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 **16 June 2011**

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Item 2 (d) of the provisional agenda
Explosives and related matters:
DDT Test and Criteria for flash compositioin

A Comparison of the results obtained for a set of pyrotechnic compositions subjected to the HSL Flash Composition Test and the US Modified DDT Test

Transmitted by the expert from the United Kingdom

Introduction

1. At its 37th session the Sub-Committee considered two papers regarding a proposed new DDT Test and Criteria for flash compositions used to produce an aural effect or used as a bursting charge, or lifting charge in fireworks (see ST/SG/AC.10/C.3/2010/31 and UN/SCETDG/37/INF.34 transmitted by the experts from the United States of America and the United Kingdom respectively).

2. In its report to the 37th session (UN/SCETDG/37/INF.73) the Explosives Working Group noted that it would be desirable to have both the HSL Flash Composition Test (T-P test) and the US Modified DDT Test (DDT test) test performed on samples so that the results could be evaluated as to whether the tests provide comparable results.

3. The Expert from the United Kingdom reports here the results of initial work that he has commissioned from HSL to compare the performance of samples of pyrotechnic compositions in both the HSL Flash Composition Test and the US Modified DDT Test.

Discussion of the Selection of the Sample Set

4. A small set of samples was selected comprising a range of types of pyrotechnic compositions. These samples (which are detailed along with the results of both the T-P test and DDT test in Annex I) were selected as:-

- Presenting a range of actual or expected performances in the T-P test;
- Representing a range of compositions with the potential to be encountered as burst charges in fireworks;
- Representing a range of particle sizes;
- Representing a mixture of granular and finely divided compositions.

5. The sample selection also took account of the comments of the Expert from the Netherlands in the report of the Explosives Working Group to the 37th Session and the comments of the Expert from the UK in the paper UN/SCETDG/37/INF.34 in relation to:-

- The broader types of composition that can be regarded as flash according to the description in Note 2 to 2.1.3.5.5 of the Model Regulations
- The potential for the proportion of flash contained within a firework to be used to determine its likely hazard division.

Comments on the test procedure given in ST/SG/AC.10/C.3/2010/31

6. The test procedure was reported by HSL as being relatively straightforward to undertake once certain discrepancies in the text of ST/SG/AC.10/C.3/2010/31 had been resolved and once materials available within the United Kingdom with properties similar to those described in the paper had been sourced.

7. The discrepancies in the paper between the description and the drawing were:-

a) The Apparatus and Materials Section of Annex 1 describes the sample tube as consisting *"of a heavy-wall cardboard convolute sample tube with an inside diameter of 25.4 mm and height 152 mm with a maximum wall thickness of 3.8 mm, closed at the base with a paper or thin cardboard cap membrane just sufficient to retain the sample"* whilst the drawing describes the sample tube as *"Tube, Convolute Craft Paper, 25-mm i.d., 3.8mm wall, 150mm long"*.

b) The Apparatus and Materials Section of Annex 1 describes the sample tube as being held within *"a rugged mild steel confinement cover or "cap" with inner walls and head section approx. 32 mm thick with an inside diameter of 38 mm, an outside diameter of 102 mm and a height of 152 mm"* whilst the drawing describes the cover or cap as *"Steel Sleeve, 38-mm i.d., 100-mm o.d., 165mm long bored from solid billet"* and Section IIIB describes the sleeve as a *"cylindrical mild steel cap of the same height [as the sample tube] with an inner diameter and head thickness of 38 mm. an outer diameter of 102 mm., an interior depth of 152 mm and an overall exterior height of approx. 190 mm."*

Comments on the Test Procedure as Employed by HSL

8. The sample tubes used were 152mm long with a 25mm internal diameter and 3.5mm wall thickness. They were formed from spiral wound card and closed off with 1mm thick card discs. The spiral wound tubes were sourced from a manufacturer of fireworks mortar tubes.

9. A mild steel tube of 38mm internal diameter, 102mm outside diameter diameter, 190mm long with an internal pocket 152mm deep was used. This type of tube was used because there were concerns that a shorter tube, would result in the end thickness of the sleeve being only 13mm thereby presenting potential safety issues.

10. A Vulcan fusehead of the type used in the T-P test was used to fire the sample.

11. A 425 micron sieve was used rather than a Number 40 mesh screen as part of the sample preparation.[1]

[1] Some of the samples were not sieved as part of the sample preparation. This change to the method was because the grain size was obviously greater than that of the mesh. Some of the sieved samples left a residue on the mesh. It is not known at this time what effect this may have had on the results.

Consideration of the Results

12. A tabulated comparison of the test results can be found at Annex I. A set of photographs showing the results of the tests on 8 of the 9 samples can be found at Annex II.

13. Although the sample set was relatively small and a limited number of tests were completed there was generally consistent agreement between the ranking of the performance of the samples in both the HSL Flash Composition Test and the US Modified DDT Test.

14. Substances that would not be traditionally regarded as flash (as described in **ST/SG/AC.10/C.3/2010/31**) gave positive results. This indicates that the modified DDT test can, like the HSL T-P test, identify compositions used in fireworks to produce an aural effect or as bursting or lifting charges that can present an enhanced hazard and that could produce an effect similar to traditional flash compositions.

15. Fine blackpowder and FO/A grade blackpowder gave positive results in the test.

Recommendations for Improvement to the Test Method

15. That granular samples be subjected to testing as is i.e. without sieving. Such an approach would allow a more appropriate assessment of the likely performance and hazard associated with a burst charge etc.

Proposals

16. That the Explosives working Group considers whether the current criteria for a positive result i.e. “if in any trial the witness plate is torn, perforated, pierced or otherwise penetrated (i.e. light is visible through the plate)” are appropriate or whether these criteria should be revised.

17. That the Explosives Working Group considers how the test could be conducted in order to determine how reproducible the test is at the margins of the go/no-go criteria. It is suggested that where the result is positive because there is a small tear in the witness plate all three runs are carried out in order to more fully assess the potential of the composition under test.

18. That the Explosives Working Group considers whether the results of the comparative tests suggest that there is the potential for the 8ms threshold for flash described in Note 2 to 2.1.3.5.5 of the Model Regulations to be reduced to a lower value.

19. The objective of the requirements of Note to 2.1.3.5.5 of the Model Regulations appears to be to ensure that fireworks classified according to the use of the default table do not include compositions that would present an enhanced hazard, different to that which would be expected purely from the fireworks’ size and construction. The Expert from the United Kingdom asks that the Explosives Working Group considers whether the results of the comparative tests indicate that the adoption of criteria in the Model Regulations combining the results from both the HSL Flash Composition Test and the US Modified DDT Test would have value.

e.g. “Flash composition” in this table refers to pyrotechnic substances in powder form or as pyrotechnic units as presented in the firework that are used to produce an aural effect or used as a bursting charge, or lifting charge unless:-

a) the time taken for the pressure rise is demonstrated to be more than xms for 0.5g of pyrotechnic substance in the HSL Flash Composition Test……

or

b) the pyrotechnic substance gives a negative result in the Modified US DDT test

or

c) the time taken for the pressure rise is demonstrated to be more than yms [where y<x] for 0.5g of pyrotechnic substance in the HSL Flash Composition Test and the pyrotechnic substance gives an ABC result in the Modified US DDT test.

Where x < 8ms and ABC is a description of the type and frequency of a tolerable positive result e.g. a tear in the witness plate at the circumference of the supporting steel ring of no more than [20mm] in length which occurs with a frequency of no more than once in every n runs.

Annex I

Results of Comparative Tests

Sample	Description	Chemical Composition	Grain Size	T-P Test Result (ms)				DDT Test Result	
				Min	Average	SD	Run 1	Run 2	Run 3
Sample 1	Flashpowder 1	Potassium Perchlorate (50%), Aluminium Dark Pyro (40%), Magnesium #6 - Active (10%)	N/A	0.67	0.72	0.06	+	Not Run	Not Run
Sample 2	Flashpowder 2	Potassium Perchlorate (40%), Magnesium #6 - Active (60%)	N/A	1.41	1.57	0.23	+	Not Run	Not Run
Sample 3	Number 1 Blackpowder	N/A	0.25 – 0.50mm	2.14	2.84	0.47	-	+[2]	+
Sample 4	Flashpowder 3	Potassium Nitrate (60%), Magnesium #5 (40%)	N/A	2.31	2.40	0.14	+	Not Run	Not Run
Sample 5	Blackpowder Substitute[3]	Proprietary product marketed as providing an equivalent performance to FFFG	<425 micron	3.08	3.62	0.38	-	-	-

[2] Small tear which may have resulted from the cap falling back onto the plate after the shot.

		blackpowder							
Sample 6	Flashpowder 4	Potassium Perchlorate (64.2%), Aluminium – High Grade (20%), Magnesium # 5 (10%), Graphite (5.8%)	N/A	3.11	5.40	1.50	+	Not Run	Not Run
Sample 7	Comet Composition	Potassium Perchlorate (64%), Barium Nitrate (2%), Magnesium #5 (10%), Acaroid Resin (18%)	N/A	4.36	5.37	0.97	-	-	-
Sample 8	FO/A Blackpowder	N/A	0.25 – 0.8mm	4.83	5.83	0.84	-	+[4]	-
Sample 9	5FA Blackpowder	N/A	0.425 – 1mm	No Value	No Value	No Value	-	-	-

[3] This sample was run using a 1.5mm thick sample tube as part of the setting up of the equipment.
[4] Split along the stand edge

Annex II

Witness Plate Images

Sample 1 – Flashpowder 1

Sample 2 – Flashpowder 2

Sample 3 – Number 1 Blackpowder

Sample 3 – Number 1 Blackpowder – Run 2

Sample 3 – Number 1 Blackpowder – Run3

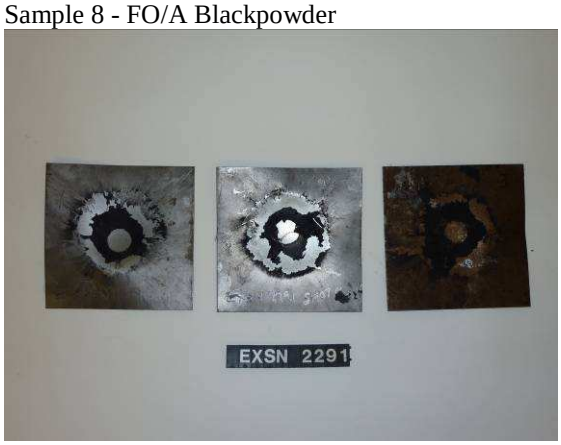
Sample 4 – Flashpowder 3

Sample 6 – Flashpowder 4

Sample 7 – Comet Composition

Sample 8 - FO/A Blackpowder

Sample 8 - FO/A Blackpowder – Run 2



Sample 9 - 5FA Blackpowder