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 2 of the provisional agenda

EXPLOSIVES AND RELATED MATTERS

Modifications to the time/ pressure test for defining flash powders

Transmitted by the expert from the United Kingdom

Background

1. At the 30th session of the Sub-Committee of Experts on the Transport of Dangerous goods the Expert from the United Kingdom presented ST/SG/AC.10/C.3/2006/84 and UN/SCETGD/30/INF3 which advocated changes to the time pressure test. During this session, it was agreed by the Sub-Committee that the definition of flash composition in 2.1.3.5.5 be changed because novel pyrotechnic compositions that had the same behaviour as flash compositions, but did not meet the definition, were being introduced (e.g. removing metal powder fuel and replacing with organic powder fuel). To address this problem, the Sub-Committee decided to amend Note 2 to 2.1.3.5.5 so firework compositions designed to produce an aural effect, or used as a bursting charge or lifting charge, should be considered as flash composition unless the time taken for the pressure rise is demonstrated to be more than 8ms for 0.5g of pyrotechnic composition in Test 2(c)(i) "Time pressure test" of the Manual of Test and Criteria.

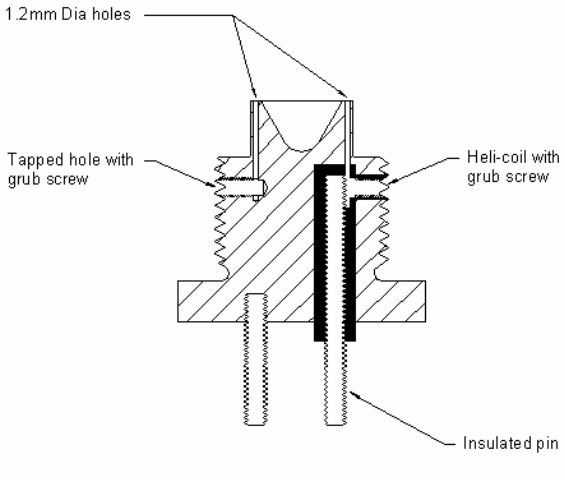
2. The expert from United Kingdom acknowledged the concerns of other experts, that the Time pressure test can in some cases give large standard deviations in the results, see for example (UN/SCETDG/30/INF.24)produced by the expert from the Netherlands. In the report of the Explosives Working Group at the July 2007session (UN/SCETDG/31/INF.45), the expert from the United Kingdom gave information on work undertaken to improve the Time pressure test and reduce the standard deviation. He also said this work would be completed and shared with the Working Group before the end of the year (2007). The research was conducted by the Health and Safety Laboratory and was presented at the 10th International Symposium on Fireworks, October 2007 and in UN/SCETDG/32/INF.36 "Modifications to the time/ pressure test for defining flash powders". The Sub-Committee was asked to provide comments to the UK expert on the modifications to the time pressure test (para. 36, ST/SG/AC.10/C.3/64). No comments were received by the expert from the United Kingdom.

Discussion

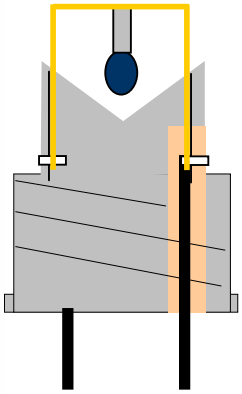
3. UN/SCETDG/32/INF.36 gave details of the proposed changes to the "Time pressure test" in Test Series 2.1(c) to improve the repeatability of the results obtained.

4. The research has focused on improving the ignition system and reducing the standard deviation of the results of the time/pressure test. The new 'Cone in plug' firing plug incorporates a cone into the plug body in which the pyrotechnic sample rests. This allows for the sample to be retained at the base of the test chamber. Retaining the sample in this way allows the ignition to initiate from the top of the sample. The ignition system consists of a single Vulcan fuse head, with leads to allow the fuse head to be consistently positioned above the pyrotechnic composition in the test chamber. The fuse head is inverted above the sample, ensuring consistent ignition. Primed Cambric is used in the ignition system of Test 2.1(c) "Time pressure test" but is not required in the "cone in plug" version to ignite pyrotechnic compositions.

Figure 1: Diagram of 'cone in plug' firing plug.



(a) Cone in firing plug design.



(b) Location of Vulcan fuse head in firing plug.

5. The firing plug was validated with the more energetic pyrotechnic substances, flash powders and black powders. It is envisaged that the modified test method would not be routinely used for the less energetic pyrotechnic substances (e.g. Black powder/large particle metal fountain compositions), but only for determining whether the composition used to produce aural effect, or used as a bursting charge or lifting charge is energetic enough to be flash powder (see Note 2 to 2.1.3.5.5).

6. Tables 1 and 2 below detail the results obtained both with the original test method and with the new firing plug. The original test method included the original ignition system with Primed Cambric. Both test methods used 0.5 g of material. The mean average rise time is quoted here rather than the fastest rise.

(a) Table 1 Comparison of original test method and new firing plug.

Composition	Mean rise time (ms) / Standard Deviation		Comments
	Original method	New method	
1	0.78 / 0.14#	0.70 / 0.10*	*5 repeats
2	5.10 / 1.18#	4.98 / 0.65*	*10 repeats
3	11.18 / 3.35#	11.98 / 3.46*	*5 repeats Homemade powder
4	3.11 / 3.31	1.51 / 0.47	
5	0.74 / 0.17	0.84 / 0.08	

Note: Data previously presented in UN/SCETDG/30/INF.3

(b) Table 2 Composition data

Composition	HSL reference	Composition (% mass)							
		Potassium perchlorate	Magnalium	Sulfur	Aluminium	Barium nitrate	Carbon	Potassium nitrate	Titanium
1	Comp AB Flash 1	45	22	11	22	-	-	-	-
2	Comp W HKF BP	-	-	10.4	-	-	15.6	74.0	
3	Comp V Flash 2	-	-	-	30	70	-	-	-
4	NN/6 Flash	60	-	-	25	-	-	-	15
5	NN/13 Flash	45	22	11	22	-	-	-	-

6. Compositions 1, 2, 4 and 5 all have similar mean average rise times for both test methods. The standard deviation is lower for the modified test method. Composition 3 was a ‘flash’ powder that was made in the laboratory. As such it was not representative of commercially manufactured compositions. It is suspected that it may have degraded between the original work reported in UN/SCETDG/30/INF.3 and this research.

Proposal

7. The expert from the United Kingdom proposes that the "cone in plug" modification of the Time/Pressure test and the modification of the ignition system, referred to above in Figure 1, is used to determine whether a pyrotechnic composition, used as aural effect, or used as a bursting

charge or lifting charge, is considered to be flash composition. A detailed technical drawing of the "Cone in plug" apparatus will be submitted separately in an INF paper.

8. Because of this modification to the ignition system, expert from the United Kingdom proposes that the test is published as an appendix to the Manual of Test and Criteria as this would avoid having two ignition systems and test methods listed in Test 2(c)(i). If this is accepted, then Note 2 to 2.1.3.5.5 should be amended to read:

> "***NOTE 2:*** *"Flash composition" in this table refers to pyrotechnic compositions in powder form or as pyrotechnic units as presented in the fireworks, that are used to produce an aural effect, or used as a bursting charge or lifting charge, unless the time taken for the pressure rise is demonstrated to be more than 8 ms for 0.5 g of pyrotechnic composition in the Flash Composition Test in Appendix X of the Manual of Tests and Criteria"*

Appendix X

Flash Composition Test

X.1 *Introduction*

This test is used to determine whether pyrotechnic compositions in powder form or as pyrotechnic units as presented in the fireworks, that are used to produce an aural effect, or used as a bursting charge or lifting charge, are considered to be flash compositions for the purposes of determining the classification of fireworks using the UN default fireworks classification table in 2.1.3.5.5.

X.2 *Apparatus and materials*

X.2.1 The time/pressure apparatus (Figure XX) consists of a cylindrical steel pressure vessel 89 mm in length and 60 mm in external diameter. Two flats are machined on opposite sides (reducing the cross-section of the vessel to 50 mm) to facilitate holding whilst fitting the "cone in plug" firing plug and vent plug. The vessel, which has a bore of 20 mm diameter, is internally rebated at either end to a depth of 19 mm and threaded to accept 1" British Standard Pipe (BSP). A pressure take-off, in the form of a side-arm, is screwed into the curved face of the pressure vessel 35 mm from one end and at 90° to the machined flats. The socket for this is bored to a depth of 12 mm and threaded to accept the 1/2" BSP thread on the end of the side-arm. A washer is fitted to ensure a gastight seal. The side-arm extends 55 mm beyond the pressure vessel body and has a bore of 6 mm. The end of the side-arm is rebated and threaded to accept a diaphragm type pressure transducer. Any pressure-measuring device may be used provided that it is not affected by the hot gases or decomposition products and is capable of responding to rates of pressure rise of 690-2070 kPa in not more than 5 ms.

X.2.2 The end of the pressure vessel furthest from the side-arm is closed with a "cone in plug" firing plug which is fitted with two electrodes, one insulated from and the other earthed to, the plug body. The other end of the pressure vessel is closed by an aluminium bursting disk 0.2 mm thick (bursting pressure approximately 2200 kPa) held in place with a retaining plug which has a 20 mm bore. A soft lead washer is used with both plugs to ensure a good seal. A support stand (Figure 12.6.1.2) holds the assembly in the correct attitude during use. This comprises a mild steel base plate measuring 235 mm × 184 mm × 6 mm and a 185 mm length of square hollow section (S.H.S.) 70 × 70 × 4 mm.

X.2.3 A section is cut from each of two opposite sides at one end of the length of S.H.S. so that a structure having two flat sided legs surmounted by an 86 mm length of intact box section results. The ends of these flat sides are cut to an angle of 60° to the horizontal and welded to the base plate.

X.2.4 A slot measuring 22 mm wide × 46 mm deep is machined in one side of the upper end of the base section such that when the pressure vessel assembly is lowered, firing plug end first, into the box section support, the side-arm is accommodated in this slot. A packing piece of steel 30 mm wide and 6 mm thick is welded to the lower internal face of the box section to act as a spacer. Two 7 mm thumb screws, tapped into the opposite face, serve to hold the pressure vessel

firmly in place. Two 12 mm wide strips of 6 mm thick steel, welded to the side pieces abutting the base of the box section, support the pressure vessel from beneath.

X.2.5 The ignition system consists of a Vulcan electric fusehead of the type commonly used in low tension detonators. Fuseheads with equivalent properties may be used.

X.2.6 The procedure for the preparation of the ignition assembly starts with separation of the brass foil contacts of an electric fusehead from its insulator, (see Figure 12.6.1.3). The exposed portion of insulation is then cut off. The fusehead is then fixed onto the terminals of the firing plug by means of the brass contacts such that the tip of the fusehead is above the surface of the pyrotechnic composition.

X.3 *Procedure*

X.3.1 The apparatus, assembled complete with pressure transducer but without the aluminium bursting disk in position, is supported firing plug end down. 0.5g of the substance is introduced into the apparatus so as to be in contact with the ignition system. No tamping is carried out when filling the apparatus. Note should be taken of the charge weight used. The lead washer and aluminium bursting disk are placed in position and the retaining plug is screwed in tightly. The charged vessel is transferred to the firing support stand, bursting disk uppermost, which should be contained in a suitable, armoured fume cupboard or firing cell. An exploder dynamo is connected to the external terminals of the firing plug and the charge is fired. The signal produced by the pressure transducer is recorded on a suitable system which allows both evaluation and a permanent record of the time/pressure profile to be achieved (e.g. transient recorder coupled to a chart-recorder).

X.3.2 The test is carried out three times. The time taken for the pressure to rise from 690 kPa to 2070 kPa above atmospheric is noted. The average of three firings should be used for classification.

X.4 *Test criteria and method of assessing results*

The test results are interpreted in terms of whether a gauge pressure of 2070 kPa is reached and, if so, the time taken for the pressure to rise from 690 kPa to 2070 kPa gauge. The pyrotechnic compositions in powder form or as pyrotechnic units as presented in the fireworks, that are used to produce an aural effect, or used as a bursting charge or lifting charge, is to be considered as flash composition if the average pressure rise is shown to be less than, or equal to, 8ms for 0.5g of pyrotechnic composition.

Example of Results:

Substance	**Maximum Pressure Rise (kPa)**	**Mean time for a pressure rise from 690 to 2070kPa (ms) / Standard Deviation**	**Result**
1	>2070	0.70 / 0.10	Flash Composition
2	>2070	4.98 / 0.65	Flash Composition
4	>2070	1.51 / 0.47	Flash Composition
5	>2070	0.84 / 0.08	Flash Composition

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