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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

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Item 3 (b) of the provisional agenda

EXPLOSIVES, SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES

Ammonium nitrate emulsions, suspensions and gels

Transmitted by the expert from the United Kingdom

Introduction

1. In ST/SG/AC.10/C.3/2004/25, The Expert from Sweden has proposed a new UN number for sensitised emulsions, suspensions and gels. The United Kingdom expert supports the call for a new UN number but proposes that UN 3375 be used for ammonium nitrate emulsions and the new number is used for ammonium nitrate suspensions and gels. The United Kingdom Expert also proposes a new Test Series for the new UN number which is believed to be more appropriate for these substances than Test Series 8.

Background

2. The Experts met in Engene in 1999 and were given a mandate by the Committee *"to analyse the properties of the emulsions so as to determine test methods that could be used classifying them"*(ST/SG/AC.10/C.3/2000/21). The Experts agreed the definition for these substances based on industry and regulators knowledge and experience of ammonium nitrate emulsions, and a number of tests for assigning classification to UN 3375 were agreed. At the July 2000 working group meeting of Experts Test Series 8 was further refined, and these tests were designed specifically for ammonium nitrate emulsions. At that working group meeting, the United Kingdom made proposals for an elevated temperature test (Test 8(a)) that was designed to verify an emulsion of ammonium nitrate liquor, fuel, emulsifier, and trace ingredients complying with the definition in SP309 would not self react above its transport temperature. The United Kingdom made proposals for a new test, to replace the cap sensitivity test suggested at the Engene meeting, as a means of showing that ammonium nitrate emulsions are not sensitised to a specified shock. The working group agreed the ANE UN Gap test as Test Series 8(b) and this test is performed on the ammonium nitrate emulsion at its transport temperature. Test 8(c), Keonen Test, is used to determine the sensitiveness of ammonium nitrate emulsions to intense heat under high confinement (ST/SG/AC.10/2000/20).

3. In December 2000, UN 3375 was amended to include ammonium nitrate suspensions and gels and SP309 was also amended to include these substances within the definition (ST/SG/AC.10/27, ST/SG/AC.10/27/Add.1).

4. Working group meetings of Experts were held in March 2001, July 2001 and July 2002 at which test results for ammonium nitrate emulsions were presented and included in the table of results for Test Series 8 (UN/SCETDG/19/INF.19, UN/SCETDG/21/INF.69). At each of these meetings the Chairman of the working group asked for test results for ammonium nitrate suspensions and gels to be presented for inclusion in the table of results in the Test and Criteria manual.

5. In December 2002 the Spanish Expert presented to the sub-committee Test Series 8 results for inclusion in the Test and Criteria manual (UN/SCETDG/22/INF.4) but the substances tested did not meet the definition in SP309.

6. At the July 2003 Working Group meeting the Spanish Expert presented proposals for the SP309 definition to be amended to include ammonium nitrate suspensions and gels to include potassium perchlorate, methylamine nitrate and hexamine nitrate (ST/SG/AC.10/C.3/2003/13). There were a number of questions asked by Experts concerning the inclusion of these additional energetic chemical ingredients to the SP309 definition. The United Kingdom Expert was concerned about the formation of new crystal forms of the ingredients or new chemical species produced during thermal cycling of the substance during transport. These concerns were included in the Working Group Chairman's report to the sub-committee.

7. At the December 2003 meeting an unplanned working group meeting was held and the Spanish Expert presented a new SP309 definition which split the formulations into two parts; the first covering ammonium nitrate emulsions and the second dealing with ammonium nitrate suspensions and gels. The Spanish Expert also presented answers to concerns expressed at the previous meeting. The United Kingdom Expert is concerned that during transport the ammonium nitrate suspension or gel may undergo physical and chemical changes caused by temperature changes in tanks or IBC's. The United Kingdom Expert has carried out thermal cycling experiments on an ammonium nitrate emulsions and an ammonium nitrate suspensions. These materials tested were cycled 5 times and then subjected to Test Series 3, BAM fall hammer. This gave negative results for all substances tested. There was no physical change discernable in the ammonium nitrate emulsion. However, there was evidence that the ammonium nitrate suspension had new or different crystalline substances suspended in the liquid phase. It was not possible to determine the nature of the crystals formed after the thermal cycling.

8. The Spanish Expert also presented data on thermal cycling on ammonium nitrate suspensions followed by either the Test Series 1(a) at 0ºC and 40ºC and Test Series 8(b) at 40ºC (UN/SCETDG/24/INF.18). The paper presented density values for three temperatures 0ºC, 20ºC and 40ºC and these showed a 2.3% density difference between 0ºC and 40ºC for ammonium nitrate emulsion (EM1) but 3.5% for a perchlorate suspension (SP1), 4.3% or a methylamine nitrate suspension (SP3), 4.2% for hexamine nitrate suspension (SP4) and 3.5% for the hexamine and perchlorate suspension (SP5). This appears to suggest that ammonium nitrate suspensions are subject to physical and/or chemical characteristics change with temperature. This is not a factor when considering the properties of ammonium nitrate emulsions.

Proposal

9. The Swedish proposal recommends a new UN number but suggests that Test Series 8 is retained as part of the testing regime for both UN 3375 and the proposed UN number for at least the current biennium.

10. The United Kingdom Expert believes that Test Series 8(a) and 8(b) are unsuitable to assess the safety of ammonium nitrate suspensions and gels because of their physical and chemical properties.

11. Test Series 8(a) was designed to demonstrate ingredients of ammonium nitrate emulsions do not self react or crystallise producing heat at 20ºC above their transport temperature. This

test will always give a negative result for ammonium nitrate suspensions described in the Spanish paper. Test 8(b) was designed to test the sensitiveness of ammonium nitrate emulsions at their transport temperature to an explosive shock. The emulsions do not change physical or chemical form with changing temperature. There is evidence that ammonium nitrate suspensions are subject to physical change and that new or different chemical solids are formed when these substances are thermally cycled. Test Series 8(b) does not include a thermally cycling requirement as part of the test.

12. The United Kingdom Expert proposes that, in addition to a new UN number, UN 3375 reverts to its original Name – “Ammonium nitrate emulsion, intermediate for blasting intermediate” and SP 309 is also amended. That UN 3XXX Name is “Ammonium nitrate suspensions and gels, intermediate for blasting explosive” and a new definition is used for these substances ".

"This entry applies to non sensitised emulsions, suspensions and gels consisting primarily of a mixture of ammonium nitrate and a fuel phase, intended to produce a Type E blasting explosive only after further processing prior to use.

The mixture for suspensions and gels typically has the following composition: 60-85% ammonium nitrate; 0-5% sodium or potassium perchlorate; 0-17% hexamine nitrate or monomethylamine nitrate; 5-30% water; 2-15% fuel; 0.5-4% thickening agent; 0-10% soluble flame suppressants; and trace additives. Other inorganic nitrate salts may replace part of the ammonium nitrate.

Substances shall satisfactorily pass Test Series 8 of the Manual of Tests and Criteria, Part I, Section 18 and be approved by the competent authority”

13. The United Kingdom also proposes that Box 2a is amended to remove "ANE" at the end of the text and a modified Box 19 in Figure 10.2 be changed to include Test Series 8 and a new Test Series 9 which will be used to classify ammonium nitrate suspensions and gels into Class 5.1. The flow chart for this proposed test is described in Annex 1 of this document

14. The proposed Test Series 9 should include a test which subjects the ammonium nitrate suspension or gel to five thermal cycles +/-25ºC of the transport temperature followed by an impact/friction test. The method described in UN/SCETDG/24/INF.54 could be used as the Proposed Test 9(a).

15. Alternatively, if the proposal outlined above is not suitable then dried ammonium nitrate suspension or gel should be subjected to the test described in the Spanish paper (UN/SCETDG/24/INF.18). *"Commercial suspensions were dried so as to obtain humidity levels between 1.1 and 1.3 %, respectively. The crystals obtained were submitted to Test 3(a)(ii) BAM fallhammer obtaining limiting impact energies between 39 and 49 J. An emulsion dried to obtain a humidity level of 1.2 was also tested, obtaining a limiting impact energy of 49 J. These values are above those obtained for ANFO (20-25 J)."*

16. The proposed Test 9(b) should subject the ammonium nitrate suspension or gel to five thermal cycles +/-25ºC of the transport temperature followed by a detonation test such as the ANE gap test.

17. The proposed Test 9(c) should be the Keonen Test using the same procedure as Test 8(c). The procedures for these Tests 9(a), (b) and (c) are described in Annex 1.

18. The United Kingdom Expert has studied the research on the modified vented pipe test and is concerned that the test does not adequately reflect the hazards posed by transport in tanks. The United Kingdom Expert suggests that Canadian work on the Minimum Burning Pressure Test (MBP) described in UN/SCETDG/21/INF.22 continues and is considered as a candidate test to replace the Modified Vented Pipe Test in the near future.

Annex 1

Revised Box 2a

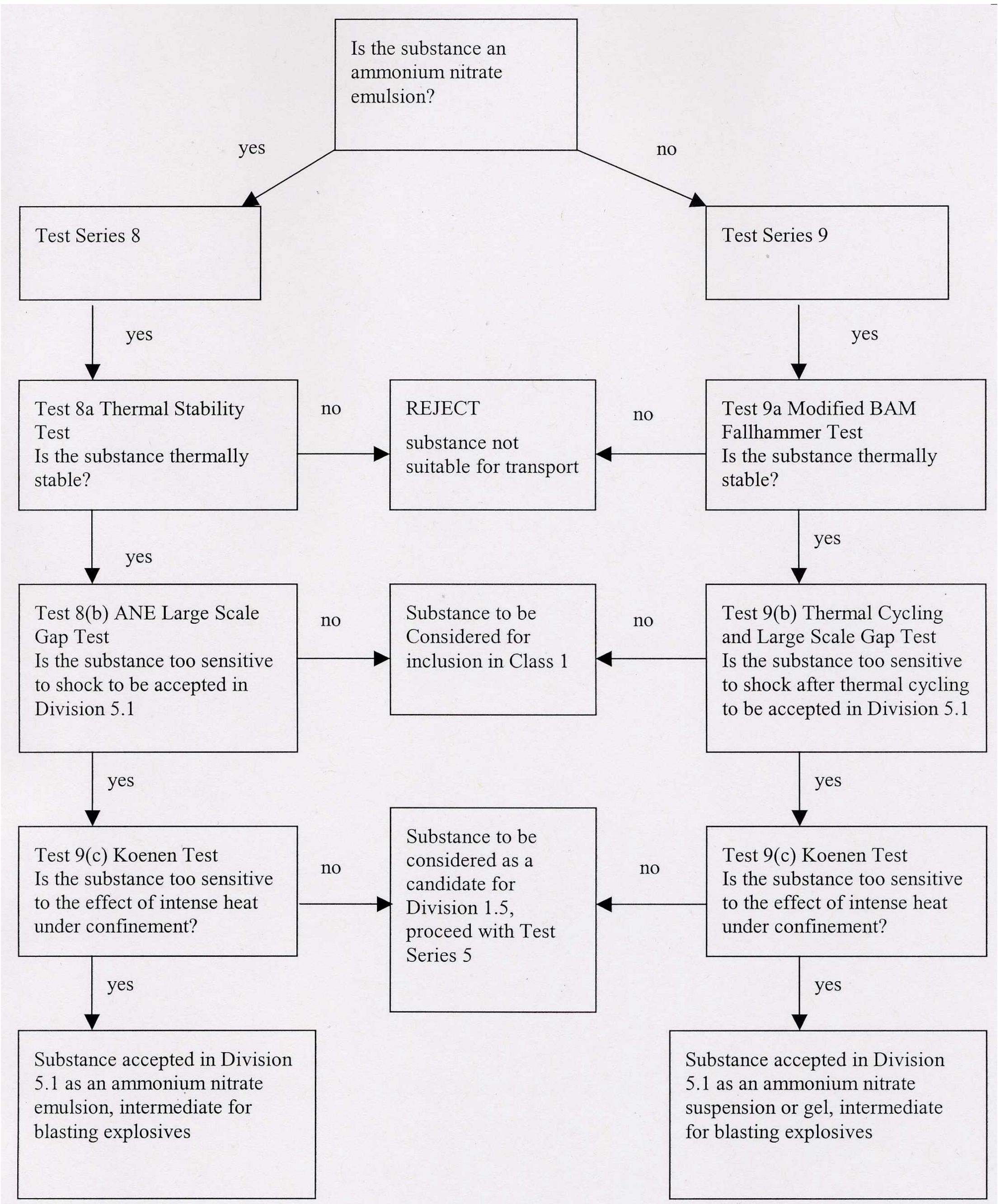
Is the substance a candidate for ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosive?

Revised Box 19

Test Series 8 & 9
(go to Figure 10.4)

New Figure 10.4

Figure 10.4 Procedure for ammonium nitrate emulsion, suspension and gel, intermediate for blasting explosives



Annex 2

TEST SERIES 9

X.1 Introduction

The assessment whether a candidate for "ammonium nitrate suspension or gel, intermediate for blasting explosives (ANE)" is insensitive enough for inclusion in Division 5.1 is answered by series 9 tests and any such candidate for inclusion in Division 5.1 should pass each of the three types of tests comprising the series. The three test types are:

type 9 (a) - a test to determine the thermal stability;
type 9 (b) - a shock test to determine sensitivity to intense shock;
type 9 (c) - a test to determine the effect of heating under confinement;

X.2 Test methods

The test methods currently used are listed in table X.1.

Table X.1: TEST METHODS FOR TEST SERIES 9

Test code	Name of Test	Section
9 (a)	Modified BAM Fallhammer Test */	X.4.1
9 (b)	Thermal Cycling and Large Scale Gap Test */	X.4.2
9 (c)	Koenen test */	X.4.3

*/ Recommended test.

X.3 Test conditions

X.3.1 The substance should be tested as offered for transport, at the highest transport temperature (see 1.5.4 of this Manual).

X.4 Series 9 Type (a) test prescription

X.4.1 Test 9 (a): Modified BAM Fallhammer Test

X.4.1.1 *Introduction*

X.4.1.1.1 This test is used to measure the sensitiveness of ammonium nitrate suspensions and gels to thermal cycling followed by drop-weight impact and to determine if the substance is too dangerous to transport in the form tested.

X.4.1.2 *Procedure*

X.4.1.2.1 The essential parts of the fallhammer are the cast steel block with base, the anvil, the column, the guides, the drop weights with release device and the impact device. A steel anvil is screwed onto the steel block and cast base. The support, into which is fixed the column (made from a seamless drawn steel tube), is bolted to the back of the steel block. The dimensions of the anvil, the steel block, the base and the column are given in Figure X.4.1.1. The two guides which are fixed to the column by means of three cross-pieces are fitted with a toothed rack to limit the rebound of the drop weight and a movable graduated scale for adjusting the height of the fall. The drop weight release mechanism is adjustable between the guides and is clamped to them by the operation of a lever nut on two jaws. The apparatus is fixed onto a concrete block (600 x 600 mm) by means of four anchoring screws secured in the concrete, so that the base is in contact with the concrete over its whole area and the guides are exactly vertical. A wooden protective box with inner protective lining and which can be opened easily, surrounds the apparatus up to the level of the bottom cross-bar. An extraction system allows removal of any explosion gases or dust from the box.

X.4.1.2.2 The drop weights are shown in Figure X.4.1.1. Each drop weight is provided with two locating grooves holding it between the guides as it drops, a suspension spigot, a removable cylindrical striking head and a rebound catch which are screwed on to the drop weight. The striking head is of hardened steel (HRC hardness of 60 to 63); its minimum diameter is 25 mm; it has a shoulder piece preventing it from being forced into the drop weight by the impact. Three drop weights are available with the following masses, 1.00 kg, 5.00 kg and 10.00 kg. The 1 kg-drop weight has a heavy steel centre fitted with the striking head. The 5 kg and 10 kg drop weights are of massive and compact steel, e.g. material specification at least St 37-1 in accordance with DIN 1700.

X.4.1.2.3 The sample of the substance under test is enclosed in an impact device consisting of two coaxial steel cylinders, one above the other in a hollow cylindrical steel guide ring. The cylinders are steel rollers from roller bearings with polished surfaces and rounded edges and a HRC hardness between 58 and 65. The dimensions of the cylinders and the ring are given in Figure X.4.2.3 *(not included)*. The impact device is placed on an intermediate anvil and centred by a locating ring with a ring of vent-holes to permit the escape of gases. The dimensions of the intermediate anvil are given in Figure X.4.2.4 and those of the locating ring in Figure X.4.2.3*(not included)*.

X.4.1.3 *Apparatus and materials*

X.4.1.3.1 A nominal 30 g and a nominal 400 g sample of each substance, in closed containers fitted with a pinhole vent, are cycled five times at 25°C above and below their transport temperature. The samples are maintained at the holding temperatures for no less than 18 and no more than 24 hours; all of the samples experience five excursions up to the higher temperature and five excursions down to the lower temperature. Aliquots of each sample, maintained at 20 + 5 °C, are used as controls for comparative purposes.

X.4.1.3.2 The cylinders and the guide ring should be degreased with acetone before use. The cylinders and guide ring should only be used once.

X.4.1.3.3 For ammonium nitrate suspensions and gels, a cylindrical tube of the same capacity is inserted into the substance and after levelling off the surplus, the sample is removed from the tube by means of a wooden rod. For liquid substances, a fine-drawn pipette of 40 mm3 capacity is used. The upper steel cylinder is gently pressed until it touches the sample without flattening it. The filled impact device is placed centrally on the main anvil, the protective wooden box is closed and the appropriate drop weight, suspended at the required height, is released. In the interpretation of the results of the trial, distinction is made between "no reaction", "decomposition" (without flame or explosion) recognisable by change of colour or odour and "explosion" (with weak to strong report or inflammation). In some cases it is advisable to perform trials with appropriate inert reference substances to allow a better judgement of whether or not an audible report has occurred.

X.4.1.3.4 The limiting impact energy, characterising the impact sensitiveness of a substance, is defined as that lowest impact energy at which the result "explosion" is obtained from at least one out of at least six trials. The impact energy used is calculated from the mass of the drop weight and the fall height (e.g. 1 kg x 0.5 m 5 J). The 1 kg drop weight is used at fall heights of 10, 20, 30, 40 and 50 cm (impact energy 1 to 5 J); the 5 kg drop weight for fall heights of 15, 20, 30, 40, 50 and 60 cm (impact energy 7.5 to 30 J) and the 10 kg drop weight for fall heights of 35, 40 and 50 cm (impact energy 35 to 50 J). The series of trials is started with a single trial at 10 J. If at this trial the result "explosion" is observed, the series is continued with trials at stepwise lower impact energies until the result "decomposition" or "no reaction" is observed. At this impact energy-level, the trial is repeated up to the total number of six if no "explosion" occurs; otherwise the impact energy is reduced in steps until the limiting impact energy is determined. If at the impact energy level of 10 J the result “decomposition” or “no reaction” (i.e. no explosion) was observed, the test series is continued by trials at stepwise increased impact energies until for the first time the result “explosion” is obtained. Now the impact energy is lowered again until the limiting impact energy is determined.

X.4.1.4 Test criteria and method of assessing results

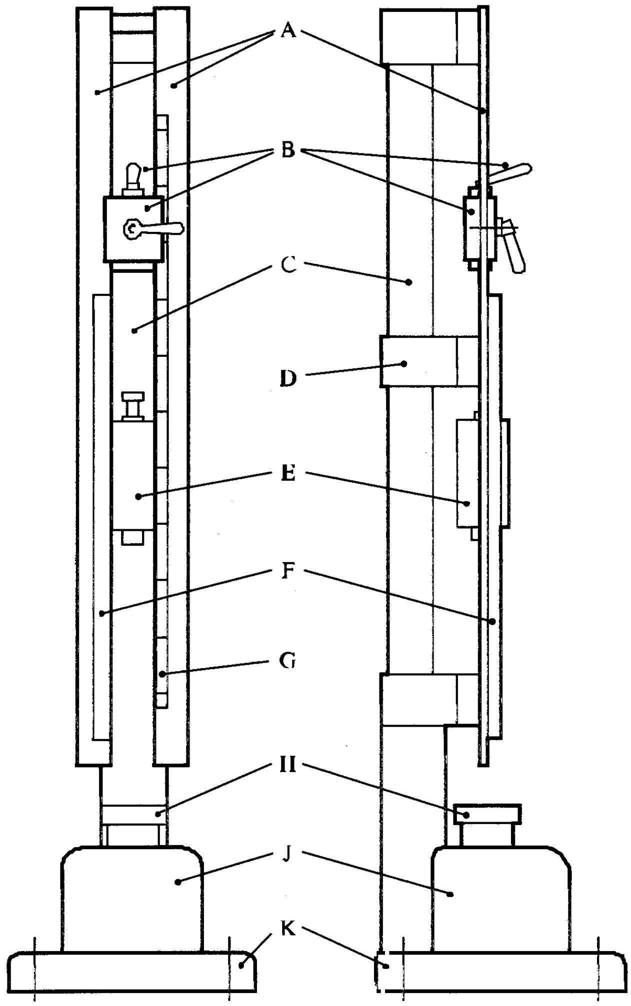
The test results are assessed on the basis of:

(a) Whether an “explosion” occurs in any of up to six trials at a particular impact energy; and
(b) The lowest impact energy at which at least one “explosion” occurs in six trials.

The test result is considered "+" if the lowest impact energy at which at least one “explosion” occurs in six trials is 2 J or less and the substance is considered too dangerous for transport in the form in which it was tested. Otherwise, the result is considered "-".

X.4.1.5 *Examples of results*

Sample	**Appearance**		**Mass Loss after cycling (%)[(a)]**	**Limiting Impact Energy (J)**
	Control	**After Cycling**		
AN Suspension Type SP1	Viscous, opaque fluid with two phases: Lower phase, white solid dispersed in viscous liquid	Uniform fine-grained non-mobile suspension, opaque white. Some crystal growth on surface with a 'skin'. Rust coloured spot in both 'large' and 'small' samples	0.3 and 1.1	>50
AN Suspension Type SP5	Viscous but fluid and opaque; two phases, upper 'syrup-like' Lower phase, as upper phase with dispersed white grainy solid	Layered, non-mobile suspension, opaque white. Some crystal growth on surface with a 'skin' of clear crystals. Thin layer of a viscous liquid on top of a similar phase containing white granules. The bottom layer is gritty white crystalline. Rust-coloured spot in both 'large' and 'small' samples	0.3 and 1.1	>50



(A)	Two guides	(B)	Holding and releasing device
(C)	Column	(D)	Middle cross-piece
(E)	Drop weight	(F)	Toothed rack
(G)	Graduated scale	(H)	Anvil 100 mm diameter x 70 mm
(J)	Steel block 230 x 250 x 200 mm	(K)	Base 450 x 450 x 60 mm

Figure X.4.1.1: BAM FALLHAMMER GENERAL VIEW, FRONT AND SIDE DIMENSIONS

X.4.2 Series 9 Type (b) Test prescription

X.4.2.1 Thermal Cycling and Large Scale Gap Test

X.4.2.1.1 Introduction

This test is used to determine whether the suspension or gel is stable at temperatures encountered during transport (i.e. thermal cycling) and to measure the sensitivity of a candidate for "ammonium nitrate suspension or gel, intermediate for blasting explosives" to a specified shock level (i.e. a specified donor charge and gap) to determine if the candidate is too dangerous for transport.

X.4.2.1.2 *Apparatus and materials*

X.4.2.1.2.1 The Thermal Cycling of the sample for this test consists of holding a sample of the candidate composition at 20ºC above and below the transport temperature for 5 cycles each lasting 24 hours. The set-up for the sensitivity test consists of an explosive charge (donor), a barrier (gap), a container holding the test charge (acceptor), and a steel witness plate (target).

X.4.2.1.2.2 *Thermal cycling apparatus and materials*

X.4.2.1.2.2.1 2kg of the ammonium nitrate suspension or gel is placed in a large measuring cylinder or vessel with a large length to diameter ratio. The vessel and sample is placed in an insulated environment chamber capable of altering the transport temperature of the sample by ± 25ºC. The sample is held at each extreme temperature for a minimum of 3 hours. The sample is visually examined to determine whether there has been deterioration or segregation of the sample's components. The sample is to be subjected to the sensitivity test

X.4.2.1.2.3 *Sensitivity Test*

X.4.2.1.2.3.1 The following materials are to be used for the sensitivity test:

(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) the thermally cycled 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 suspension or gel somewhere between 3.5 and 4 GPa, depending on the type of donor used (see table X.5.1.1 and figure X.4.1.2);

(f) Mild steel plate, 200 mm x 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.

X.4.2.3 *Procedure*

X.4.2.3.1 As shown in figure X.4.2.1.1, 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.

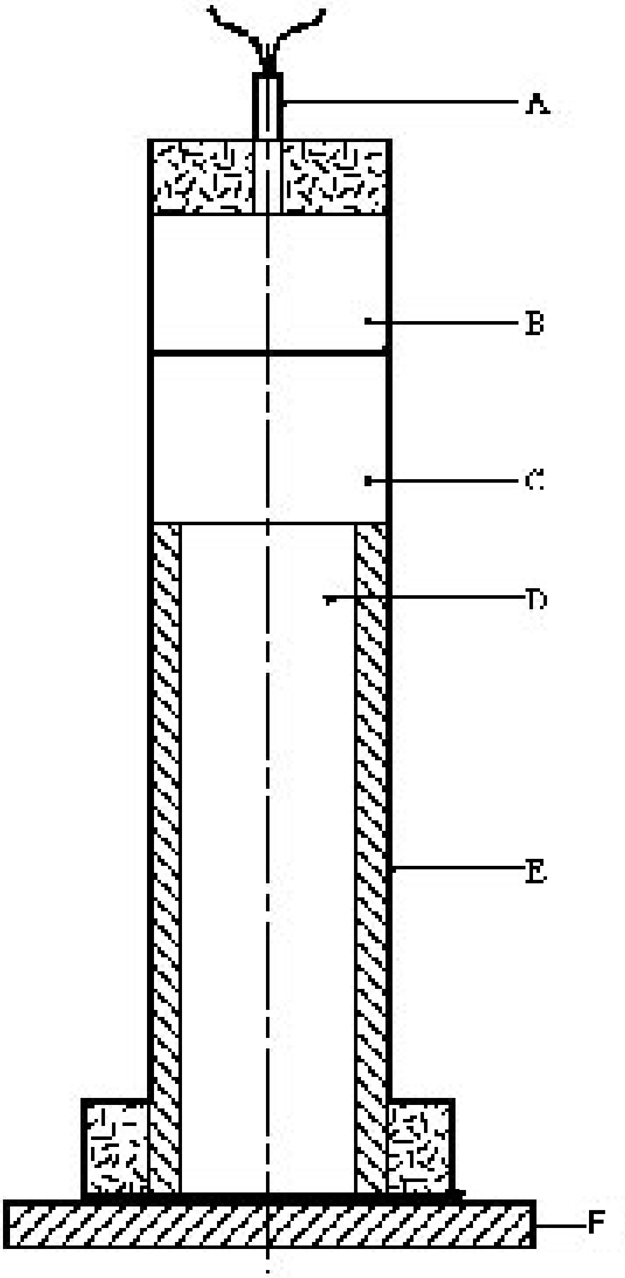
X.4.2.3.2 To assist in collecting the remains of the witness plate, the whole assembly may be mounted over a container of water with at least a 10 cm air gap between the surface of the water and the bottom surface of the witness plate which should be supported along two edges only.

X.4.2.3.4 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.

X.4.2.4 *Test criteria and method of assessing results*

A note is made if there is any deterioration or segregation of the components of the sample during the thermal cycling procedure.
In the sensitivity test, 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 suspension or gel, intermediate for blasting explosives" and the result is noted as "+".

Substances	Density g/cm^3	Gap mm	Result	Comments



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

Figure X.4.2.1.1: THERMAL CYCLING AND SENSITIVY TEST

Table X.4.2.1.1 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

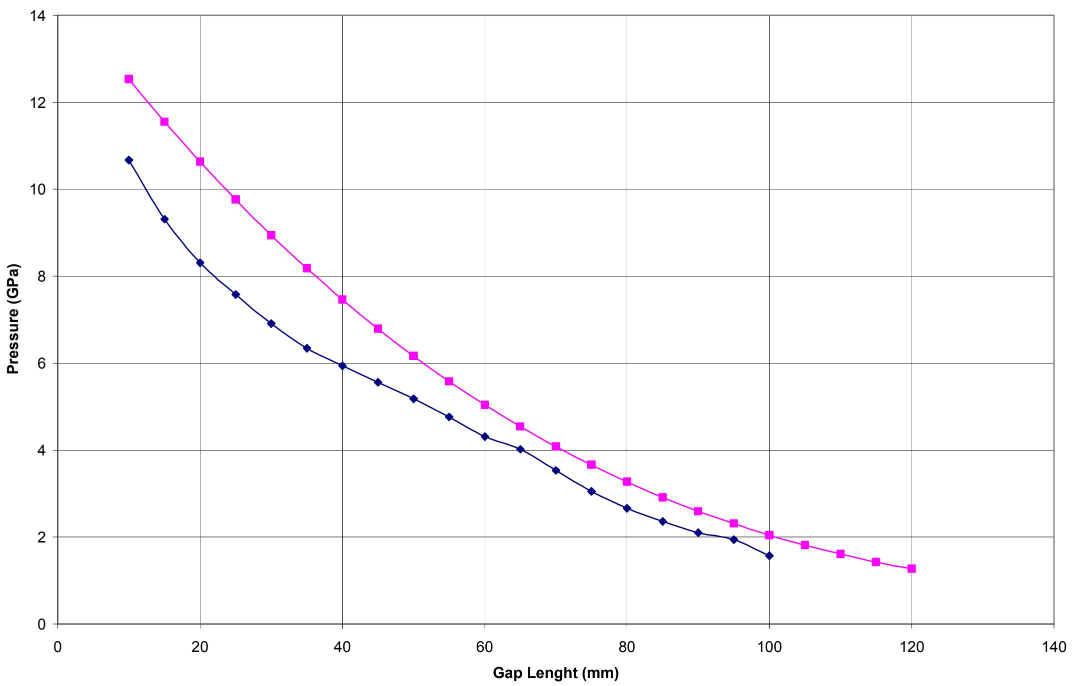


Figure X.4.2.1.2 : Test Calibration Data

X.4.3 Series 9 Type (b) Test prescription

X.4.3.1 Test 8(c): Koenen test

X.4.3.1 *Introduction*

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

X.4.3.2 *Apparatus and materials*

X.4.3.2.1 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 X.4.3.1.1. 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 X.4.3.1.1.

X.4.3.2.2 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 cm3 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.

X.4.3.2.3 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 X.4.3.1.2. 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 X.4.3.1.2. 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.

X.4.3.3 *Procedure*

X.4.3.3.1 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.

X.4.3.3.2 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.

X.4.3.3.3 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.

X.4.3.3.4 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 X.4.3.1.3 *(not included)*. 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".

X.4.3.3.5 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 X.4.3.2.1, 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.

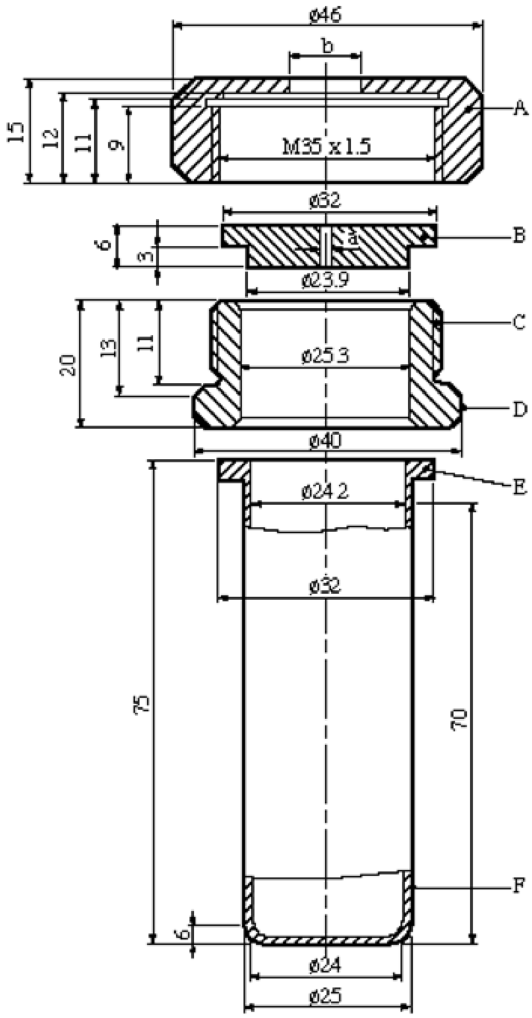
X.4.3.4 *Test criteria and method of assessing results*

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.

X.4.3.5 Examples of results

Substances	**Mass (g)**	**Hole diameter (mm)**				**Limiting diameter (mm)**
		1.0	**1.5**	**2.0**	**3.0**	
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	39.2-39.7	O,O,O	O			<1.0
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	39.5-41.1	F	O,O,O			1.0
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	39.5-41.1	O,F	O,O,O			1.0
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	39.0-41.0		F	O,O,O		1.5
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	39.6-39.9	F	O,O,O			1.0
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	39.9-40.1	O,O,O	O			<1.0

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



(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 X.4.3.1.1: TEST TUBE ASSEMBLY

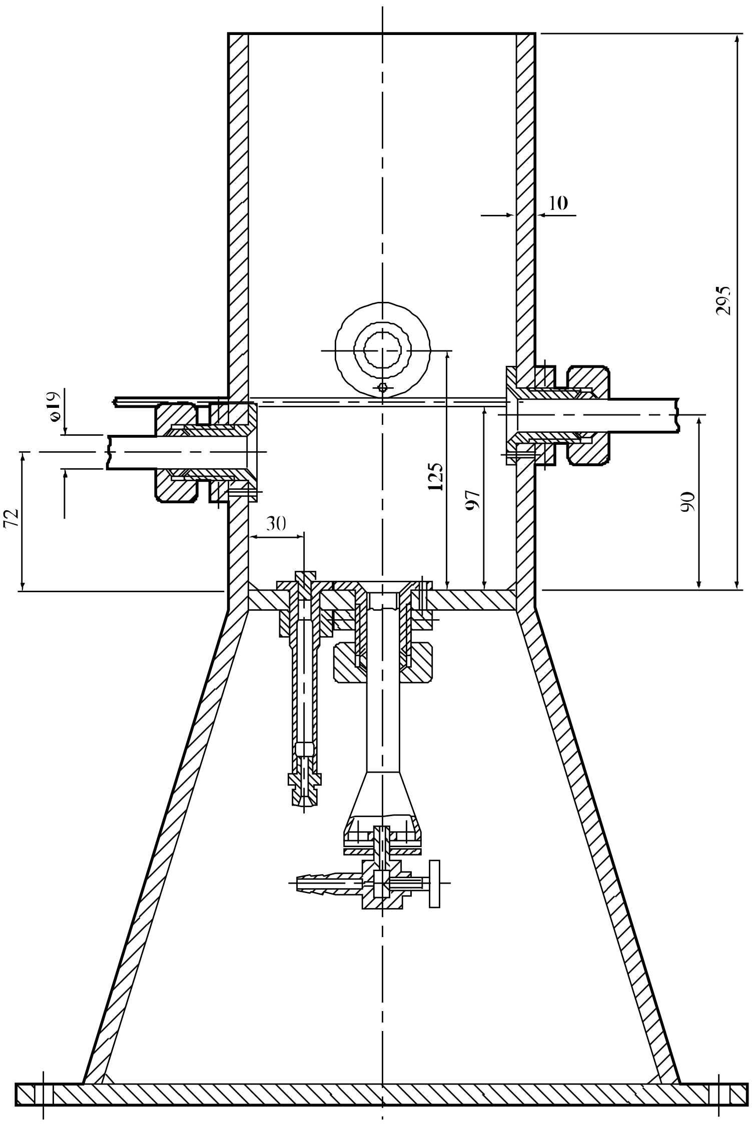


Figure X.4.3.1.2: HEATING AND PROTECTIVE DEVICE