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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 **13 June 2013**

Forty-third session
Geneva, 24–28 June 2013
Item 2 (c) of the provisional agenda:
Explosives and related matters: review of tests in parts I and II of the Manual of Tests and Criteria

Manual of Tests and Criteria

Review of Test Series 8

Transmitted by the Australian Explosives Industry and Safety Group Inc. (AEISG)

Introduction

1. At the 41st session of the Sub-Committee of Experts on the Transport of Dangerous Goods (TDG), 25 June - 4 July 2012, the Chairman of the EWG submitted a paper on the 'Difficulties in carrying out classification tests' (refer INF.26, 2012), outlining:

- there was agreement that the problem of specifications in the test procedures was real and should be corrected;

- there could be other problems such as errors in procedure, incorrect use of the examples in the procedures, and a difficulty in identifying the key parameters of the tests;

- the Sub-Committee had already agreed that it should conduct a review of the tests mentioned in Parts I and II of the Manual with a view to:

  (a) Better defining the specifications of the tests;

  (b) Better defining the tolerances associated with those specifications; and

  (c) Removing any unnecessary or over-specifications;

2. This position was endorsed by the Sub-Committee of Experts on the Globally Harmonized System of Classification and Labelling of Chemicals (GHS) (see ST/SG/AC.10/C.4/42; paragraph 9).

3. The Explosives Working Group (EWG) discussed the issues raised in INF.26 and reported back to TDG (refer INF.67, 2012) recommending:

- there was general support for the way forward proposed by the chairman of the working group. The working group noted that IME (with the USA and Canada) is already underway in regards to reviewing Test Series 6 and that AEISG had committed to work on Test Series 8. The UK commented that they may also have resources that could be devoted to the project. The working group agreed with the priorities put forward by the chairman. The chairman of the working group reconfirmed his willingness to coordinate this effort.

4. The working group endorsed the proposal of INF.26 that the sub-committees on TDG and GHS accept the general principle outlined in regards to Parts I and II of the Test Manual, and include this activity in their next programme of work, and take action as deemed appropriate.

5. TDG considered the EWG report (INF.67) and accepted the general principle outlined as regards Parts I and II of the Manual of Tests and Criteria and agreed to include this activity in its next programme of work, subject to concurrence by the GHS Sub-Committee (ST/SG/AC.10/C.3/82, 3 August 2012).

Discussion

6. In line with the principles outlined above, the various tests of Test Series 8, in Section 18 of the Manual of Tests and Criteria, have been reviewed with amended versions presented in the attached Appendix for consideration by the Explosives Working Group as part of their program of work.

(a) Thermal Stability Test 8(a)

- clause added to indicate that test is unnecessary if the ANE is manufactured at temperatures higher than 20 C above any temperature experienced during transport;
- reference to Dewar vessel (and associated Figure 18.4.1.1) is removed, replaced with 'insulated test vessel' meeting the heat loss characteristics specified;
- the specification for the test chamber (oven) has been increased to +/- 2 C to remove overspecification when looking for sample temperature rise of more than 6 C (refer Test 3(c) which is +/- 2 C when looking for 3 C rise);
- changes agreed at the 41st session have been included;
- it is still considered that this test could be removed completely given the nature of ANEs and the manufacturing processes involved.

(b) ANE Gap Test 8(b)

- changes agreed at 41st session have been included;
- the 'seamless' specification for the steel tubing has been removed as this is considered an over-specification for what is a sensitivity test (ie fragmentation of tubing not an issue here);
- reference to 'UN standard detonator' amended;
- reference to conducting test over container of water removed (over specified) and replaced with the witness test plate needing to be off the ground (100 mm);
- calibration data removed (Table 18.5.1.1 and Figure 18.5.1.2) as not relevant here (could be captured in the Appendices if considered useful);
- Figure 18.5.1.1 amended to reflect test set up.

(c) Koenen Test 8(c)

- written as a go/no go test using a 2.0 mm orifice plate (ie removing all other orifice plates, other than the 1.5 mm calibration orifice plate);
- molybdenum disulphide used as one example of a high temperature anti-seize compound;
- pictures of equipment added;
- pictures of resultant effects added;
- source of supply of tubes included;
- criteria allowance for 'false positives' from orifice blockages.

(d) Vented Pipe Test 8(d)(i)

- anemometer added;
- safety issues/precautions added;
- observations increased;
- clarification of criteria.

Modified Vented Pipe Test 8(d)(ii)

- flexibility added for insignificant specifications e.g. heat shield;
- flexibility for fuel gas type used;
- safety issues/precautions added;
- dual tests changed to one test only, as in 8(d)(i).

Consideration

7. AEISG presents the amended versions of UN Test Series 8 tests for consideration and discussion.

8. The contents and results of discussions in this regard may be used by AEISG in formulating specific proposals for amending the Manual of Tests and Criteria at future meetings.

APPENDIX: AMENDED VERSIONS OF UN TEST SERIES 8 TESTS (SECTION 18 OF THE MTC)

18.3 Test conditions

18.3.1 The substance should be tested as offered for transport, at the maximum temperature which may occur during transport (see 1.5.4 of this Manual), unless it is intended for transport in portable tanks (see 18.4.1.1.1).

18.4 Series 8 Type (a) test prescription

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

18.4.1.1 Introduction

18.4.1.1.1 This test is used to determine whether a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" is thermally 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 insulated test vessel is only representative for packagings, IBC's and small tanks. For the transport of ammonium nitrate emulsions, suspensions or gels the test is used to measure their thermal stability during tank transport. The test is carried out at a temperature 20 °C higher than the maximum temperature which may occur during transport, or, if higher, at the temperature at the time of loading.

18.4.1.1.2 This test is not considered necessary where the candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" is manufactured at a temperature which is 20 °C, or higher, than the maximum temperature which may occur during transport.

18.4.1.2 *Apparatus and materials*

18.4.1.2.1 The experimental equipment consists of a suitable thermostatically controlled test chamber (which may be fan-assisted), appropriate insulated test vessels with closures, temperature probes and recording equipment.

18.4.1.2.2 The test should be performed following a risk assessment, taking account of the potential for fire and/or explosion in the test chamber, and application of appropriate control measures for the protection of persons and property. A number of tests may be run concurrently. The recording system should be housed in a separate observation area.

18.4.1.2.3 The test chamber must be large enough to allow air circulation on all sides of the insulated test vessels. The air temperature in the test chamber should be controlled so that the desired temperature of a liquid inert sample in the insulated test vessel can be maintained with a deviation of not more than ± 2 °C for up to 10 days. The air temperature in the test chamber should be measured and recorded.

18.4.1.2.4 Insulated test vessels with a volume of approximately 500 ml with a closure system are used. The closure of the test vessel should be inert.

18.4.1.2.5 The heat loss characteristics of the system used (i.e. insulated test vessel and closure), must 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 are determined by measuring the half time of cooling of the vessel filled with a known inert liquid substance(e.g. distilled water).

The heat loss per unit of mass, L (W/kg.K) is calculated from the half time of cooling, t1/2 (s), and the specific heat, Cp (J/kg.K), of the substance using the formula:

$$L = \ln 2 \times (Cp / t1/2)$$

18.4.1.2.6 Test vessels filled with 400 ml of inert substance (eg water), with a heat loss of 100 mW/kg.K or less are suitable.

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, at the temperature at the time of loading. Fill the test vessel with the substance under test to about 80% of the capacity of the test vessel, or approximately 400 ml. Insert the temperature probe into the centre of the sample. Seal the lid of the test vessel and insert the vessel in the test chamber, connect the temperature recording system and close the test chamber.

18.4.1.3.2 The temperature of the sample and of the test chamber are 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.

18.4.1.3.3 At the end of the test, allow the sample to cool, remove it from the test chamber and carefully dispose of it as soon as possible.

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 within the seven day period 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) 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%

ET AL

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 booster charge (donor), a barrier (gap), a container holding the sample substance (acceptor charge), and a steel witness plate (target).

The following materials are to be used:

(a) Detonators of sufficient strength to effectively initiate the booster charge (e.g. United Nations Standard detonator or equivalent);

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

(c) Tubing, steel, with an outer diameter of 95.0 ± 7.0mm, a wall thickness of 9.75 ± 2.75mm and an inner diameter of 73.0 ± 7.0mm, and with a length of 280mm;

(d) Sample substances (acceptor charges);

(e) Solid polymethyl methacrylate (PMMA) cylinders, 95mm diameter by 70mm long;

(f) Mild steel witness plates, approximately 200mm × 200mm × 20mm;

(g) Wood blocks, 95mm diameter and approximately 25mm thick with a hole drilled through the centre to hold the detonators in place against the booster charge.

(h) Wood blocks or similar to stand the assembly at least 100mm off the ground.

18.5.1.3 *Procedure*

18.5.1.3.1 As shown in Figure 18.5.1.1, the detonator, booster charge, PMMA gap and acceptor charge are coaxially aligned above the centre of the witness plate.

The bottom end of the tube is sealed with a single layer of cloth adhesive tape, or equivalent, to contain the sample substance, which is carefully loaded so as to avoid the formation of voids within the sample or between the sample and the tube walls. The surface of the sample should be level with the rim of the tube.

Care should be taken to ensure good contact between the detonator, the booster charge, the PMMA cylinder and the acceptor charge. The sample substance should be at ambient temperature or not more than 30°C. The wood block holding the detonator, the booster charge, the PMMA cylinder and the steel tube should be held firmly in alignment (e.g. by using a band of adhesive tape at each intersection).

18.5.1.3.2 The whole assembly, including the witness plate, is raised above the ground, with at least a 100mm air gap between the ground and the bottom surface of the witness plate which is supported along two edges

only with wooden blocks, or similar, as shown in Figure 18.5.1.1. The location of the blocks must ensure that there is a clear space under where the tube is standing on the witness plate. To assist in collecting the remains of the witness plate, the whole assembly should be vertical (e.g. checked with a spirit level).

18.5.1.3.3 The test is performed three times unless a positive result is observed earlier.

18.5.1.4 *Test criteria and method of assessing results*

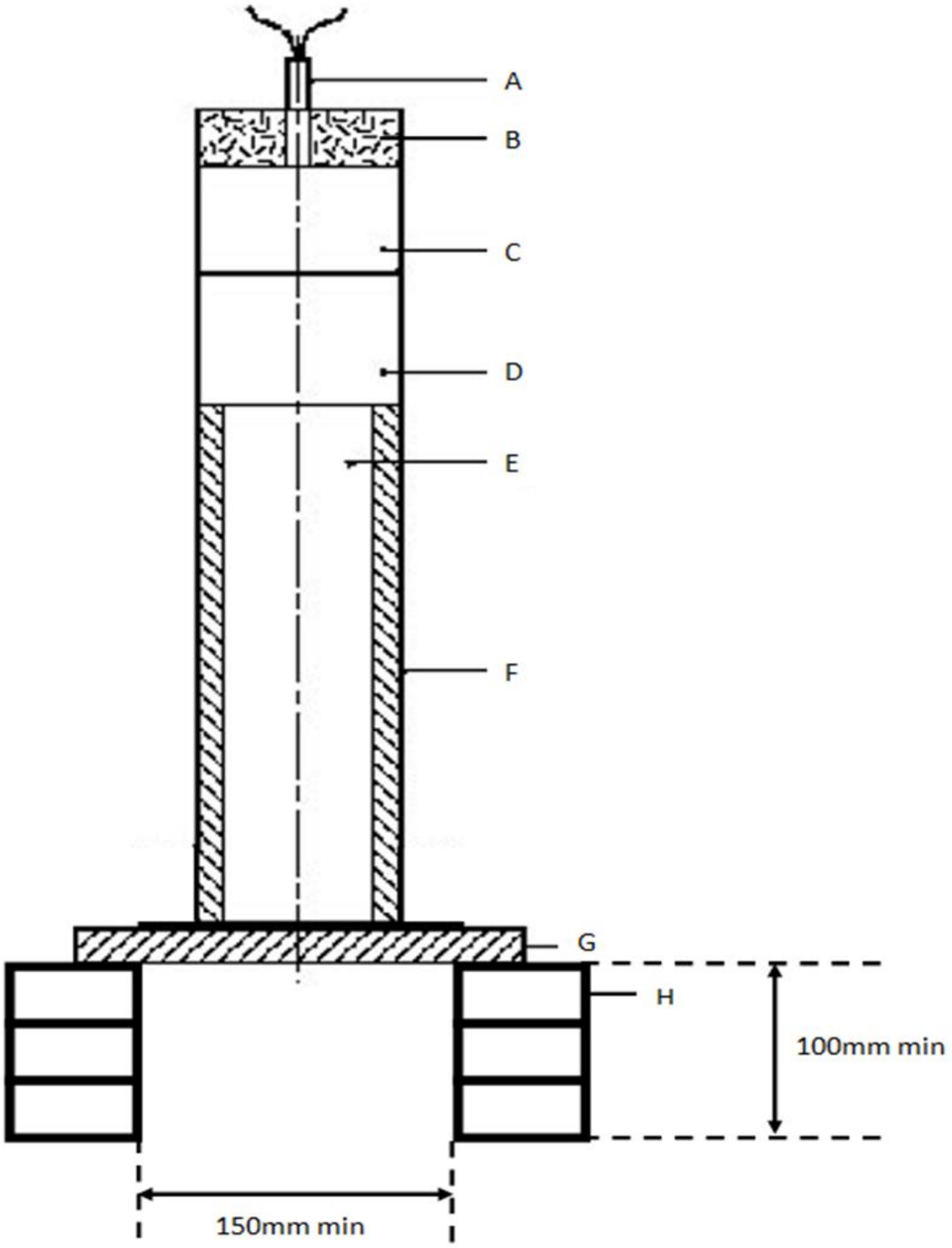
A clean hole punched through the plate indicates that a detonation was initiated in the sample. A substance which detonates in any trial is not to be classified as "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" and the result is noted as "+".

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

Substances	Density g/cm^3	Gap mm	Result	Comments
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%, Fuel/emulsifier 9%	1.29	70	-	Tube fragmented 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

Substances	Density g/cm^3	Gap mm	Result	Comments
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) Wooden detonator holder	(C) Booster charge
(D) PMMA gap	(E) Substance under test	(F) Steel Tube
(G) Witness plate	(H) Wooden blocks	

Figure 18.5.1.1: ANE GAP TEST

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 for "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[1] 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:

- 1.5mm for the closing plate used in the heating calibration procedure; and
- 2.0mm for the closing plate used in the test.

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 randomly selected and subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be 26.5 ± 1.5g;

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

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

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

18.6.1.2.2 Heating is provided by a gaseous fuel (eg propane), from an industrial cylinder fitted with a pressure regulator, via a flow meter and distributed by a manifold to the four burners. 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 1.5mm orifice plate) filled with 27cm^3 of dibutyl phthalate. The time taken for the temperature of the liquid (measured with a 1 mm diameter thermocouple centrally placed 43mm below the rim of the tube and inserted through the orifice plate) 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. A suitable arrangement of the construction and dimensions of the box is 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 draught does not affect the burner flames. Provision should be made for extracting any gases or smoke resulting from the test.

18.6.1.2.4 A video camera should be provided to record the test and to ensure all burners are functional during the test.

18.6.1.3 *Procedure*

[1] *For the Koenen Test, nozzle plates, closing devices, steel shells, burners, etc. are available from Swann Technologies, UK. Phone: 44 1763 249967. email: sales@swanntech.com.*

18.6.1.3.1 The substance is loaded into the tube to a height of 60mm taking particular care to prevent the formation of voids. The threaded collar is slipped onto the tube from below, the 2mm orifice plate is inserted and the nut tightened by hand after applying some high temperature anti-seize compound (e.g. 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 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. 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 to ensure all pieces have been recovered.

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[2] fragments;

"F": Tube fragmented into three[2] 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 test is performed three times unless a positive result is observed earlier.

18.6.1.4 *Test criteria and method of assessing results*

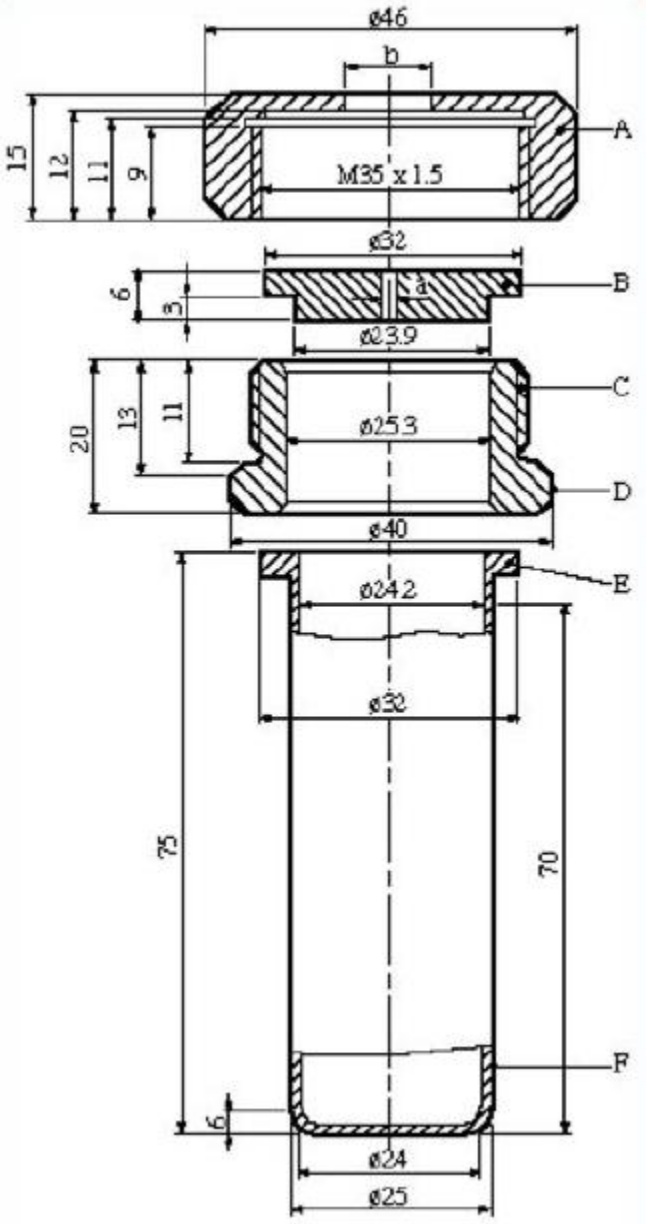
The result is considered "+" and the substance should not be classified in Division 5.1 if the result “explosion” occurs in any of the tests.

Note: Given the nature of ammonium nitrate emulsions, suspensions or gels and the possibility of varying percentages of solids present, blockages of the orifices may occur during testing potentially leading to a false "+" result. Where this is observed the test may be repeated (maximum twice).

18.6.1.5 *Examples of results*

Substances	**Result**	**Comments**
Ammonium nitrate (low density) ET AL	-	Limiting diameter: <1 mm

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



(A) Nut (b = 10 mm) with flats for size 41 spanner
(B) Orifice plate (a = 1.5 or 2.0 mm)
(C) Threaded collar
(D) Flats for size 36 spanner
(E) Flange
(F) Tube

Figure 18.6.1.1: TEST TUBE ASSEMBLY

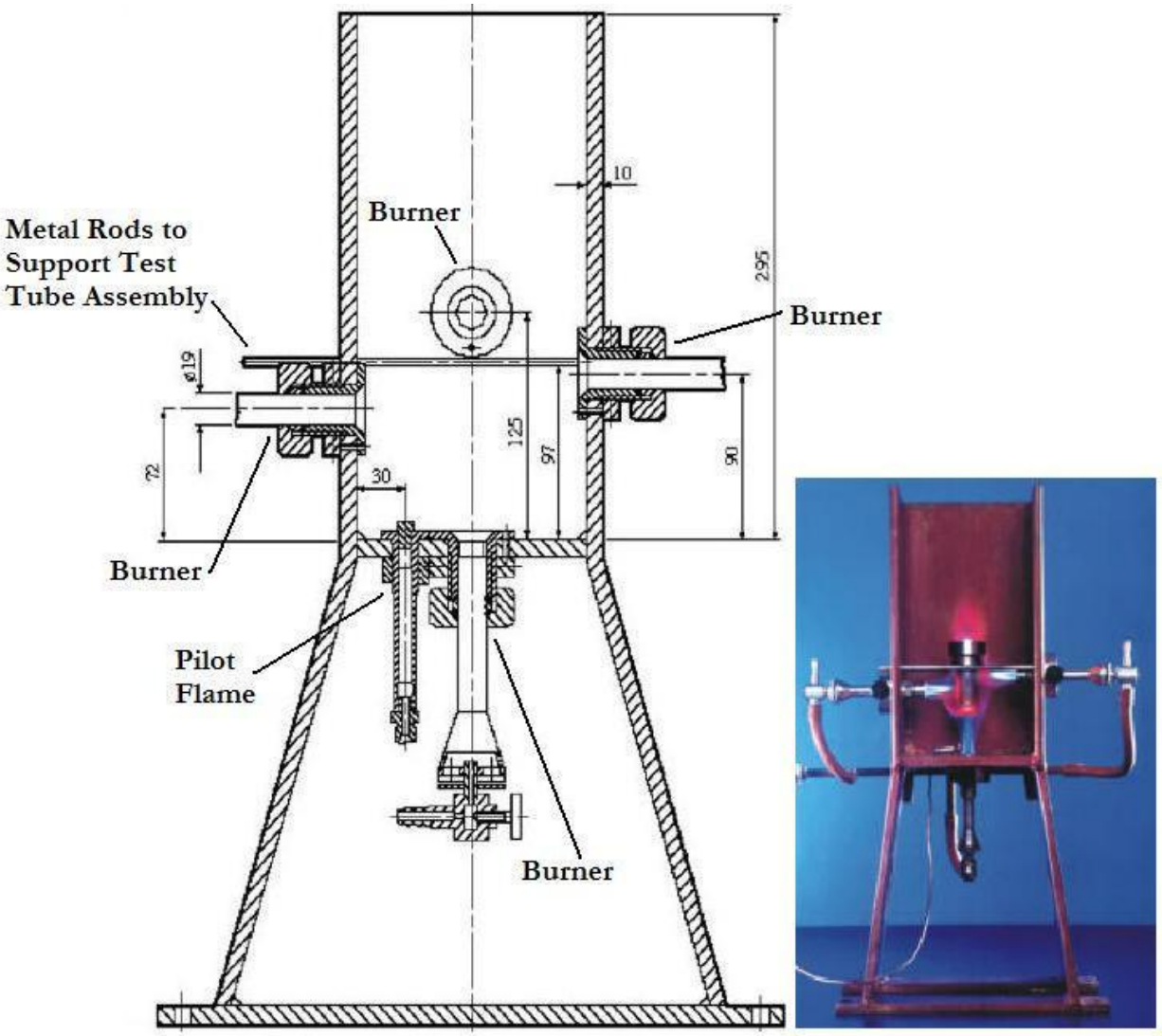


Figure 18.6.1.2 HEATING DEVICE

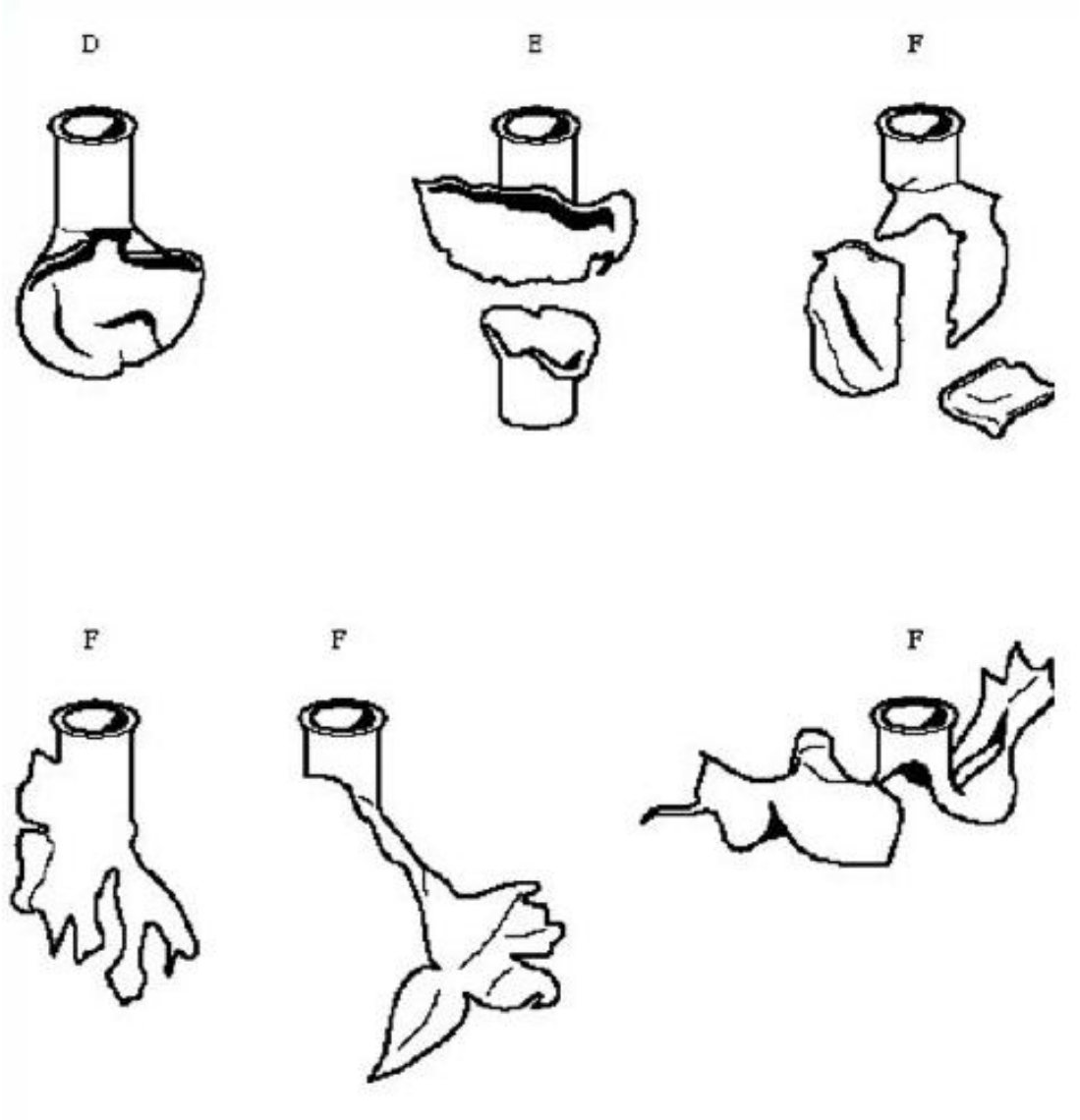


Figure 18.6.1.3 EXAMPLES OF EFFECT TYPES D, E AND F

Examples of Koenen test results

"O": Tube unchanged

“A”: Bottom of the tube bulged out

“B”: Bottom and wall of the tube bulged out

Classification “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 smaller pieces, closing device undamaged




18.7 Series 8 Type (d) Test prescription

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 of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" for transport in portable tanks as a dangerous substance of Division 5.1.

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 ± 10mm diameter and 610 ± 10mm long, welded close at the bottom with a 380mm square 10 ± 0.5mm thick mild steel plate. The top of the pipe is welded to a 380mm square, 10 ± 0.5mm thick, mild steel plate that contains a 78mm diameter vent hole centrally located in the plate to which a 152mm long steel pipe nipple of 78mm internal diameter is welded (see Figure 18.7.1.1). All welding must be to AS/NZS1554.1 or equivalent ISO standard. All steel components are to be Schedule 40 carbon steel (A53 Grade B) or equivalent;

(b) A metal grid to support the filled pipe above the fire and to allow adequate heating. If a wooden crib fire is used, the grid should be approximately 1 m above the ground. If a liquid hydrocarbon pool fire is used then the grid should be approximately 0.5 m above the ground;

(c) Enough fuel to produce a fire, reaching 800 °C (measured at the external base of the pipe), and burning for at least 30 minutes or, if necessary, until the substance has clearly had enough time to react to the fire, evidenced by ejection of material, smoke, fumes, flames, etc., from the top of the pipe.

The fuel must extend laterally beyond the pipe for 1m in every direction.

Where a wooden crib is used, it should be made up of 50mm square air-dried pine laths. To form each layer of the crib the following timber lengths will be required. The laths should be stacked from the ground up to form a lattice as close as possible to the base of the grid supporting the pipe. The laths should extend beyond the pipe to a distance of at least 1m in every direction and the lateral distance between the laths should be about 100mm. A suitable construction is shown in Figure 18.7.1.2.

Lath length (m)	Number of laths required
2.4	5
2.1	4
1.8	2
1.5	2
0.9	2

(d) Suitable means of ignition to effectively ignite the fuel (e.g. fuel oil to soak the wood and igniters on two sides);

(e) At least one normal speed video camera to record events in colour;

(f) Means of measuring and recording temperature, up to and above 800°C, with a thermocouple located at the external base of the pipe;

(g) A means of measuring wind, such as an anemometer.

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. The pipe must be engulfed by the fire. Precautions against side winds may be required to avoid dissipation of the heat.

18.7.1.3.2 The test should not be commenced under conditions where the wind speed exceeds 6m/s, unless precautions are taken against side wind.

18.7.1.3.3 Observations are made on the following:

Wind speed at commencement of the test as per Section 18.7.1.3.2;

Fire duration of at least 30 minutes, with 800°C reached at the external base of the pipe;

Temperature at the external base of pipe;

Substance reacting to the fire as described in 18.7.1.2(c);

Evidence of explosion (e.g. fragmentation of the pipe into two or more pieces) within 30 minutes of the fire reaching 800°C at the base of the pipe;

Projection of fragments of the cylindrical pipe section from the fire area within 30 minutes of the fire reaching 800°C at the external base of the pipe;

Evidence of a rupture (e.g. a split of the pipe or separation of the pipe from the base plate at the weld) within 30 minutes of the fire reaching 800°C at the base of the pipe;

18.7.1.4 *Safety Issues*

18.7.1.4.1 Suitable test site selection criteria

(a) a recommended minimum safety distance of 1km radius from the test area if the test is conducted unprotected;

(b) sufficient distance from public or private areas to eliminate the risk of public exposure to noise, fume and shrapnel;

(c) remote and/or secure enough to prevent unauthorised entry;

(d) minimal significant fire risk in the event of a positive result;

(e) minimal risk from loss of containment issues;

(f) adequate capacity for safe storage of test products; and

(g) appropriate approvals, notifications and awareness.

18.7.1.4.2 Test precautions

(a) if line of sight to the test is not possible, a remote observation system (e.g. video surveillance) should be used to observe the test from a safe distance;

(b) re-entry time to the test area should be set at a recommended minimum of 150 minutes if the test remains active;

(c) appropriate personal protective equipment (PPE) should be provided. Precautions should be taken to minimise risk of exposure to elements, fire, hot objects, fumes, etc.;

(d) risk of vehicle damage due to shrapnel on or protruding from the ground during re-entry to the test area.

(e) 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. Ideally this should be left overnight.

18.7.1.5 *Test criteria and method of assessing results*

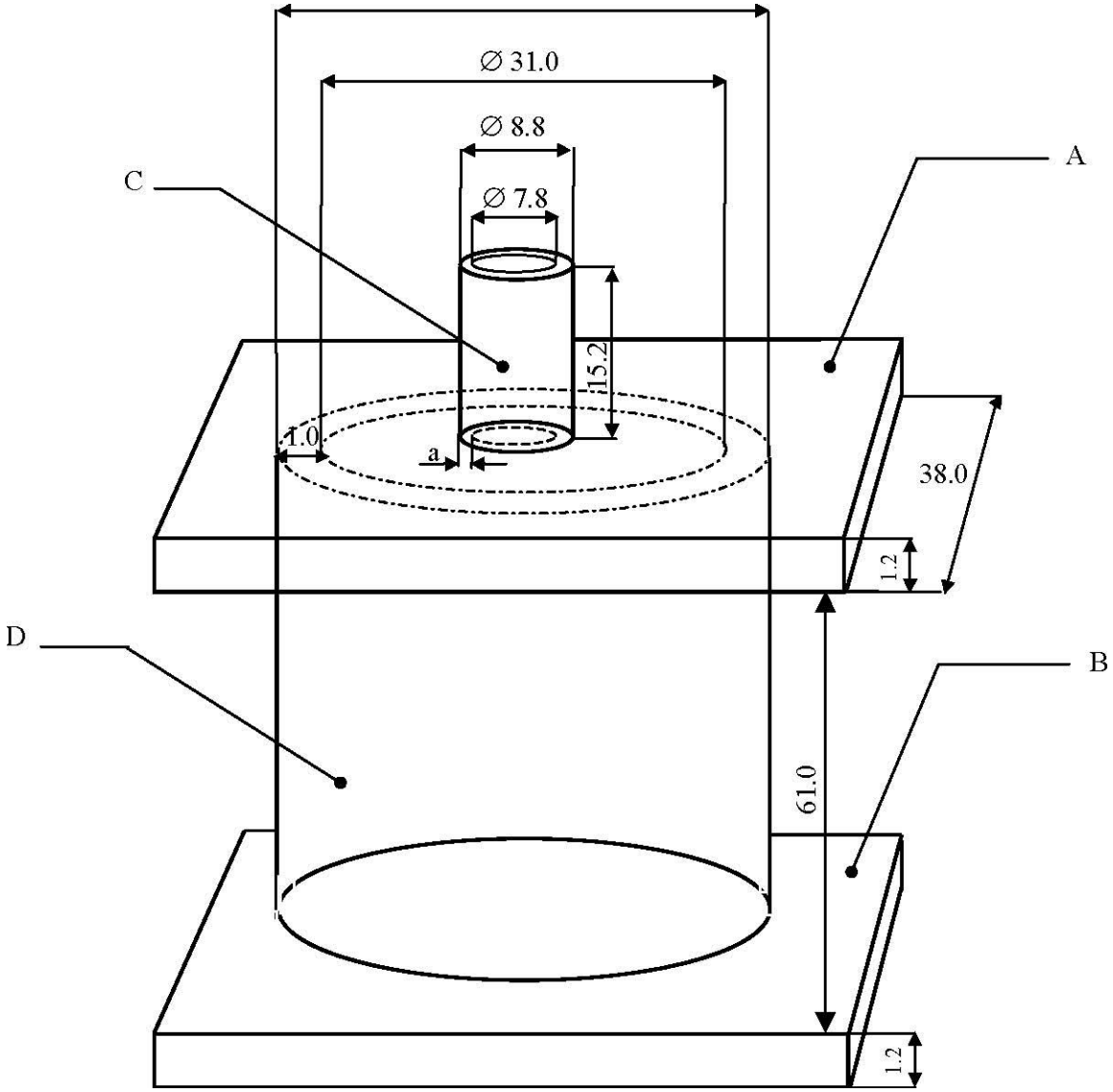
A test is considered valid if observation criteria Section 18.7.1.3.3 (a) to (d) have been met.

The test result is considered "+" and the substance should not be transported in portable tanks as a dangerous substance of Division 5.1 if an explosion and/or fragmentation of the cylindrical pipe, as specified in Section 18.7.1.3.3 (e) and (f) is observed within 30 minutes of the fire reaching 800°C at the external base of the pipe.

The result is considered a "-" if no explosion and/or fragmentation of the cylindrical pipe into two or more pieces is observed within 30 minutes of the fire reaching 800°C at the external base of the pipe. Splitting of the cylindrical pipe or its separation from the end plates, as specified in Section 18.7.1.3.3 (f) is evidence of a "-" result.

18.7.1.5 *Examples of results*

Substance	**Result**
to be added	



(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) Cylindrical Steel pipe Section (Schedule 40 carbon (A53 grade B))

Figure 18.7.1.1: VENTED PIPE TEST

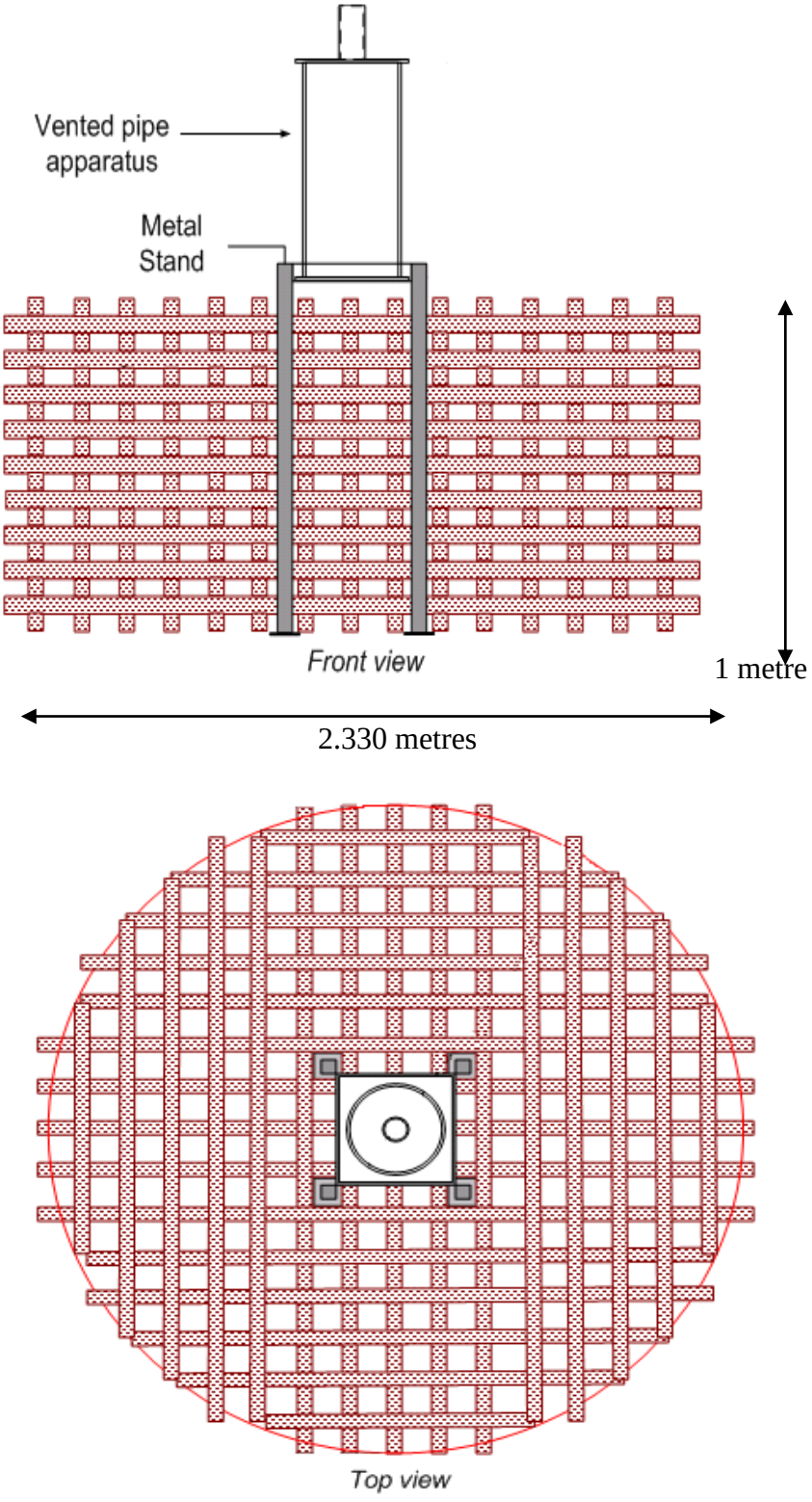


Figure 18.7.1.2: A SUITABLE WOOD FIRE FOR THE VENTED PIPE TEST

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 a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives", to be transported in portable tanks as a dangerous substance of Division 5.1.

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, 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 approximately 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. All welding should be to a relevant ISO standard. 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 neatly accommodate thermocouple robes;

(b) A concrete block, or similar solid base, about 400 mm square and 50 to 75 mm thick;

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

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

(f) Fuel gas bottle(s) connected via a manifold and fed into a pressure regulator. The pressure regulator should reduce the fuel gas 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, and a needle valve. An electrical solenoid valve is used to switch the fuel gas flow on and off remotely. Typically three 9 kg fuel gas 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 approximately 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) A means of measuring wind speed at the commencement of the test, such as an anemometer;

(k) Pure water for calibration;

(l) The candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives to be tested;

(m) Blast gauges, radiometers and other 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 approximately 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 test substance, 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 leveled using a spirit level. The fuel gas 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 approximately 435 mm) with the substance under test without tamping during loading. The initial temperature of the test substance must be recorded. The substance 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 Fuel gas 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, unless additional precautions against side winds are taken to avoid dissipation of the heat.

18.7.2.4.5 The fuel gas 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.

18.7.2.5 *Safety Issues*

18.7.2.5.1 Suitable test site selection criteria –

a) a recommended minimum safety distance of 1km radius from the test area if the test is conducted unprotected;

b) sufficient distance from public or private areas to eliminate the risk of public exposure to noise, fume and shrapnel;

c) remote and/or secure enough to prevent unauthorised entry;

d) minimal significant fire risk in the event of a positive result;

e) minimal risk from loss of containment issues;

f) adequate capacity for safe storage of test products; and

g) appropriate approvals, notifications and awareness.

18.7.2.5.2 Test precautions –

a) if line of sight to the test is not possible, a remote observation system should be used to observe the test from the recommended safe distance;

b) re-entry time to the test area should be set at a recommended minimum of 150 minutes if the test is considered still active;

c) appropriate personal protective equipment (PPE) should be provided. Precautions should be taken to minimise risk of exposure to elements, fire, fuels, hot objects, fumes, etc.;

d) risk of vehicle damage due to shrapnel on the ground during re-entry to test area.

18.7.2.6 *Test criteria and method of assessing results*

The test result is considered "+" and the substance should not be transported in tanks as a dangerous substance of Division 5.1 if an explosion is observed in any trial. Explosion is evidenced by rupture of the vessel. Once the substance is consumed and no rupture of the vessel is observed, then the result is considered "-".

18.7.2.7 *Examples of results*

Substances	**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	-