.2.4 Dewar vessels with a volume of 500 ml with a closure system are used. The closure of the Dewar vessel should be inert. A closure system is illustrated in Figure 18.4.1.1.

18.4.1.2.5 The heat loss characteristics of the system used, i.e. Dewar vessel and closure, should be established prior to performance of the test. Since the closure system has a significant effect on the heat loss characteristics, these can be adjusted to some extent by varying the closure system. The heat loss characteristics can be determined by measuring the half time of cooling of the vessel filled with an inert substance having similar physical properties. The heat loss per unit of mass, L (W/kg.K) can be calculated from the half time of cooling, $t_{1/2}$ (s), and the specific heat, Cp (J/kg.K), of the substance using the formula:

~~$L = \ln 2 \times C_p / t_{1/2}$~~

$$L = \ln 2 \times \left(C_p / t_{1/2} \right)$$

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.4.1.2.6 Dewar vessels filled with 400 ml of inert substance, with a heat loss of ~~80 to~~ 100 mW/kg.K or less are suitable.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.4.1.2.7 The Dewar vessel shall be filled to about 80% of its capacity. In case of a sample with very high viscosity it may be required to have the sample provided with a shape which just fits into the Dewar vessel. The diameter of such a preshaed sample shall be just under the inner diameter of the Dewar vessel. The hollow lower end of the Dewar vessel may be filled with an inert solid substance prior to loading the sample into the vessel to facilitate the use of cylindrically shaped sample substances.

18.4.1.3 *Procedure*

18.4.1.3.1 Set the test chamber at a temperature which is 20 °C higher than the maximum temperature which may occur during transport [RG19]or, if higher, the temperature at the time of loading. Fill the Dewar vessel with the substance under test and note the mass of the sample. Make sure the sample is filled to about 80% of its height. Insert the temperature probe into the centre of the sample. Seal the lid of the Dewar in place and insert the Dewar vessel in the test chamber, connect the temperature recording system and close the test chamber.

18.4.1.3.2 The sample is heated and the temperature of the sample and test chamber continuously monitored. The time is noted at which the sample temperature reaches a temperature 2 °C below the test chamber temperature. The test is then continued for a further seven days or until the sample temperature rises to 6 °C or more above the test chamber temperature if this occurs sooner. Note the time taken for the sample to rise from 2 °C below the test chamber temperature to its maximum temperature.

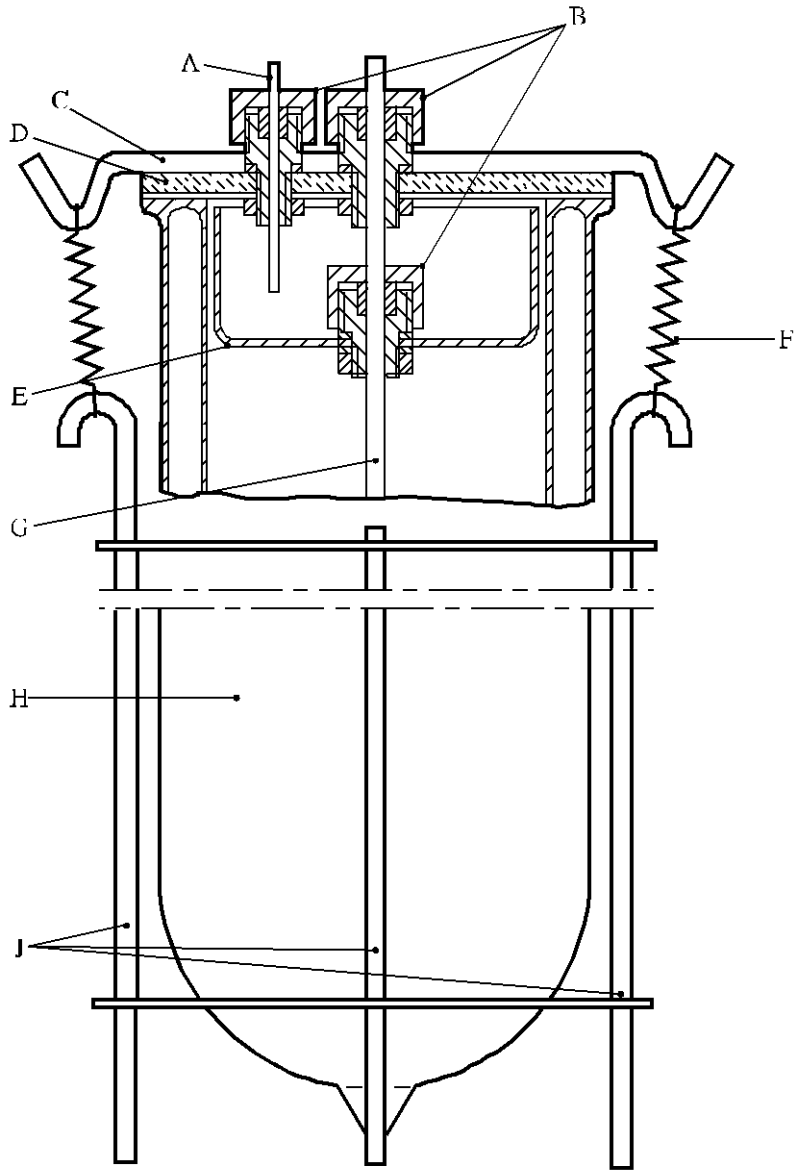
18.4.1.3.3 If the sample survives, cool and remove it from the test chamber and carefully dispose of it as soon as possible. The percentage mass loss and change in composition may be determined.

18.4.1.4 *Test criteria and method of assessing results*

18.4.1.4.1 If the sample temperature does not exceed the test chamber temperature by 6 °C or more in any test, the ammonium nitrate emulsion, suspension or gel is considered to be thermally stable and can be further tested as a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives".

18.4.1.5 *Examples of results*

Substance~~s~~/Mixture	**Sample mass (g)**	**Test T (°C)**	**Result**	**Comments**
Ammonium nitrate	408	102	-	slight discolouration, hardened into lump Mass loss 0.5%
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	551	102	-	separation of oil and crystallized salts. Mass loss 0.8%
ANE-2 (sensitized) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	501	102	-	Some discolouration Mass loss 0.8%
ANE-Y Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	500	85	-	Mass loss 0.1%
ANE-Z Ammonium nitrate 75%, Water 20%, Fuel/emulsifier 5%	510	95	-	Mass loss 0.2%
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	553	85	-	no rise in temperature
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	540	85	-	no rise in temperature
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	613	80	-	Mass loss 0.1%
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	605	80	-	Mass loss 0.3%
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	602	80	-	Mass loss 0.1%



(A)	PTFE capillary tube	(B)	Special screw fittings (PTFE or Al) with O-ring seal
(C)	Metal strip	(D)	Glass lid
(E)	Glass beaker base	(F)	Spring
(G)	Glass protective tube	(H)	Dewar vessel
(J)	Steel retaining device		

Figure 18.4.1.1: DEWAR vessel with closure

18.5 Series 8 Type (b) Test prescription

18.5.1 *Test 8 (b): ANE Gap Test*

18.5.1.1 *Introduction*

This test is used to measure the sensitivity of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a specified shock level, i.e. a specified donor charge and gap.

18.5.1.2 *Apparatus and materials*

18.5.1.2.1 The set-up for this test consists of an explosive charge (donor), a barrier (gap), a container holding the test charge (acceptor), and a steel witness plate (target).

The following materials are to be used:

(a) United Nations Standard detonator or equivalent;

(b) 95 mm diameter by 95 mm long ~~pressed 50/50 pentolite or 95/5 RDX/WAX~~ pellet with a density of 1 600 kg/m^3 ± 50 kg/m^3 of either 50/50 pentolite or 95/5 RDX/WAX;
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(c) Tubing, steel, ~~cold drawn~~ seamless, with an outer diameter of 95.0 ± 7.0 mm, a wall thickness of 9.75 ± 2.75 mm and an inner diameter of 73.0 ± 7.0 mm, and with a length of 280 mm;
~~95 mm outer diameter, 11.1 mm wall thickness ± 10% variations, by 280 mm long having the following mechanical properties:~~

~~tensile strength = 420 MPa (± 20% variation)~~

~~elongation (%) = 22 (± 20% variation)~~

~~Brinell hardness = 125 (± 20% variation)~~
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(d) Sample substances, with a diameter which is just under the inner diameter of the steel tubing. The air gap between the sample and tubing wall should be as small as possible;

(e) ~~Cast p~~Polymethyl methacrylate (PMMA) rod, of 95 mm diameter by 70 mm long. A gap length of 70 mm results in ~~a~~ an incident shock pressure at the ANE interface ~~applied to the emulsion~~ somewhere between 3.5 and 4 GPa, depending on the type of donor used (see Table 18.5.1.1 and Figure 18.5.1.2);
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(f) Mild steel plate, 200 mm × 200 mm × 20 mm~~, having the following mechanical properties:~~

~~tensile strength = 580 MPa (± 20% variation)~~

~~elongation (%) = 21 (± 20% variation)~~

~~Brinell hardness = 160 (± 20% variation)~~
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

~~(g) Cardboard tubing, 97 mm inner diameter by 443 mm long;~~

(~~h~~g) Wood block, 95 mm diameter and 25 mm thick, with a hole drilled through the centre to hold the detonator.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.5.1.3 *Procedure*

18.5.1.3.1 As shown in Figure 18.5.1.1, the detonator, donor, gap and acceptor charge are coaxially aligned above the centre of the witness plate. Care should be taken to ensure good contact between the detonator and donor, donor and gap and gap and acceptor charge. The test sample and booster should be at ambient temperature for the test.

18.5.1.3.2 To assist in collecting the remains of the witness plate, the whole assembly may be mounted over a container of water with at least a 10 cm air gap between the surface of the water and the bottom surface of the witness plate which should be supported along two edges only.

18.5.1.3.3 Alternative collection methods may be used but it is important to allow sufficient free space below the witness plate so as not to impede plate puncture. The test is performed three times unless a positive result is observed earlier.

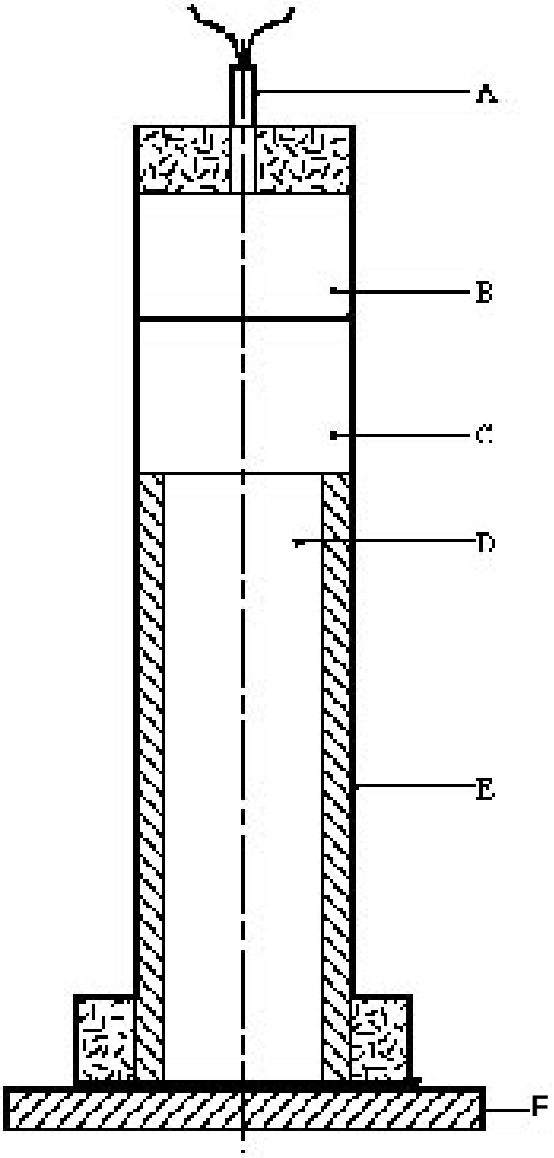
18.5.1.4 *Test criteria and method of assessing results*

A clean hole punched through the plate indicates that a detonation was initiated in the sample. A substance or mixture which detonates in any trial at a gap length of 70 mm is not to be classified as "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" and the result is noted as "+".

18.5.1.5 *Examples of results*

Substance~~s~~/Mixture	**Density g/cm^3**	**Gap mm**	**Result**	**Comments**
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate bent VOD 2.3-2.8 km/s
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate fractured
ANE-FA Ammonium nitrate 69%, Sodium nitrate 12%, Water 10%, Fuel/emulsifier 8%	1.4	50	-	Tube fragmented (large fragments) Plate not perforated
ANE-FA	1.44	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FB Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 7%	ca 1.40	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FC (sensitized) Ammonium nitrate 75%, Water 13%, Fuel/emulsifier 10%	1.17	70	+	Tube fragmented (fine fragments) Plate perforated
ANE-FD (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	ca 1.22	70	+	Tube fragmented (fine fragments) Plate perforated
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.4	35	-	Tube fragmented into large pieces. Plate dented VOD: 3.1 km/s
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	35	+	Tube fragmented into small pieces Plate perforated VOD: 6.7 km/s
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	70	+	Tube fragmented into small pieces Plate perforated VOD: 6.2 km/s
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%,	1.29	70	-	Tube fragmented

Substances/Mixture	Density g/cm^3	Gap mm	Result	Comments
Fuel/emulsifier 9%				Plate indented VOD 1 968 m/s
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	1.32	70	-	Tube fragmented Plate indented
ANE-G3 (sensitized by gassing) Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	1.17	70	+	Tube fragmented Plate punctured
ANE-G4 (sensitized by microballoons) Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	1.23	70	+	Tube fragmented Plate punctured
ANE-G5 Ammonium nitrate 70%, Calcium nitrate 8%, Water 16%, Fuel/emulsifier 7%	1.41	70	-	Tube fragmented Plate indented VOD 2 061 m/s
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	1.39	70	-	Tube fragmented Plate indented
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.42	70	-	Tube fragmented Plate indented
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	1.40	70	-	Tube fragmented Plate indented
ANE-J5 (sensitized by microballoons) Ammonium nitrate 71%, Sodium nitrate 5%, Water 18%, Fuel/emulsifier 6%	1.20	70	+	Tube fragmented Plate perforated VOD 5.7 km/s
ANE-J6 (sensitized by microballoons) Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	1.26	70	+	Tube fragmented Plate perforated VOD 6.3 km/s



(A)	Detonator	(B)	Booster charge
(C)	PMMA gap	(D)	Substance under test
(E)	Steel tube	(F)	Witness plate

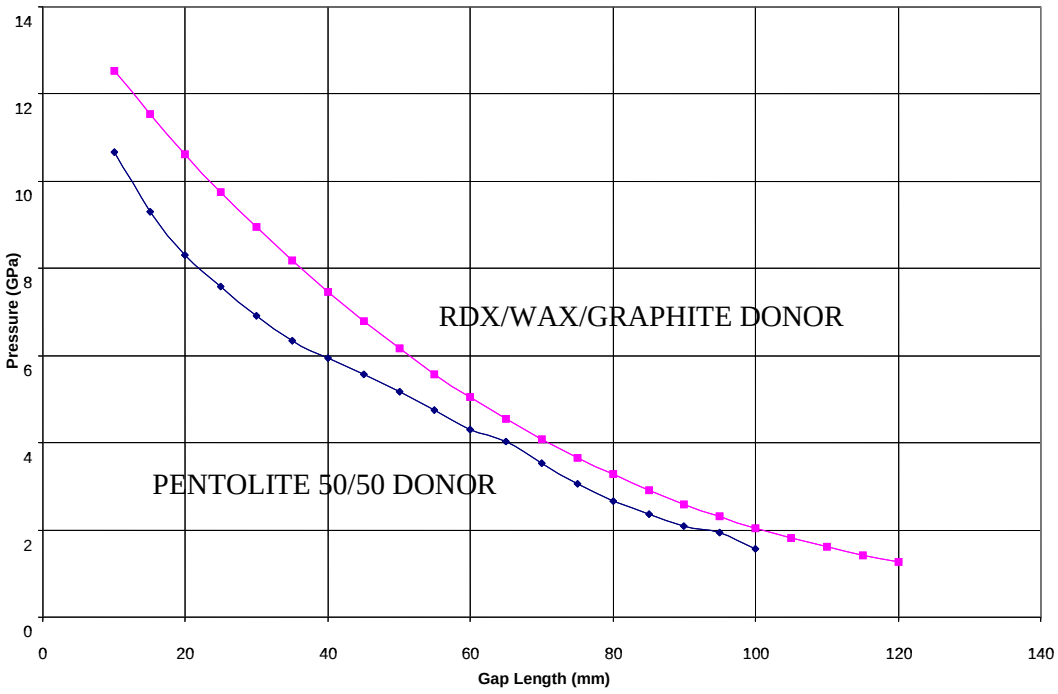
Figure 18.5.1.1: ANE gap test

Table 18.5.1.1: ANE gap test calibration data

PENTOLITE 50/50 DONOR		RDX/WAX/GRAPHITE DONOR	
Gap length (mm)	Barrier pressure (GPa)	Gap length (mm)	Barrier pressure (GPa)
10	10.67	10	12.53
15	9.31	15	11.55
20	8.31	20	10.63
25	7.58	25	9.76
30	6.91	30	8.94
35	6.34	35	8.18
40	5.94	40	7.46
45	5.56	45	6.79
50	5.18	50	6.16
55	~~4.76~~4.91	55	5.58
60	~~4.31~~4.51	60	5.04
65	4.02	65	4.54
70	3.53	70	4.08
75	3.05	75	3.66
80	2.66	80	3.27
85	2.36	85	2.91
90	2.10	90	2.59
95	1.94	95	2.31
100	1.57	100	2.04
		105	1.81
		110	1.61
		115	1.42
		120	1.27

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

Figure 18.5.1.2: ANE Gap Test Calibration Data



18.6 Series 8 Type (c) Test prescription

18.6.1 *Test 8 (c): Koenen test*

18.6.1.1 *Introduction*

This test is used to determine the sensitiveness of a candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosive, to the effect of intense heat under high confinement.

18.6.1.2 *Apparatus and materials*

18.6.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS G 3141). The dimensions are given in Figure 18.6.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with the following diameter holes: 1.0 - 1.5 - 2.0 - 2.5 - 3.0 - 5.0 - 8.0 - 12.0 - 20.0 mm. The dimensions of the threaded collar and the nut (closing device) are given in Figure 18.6.1.1.

For quality control of the steel tubes, 1% of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be 26.5 ± 1.5 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be 75 ± 0.5 mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be 0.5 ± 0.05 mm; and

(d) The bursting pressure as determined by quasi-static load through an incompressible fluid shall be 30 ± 3 MPa.

18.6.1.2.2 Heating is provided by propane, from an industrial cylinder fitted with a pressure regulator, via a flow meter and distributed by a manifold to the four burners. Other fuel gases may be used providing the specified heating rate is obtained. The gas pressure is regulated to give a heating rate of 3.3 ± 0.3 K/s when measured by the calibration procedure. Calibration involves heating a tube (fitted with a 1.5 mm orifice plate) filled with 27 cm^3 of dibutyl phthalate. The time taken for the temperature of the liquid (measured with a 1 mm diameter thermocouple centrally placed 43 mm below the rim of the tube) to rise from 135 °C to 285 °C is recorded and the heating rate calculated.

18.6.1.2.3 Because the tube is likely to be destroyed in the test, heating is undertaken in a protective welded box, the construction and dimensions of which are given in Figure 18.6.1.2. The tube is suspended between two rods placed through holes drilled in opposite walls of the box. The arrangement of the burners is given in Figure 18.6.1.2. The burners are lit simultaneously by a pilot flame or an electrical ignition device. ***The test apparatus is placed in a protective area***. Measures should be taken to ensure that any draughts does not affect the burner flames. Provision should be made for extracting any gases or smoke resulting from the test.

18.6.1.3 *Procedure*

18.6.1.3.1 The substance or mixture is loaded into the tube to a height of 60 mm taking particular care to prevent the formation of voids. The threaded collar is slipped onto the tube from below, the appropriate orifice plate is inserted and the nut tightened by hand after applying some molybdenum disulphide based lubricant. It is essential to check that none of the substance is trapped between the flange and the plate, or in the threads.

18.6.1.3.2 With orifice plates from 1.0 mm to 8.0 mm diameter, nuts with an orifice of 10.0 mm diameter should be used; if the diameter of the orifice is above 8.0 mm, that of the nut should be 20.0 mm. Each tube is used for one trial only. The orifice plates, threaded collars and nuts may be used again provided they are undamaged.

18.6.1.3.3 The tube is placed in a rigidly mounted vice and the nut tightened with a spanner. The tube is then suspended between the two rods in the protective box. The test area is vacated, the gas supply turned on and the burners lit. The time to reaction and duration of reaction can provide additional information useful in interpreting the results. If rupture of the tube does not occur, heating is to be continued for at least five minutes before the trial is finished. After each trial the fragments of the tube, if any, should be collected and weighed.

18.6.1.3.4 The following effects are differentiated:

"O": Tube unchanged;
"A": Bottom of tube bulged out;
"B": Bottom and wall of the tube bulged out;
"C": Bottom of tube split;
"D": Wall of tube split;
"E": Tube split into two[1] fragments;
"F": Tube fragmented into three[1] or more mainly large pieces which in some cases may be connected with each other by a narrow strip;
"G": Tube fragmented into many mainly small pieces, closing device undamaged; and
"H": Tube fragmented into many very small pieces, closing device bulged out or fragmented.

Examples for the effect types "D", "E" and "F" are shown in Figure 18.6.1.3. If a trial results in any of the effects "O" to "E", the result is regarded as "no explosion". If a trial gives the effect "F", "G" or "H", the result is evaluated as "explosion".

18.6.1.3.5 The series of trials is started with a single trial using an orifice plate of 20.0 mm. If, in this trial, the result "explosion" is observed, the series is continued with trials using tubes without orifice plates and nuts but with threaded collars (orifice 24.0 mm). If at 20.0 mm "no explosion" occurs, the series is continued with single trials using plates with the following orifices 12.0 - 8.0 - 5.0 - 3.0 - 2.0 - 1.5 and finally 1.0 mm until, at one of these diameters, the result "explosion" is obtained. Subsequently, trials are carried out at increasing diameters, according to the sequence given in 18.6.1.2.1, until only negative results in three tests at the same level are obtained. The limiting diameter of a substance or mixture is the largest diameter of the orifice at which the result "explosion" is obtained. If no "explosion" is obtained with a diameter of 1.0 mm, the limiting diameter is recorded as being less than 1.0 mm.

18.6.1.4 *Test criteria and method of assessing results*

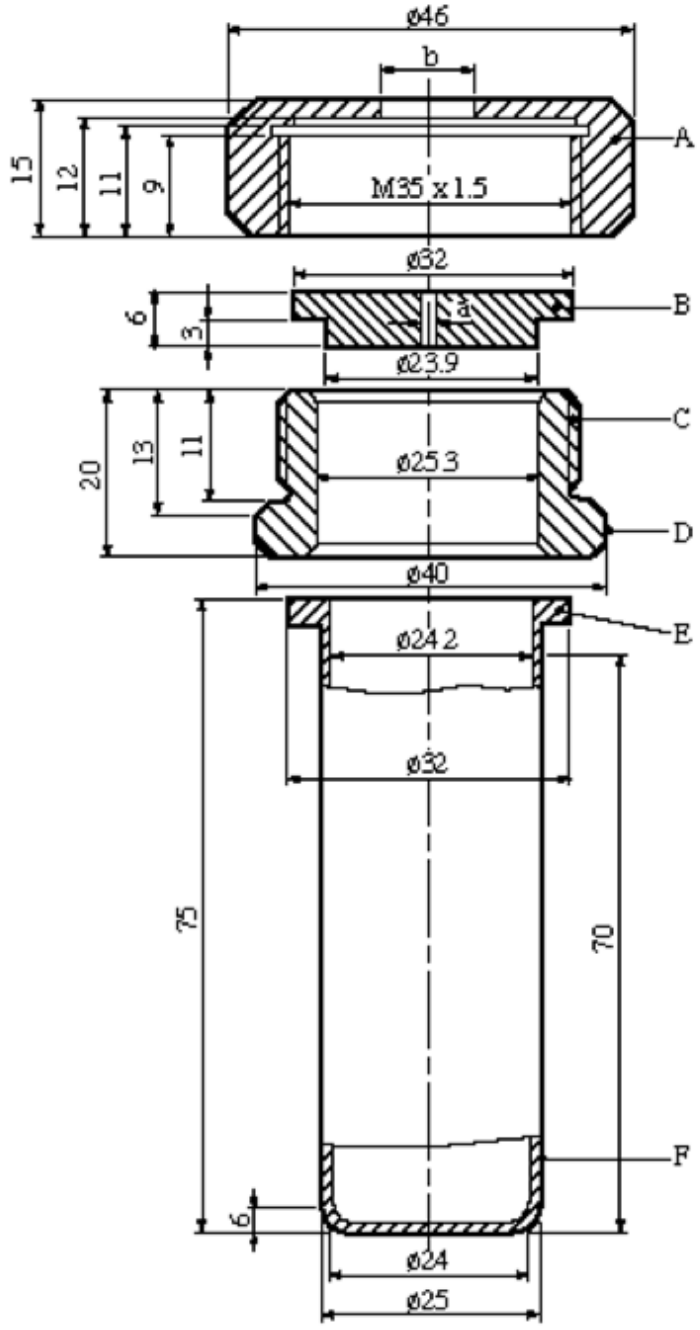
The result is considered "+" and the substance or mixture should not be classified in Division 5.1 if the limiting diameter is 2.0 mm or more. The result is considered "—" if the limiting diameter is less than 2.0 mm.

18.6.1.5 *Examples of results*

Substance~~s~~/Mixture	**Result**	**Comments**
Ammonium nitrate (low density)	-	Limiting diameter: <1 mm
ANE-F1 Ammonium nitrate 71%, Water 21%, Fuel/emulsifier 7%	-	
ANE-F2 Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	-	
ANE-F3 Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 7%	-	
ANE-F4 Ammonium nitrate 42%, Calcium nitrate 35%, Water 16%, Fuel/emulsifier 7%	-	
ANE-F5 Ammonium nitrate 69%, Sodium nitrate 13%, Water 10%, Fuel/emulsifier 8%	-	

[1] The upper part of the tube remaining in the closing device is counted as one fragment.

Substances/Mixture	Result	Comments
ANE-F6 Ammonium nitrate 72%, Sodium nitrate 11%, Water 10%, Fuel/emulsifier 6%	-	
ANE-F7 Ammonium nitrate 76%, Water 13%, Fuel/emulsifier 10%	-	
ANE-F8 Ammonium nitrate 77%, Water 16%, Fuel/emulsifier 6%	-	
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	-	Limiting diameter: 1.5 mm
ANE-2 (sensitized by microballoons) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	+	Limiting diameter: 2 mm
ANE-4 (sensitized by microballoons) Ammonium nitrate 70%, Sodium nitrate 11%, Water 9%, Fuel/emulsifier 5.5%	+	Limiting diameter: 2 mm
ANE-G1 Ammonium nitrate 74%, **Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%**	-	
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	-	
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	-	Effect type "O"
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	-	Effect type "O"
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	-	Effect type "A"



(A)	Nut (b =10.0 or 20.0 mm) with flats for size 41 spanner	(B)	Orifice plate (a = 1.0 to 20.0 mm diameter)
(C)	Threaded collar	(D)	Flats for size 36 spanner
(E)	Flange	(F)	Tube

Figure 18.6.1.1: Test tube assembly

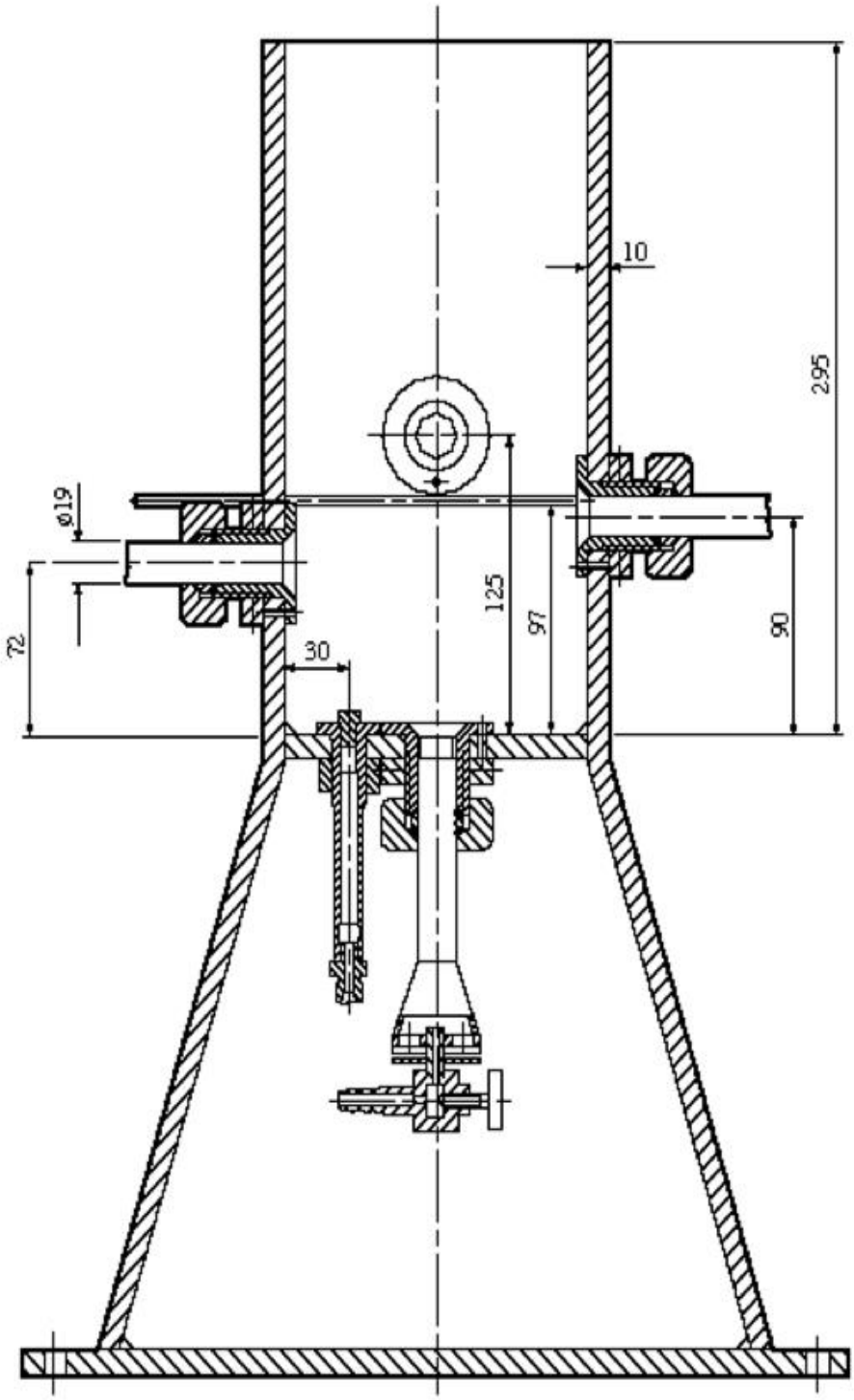


Figure 18.6.1.2: Heating and protective device

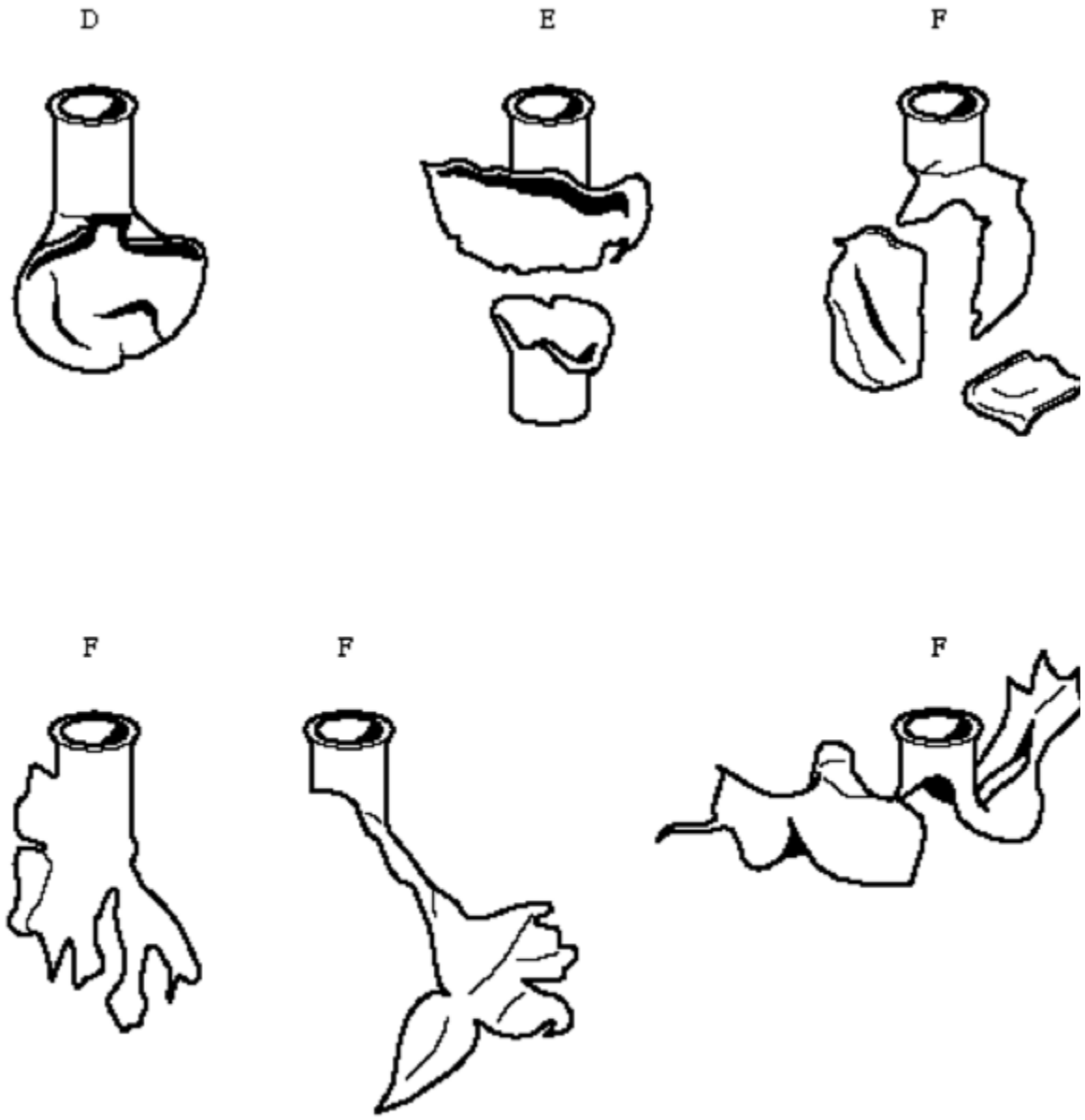


Figure 18.6.1.3: examples of effect types “D”, “E” and “F”

18.7 Series 8 Type (d) Test prescriptions

18.7.1 *Test 8 (d) (i): Vented pipe test*

18.7.1.1 *Introduction*

This test is not intended for classification but is included in this Manual for evaluating the suitability for transport in tanks.

The vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

18.7.1.2 *Apparatus and materials*

The following items are needed:

(a) A steel pipe 310 ± 10 mm diameter and 610 ± 10 mm long, welded close at the bottom with a 380 mm square, 10 ± 0.5 mm thick mild steel plate. The top of the pipe is welded to a 380 mm square, 10 ± 0.5 mm thick mild steel plate that contains a 78 mm diameter vent hole centrally located in the plate to which a 152 mm long steel pipe nipple of 78 mm internal diameter is welded (see Figure 18.7.1.1);

(b) A metal grid to support the filled pipe above the fuel and allow adequate heating. If a wooden crib fire is used, the grid should be 1.0 m above the ground and if a liquid hydrocarbon pool fire is used then the grid shall be 0.5 m above the ground;

(c) Enough fuel to keep a fire burning for at least 30 minutes or, if necessary, until the substance has clearly had enough time to react to the fire;

(d) Suitable means of ignition to ignite the fuel from two sides e.g. for a wood fire, kerosene to soak the wood and pyrotechnic igniters with wood wool;

(e) Cine or video cameras, preferably high speed and normal speed, to record events in colour;

(f) Blast gauges, radiometers and associated recording equipment may also be used.

18.7.1.3 *Procedure*

18.7.1.3.1 The pipe is filled with the substance or mixture under test without tamping during loading. The substance or mixture is carefully packed to prevent adding voids. The steel pipe is placed vertically on the grid and secured from tipping over. Fuel is placed beneath the grid so that the fire will engulf the pipe. Precautions against side winds may be required to avoid dissipation of the heat. Suitable methods of heating include a wood fire using a lattice of wooden laths, a liquid or gas fuel fire that produces a flame temperature of at least 800 °C.

18.7.1.3.2 One method is to use a wood fire which has a balanced air/fuel ratio, thereby avoiding too much smoke which would obscure the events, and which burns with sufficient intensity and duration to bring the substance to a possible reaction. A suitable method involves using air-dried pieces of wood (approximately 50 mm square section), stacked to form a lattice beneath the grid (1 m off the ground), and up to the base of the grid supporting the pipe. The wood should extend beyond the pipe to a distance of at least 1.0 m in every direction and the lateral distance between the laths should be about 100 mm.

18.7.1.3.3 A receptacle filled with suitable liquid fuel, a combination of both wood and liquid fuel fire may be used as an alternative to the wood fire providing it is as severe. If a liquid pool fire is used, the receptacle should extend beyond the pipe to a distance of at least 1.0 m in every direction. The distance between the grid platform and the receptacle should be approximately 0.5 m. Before using this method, consideration should be given to whether any quenching action or adverse interaction between the substance and the liquid fuel can occur such as might bring the results into question.

18.7.1.3.4 If gas is to be used as a fuel, the burning area must extend beyond the pipe to a distance of 1.0 m in every direction. The gas must be supplied in such a manner to ensure that the fire is evenly distributed around the pipe. The gas reservoir should be large enough to keep the fire burning for at least 30 minutes. Ignition of the gas may be accomplished either by remotely ignited pyrotechnics or by remote release of the gas adjacent to a pre-existing source of ignition.

18.7.1.3.5 The ignition system should be put into place and the fuel ignited on two sides, one up wind, simultaneously. The test should not be performed under conditions where the wind speed exceeds 6 m/s. ***The fire shall be started from a safe place. If the pipe does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up and emptying the pipe***.

18.7.1.3.6 Observations are made on the following:

(a) Evidence of explosion;

(b) Loud noise; and

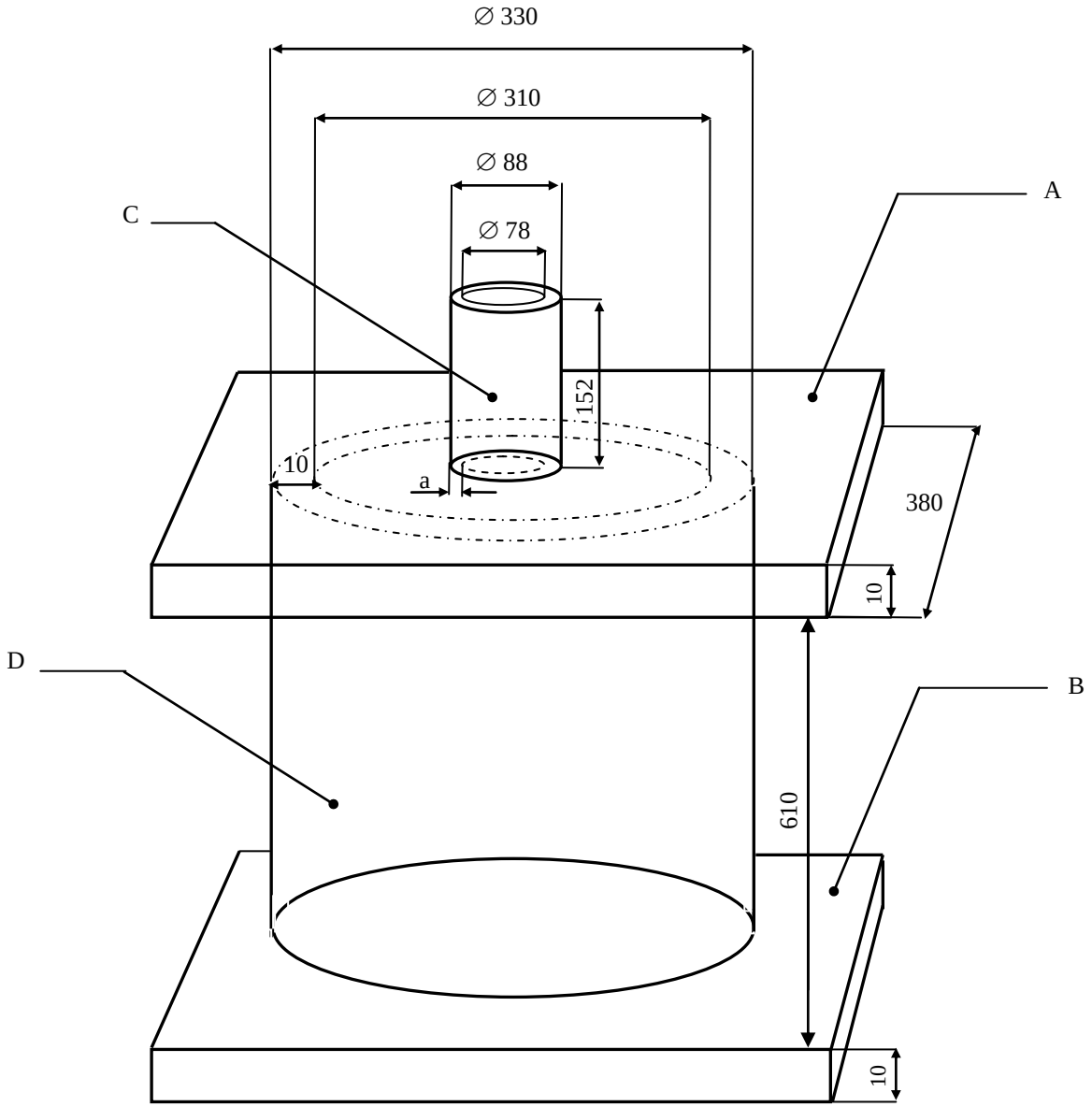
(c) Projection of fragments from the fire area.

18.7.1.4 *Test criteria and method of assessing results*

The test result is considered "+" and the substance or mixture should not be transported in tanks if an explosion and/or fragmentation of the pipe is observed. If no explosion and/or fragmentation of the pipe is observed then the result is considered "-".

18.7.1.5 *Examples of results*

Substance	**Result**
[to be added]	



All measurements are in millimetres.

(A)	Top plate (Schedule 40 carbon (A53 grade B))
(B)	Bottom plate (Schedule 40 carbon (A53 grade B))
(C)	Steel pipe nipple (a = 0.5 cm), Schedule 40 carbon (A53 grade B)
(D)	Steel pipe (Schedule 40 carbon (A53 grade B))

Figure 18.7.1.1: Vented pipe test

18.7.2 ***Test 8 (d) (ii): Modified vented pipe test***

18.7.2.1 *Introduction*

This test is not intended for classification but is included in this Manual for evaluating the suitability of bulk substances or mixtures to be transported in tanks.

The modified vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

18.7.2.2 *Apparatus and materials*

The following items are needed:

(a) A vented vessel consisting of mild drawn steel pipe with an inner diameter of 265 ± 10 mm, a length of 580 ± 10 mm and a wall thickness of 5.0 ± 0.5 mm. Both the top and the base plates are made from 300 mm square, 6.0 ± 0.5 mm thick mild steel plates. The top and base plates are fixed to the pipe with a fillet weld with a thickness of at least 5 mm. The top plate has a vent diameter of 85 mm ± 1.0 mm. A further two small holes are drilled in the top plate to accommodate neatly thermocouple probes;

(b) A concrete block about 400 mm square and 50 to 75 mm thick;

(c) A metal stand for supporting the vessel at a height of 150 mm above the concrete block;

(d) A gas burner capable of accommodating a propane flow rate of up to 60 g/min. This rests on the concrete block under the stand. A typical example of a suitable burner is a 32-jet Mongolian wok burner;

(e) A sheet metal shield to protect the propane flame from side winds. This can be fabricated from approximately 0.5 mm thick galvanised sheet metal. The diameter of the wind shield is 600 mm and the height is 250 mm. Four adjustable vents 150 mm wide and 100 mm high are spaced equally around the shield to ensure adequate air reaches the gas flame;

(f) Propane bottle(s) connected via a manifold and fed into a pressure regulator. Other fuel gases may be used providing the specified heating rate is obtained. The pressure regulator should reduce the propane bottle pressure from 600 kPa down to about 150 kPa. The gas then flows through a gas rotameter capable of measuring up to 60 g/min of propane and a needle valve. An electrical solenoid valve is used to switch the propane flow on and off remotely. Typically three 9 kg propane bottles will achieve the desired gas flow rate for the duration of up to five tests. The gas pressure and flow are regulated to give a heating rate of 3.3 ± 0.3 K/min when measured by the calibration procedure;

(g) Three thermocouples with 500 (2) and 100 (1) mm long stainless steel probes and fiber-glass coated lead wires;

(h) A data-logger capable of recording the output from the thermocouples;

(i) Cine-cameras or video cameras, preferably high speed and normal speed, to record events in colour;

(j) Pure water for calibration;

(k) The ANE to be tested.

Blast gauges, radiometers and associated recording equipment may also be used.

18.7.2.3 *Calibration*

18.7.2.3.1 The vessel is filled to the 75% level (i.e. to a depth of 435 mm) with the pure water, and heated using the procedure specified in 18.7.2.4. Water is heated from ambient temperature up to 90 ºC, monitoring temperature by the thermocouple in the water. Temperature-time data must fit a straight line whose slope will be the "calibration heating rate" for the given combination of vessel and heat source.

18.7.2.3.2 The gas pressure and flow must be regulated to give a heating rate of 3.3 ± 0.3 K/min.

18.7.2.3.3 This calibration must be performed prior to the testing of any ANE substance or mixture, though the same calibration can be applied to any test conducted within a day of the calibration provided no change is made to the vessel construction or gas supply. New calibration has to be made every time that the burner is changed.

18.7.2.4 *Procedure*

18.7.2.4.1 The concrete block is placed on a sandy base and levelled using a spirit level. The propane burner is positioned in the centre of the concrete block and connected to the gas supply line. The metal stand is placed over the burner.

18.7.2.4.2 The vessel is placed vertically on the stand and secured from tipping over. The vessel is filled to 75% of its volume (to a height of 435 mm) with the ANE under test without tamping during loading. The initial temperature of the ANE must be recorded. The substance or mixture is carefully packed to prevent adding voids. The wind shield is positioned around the base of the assembly to protect the propane flame from heat dissipation due to side winds.

18.7.2.4.3 The thermocouple positions are as follows:

(a) The first 500 mm long probe (T1) in the gas flame;

(b) The second 500 mm long probe (T2) extending all the way into the vessel so that the tip is positioned 80 to 90 mm from the bottom of the vessel;

(c) The third 100 mm long probe (T3) in the headspace 20 mm into the vessel.

The thermocouples are connected to the data-logger and the thermocouple leads and data-logger are adequately protected from the test apparatus in case of explosion.

18.7.2.4.4 Propane pressure and flow is checked and adjusted to the values used during the water calibration described in 18.7.2.3. Video cameras and any other recording equipment are checked and started. Thermocouple functioning is checked and data logging is started, with a time set between thermocouple readings not exceeding 10 seconds, and preferably shorter. The test should not be performed under conditions where the wind speed exceeds 6 m/s. With higher wind speed, additional precautions against side winds are required to avoid dissipation of the heat.

18.7.2.4.5 The propane burner may be started locally or remotely and all workers immediately retreat to a safe location. Progress of the test is followed by monitoring thermocouple readings and closed circuit television images. The start time of the trial is defined by the time at which the flame thermocouple trace T1 first begins to rise.

18.7.2.4.6 The gas reservoir should be large enough to bring the substance to a possible reaction and provide a fire duration lasting beyond total consumption of the test sample. If the vessel does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up.

18.7.2.4.7 The test outcome is determined by whether or not a rupture of the vessel is observed when the test reaches conclusion. Evidence of test conclusion is based on:

(a) The visual and aural observation of vessel rupture accompanied by loss of thermocouple traces;

(b) The visual and aural observation of vigorous venting accompanied by peaking of both vessel thermocouple traces and no substance remains in the vessel; or

(c) The visual observation of decreased levels of fuming following the peaking of both vessel thermocouple traces at temperatures in excess of 300 ºC and no substance remains in the vessel.

For the purposes of assessing results, the term “rupture” includes any failure of welds and any fracture of metal in the vessel.

18.7.2.4.8 The test is performed two times unless a positive result is observed.

18.7.2.5 *Test criteria and method of assessing results*

The test result is considered "+" and the substance or mixture should not be transported in tanks as an oxidizing liquid or solid (~~dangerous substance of~~ Division 5.1 for transport) if an explosion is observed in any trial. Explosion is evidenced by rupture of the vessel. Once the substance or mixture ~~–~~is consumed in both trials and no rupture of the vessel is observed, then the result is considered "–".

18.7.2.6 *Examples of results*

Substance/~~s~~Mixture	**Result**
76.0 ammonium nitrate/17.0 water/5.6 paraffin oil/1.4 PIBSA emulsifier	–
84.0 ammonium nitrate/9.0 water/5.6 paraffin oil/1.4 PIBSA emulsifier	+
67.7 ammonium nitrate/12.2 sodium nitrate/14.1 water/4.8 paraffin oil/1.2 PIBSA emulsifier	–
67.4 ammonium nitrate/15.0 methylamine nitrate/12.0 water/5.0 glycol/0.6 thickener	–
71.4 ammonium nitrate/14.0 hexamine nitrate/14.0 water/0.6 thickener	–

~~SECTION~~ CHAPTER 18

TEST SERIES 8

18.1 Introduction

The assessment whether a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives (ANE)" is insensitive enough ~~for inclusion~~to be classified as an oxidizing liquid or solid (~~in~~ Division 5.1 for transport) is answered by series 8 tests and any such candidate for inclusion in this hazard class ~~Division 5.1~~ should pass each of the three types of tests comprising the series. The three test types are:

Type 8 (a): a test to determine the thermal stability;
Type 8 (b): a shock test to determine sensitivity to intense shock;
Type 8 (c): a test to determine the effect of heating under confinement;

Test series 8 (d) has been included in this section as one method to evaluate the suitability for the transport in tanks.

18.2 Test methods

The test methods currently used are listed in Table 18.1.

Table 18.1: Test methods for Test Series 8

Test code	Name of Test	Section
8 (a)	Thermal Stability Test for ANE[a]	18.4
8 (b)	ANE Gap Test[a]	18.5
8 (c)	Koenen test[a]	18.6
8 (d)	Vented pipe tests[b]	18.7

[a] *This test is intended for classification.*
[b] *These tests are intended for evaluating the suitability for transport in tanks.*

18.3 Test conditions

18.3.1 The substance or mixture should be tested as offered for transport, at the highest transport temperature (see 1.5.4 of this Manual)[RG20].

18.4 Series 8 Type (a) test prescription

18.4.1 *Test 8 (a): Thermal stability test for ammonium nitrate emulsions, suspension or gels*

18.4.1.1 *Introduction*

18.4.1.1.1 This test is used to measure the stability of a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" when subjected to elevated thermal conditions to determine if the emulsion is too dangerous to transport[RG21].

18.4.1.1.2 This test is used to determine whether the emulsion, suspension or gel is stable at temperatures encountered during transport[RG22]. In the way this type of test is normally carried out (see 28.4.4), the 0.5 litre Dewar vessel is only representative for packagings, IBC's and small tanks. For the transport of ammonium nitrate emulsions, suspensions or gels the test can be used to measure its stability during tank transport if the test is carried out at a temperature 20 °C higher than the maximum temperature which may occur during transport, including the temperature at the time of loading.

18.4.1.2 *Apparatus and materials*

18.4.1.2.1 The experimental equipment consists of a suitable test chamber, appropriate Dewar vessels with closures, temperature probes and measuring equipment.

18.4.1.2.2 ***The test should be performed in a test cell capable of withstanding fire and overpressure and, preferably, should be fitted with a pressure relief system e.g. a blow out panel.*** The recording system should be housed in a separate observation area.

18.4.1.2.3 A thermostatically controlled drying oven (which may be fan-assisted) large enough to allow air circulation on all sides of the Dewar vessel may be used. The air temperature in the oven should be controlled so that the desired temperature for a liquid inert sample in the Dewar vessel can be maintained with a deviation of not more than ± 1 °C for up to 10 days. The air temperature in the oven should be measured and recorded. It is recommended that the door of the oven be fitted with a magnetic catch or replaced by a loosely fitting insulated cover. The oven may be protected by an appropriate steel liner and the Dewar vessel housed in a wire mesh cage.

18.4.1.2.4 Dewar vessels with a volume of 500 ml with a closure system are used. The closure of the Dewar vessel should be inert. A closure system is illustrated in Figure 18.4.1.1.

18.4.1.2.5 The heat loss characteristics of the system used, i.e. Dewar vessel and closure, should be established prior to performance of the test. Since the closure system has a significant effect on the heat loss characteristics, these can be adjusted to some extent by varying the closure system. The heat loss characteristics can be determined by measuring the half time of cooling of the vessel filled with an inert substance having similar physical properties. The heat loss per unit of mass, L (W/kg.K) can be calculated from the half time of cooling, $t_{1/2}$ (s), and the specific heat, Cp (J/kg.K), of the substance using the formula:

~~$L = \ln 2 \times C_p / t_{1/2}$~~

$$L = \ln 2 \times \left(C_p / t_{1/2} \right)$$

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.4.1.2.6 Dewar vessels filled with 400 ml of inert substance, with a heat loss of ~~80 to~~ 100 mW/kg.K or less are suitable.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.4.1.2.7 The Dewar vessel shall be filled to about 80% of its capacity. In case of a sample with very high viscosity it may be required to have the sample provided with a shape which just fits into the Dewar vessel. The diameter of such a preshaed sample shall be just under the inner diameter of the Dewar vessel. The hollow lower end of the Dewar vessel may be filled with an inert solid substance prior to loading the sample into the vessel to facilitate the use of cylindrically shaped sample substances.

18.4.1.3 *Procedure*

18.4.1.3.1 Set the test chamber at a temperature which is 20 °C higher than the maximum temperature which may occur during transport [RG23]or, if higher, the temperature at the time of loading. Fill the Dewar vessel with the substance under test and note the mass of the sample. Make sure the sample is filled to about 80% of its height. Insert the temperature probe into the centre of the sample. Seal the lid of the Dewar in place and insert the Dewar vessel in the test chamber, connect the temperature recording system and close the test chamber.

18.4.1.3.2 The sample is heated and the temperature of the sample and test chamber continuously monitored. The time is noted at which the sample temperature reaches a temperature 2 °C below the test chamber temperature. The test is then continued for a further seven days or until the sample temperature rises to 6 °C or more above the test chamber temperature if this occurs sooner. Note the time taken for the sample to rise from 2 °C below the test chamber temperature to its maximum temperature.

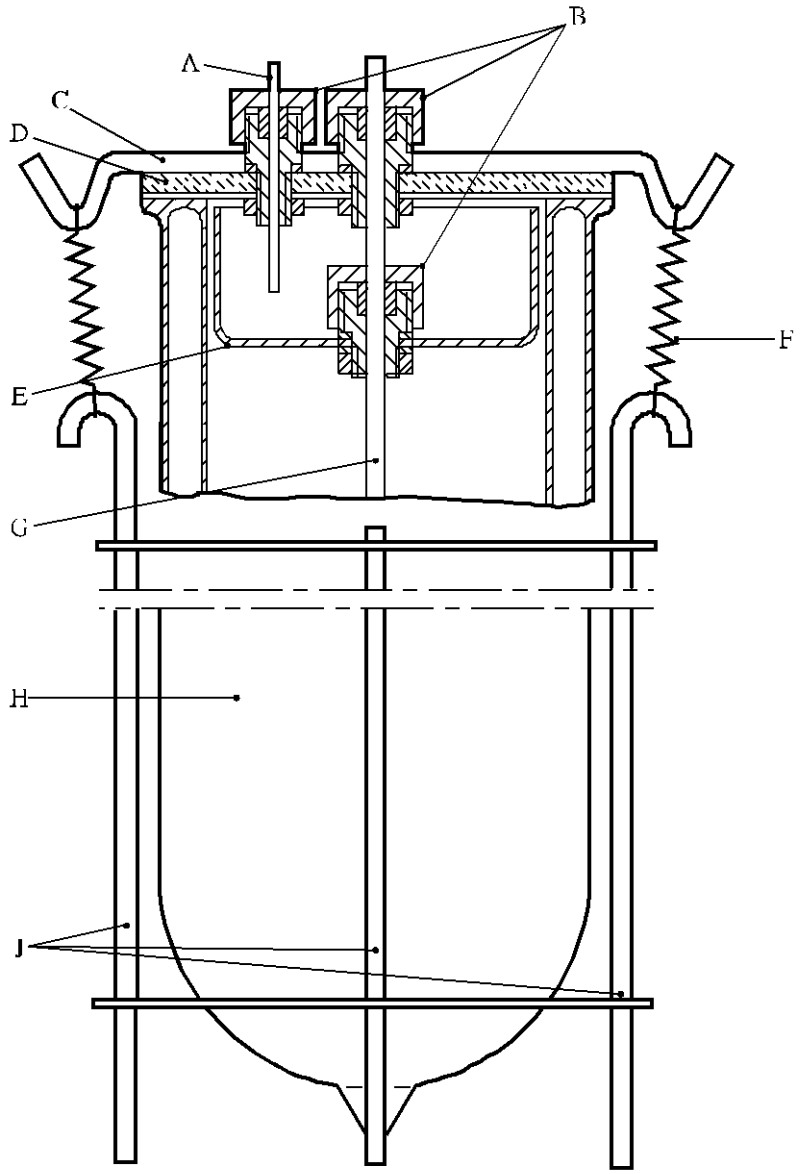
18.4.1.3.3 If the sample survives, cool and remove it from the test chamber and carefully dispose of it as soon as possible. The percentage mass loss and change in composition may be determined.

18.4.1.4 *Test criteria and method of assessing results*

18.4.1.4.1 If the sample temperature does not exceed the test chamber temperature by 6 °C or more in any test, the ammonium nitrate emulsion, suspension or gel is considered to be thermally stable and can be further tested as a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives".

18.4.1.5 *Examples of results*

Substances/Mixture	**Sample mass (g)**	**Test T (°C)**	**Result**	**Comments**
Ammonium nitrate	408	102	-	slight discolouration, hardened into lump Mass loss 0.5%
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	551	102	-	separation of oil and crystallized salts. Mass loss 0.8%
ANE-2 (sensitized) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	501	102	-	Some discolouration Mass loss 0.8%
ANE-Y Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	500	85	-	Mass loss 0.1%
ANE-Z Ammonium nitrate 75%, Water 20%, Fuel/emulsifier 5%	510	95	-	Mass loss 0.2%
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	553	85	-	no rise in temperature
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	540	85	-	no rise in temperature
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	613	80	-	Mass loss 0.1%
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	605	80	-	Mass loss 0.3%
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	602	80	-	Mass loss 0.1%



(A)	PTFE capillary tube	(B)	Special screw fittings (PTFE or Al) with O-ring seal
(C)	Metal strip	(D)	Glass lid
(E)	Glass beaker base	(F)	Spring
(G)	Glass protective tube	(H)	Dewar vessel
(J)	Steel retaining device		

Figure 18.4.1.1: DEWAR vessel with closure

18.5 Series 8 Type (b) Test prescription

18.5.1 *Test 8 (b): ANE Gap Test*

18.5.1.1 *Introduction*

This test is used to measure the sensitivity of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a specified shock level, i.e. a specified donor charge and gap.

18.5.1.2 *Apparatus and materials*

18.5.1.2.1 The set-up for this test consists of an explosive charge (donor), a barrier (gap), a container holding the test charge (acceptor), and a steel witness plate (target).

The following materials are to be used:

(a) United Nations Standard detonator or equivalent;

(b) 95 mm diameter by 95 mm long ~~pressed 50/50 pentolite or 95/5 RDX/WAX~~ pellet with a density of 1 600 kg/m^3 ± 50 kg/m^3 of either 50/50 pentolite or 95/5 RDX/WAX;
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(c) Tubing, steel, ~~cold drawn~~ seamless, with an outer diameter of 95.0 ± 7.0 mm, a wall thickness of 9.75 ± 2.75 mm and an inner diameter of 73.0 ± 7.0 mm, and with a length of 280 mm;
~~95 mm outer diameter, 11.1 mm wall thickness ± 10% variations, by 280 mm long having the following mechanical properties:~~

~~tensile strength – 420 MPa (± 20% variation)~~

~~elongation (%) – 22 (± 20% variation)~~

~~Brinell hardness – 125 (± 20% variation)~~
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(d) Sample substances, with a diameter which is just under the inner diameter of the steel tubing. The air gap between the sample and tubing wall should be as small as possible;

(e) ~~Cast p~~Polymethyl methacrylate (PMMA) rod, of 95 mm diameter by 70 mm long. A gap length of 70 mm results in ~~a~~ an incident shock pressure at the ANE interface ~~applied to the emulsion~~ somewhere between 3.5 and 4 GPa, depending on the type of donor used (see Table 18.5.1.1 and Figure 18.5.1.2);
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

(f) Mild steel plate, 200 mm × 200 mm × 20 mm~~, having the following mechanical properties:~~

~~tensile strength – 580 MPa (± 20% variation)~~

~~elongation (%) – 21 (± 20% variation)~~

~~Brinell hardness – 160 (± 20% variation)~~
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

~~(g) Cardboard tubing, 97 mm inner diameter by 443 mm long;~~

(~~h~~g) Wood block, 95 mm diameter and 25 mm thick, with a hole drilled through the centre to hold the detonator.
(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

18.5.1.3 *Procedure*

18.5.1.3.1 As shown in Figure 18.5.1.1, the detonator, donor, gap and acceptor charge are coaxially aligned above the centre of the witness plate. Care should be taken to ensure good contact between the detonator and donor, donor and gap and gap and acceptor charge. The test sample and booster should be at ambient temperature for the test.

18.5.1.3.2 To assist in collecting the remains of the witness plate, the whole assembly may be mounted over a container of water with at least a 10 cm air gap between the surface of the water and the bottom surface of the witness plate which should be supported along two edges only.

18.5.1.3.3 Alternative collection methods may be used but it is important to allow sufficient free space below the witness plate so as not to impede plate puncture. The test is performed three times unless a positive result is observed earlier.

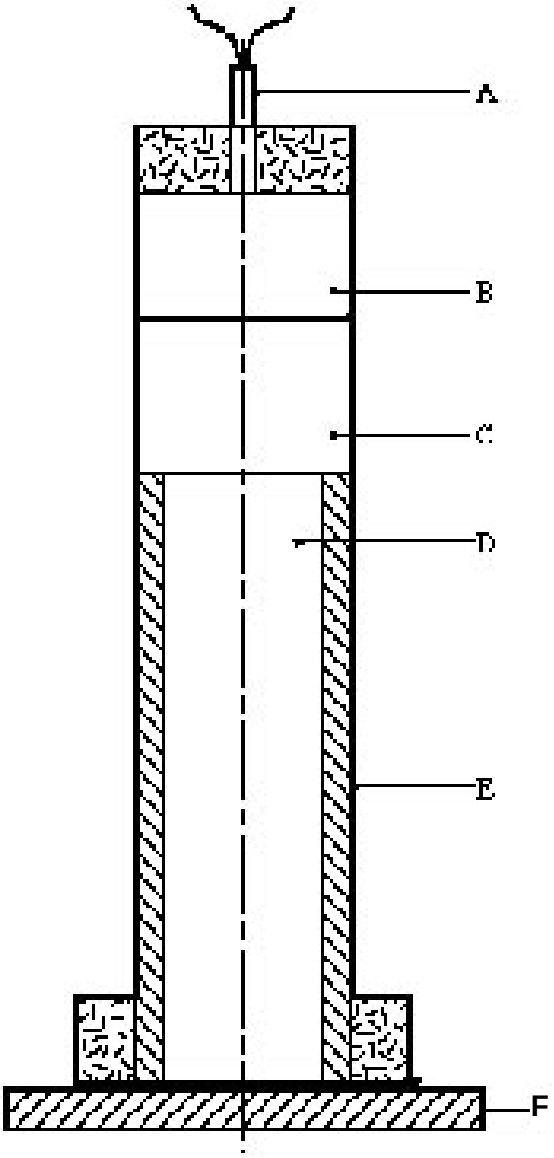
18.5.1.4 *Test criteria and method of assessing results*

A clean hole punched through the plate indicates that a detonation was initiated in the sample. A substance or mixture which detonates in any trial at a gap length of 70 mm is not to be classified as "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" and the result is noted as "+".

18.5.1.5 *Examples of results*

Substance~~s~~/Mixture	**Density g/cm^3**	**Gap mm**	**Result**	**Comments**
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate bent VOD 2.3-2.8 km/s
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate fractured
ANE-FA Ammonium nitrate 69%, Sodium nitrate 12%, Water 10%, Fuel/emulsifier 8%	1.4	50	-	Tube fragmented (large fragments) Plate not perforated
ANE-FA	1.44	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FB Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 7%	ca 1.40	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FC (sensitized) Ammonium nitrate 75%, Water 13%, Fuel/emulsifier 10%	1.17	70	+	Tube fragmented (fine fragments) Plate perforated
ANE-FD (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	ca 1.22	70	+	Tube fragmented (fine fragments) Plate perforated
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.4	35	-	Tube fragmented into large pieces. Plate dented VOD: 3.1 km/s
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	35	+	Tube fragmented into small pieces Plate perforated VOD: 6.7 km/s
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	70	+	Tube fragmented into small pieces Plate perforated VOD: 6.2 km/s
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%,	1.29	70	-	Tube fragmented Plate indented

Substances/Mixture	Density g/cm^3	Gap mm	Result	Comments
Fuel/emulsifier 9%				VOD 1 968 m/s
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	1.32	70	-	Tube fragmented Plate indented
ANE-G3 (sensitized by gassing) Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	1.17	70	+	Tube fragmented Plate punctured
ANE-G4 (sensitized by microballoons) Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	1.23	70	+	Tube fragmented Plate punctured
ANE-G5 Ammonium nitrate 70%, Calcium nitrate 8%, Water 16%, Fuel/emulsifier 7%	1.41	70	-	Tube fragmented Plate indented VOD 2 061 m/s
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	1.39	70	-	Tube fragmented Plate indented
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.42	70	-	Tube fragmented Plate indented
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	1.40	70	-	Tube fragmented Plate indented
ANE-J5 (sensitized by microballoons) Ammonium nitrate 71%, Sodium nitrate 5%, Water 18%, Fuel/emulsifier 6%	1.20	70	+	Tube fragmented Plate perforated VOD 5.7 km/s
ANE-J6 (sensitized by microballoons) Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	1.26	70	+	Tube fragmented Plate perforated VOD 6.3 km/s



(A)	Detonator	(B)	Booster charge
(C)	PMMA gap	(D)	Substance under test
(E)	Steel tube	(F)	Witness plate

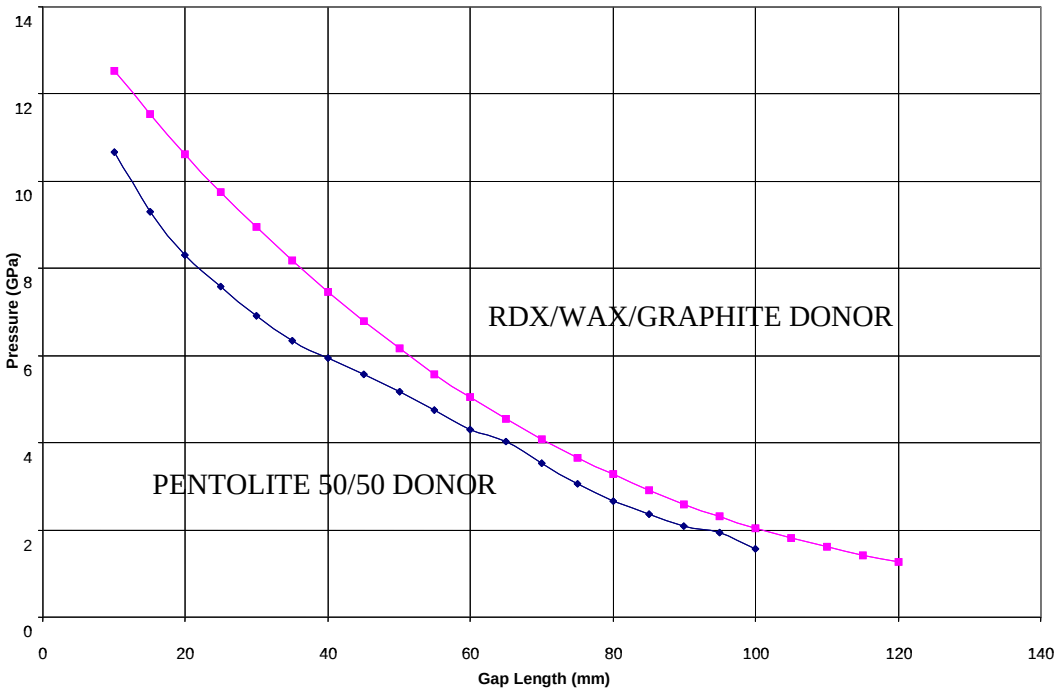
Figure 18.5.1.1: ANE gap test

Table 18.5.1.1: ANE gap test calibration data

PENTOLITE 50/50 DONOR		RDX/WAX/GRAPHITE DONOR	
Gap length (mm)	Barrier pressure (GPa)	Gap length (mm)	Barrier pressure (GPa)
10	10.67	10	12.53
15	9.31	15	11.55
20	8.31	20	10.63
25	7.58	25	9.76
30	6.91	30	8.94
35	6.34	35	8.18
40	5.94	40	7.46
45	5.56	45	6.79
50	5.18	50	6.16
55	~~4.76~~4.91	55	5.58
60	~~4.31~~4.51	60	5.04
65	4.02	65	4.54
70	3.53	70	4.08
75	3.05	75	3.66
80	2.66	80	3.27
85	2.36	85	2.91
90	2.10	90	2.59
95	1.94	95	2.31
100	1.57	100	2.04
		105	1.81
		110	1.61
		115	1.42
		120	1.27

(Ref.Doc: ST/SG/AC.10/11/Rev.5/Amend.2)

Figure 18.5.1.2: ANE Gap Test Calibration Data



18.6 Series 8 Type (c) Test prescription

18.6.1 *Test 8 (c): Koenen test*

18.6.1.1 *Introduction*

This test is used to determine the sensitiveness of a candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosive, to the effect of intense heat under high confinement.

18.6.1.2 *Apparatus and materials*

18.6.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS G 3141). The dimensions are given in Figure 18.6.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with the following diameter holes: 1.0 - 1.5 - 2.0 - 2.5 - 3.0 - 5.0 - 8.0 - 12.0 - 20.0 mm. The dimensions of the threaded collar and the nut (closing device) are given in Figure 18.6.1.1.

For quality control of the steel tubes, 1% of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be 26.5 ± 1.5 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be 75 ± 0.5 mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be 0.5 ± 0.05 mm; and

(d) The bursting pressure as determined by quasi-static load through an incompressible fluid shall be 30 ± 3 MPa.

18.6.1.2.2 Heating is provided by propane, from an industrial cylinder fitted with a pressure regulator, via a flow meter and distributed by a manifold to the four burners. Other fuel gases may be used providing the specified heating rate is obtained. The gas pressure is regulated to give a heating rate of 3.3 ± 0.3 K/s when measured by the calibration procedure. Calibration involves heating a tube (fitted with a 1.5 mm orifice plate) filled with 27 cm^3 of dibutyl phthalate. The time taken for the temperature of the liquid (measured with a 1 mm diameter thermocouple centrally placed 43 mm below the rim of the tube) to rise from 135 °C to 285 °C is recorded and the heating rate calculated.

18.6.1.2.3 Because the tube is likely to be destroyed in the test, heating is undertaken in a protective welded box, the construction and dimensions of which are given in Figure 18.6.1.2. The tube is suspended between two rods placed through holes drilled in opposite walls of the box. The arrangement of the burners is given in Figure 18.6.1.2. The burners are lit simultaneously by a pilot flame or an electrical ignition device. ***The test apparatus is placed in a protective area***. Measures should be taken to ensure that any draughts does not affect the burner flames. Provision should be made for extracting any gases or smoke resulting from the test.

18.6.1.3 *Procedure*

18.6.1.3.1 The substance or mixture is loaded into the tube to a height of 60 mm taking particular care to prevent the formation of voids. The threaded collar is slipped onto the tube from below, the appropriate orifice plate is inserted and the nut tightened by hand after applying some molybdenum disulphide based lubricant. It is essential to check that none of the substance is trapped between the flange and the plate, or in the threads.

18.6.1.3.2 With orifice plates from 1.0 mm to 8.0 mm diameter, nuts with an orifice of 10.0 mm diameter should be used; if the diameter of the orifice is above 8.0 mm, that of the nut should be 20.0 mm. Each tube is used for one trial only. The orifice plates, threaded collars and nuts may be used again provided they are undamaged.

18.6.1.3.3 The tube is placed in a rigidly mounted vice and the nut tightened with a spanner. The tube is then suspended between the two rods in the protective box. The test area is vacated, the gas supply turned on and the burners lit. The time to reaction and duration of reaction can provide additional information useful in interpreting the results. If rupture of the tube does not occur, heating is to be continued for at least five minutes before the trial is finished. After each trial the fragments of the tube, if any, should be collected and weighed.

18.6.1.3.4 The following effects are differentiated:

"O": Tube unchanged;
"A": Bottom of tube bulged out;
"B": Bottom and wall of the tube bulged out;
"C": Bottom of tube split;
"D": Wall of tube split;
"E": Tube split into two[1] fragments;
"F": Tube fragmented into three[1] or more mainly large pieces which in some cases may be connected with each other by a narrow strip;
"G": Tube fragmented into many mainly small pieces, closing device undamaged; and
"H": Tube fragmented into many very small pieces, closing device bulged out or fragmented.

Examples for the effect types "D", "E" and "F" are shown in Figure 18.6.1.3. If a trial results in any of the effects "O" to "E", the result is regarded as "no explosion". If a trial gives the effect "F", "G" or "H", the result is evaluated as "explosion".

18.6.1.3.5 The series of trials is started with a single trial using an orifice plate of 20.0 mm. If, in this trial, the result "explosion" is observed, the series is continued with trials using tubes without orifice plates and nuts but with threaded collars (orifice 24.0 mm). If at 20.0 mm "no explosion" occurs, the series is continued with single trials using plates with the following orifices 12.0 - 8.0 - 5.0 - 3.0 - 2.0 - 1.5 and finally 1.0 mm until, at one of these diameters, the result "explosion" is obtained. Subsequently, trials are carried out at increasing diameters, according to the sequence given in 18.6.1.2.1, until only negative results in three tests at the same level are obtained. The limiting diameter of a substance or mixture is the largest diameter of the orifice at which the result "explosion" is obtained. If no "explosion" is obtained with a diameter of 1.0 mm, the limiting diameter is recorded as being less than 1.0 mm.

18.6.1.4 *Test criteria and method of assessing results*

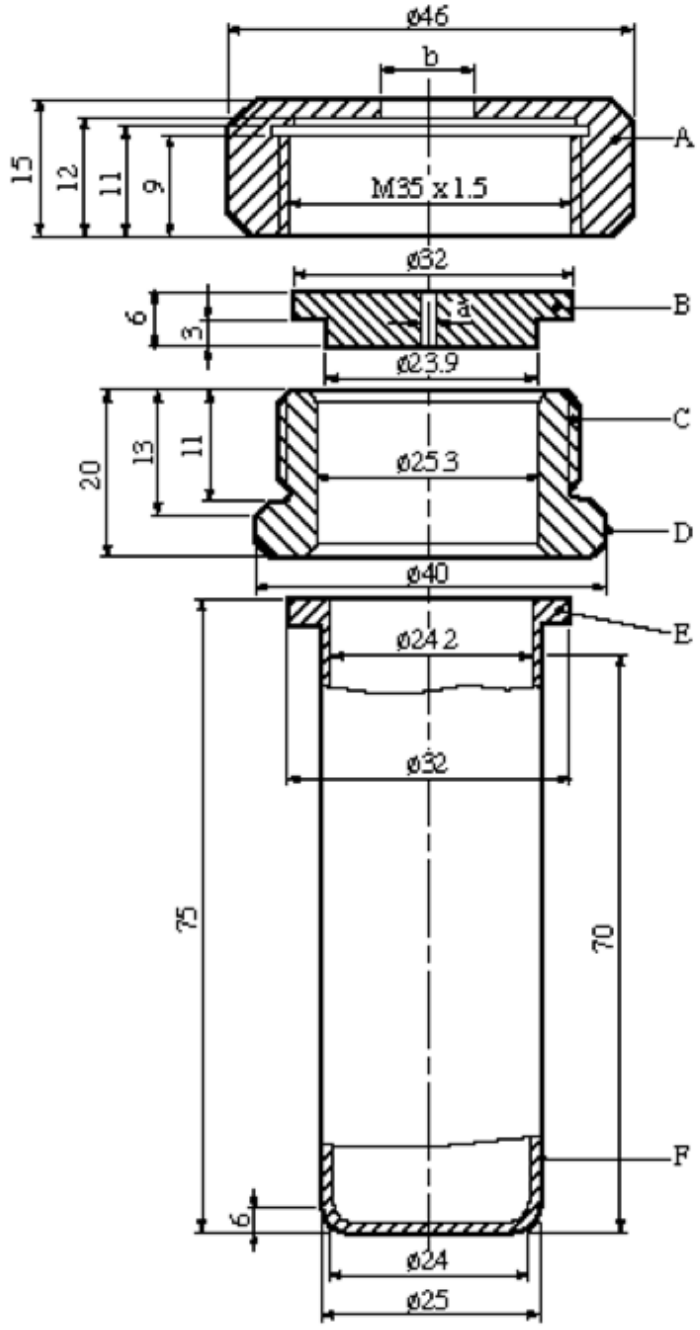
The result is considered "+" and the substance or mixture should not be classified in Division 5.1 if the limiting diameter is 2.0 mm or more. The result is considered "—" if the limiting diameter is less than 2.0 mm.

18.6.1.5 *Examples of results*

Substance~~s~~/Mixture	**Result**	**Comments**
Ammonium nitrate (low density)	-	Limiting diameter: <1 mm
ANE-F1 Ammonium nitrate 71%, Water 21%, Fuel/emulsifier 7%	-	
ANE-F2 Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	-	
ANE-F3 Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 7%	-	
ANE-F4 Ammonium nitrate 42%, Calcium nitrate 35%, Water 16%, Fuel/emulsifier 7%	-	

[1] The upper part of the tube remaining in the closing device is counted as one fragment.

Substance~~s~~/Mixture	Result	Comments
ANE-F5 Ammonium nitrate 69%, Sodium nitrate 13%, Water 10%, Fuel/emulsifier 8%	-	
ANE-F6 Ammonium nitrate 72%, Sodium nitrate 11%, Water 10%, Fuel/emulsifier 6%	-	
ANE-F7 Ammonium nitrate 76%, Water 13%, Fuel/emulsifier 10%	-	
ANE-F8 Ammonium nitrate 77%, Water 16%, Fuel/emulsifier 6%	-	
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	-	Limiting diameter: 1.5 mm
ANE-2 (sensitized by microballoons) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	+	Limiting diameter: 2 mm
ANE-4 (sensitized by microballoons) Ammonium nitrate 70%, Sodium nitrate 11%, Water 9%, Fuel/emulsifier 5.5%	+	Limiting diameter: 2 mm
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	-	
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	-	
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	-	Effect type "O"
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	-	Effect type "O"
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%	-	Effect type "A"



(A)	Nut (b =10.0 or 20.0 mm) with flats for size 41 spanner	(B)	Orifice plate (a = 1.0 to 20.0 mm diameter)
(C)	Threaded collar	(D)	Flats for size 36 spanner
(E)	Flange	(F)	Tube

Figure 18.6.1.1: Test tube assembly

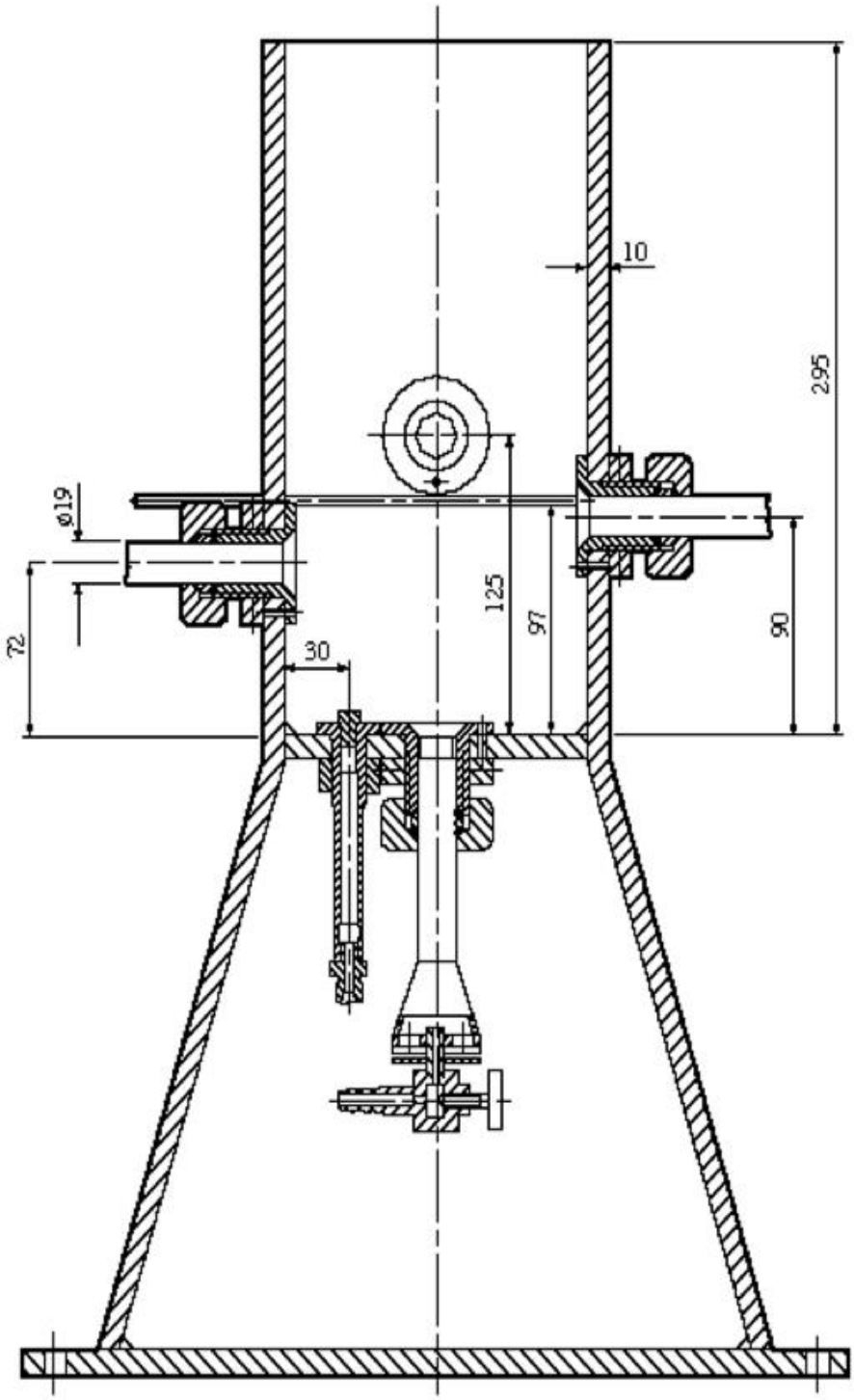


Figure 18.6.1.2: Heating and protective device

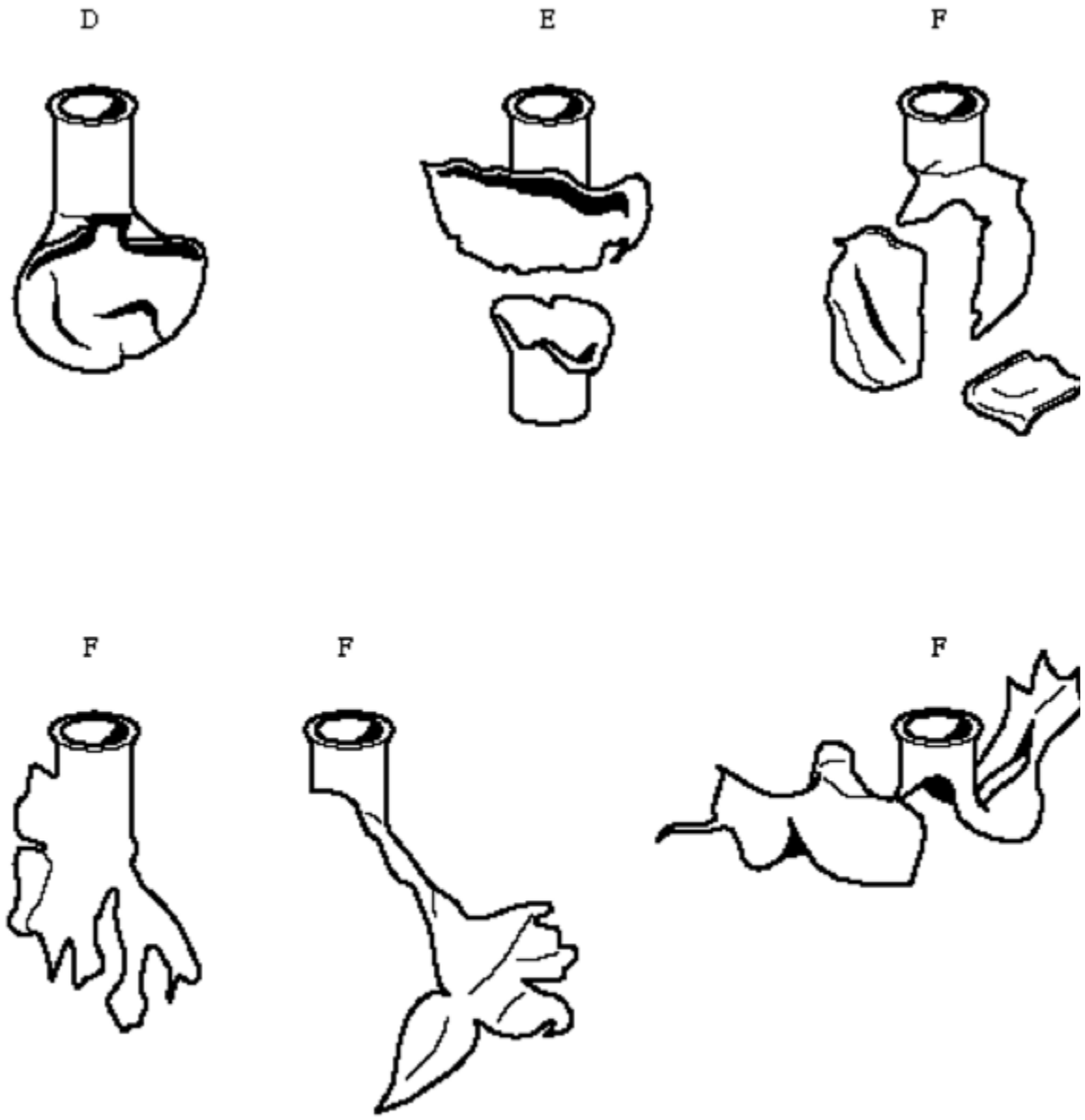


Figure 18.6.1.3: examples of effect types “D”, “E” and “F”

18.7 Series 8 Type (d) Test prescriptions

18.7.1 *Test 8 (d) (i): Vented pipe test*

18.7.1.1 *Introduction*

This test is not intended for classification but is included in this Manual for evaluating the suitability for transport in tanks.

The vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

18.7.1.2 *Apparatus and materials*

The following items are needed:

(a) A steel pipe 310 ± 10 mm diameter and 610 ± 10 mm long, welded close at the bottom with a 380 mm square, 10 ± 0.5 mm thick mild steel plate. The top of the pipe is welded to a 380 mm square, 10 ± 0.5 mm thick mild steel plate that contains a 78 mm diameter vent hole centrally located in the plate to which a 152 mm long steel pipe nipple of 78 mm internal diameter is welded (see Figure 18.7.1.1);

(b) A metal grid to support the filled pipe above the fuel and allow adequate heating. If a wooden crib fire is used, the grid should be 1.0 m above the ground and if a liquid hydrocarbon pool fire is used then the grid shall be 0.5 m above the ground;

(c) Enough fuel to keep a fire burning for at least 30 minutes or, if necessary, until the substance has clearly had enough time to react to the fire;

(d) Suitable means of ignition to ignite the fuel from two sides e.g. for a wood fire, kerosene to soak the wood and pyrotechnic igniters with wood wool;

(e) Cine or video cameras, preferably high speed and normal speed, to record events in colour;

(f) Blast gauges, radiometers and associated recording equipment may also be used.

18.7.1.3 *Procedure*

18.7.1.3.1 The pipe is filled with the substance or mixture under test without tamping during loading. The substance or mixture is carefully packed to prevent adding voids. The steel pipe is placed vertically on the grid and secured from tipping over. Fuel is placed beneath the grid so that the fire will engulf the pipe. Precautions against side winds may be required to avoid dissipation of the heat. Suitable methods of heating include a wood fire using a lattice of wooden laths, a liquid or gas fuel fire that produces a flame temperature of at least 800 °C.

18.7.1.3.2 One method is to use a wood fire which has a balanced air/fuel ratio, thereby avoiding too much smoke which would obscure the events, and which burns with sufficient intensity and duration to bring the substance to a possible reaction. A suitable method involves using air-dried pieces of wood (approximately 50 mm square section), stacked to form a lattice beneath the grid (1 m off the ground), and up to the base of the grid supporting the pipe. The wood should extend beyond the pipe to a distance of at least 1.0 m in every direction and the lateral distance between the laths should be about 100 mm.

18.7.1.3.3 A receptacle filled with suitable liquid fuel, a combination of both wood and liquid fuel fire may be used as an alternative to the wood fire providing it is as severe. If a liquid pool fire is used, the receptacle should extend beyond the pipe to a distance of at least 1.0 m in every direction. The distance between the grid platform and the receptacle should be approximately 0.5 m. Before using this method, consideration should be given to whether any quenching action or adverse interaction between the substance and the liquid fuel can occur such as might bring the results into question.

18.7.1.3.4 If gas is to be used as a fuel, the burning area must extend beyond the pipe to a distance of 1.0 m in every direction. The gas must be supplied in such a manner to ensure that the fire is evenly distributed around the pipe. The gas reservoir should be large enough to keep the fire burning for at least 30 minutes. Ignition of the gas may be accomplished either by remotely ignited pyrotechnics or by remote release of the gas adjacent to a pre-existing source of ignition.

18.7.1.3.5 The ignition system should be put into place and the fuel ignited on two sides, one up wind, simultaneously. The test should not be performed under conditions where the wind speed exceeds 6 m/s. ***The fire shall be started from a safe place. If the pipe does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up and emptying the pipe***.

18.7.1.3.6 Observations are made on the following:

(a) Evidence of explosion;

(b) Loud noise; and

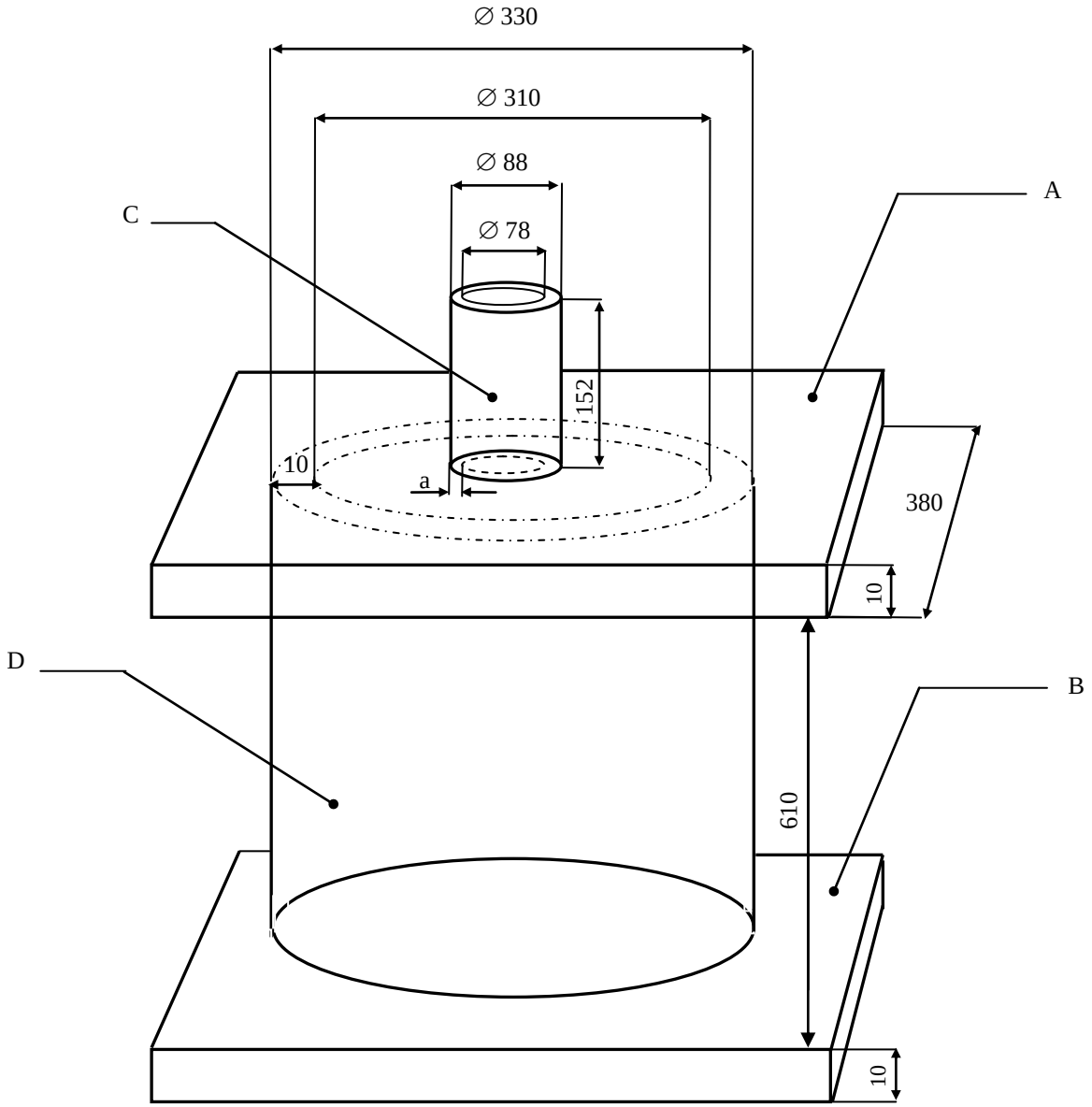
(c) Projection of fragments from the fire area.

18.7.1.4 *Test criteria and method of assessing results*

The test result is considered "+" and the substance or mixture should not be transported in tanks if an explosion and/or fragmentation of the pipe is observed. If no explosion and/or fragmentation of the pipe is observed then the result is considered "-".

18.7.1.5 *Examples of results*

Substance	**Result**
[to be added]	



All measurements are in millimetres.

(A)	Top plate (Schedule 40 carbon (A53 grade B))
(B)	Bottom plate (Schedule 40 carbon (A53 grade B))
(C)	Steel pipe nipple (a = 0.5 cm), Schedule 40 carbon (A53 grade B)
(D)	Steel pipe (Schedule 40 carbon (A53 grade B))

Figure 18.7.1.1: Vented pipe test

18.7.2 *Test 8 (d) (ii): Modified vented pipe test*

18.7.2.1 *Introduction*

This test is not intended for classification but is included in this Manual for evaluating the suitability of bulk substances or mixtures to be transported in tanks.

The modified vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

18.7.2.2 *Apparatus and materials*

The following items are needed:

(a) A vented vessel consisting of mild drawn steel pipe with an inner diameter of 265 ± 10 mm, a length of 580 ± 10 mm and a wall thickness of 5.0 ± 0.5 mm. Both the top and the base plates are made from 300 mm square, 6.0 ± 0.5 mm thick mild steel plates. The top and base plates are fixed to the pipe with a fillet weld with a thickness of at least 5 mm. The top plate has a vent diameter of 85 mm ± 1.0 mm. A further two small holes are drilled in the top plate to accommodate neatly thermocouple probes;

(b) A concrete block about 400 mm square and 50 to 75 mm thick;

(c) A metal stand for supporting the vessel at a height of 150 mm above the concrete block;

(d) A gas burner capable of accommodating a propane flow rate of up to 60 g/min. This rests on the concrete block under the stand. A typical example of a suitable burner is a 32-jet Mongolian wok burner;

(e) A sheet metal shield to protect the propane flame from side winds. This can be fabricated from approximately 0.5 mm thick galvanised sheet metal. The diameter of the wind shield is 600 mm and the height is 250 mm. Four adjustable vents 150 mm wide and 100 mm high are spaced equally around the shield to ensure adequate air reaches the gas flame;

(f) Propane bottle(s) connected via a manifold and fed into a pressure regulator. Other fuel gases may be used providing the specified heating rate is obtained. The pressure regulator should reduce the propane bottle pressure from 600 kPa down to about 150 kPa. The gas then flows through a gas rotameter capable of measuring up to 60 g/min of propane and a needle valve. An electrical solenoid valve is used to switch the propane flow on and off remotely. Typically three 9 kg propane bottles will achieve the desired gas flow rate for the duration of up to five tests. The gas pressure and flow are regulated to give a heating rate of 3.3 ± 0.3 K/min when measured by the calibration procedure;

(g) Three thermocouples with 500 (2) and 100 (1) mm long stainless steel probes and fiber-glass coated lead wires;

(h) A data-logger capable of recording the output from the thermocouples;

(i) Cine-cameras or video cameras, preferably high speed and normal speed, to record events in colour;

(j) Pure water for calibration;

(k) The ANE to be tested.

Blast gauges, radiometers and associated recording equipment may also be used.

18.7.2.3 *Calibration*

18.7.2.3.1 The vessel is filled to the 75% level (i.e. to a depth of 435 mm) with the pure water, and heated using the procedure specified in 18.7.2.4. Water is heated from ambient temperature up to 90 ºC, monitoring temperature by the thermocouple in the water. Temperature-time data must fit a straight line whose slope will be the "calibration heating rate" for the given combination of vessel and heat source.

18.7.2.3.2 The gas pressure and flow must be regulated to give a heating rate of 3.3 ± 0.3 K/min.

18.7.2.3.3 This calibration must be performed prior to the testing of any ANE substance or mixture, though the same calibration can be applied to any test conducted within a day of the calibration provided no change is made to the vessel construction or gas supply. New calibration has to be made every time that the burner is changed.

18.7.2.4 *Procedure*

18.7.2.4.1 The concrete block is placed on a sandy base and levelled using a spirit level. The propane burner is positioned in the centre of the concrete block and connected to the gas supply line. The metal stand is placed over the burner.

18.7.2.4.2 The vessel is placed vertically on the stand and secured from tipping over. The vessel is filled to 75% of its volume (to a height of 435 mm) with the ANE under test without tamping during loading. The initial temperature of the ANE must be recorded. The substance or mixture is carefully packed to prevent adding voids. The wind shield is positioned around the base of the assembly to protect the propane flame from heat dissipation due to side winds.

18.7.2.4.3 The thermocouple positions are as follows:

(a) The first 500 mm long probe (T1) in the gas flame;

(b) The second 500 mm long probe (T2) extending all the way into the vessel so that the tip is positioned 80 to 90 mm from the bottom of the vessel;

(c) The third 100 mm long probe (T3) in the headspace 20 mm into the vessel.

The thermocouples are connected to the data-logger and the thermocouple leads and data-logger are adequately protected from the test apparatus in case of explosion.

18.7.2.4.4 Propane pressure and flow is checked and adjusted to the values used during the water calibration described in 18.7.2.3. Video cameras and any other recording equipment are checked and started. Thermocouple functioning is checked and data logging is started, with a time set between thermocouple readings not exceeding 10 seconds, and preferably shorter. The test should not be performed under conditions where the wind speed exceeds 6 m/s. With higher wind speed, additional precautions against side winds are required to avoid dissipation of the heat.

18.7.2.4.5 The propane burner may be started locally or remotely and all workers immediately retreat to a safe location. Progress of the test is followed by monitoring thermocouple readings and closed circuit television images. The start time of the trial is defined by the time at which the flame thermocouple trace T1 first begins to rise.

18.7.2.4.6 The gas reservoir should be large enough to bring the substance to a possible reaction and provide a fire duration lasting beyond total consumption of the test sample. If the vessel does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up.

18.7.2.4.7 The test outcome is determined by whether or not a rupture of the vessel is observed when the test reaches conclusion. Evidence of test conclusion is based on:

(a) The visual and aural observation of vessel rupture accompanied by loss of thermocouple traces;

(b) The visual and aural observation of vigorous venting accompanied by peaking of both vessel thermocouple traces and no substance remains in the vessel; or

(c) The visual observation of decreased levels of fuming following the peaking of both vessel thermocouple traces at temperatures in excess of 300 ºC and no substance remains in the vessel.

For the purposes of assessing results, the term “rupture” includes any failure of welds and any fracture of metal in the vessel.

18.7.2.4.8 The test is performed two times unless a positive result is observed.

18.7.2.5 *Test criteria and method of assessing results*

The test result is considered "+" and the substance or mixture should not be transported in tanks as an oxidizing liquid or solid (~~dangerous substance of~~ Division 5.1 for transport) if an explosion is observed in any trial. Explosion is evidenced by rupture of the vessel. Once the substance or mixture ~~–~~is consumed in both trials and no rupture of the vessel is observed, then the result is considered "–".

18.7.2.6 *Examples of results*

Substance~~/s~~Mixture	**Result**
76.0 ammonium nitrate/17.0 water/5.6 paraffin oil/1.4 PIBSA emulsifier	–
84.0 ammonium nitrate/9.0 water/5.6 paraffin oil/1.4 PIBSA emulsifier	+
67.7 ammonium nitrate/12.2 sodium nitrate/14.1 water/4.8 paraffin oil/1.2 PIBSA emulsifier	–
67.4 ammonium nitrate/15.0 methylamine nitrate/12.0 water/5.0 glycol/0.6 thickener	–
71.4 ammonium nitrate/14.0 hexamine nitrate/14.0 water/0.6 thickener	–