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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

Forty-sixth session
Geneva, 1 – 9 December 2014
Item 2 (a) of the provisional agenda
Recommendations made by the Sub-Committee on its forty-third, forty-fourth and forty-fifth sessions and pending issues: explosives and related matters

Proposals on the apparatus, materials and criteria of US- and HSL Flash Composition Tests

Transmitted by the expert from Japan[1]

Introduction

1. The "US Flash Composition Test" proposed by the United States of America has been developed by the Sub-Committee on the Transport of Dangerous Goods as an alternative to the HSL Flash Composition Test.

2. At its forty-second session, the Sub-Committee agreed that further work would be undertaken to ensure comparability between US Flash Composition Test (US test) and HSL Flash Composition Test (HSL test) before the US test is formally accepted.

3. At the forty-third session, new proposals were submitted by the expert from the United States of America on materials and criteria of US test (ST/SG/AC.10/C.3/2013/24). The Working Group on explosives concluded that further data should be collected by the 45th session to finalize the refinement of the US test (informal document INF.61/Rev.1, 43rd session).

4. As a member of the study group of US test, Japan has conducted experiments of US- and HSL tests for various fireworks compositions and provided possible solutions for issues on the apparatus, materials and appropriate criteria of US- and HSL tests at the 45th session

[1] In accordance with the programme of work of the Sub-Committee for 2013-2014 approved by the Committee at its sixth session (refer to ST/SG/AC.10/C.3/84, para. 86 and ST/SG/AC.10/40, para. 14).

(informal document INF.19, 45th session). The Working Group on explosives generally supported the proposals in informal document INF.19 and concluded to ask Japan to prepare a formal proposal by taking account of its comments (informal document INF.61, 45th session).

Discussion

5. The proposals in this document are mainly based on the text and figure originally submitted by the expert from the United States of America (ST/SG/AC.10/C.3/2013/24) that intended to modify the US Flash Composition Test method tentatively adopted at the June 2012 session (ST/SG/AC.10/C.3/82/Add.1, Annexes I and II). According to the discussion in the Working Group of explosives at the forty-fifth session and comments received afterward, the text and figure in the original proposal from the United States of America are revised in the following respects:

(a) To avoid unnecessary air gap between the witness plate and the bottom end of the substance to be tested, a sentence and illustrations are added to the "Procedure" section and Figure A7.10 of US Flash Composition Test.

(b) To extend the availability of 1.0 mm thick steel witness plates, SPCC (JIS G 3141) is added to the recommended steel grades that were originally S235JR and ST37 only. Consequently, acceptable range of mechanical property, particularly a percentage elongation after fracture ("a break limit" in the original text) is fairly extended because the value of percentage elongation after fracture of SPCC is 39-46%[2] that is significantly greater than 26 % of S235JR or ST37. Based on the Japanese data using SPCC for the witness plate, an indentation depth threshold, above which the witness plate begins to be torn, was about 17 mm and this value was regarded as a possible criterion for the classification. However, it is considered that under the given pressure load, the witness plate having larger percentage elongation after fracture shows larger indentation depth without being torn. Therefore, strictly speaking, the criterion of indentation depth should be adjusted considering the type of steel grade chosen by the testing authority. In this respect the value of 15 mm proposed by USA, using S235JR or ST37 for the witness plate, can be regarded as a safe side criterion.

(c) To avoid complexity of adjusting criterion in accordance with steel grade for the witness plate, the safe side value of 15 mm is employed as a default criterion of the average depth of indentation. Then a criterion of 17 mm is optionally accepted under the condition that a percentage elongation after fracture of the selected steel grade (such as SPCC) is confirmed to exceed 40 % by tensile test in accordance with ISO 6892-1:2009. This option is provided in a note newly added at the end of "Test criteria and method" section of US Flash Composition Test.

(d) The text in subsection 3.1 of the "Procedure" section of US Flash Composition Test is modified to indicate that both density and particle size distribution of pyrotechnic substance achieved in a fireworks device during transport should be taken into account to consider the worst case of the condition of pyrotechnic substance.

[2] See Table 1 of informal document INF.19, 45th session. The data of SPCCs of unknown manufacturer were omitted. The unit of strength in this table should be corrected from kN/mm^2 to N/mm^2.

(e) The text in the test criterion (b) for US Flash Composition Test is modified to clarify that the "average" is meant to be the average of the maximum depths of indented witness plates from all three trials..

(f) In the indenting process of witness plate, the inner diameter of the steel ring is a crucial factor because it defines indented area. Furthermore, the projected area of the steel sleeve on the witness plate and the volume of the round bore of the steel sleeve are also important factors because they can influence the pressure that builds in the space between the steel sleeve and the witness plate. Thus, the inner diameter of the steel ring, the inner and outer diameters of the steel sleeve and the volume of the round bore, i.e. the depth of the bore should be specified. It is also considered that symmetrical alignment of the steel sleeve, the steel ring and, possibly, the witness plate is important to achieve a symmetrical and, hence, reproducible indentation of the witness plate. Therefore, the specifications corresponding to above mentioned crucial factors are revised or newly added. On the other hand, the original words “bored from a solid billet” in the specification of the steel sleeve is omitted because the steel sleeve can also be fabricated by using a steel tube and a steel lid that is screwed and welded on the edge of the tube.

(g) The criterion of minimum pressure rise time of the HSL Flash Composition Test shown in the Note 2 of section 2.1.3.5.5 of the Model Regulations is changed from 6 ms to 4 ms according to the agreement in the working group of explosives that black powder is generally not flash composition (informal document INF.61 43rd session). It should be noted that this criterion of 4 ms is still conservative because some kinds of black powders showed minimum pressure rise time of less than 4 ms (see ST/SG/AC.10/C.3/2012/78). Consequently, the text and table in the section 4 of the HSL Flash Composition Test in the Appendix 7 of the Manual of Tests and Criteria are amended. These consequential amendments were not included in the original proposals by the United States of America.

6. The rest of proposals are also based on the discussion in the Working Group on explosives at the 45th session and comments received afterward including change of the recommended thickness of aluminium bursting disc from “0.2 mm” to “at least 0.2 mm”, addition of a new deformable material for washer in the section 2.2 and editorial amendment of the table in the section 4 of the HSL Flash Composition Test in the Appendix 7 of the Manual of Tests and Criteria.

Proposals

7. All proposed amendments are combined and shown below.

Proposed amendments to the Manual of Tests and Criteria

Rename the title of Appendix 7 to read "**FLASH COMPOSITION TESTS**”

Insert a new subsection heading “**A. HSL Flash Composition Test**” at the beginning.

Amend 2.2 as follows,

“2.2 The end of the pressure vessel furthest from the side-arm is closed with a cone in 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 disc <u>at least</u> 0.2 mm thick (bursting pressure approximately 2 200 kPa) held in place with a retaining plug which has a 20 mm bore. A soft lead

washer or a washer of a suitable deformable material (for example, polyoxymethylene) is used with both plugs to ensure a good seal."

Amend section 4 as follows,

"4. Test criteria and method of assessing results

The test results are interpreted in terms of whether a gauge pressure of 2 070 kPa is reached and, if so, the time taken for the pressure to rise from 690 kPa to 2 070 kPa gauge. The pyrotechnic substances 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 minimum time taken for the pressure rise is shown to be less than, or equal to, 4 ms ~~8 ms~~ for 0.5 g of pyrotechnic substance.

Examples of results:

Substance	*Maximum pressure rise (kPa)*	*Minimum ~~Mean~~ time for a pressure rise from 690 to 2 070 kPa (ms)*	*Result*
1	> 2 070	0.70	Flash composition
2	> 2 070	4.98	~~Flash composition~~ Not flash composition
4	> 2 070	1.51	Flash composition
5	> 2 070	0.84	Flash composition
6	> 2 070	11.98	Not flash composition

"

Add the following new procedure at the end:

"**B. US Flash Composition Test**

1. Introduction

This test may be used to determine if pyrotechnic substances in powder form or as pyrotechnic units as presented in fireworks that are used to produce an aural effect or used as a bursting charge or propellant charge, may be considered a "flash composition" for the purposes of the default fireworks classification table in 2.1.3.5.5 of the Model Regulations.

2. Apparatus and materials

The experimental set up consists of:

- A cardboard or fibreboard sample tube with a minimum inside diameter of 25 mm and a maximum height of 154 ~~150~~ mm with a maximum wall thickness of 3.8 mm, closed at the base with a thin cardboard or paperboard disk, plug or cap just sufficient to retain the sample;
- A 1.0 mm thick 160 × 160 mm ~~steel~~ witness plate consisting of steel conforming to specification S235JR (EN10025) or ST37-2 (DIN17100) or SPCC (JIS G 3141) or equivalent having a stretch limit (or rupture strength) of 185-355 N/mm^2, an ultimate tensile strength of 336-379 N/mm^2 and a percentage elongation after fracture of 26-46% ~~Steel ST37 or Steel S235JR having a density of 7850 Kg/m2, a stretch limit of 185-355 N/mm2 , an ultimate strength of 340 N/mm2 and a break limit of 26%, or equivalent~~;

- An electric igniter, e.g. a fuse head, with lead wires of at least 30 cm in length;
- A mild steel confinement sleeve (weighing approximately 3 kg) having an outside diameter of 63 mm and a minimum length of 165 mm with a flat-bottomed round bore whose interior dimensions for diameter and depth are 38 mm and 155 mm, respectively, and ~~which is bored from a solid billet approximately 1 mm deeper than the overall sample tube length and having an inside diameter of 38 mm, an outside diameter of 63 mm and a height of 165 mm with~~ a notch or groove cut into one radius of the open end sufficient to allow the igniter lead wires to pass through (the steel sleeve might be provided with a rugged steel handle for easier handling);
- A steel ring of approximately 50 mm height with an inner diameter of ~~approximately~~ 95 mm; and
- A solid metal base, e.g. a plate of approximately 25 mm in thickness and 150 mm square.

3. Procedure

3.1 Prior to testing, the pyrotechnic substance is stored for at least 24 hours in a desiccator at a temperature of 20 - 30 °C. Twenty-five (25) g net mass of the pyrotechnic substance to be tested as a loose powder or granulated or coated onto any substrate, is pre-weighed and then poured carefully into a fibreboard sample tube with the bottom end closed with a cardboard or paperboard disk, cap or plug. After filling, the top cardboard or paperboard disk, cap or plug might be inserted lightly to protect the sample from spillage during transport to the test stand. The height of the sample substance in the tube will vary depending on its density. The sample should be first consolidated by lightly tapping the tube on a non-sparking surface. The final condition of the sample such as density and size distribution of the pyrotechnic substance in the tube should be as close as possible to the condition ~~density~~ achieved ~~when contained~~ in a fireworks device during transport.

3.2 The witness plate is placed on the supporting ring. If present, the paperboard or cardboard top disk, cap or plug of the fibreboard sample tube is removed and the electric igniter is inserted into the top of the pyrotechnic substance to be tested and visually positioned to an approximate depth of 10 mm. The paperboard or cardboard top disk, cap or plug is then inserted or re-inserted, fixing the igniter's position in the fibreboard sample tube and the depth of its match head. The lead wires are bent over and down along the sidewall and bent away at the bottom. The sample tube is placed vertically and centred on the witness plate. The steel sleeve is placed over the fibreboard sample tube. The igniter lead wires are positioned to pass through the slotted groove in the bottom edge of the steel confining sleeve and will be ready to attach to the firing circuit apparatus. Finally, the alignment of the steel sleeve and the witness plate is corrected so that their centres are aligned with the centre of the steel ring. See Figure A7.10 as an example of the test set-up. As illustrated in the figure, the cardboard or paperboard disk, cap or plug at the bottom end of the sample tube should be placed properly to avoid air gap between the witness plate and the bottom end of the substance to be tested.

3.3 The electric igniter is then initiated from a safe position. After initiation and a suitable interval the witness plate is recovered and examined. The test should be performed 3 times unless a positive result is obtained earlier.

4. Test criteria and method of assessing results

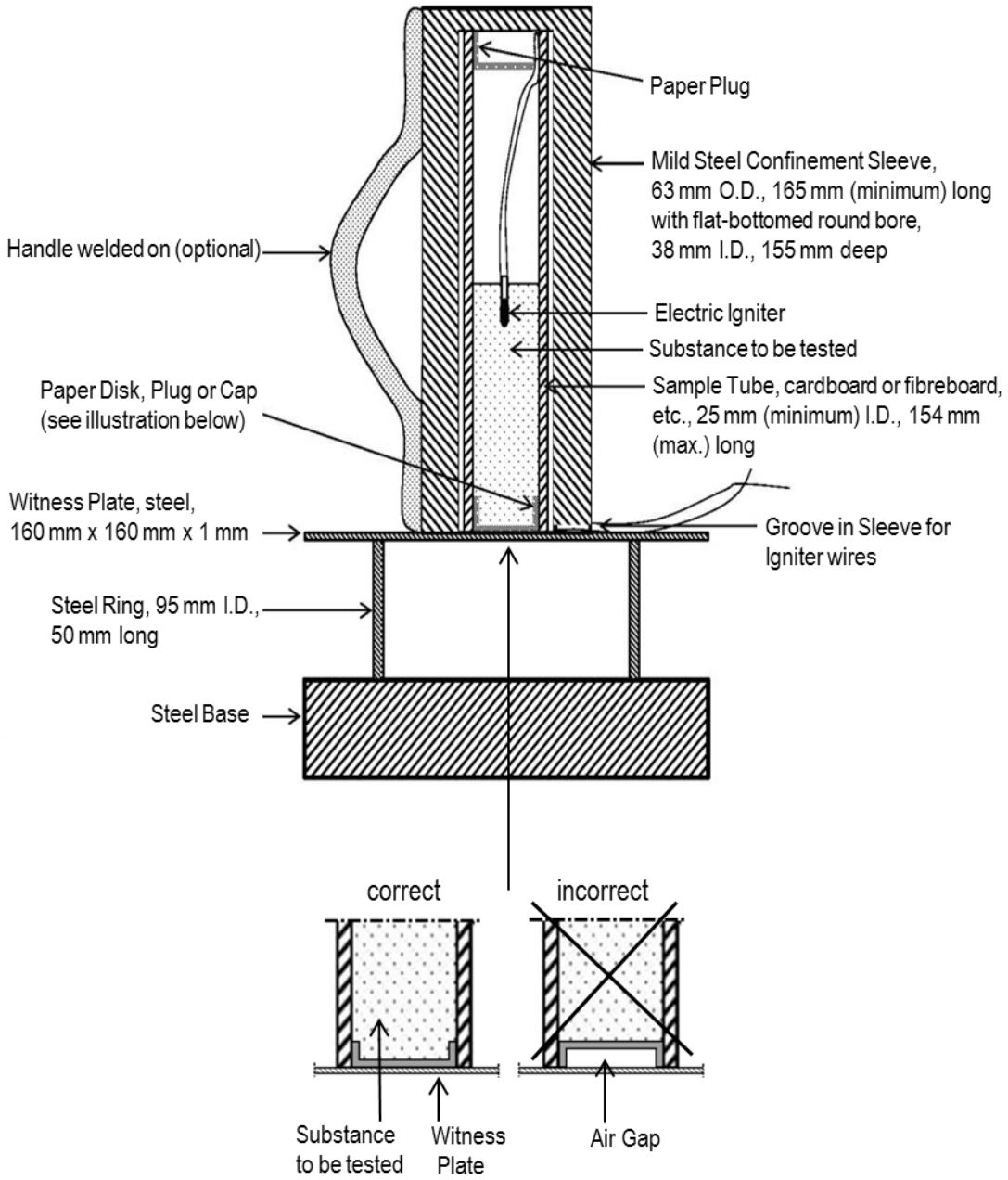
The result is considered positive "+" and the substance is considered to be a "flash composition" if:

(a) In any trial the witness plate is torn, perforated, pierced or penetrated; or;

(b) The average of the maximum depths of indented ~~depth of the indentations from the 1.0 mm thick steel~~ witness plates from all three trials exceeds 15 mm.

NOTE: *The criterion of the averaged maximum indentation depth may be increased to 17 mm if the percentage elongation after fracture of the witness plate measured in accordance with ISO 6892-1:2009 exceeds 40 %.*

Figure A7.10



Proposed amendments to the Model Regulations

Amend Note 2 in 2.1.3.5.5 to read as follows:

"***NOTE 2***: "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 propellant charge unless:

(a) The pyrotechnic substance gives a negative "-" result in the US Flash Composition Test in Appendix 7 of the Manual of Tests and Criteria; or

(b) The time taken for the pressure rise is demonstrated to be more than 4 ms ~~6 ms~~ for 0.5 g of pyrotechnic substance in the HSL Flash Composition Test in Appendix 7 of the Manual of Tests and Criteria.

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