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COMMITTEE OF EXPERTS ON THE TRANSPORT OF DANGEROUS GOODS AND ON THE GLOBALLY HARMONIZED SYSTEM OF CLASSIFICATION AND LABELLING OF CHEMICALS

Sub-committee of Experts on the Transport of Dangerous Goods

REPORT OF THE SUB-COMMITTEE OF EXPERTS ON ITS NINETEENTH SESSION

(Geneva, 2-6 July 2001)

Addendum 1

Draft amendments to the Recommendations on the Transport of Dangerous Goods (Model Regulations and Manual of Tests and Criteria)

This addendum contains the draft amendments to the Model Regulations on the Transport of Dangerous Goods (as annexed to the twelfth revised edition of the Recommendations on the Transport of Dangerous Goods, ST/SG/AC.10/1/Rev.12) and to the Manual of Tests and Criteria (ST/SG/AC.10/11/Rev.3) adopted by the Sub-Committee of Experts at its nineteenth session.

It contains two annexes:

Annex 1: Draft amendments to the Model Regulations on the Transport of Dangerous Goods;

Annex 2: Draft amendments to the Manual of Test and Criteria.

ANNEX 1

DRAFT AMENDMENTS TO THE MODEL REGULATIONS ANNEXED TO THE TWELFTH REVISED EDITION OF THE UNITED NATIONS RECOMMENDATIONS ON THE TRANSPORT OF DANGEROUS GOODS (ST/SG/AC.10/1/Rev.12)

PART 1

Chapter 1.2

1.2.1 In the definition of "portable tank", subparagraph (a), delete the words "with a capacity of more than 450 litres".

In the definition of "tank", delete the words "with a capacity of not less than 450 litres" and add at the end "and has a capacity of not less than 450 litres when used for the transport of substances of Class 2.".

PART 3

Chapter 3.2

Dangerous Goods List

In the Dangerous Goods List, assign "TP5" in column (11) to each refrigerated liquefied gas that is assigned "T75".

UN 1010 Add the following text at the end of the existing name in column (2): "or BUTADIENES AND HYDROCARBON MIXTURE, STABILIZED, containing more than 40% butadienes".

UN 1605 Replace "P601" with "P602" in column 8.

UN 2857 Amend the name in column (2) to read as follows: "Refrigerating machines containing non-flammable, non-toxic gases or ammonia solutions (UN 2672)".

UN 3375 Delete "306" in column (6).

Chapter 3.3

SP 215 Add the following text at the end:

"Homogeneous mixtures containing not more than 35 % by mass of azocarbonamide and at least 65 % of inert substance are not subject to these Regulations unless criteria of other classes or divisions are met.".

SP 247 Delete: "deviating for the requirements of Chapter 6.1," in the first paragraph.

SP 309 Amend last sentence to read as follows:

"Substances shall satisfactorily pass Test Series 8 of the *Manual of Tests and Criteria*, Part I, Section 18."

PART 4

Chapter 4.1

4.1.3.5 In the first sentence, delete "outer" (twice) and "in a combination packaging" and add ";1A2" after "4G" in the example between brackets.

In the second sentence, add ";1A2V, 1A2U or 1A2W" after "4GW" in the example between brackets.

4.1.4.1 For P001, delete "(2C1 and 2C2)" for special packing provision PP2.

For P200, in "Table 2: Liquefied and dissolved gases", for UN No. 1010:

- Under "name and description", replace "BUTADIENE, STABILIZED (mixtures of 1,3-butadiene and hydrocarbons)" with "BUTADIENES AND HYDROCARBON MIXTURE, STABILIZED, containing more than 40% butadienes".

- Under "Test pressure, bar" and "Filling ratio", delete "10" and "0.50" respectively;

- Under "Special packing provisions", add "v,"

For Packing instruction P601, add a new special packing provision PP82 to read as follows:

"PP82 For UN No.1744, glass inner packagings with a capacity of not more than 1.3 litres may be used in a permitted outer packaging with a maximum gross mass of 25 kg.".

Chapter 4.2

4.2.5.2.5 For portable tank instructions T2 and T4, delete "T6" under "Portable tank instructions also permitted".

4.2.5.2.6 Insert the following paragraph after the title:

"Portable tank instructions specify the requirements applicable to a portable tank when used for the transport of specific substances. Portable tank instructions T1 to T22 specify the applicable minimum test pressure, the minimum shell thickness (in mm reference steel), and the pressure-relief and bottom-opening requirements."

In the table for portable tank instruction "T1-T22" add a reference "*" to a footnote at the end of the heading "Pressure-relief requirements". The footnote will read as follows:

"* *When the word "Normal" is indicated all the requirements of 6.7.2.8 apply except for 6.7.2.8.3.".*

T50 In the table for portable tank instruction "T50":

- Add a reference "*" to a footnote at the end of the heading "Pressure-relief requirements", and a footnote to read as follows:

  " The word "Normal" in the pressure relief requirements column indicates that a frangible disc as specified in 6.7.3.7.3 is not required.".*

- In the heading "Max. allowable working pressure (bar) Small, Bare; Sunshield; Insulated", add at the end "respectively**" and a footnote to read as follows:

  *"** "Small" means tanks having a shell with a diameter of 1.5 metres or less; "Bare" means tanks having a shell with a diameter of more than 1.5 metres without insulation or sun shield (see 6.7.3.2.12); "Sunshield" means tanks having a shell with a diameter of more than 1.5 metres with sun shield (see 6.7.3.2.12); "Insulated" means tanks having a shell with a diameter of more than 1.5 metres with insulation (see 6.7.3.2.12); (See definition of "Design reference temperature" in 6.7.3.1)."*

- Add a new row as follows:

UN No.	**Non-refrigerated liquefied gases**	**Max. allowable working pressure (bar) Small; Bare; Sunshield; Insulated**	**Openings below liquid level**	**Pressure-relief requirements (see 6.7.3.7)**	**Maximum filling ratio**
1010	Butadienes and hydrocarbon mixture, stabilized	See MAWP definition in 6.7.3.1	Allowed	Normal	See 4.2.2.7

4.2.5.3 Amend TP5 to read as follows: "The degree of filling prescribed in 4.2.3.6 shall be met".

PART 5

Chapter 5.2

5.2.2.1.6 Amend the beginning of this paragraph to read:

"Except as provided in 5.2.2.2.1.2, each label shall:"

5.2.2.2.1.2 Add the following text at the end of the existing paragraph:

"Labels may overlap to the extent provided for by ISO 7225:1994 "Gas cylinders - Precautionary labels", however, in all cases, the labels representing the primary hazard and the numbers appearing on any label shall remain fully visible and the symbols recognisable.".

PART 6

Chapter 6.1

6.1.2.5 Replace "Wooden barrel" with "Reserved".

6.1.2.7 In the table, replace the existing row 2 ("2. Barrels C. Wooden…. 6.1.4.6") with the following: "2. Reserved".

6.1.4.1.1 Add a note at the end of the paragraph to read:

*"**NOTE**: In the case of carbon steel drums of a capacity greater than 100 litres, "suitable" steels are identified in ISO 3573:1999 "Hot-rolled carbon steel sheet of commercial and drawing qualities" and ISO 3574:1999 "Cold-reduced carbon steel sheet of commercial and drawing qualities".".*

6.1.4.6 Delete this paragraph and renumber subsequent paragraphs and subparagraphs accordingly.

6.1.5.2.1 In the second sentence, insert "other than bags" after "packagings".

Insert the following new third sentence: "Bags shall be filled to the maximum mass at which they may be used.".

6.1.5.3.3 Add a new paragraph to read as follows:

"6.1.5.3.3 Removable head packagings for liquids shall not be dropped until at least 24 hours after filling and closing to allow for any possible gasket relaxation."

Renumber subsequent paragraphs and subparagraphs accordingly.

6.1.5.3.4 Replace the sentence: "For liquids if the test is performed with water:" with "For liquids in single packagings and for inner packagings of combination packagings, if the test is performed with water:"

Add the following note at the end of this paragraph as follows:

"***NOTE**: The term water includes water/antifreeze solutions with a minimum specific gravity of 0.95 for testing at - 18 °C.*".

6.1.5.3.5.2 Insert the words "while retaining its containment function" after "closure".

6.1.5.7 Delete this paragraph and renumber subsequent paragraphs and subparagraphs accordingly.

Chapter 6.7

6.7.2.1 In the definition of "portable tank" delete the words "having a capacity of more than 450 litres".

Insert the following definitions in alphabetical order:

"*Fine grain steel* means steel which has a ferritic grain size of 6 or finer when determined in accordance with ASTM E 112-96 or as defined in EN 10028-3, Part 3.

Fusible element means a non-reclosable pressure relief device that is thermally actuated.

Offshore portable tank means a portable tank specially designed for repeated use for transport of dangerous goods to, from and between offshore facilities. An offshore portable tank is designed and constructed in accordance with the Guidelines for the Approval of Containers Handled in Open Seas specified by the International Maritime Organization in document MSC/Circ.860.".

ALPHABETICAL INDEX

Amend alphabetical index in accordance with the amendments adopted for Chapter 3.2.

ANNEX 2

DRAFT AMENDMENTS TO THE MANUAL OF TEST AND CRITERIA

Section 1

1.2.2 In Table 1.1, replace "1-7" with "1-8" in the first row under "Test series".

1.6 In Table 1.2, add the following:

Test series	Test type	Test code	Test name
8	(a)	8(a)	Thermal stability test for ANE
8	(b)	8(b)	ANA gap test
8	(c)	8(c)	Koenen test
[8	(d)	8(d)	Vented pipe test]

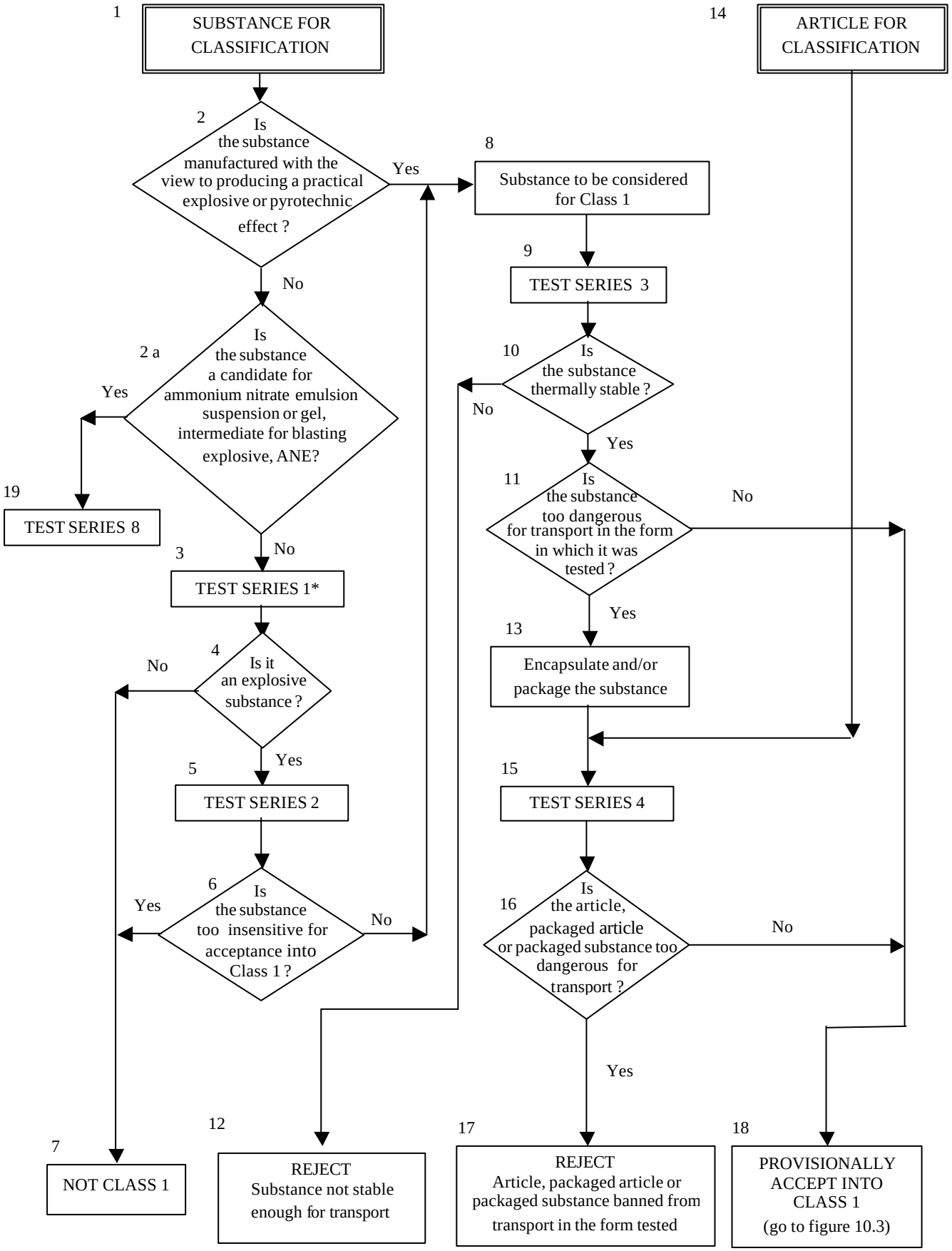
Section 10

10.1.1 Amend the last sentence to read as follows: "…in figures 10.1, 10.2, 10.3 and 10.4, the general….in sections 11 to 18 of this test manual.".

Figure 10.2 Amend this figure as shown in page 7 of this document.

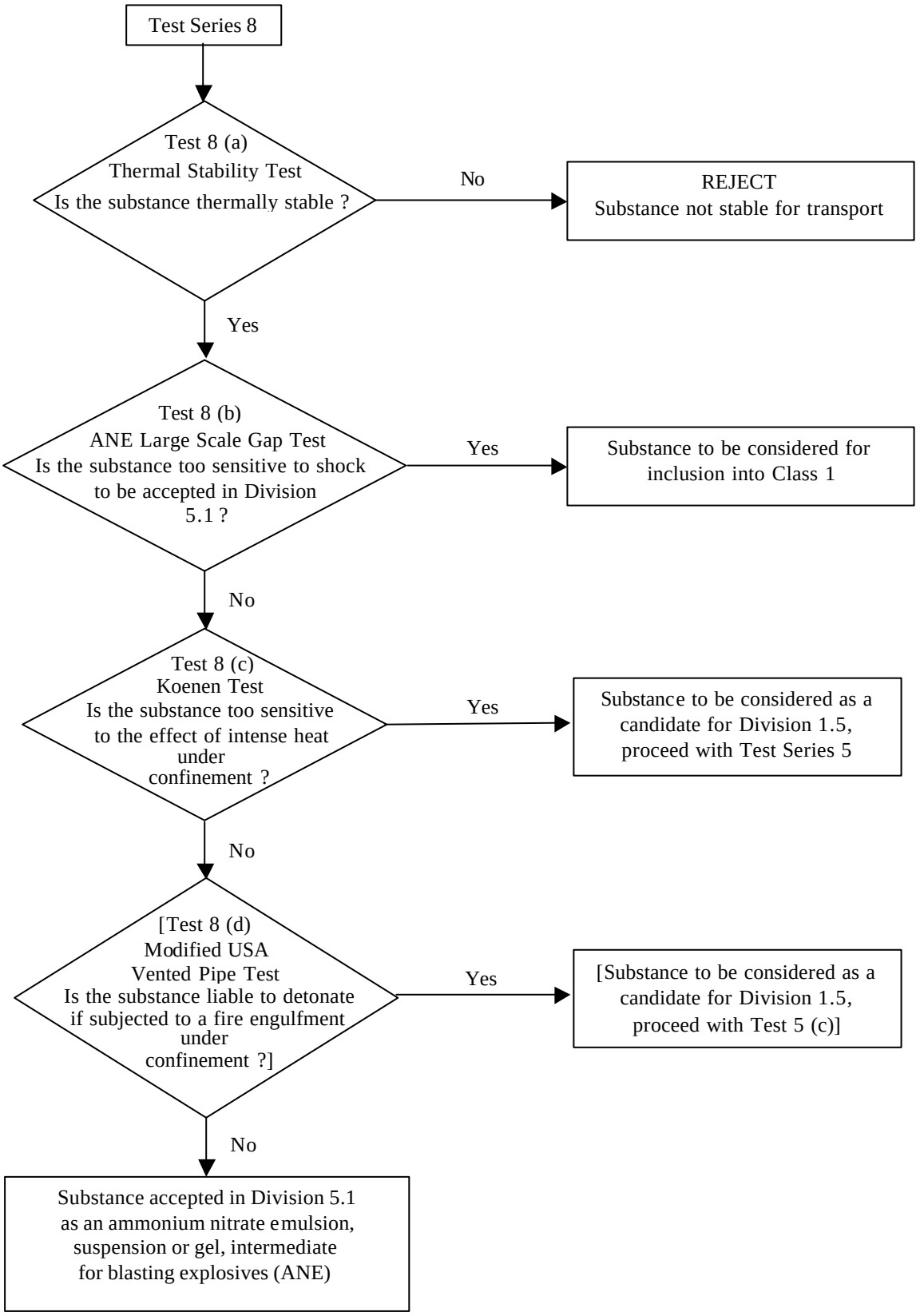
Figure 10.4 Insert a new figure 10.4, as shown in page 8 of this document, immediately after the existing Figure 10.3 and renumber the Figures 10.4 to 10.8 consequently.

Figure 10.2: PROCEDURE FOR PROVISIONAL ACCEPTANCE OF A SUBSTANCE OR ARTICLE IN CLASS 1



* *For classification purposes, start with Test Series 2.*

Figure 10.4


Test Series 8
Test 8 (a)
Thermal Stability Test
Is the substance thermally stable ?
No
REJECT
Substance not stable for transport
Yes
Test 8 (b)
ANE Large Scale Gap Test
Is the substance too sensitive to shock to be accepted in Division 5.1 ?
Yes
Substance to be considered for inclusion into Class 1
No
Test 8 (c)
Koenen Test
Is the substance too sensitive to the effect of intense heat under confinement ?
Yes
Substance to be considered as a candidate for Division 1.5, proceed with Test Series 5
No
[Test 8 (d)
Modified USA
Vented Pipe Test
Is the substance liable to detonate if subjected to a fire engulfment under confinement ?]
Yes
[Substance to be considered as a candidate for Division 1.5, proceed with Test 5 (c)]
No
Substance accepted in Division 5.1 as an ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives (ANE)

10.4.2.5 Add a new section to read as follows:

"10.4.2.5 The question "Is the substance a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives (ANE)?" (box 2(a), figure 10.2) is answered by series 8 tests and any candidate should pass each of the four tests comprising the series. The four 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;

[Type 8 (d) - a test to determine the effect of exposure to a large fire under confined, vented conditions.]".

10.4.3.7 Insert a new 10.4.3.7 to read as follows:

"10.4.3.7 Test types 8 (a) to [8 (d)] should be used to establish whether an ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives (ANE) may be assigned to Class 5.1. Substances failing any of the tests may be considered as a candidate for Class 1 in accordance with Table 10.4."

Renumber the existing 10.4.3.7 as new 10.4.3.8.

10.5.1 Amend the end of the paragraph to read: "…figures 10.5 to 10.8.".

10.5.2 Replace "figure 10.8" with "figure 10.9.".

Section 18 Insert a new Section 18, Test Series 8, as follows:

"SECTION 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 in Division 5.1 is answered by series 8 tests and any such candidate for inclusion in Division 5.1 should pass each of the four types of tests comprising the series. The four 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;
[Type 8 (d): a test to determine the effect of exposure to a large fire under confined, vented conditions.]

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 */	18.4.1
8 (b)	ANE Gap Test */	18.4.2
8 (c)	Koenen test */	18.4.3
[8 (d)	Vented pipe test */	18.4.4]

*/ Recommended test.

18.3 Test conditions

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

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.

18.4.1.1.2 This test is used to determine whether the emulsion, suspension or gel is stable at temperatures encountered during transport. 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 chracteristics 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/K), of the substance using the formula:

$$L = \ln 2 \, . \, Cp / t_{1/2}$$

18.4.1.2.6 Dewar vessels filled with 400 ml of substance, with a heat loss of 80 to 100 mW/kg.K are suitable.

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 cilindrically 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 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.

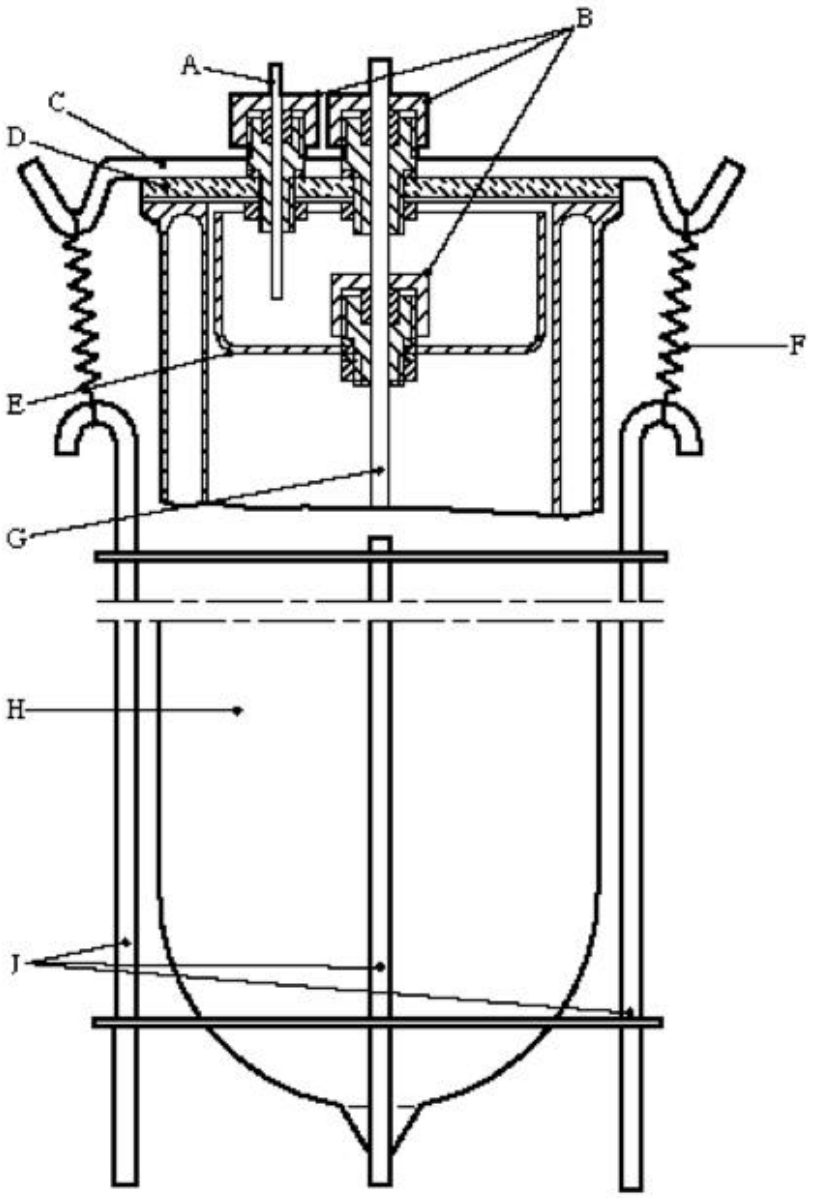
18.4.1.3.3 If the sample survives, cool and remove it from the test chamber and carefully dispose of it as soon as possible. The percentage mass loss and change in composition may be determined.

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	**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) Ammo nium 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



(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 1600 kg/m^3 ± 50 kg/m^3;

(c) Tubing, steel, cold drawn seamless, 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)

(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 polymethyl methacrylate (PMMA) rod, of 95 mm diameter by 70 mm long. A gap length of 70 mm results in a shock pressure 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);

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

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

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

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

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.

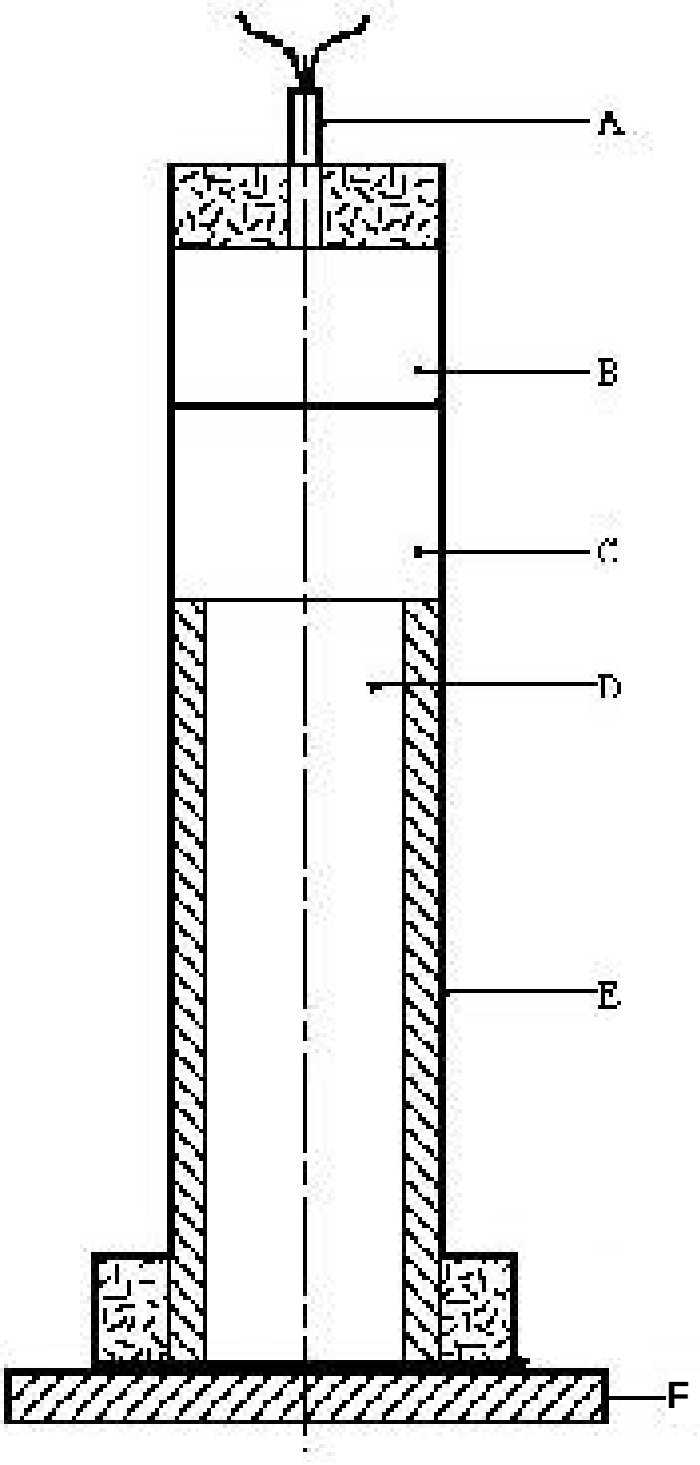
18.5.1.4 *Test criteria and method of assessing results*

A clean hole punched through the plate indicates that a detonation was initiated in the sample. A substance 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*

Substances	**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%, Sodiumnitrate 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%, Sodiumnitrate 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

Substances	Density g/cm^3	Gap mm	Result	Comments
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%, Fuel/emulsifier 9%	1.29	70	-	Tube fragmented . Plate indented. VOD 1968m/s
ANE-G2 Ammonium nitrate 74%, Sodiumnitrate 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%, Calciumnitrate 8%, Water 16%, Fuel/emulsifier 7%	1.41	70	-	Tube fragmented Plate indented. VOD 2061m/s



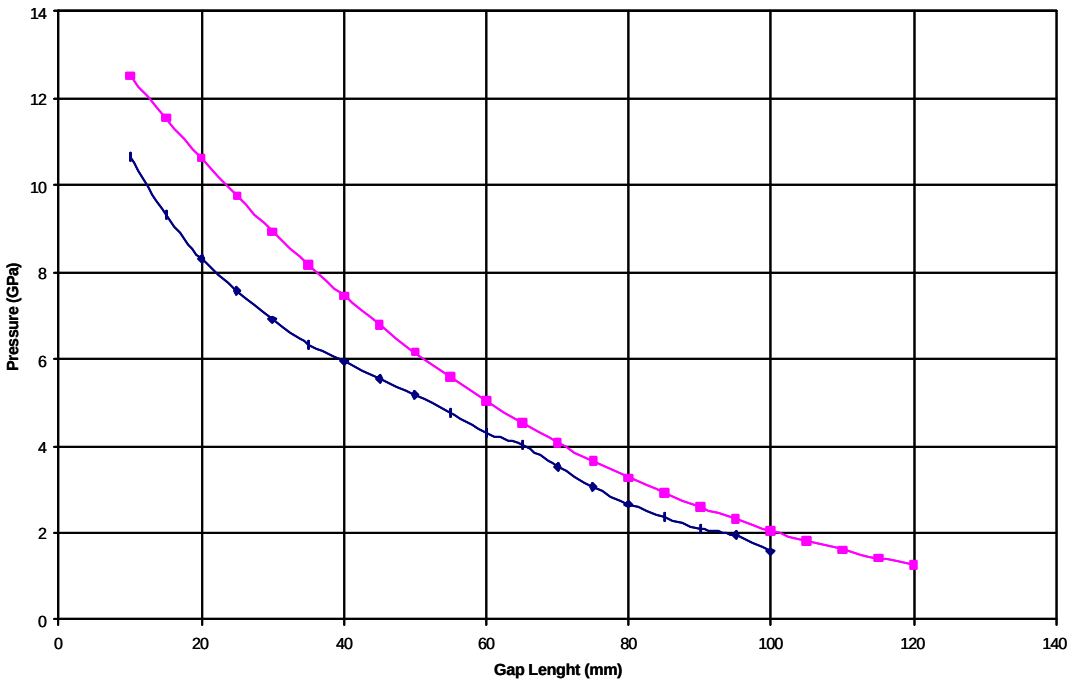
(A)	Detonator	(B)	Booster charge
(C)	PMMA gap	(D)	Substance under test
(E)	Steel tube	(F)	Witness plate

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	55	5.58
60	4.31	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

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 of suitable quality. The mass of the tube is 25.5 ± 1.0 g. 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.

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 50 °C to 250 °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 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* fragments;
"F": Tube fragmented into three* 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 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*

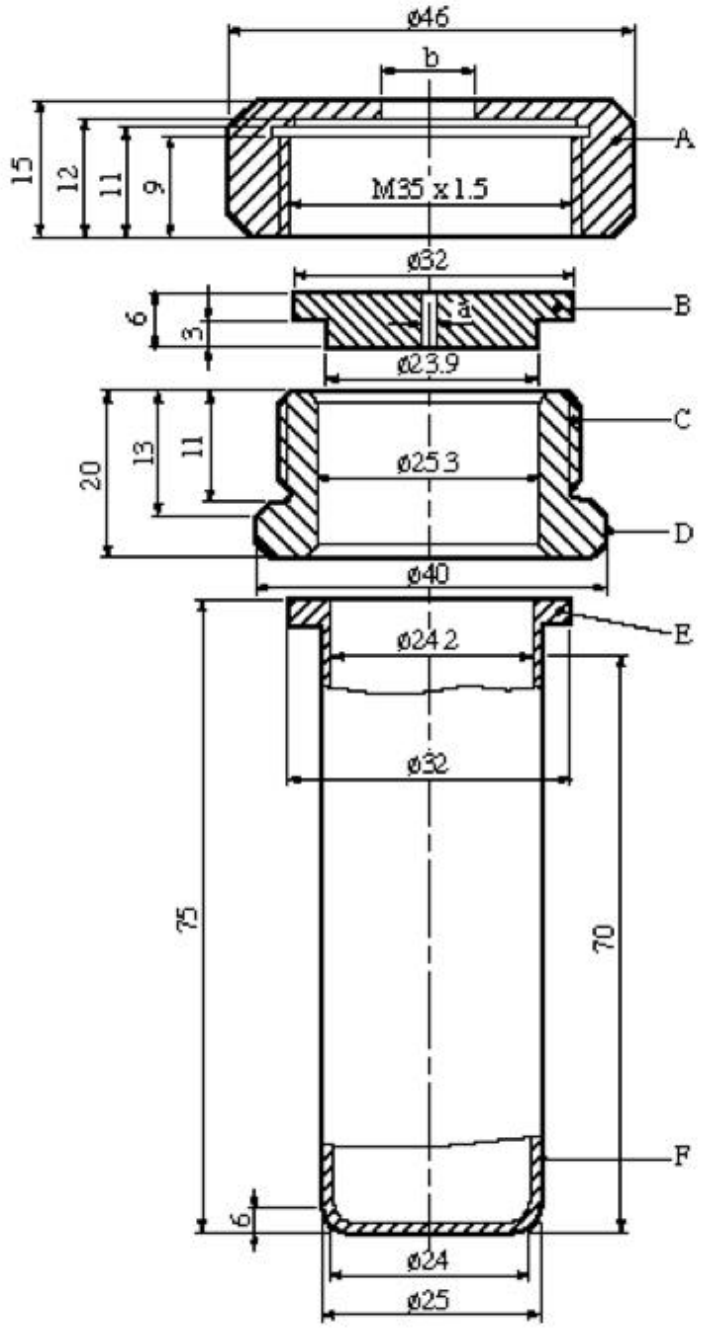
The result is considered "+" and the substance should not be classified in Division 5.1 if the limiting diameter is 2.0 mm or more. The result is considered "—" if the limiting diameter is less than 2.0 mm.

18.6.1.5 *Examples of results*

Substances	**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%	-	
ANE-F6 Ammonium nitrate 72%, Sodium nitrate 11%, Water 10%, Fuel/emulsifier 6%	-	
ANE-F7 Ammonium nitrate 76%, Water 13%, Fuel/emulsifier 10%	-	

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

Substances	Result	Comments
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%, Sodiumnitrate 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%	-	



(A)	Nut (b =10.0 or 20.0 mm) with flats for size 41 spanner	(B)	Orifice plate (a = 1.0 ÷ 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

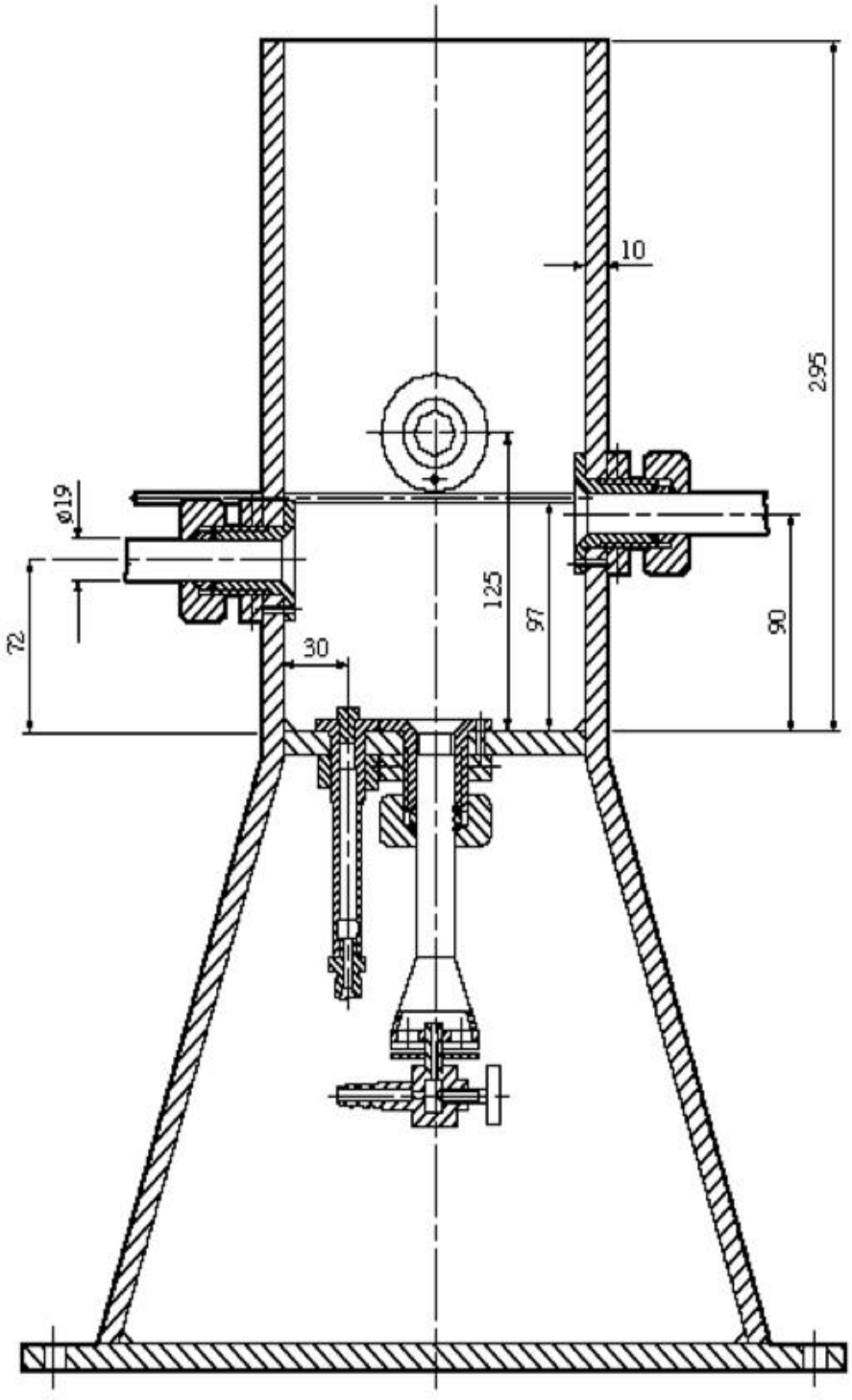


Figure 18.6.1.2: HEATING AND PROTECTIVE DEVICE

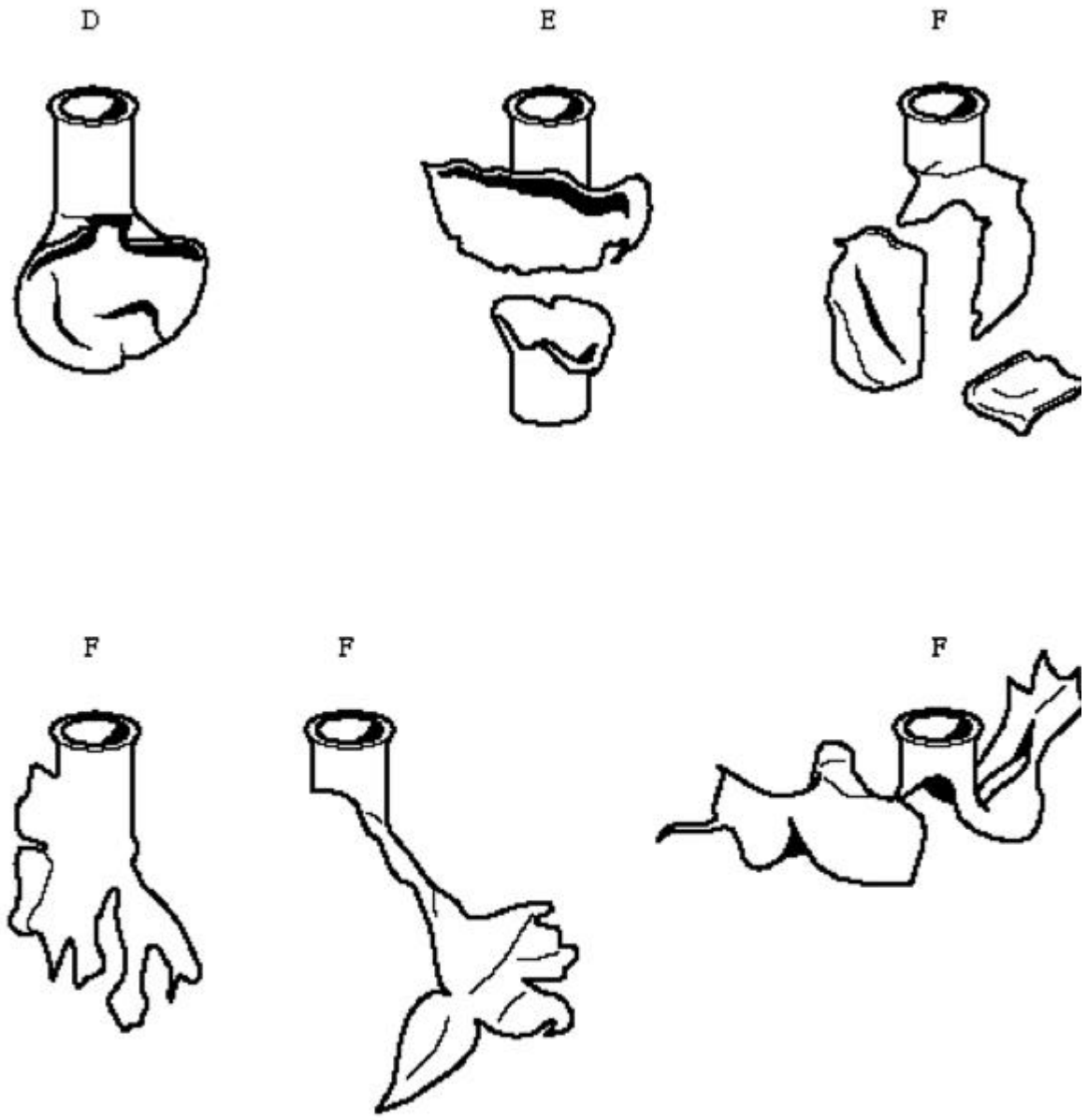


Figure 18.6.1.3 EXAMPLES OF EFFECT TYPES D, E AND F

[18.7 Series 8 Type (d) Test prescription

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

18.7.1.1 *Introduction*

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 ***(specification to be provided by USA)*** 30 ± 1 cm diameter and 60 ± 1 cm long, welded close at the bottom with a 38 cm square, 10 ± 0.5 mm thick mild steel plate. The top of the pipe is welded to a 38 cm square, 10 ± 0.5 mm thick mild steel plate that contains a 76 mm diameter vent hole centrally located in the plate to which a 150 mm long steel pipe nipple of 76 mm internal diameter is welded. (See figure 18.7.1.1) ***(Further specifications on inner/outer diameter, wall thickness and tolerances to be provided)***]

(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 under test without tamping during loading. The substance 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:

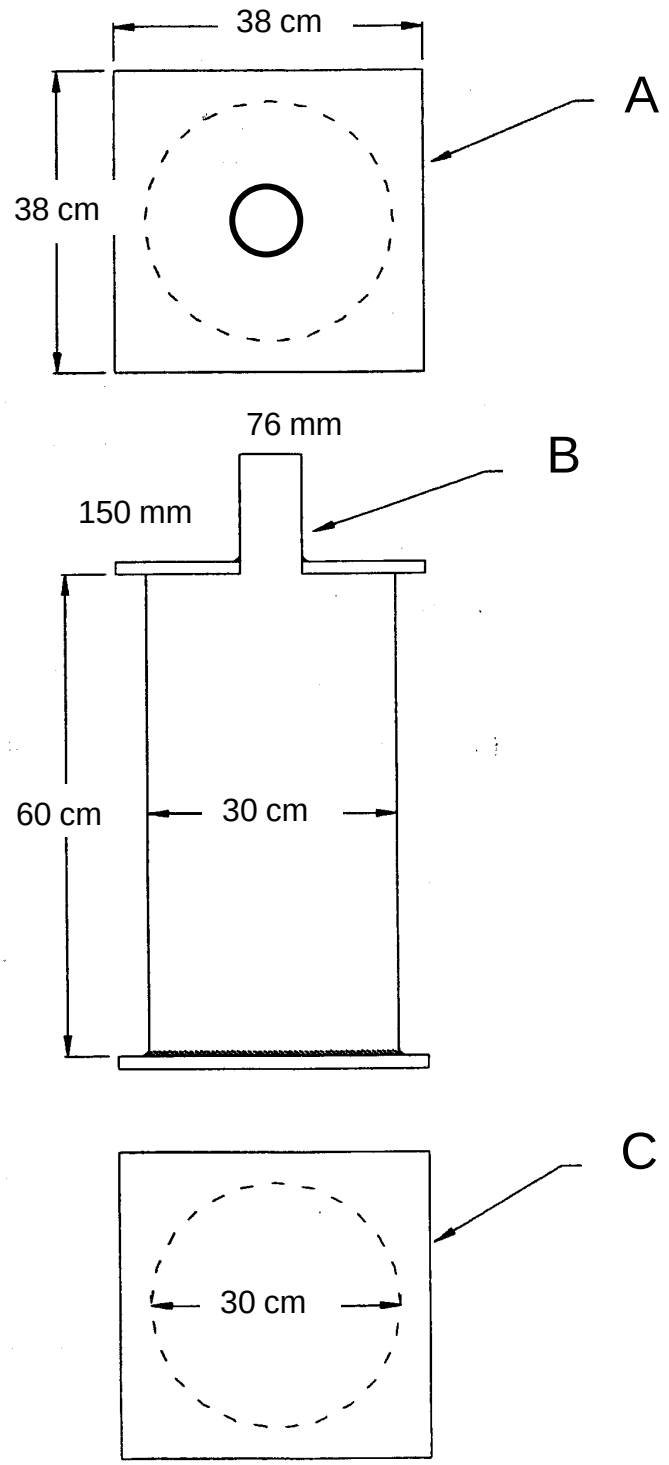
(a) evidence of explosion;
(b) loud noise; and
(c) projection of fragments from the fire area.

18.7.1.4 *Test criteria and method of assessing results*

The test result is considered "+" and the substance should not be classified in Division 5.1 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	



(A)	Top plate
(B)	Steel pipe nipple
(C)	Bottom plate

Figure 18.7.1.1: VENTED PIPE TEST