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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-third session

Geneva, 24–28 June 2013

Item 2 (a) of the provisional agenda

Explosives and related matters: tests and criteria for flash compositions

Proposed modification of the US Flash Composition Test to measure both detonation and deflagration properties

Transmitted by the expert from the United States of America¹

Introduction

- 1. At the forty-first session, it was agreed to amend Note 2 of paragraph 2.1.3.5.5 for the classification of fireworks in the Model Regulations and add a new US Flash Composition Test to Appendix 7 of Manual of Tests and Criteria (See ST/SG.AC.10/C.3/78, para. 20-21 and ST/SG/AC.10/C.3/82/Add.1, Annexes I and II). However, at the forty-second session, the expert from the Netherlands submitted ST/SG/AC.10/C.3/2012/78 questioning the comparability of the US method with the existing HSL method and proposed postponement of the final adoption of the US Flash Composition Test to the next biennium in order to expand the criteria of the US Test to consider deflagration as well as detonation characteristics of certain pyrotechnic mixtures.
- 2. At the forty-second session, the expert from the United States of America also submitted new comparability data in informal document INF.28 commenting on the similarity of results of the HSL Flash Composition Test and the US Flash Composition Test. After a lengthy discussion, a majority vote decision was taken to postpone the adoption of the US Flash Composition Method pending further studies.
- 3. The expert from the United States does not disagree with the premise that a higher degree of agreement between the existing HSL Flash Composition Test Method and the

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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).

proposed new method would be beneficial to both manufacturers and importers seeking to arrive at the correct classification of their products and enforcing authorities seeking to evaluate suspect materials. Differing classifications based on different test methods applied could be detrimental to the credibility of the Fireworks Default System which would neither enhance public safety in transport nor simplify the regulatory burden on the commerce of fireworks.

4. This working paper further explores ways of modifying the US Flash Composition Test to measure both deflagration and detonation properties and offers further test data for both test methods showing how they may become comparable to a higher degree.

Discussion

- 5. Expanding on the conceptual idea of additionally measuring plate indentation effects (as well as the puncture or piercing phenomenon) on the steel witness plate in the US Test, answers to the following four questions were sought in this work:
 - (a) Which measurement is better indentation depth or volume displacement?
 - (b) Is it better to use the average of three measurements or the maximum of three measurements?
 - (c) What effects do the properties of the steel witness plate play?
 - (d) How to choose the indentation criteria to maximize comparability to the existing HSL Test?

These questions are discussed in the annex to this document (English only)

Proposals

- 6. Modify the US Flash Composition Method tentatively adopted at the June 2012 meeting of the subcommittee in two respects:
 - (a) The specifications for 1.0mm thick steel witness plate shall be detailed; and
 - (b) There be an second pass/fail criteria added for when the plate is not punctured or pierced, then the average indentation depth (of three witness plates) shall not be greater than 15 mm for the substance to be considered a flash composition.

The specific text changes to ST/SG.AC.10/C.3/2012/78, para. 20-21 and ST/SG/AC.10/C.3/82/Add.1, Annexes I and II are shown below in bold italics:

7. Proposed amendments to Appendix 7 of the Manual of Tests and Criteria (Refer to ST/SG/AC.10/C.3/82, annex I; changes are shown in bold).

Rename Appendix 7 to read "Flash Composition Tests"

Insert a new subsection heading "A. HSL Flash Composition Tests" at the beginning.

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 height 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 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 length;
- a mild steel confinement sleeve (weighing approximately 3 kg) 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 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 preweighed 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 density of the pyrotechnic substance in the tube should be as close as possible to the density achieved when contained in a fireworks device.
- 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. See Figure A7.10 as an example of the test set-up.

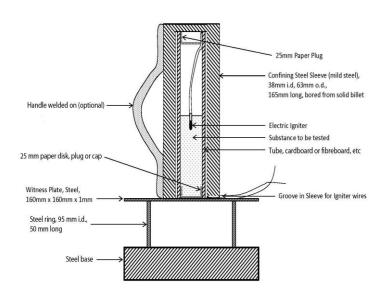
3.3 The electric ignite is then initiated from a safe position. After initiation and a suitable interval the witness plate is recovered and examined. The test should be performed 3 times unless a positive result is obtained earlier.

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 depth of the indentations from the 1.0 mm thick steel witness plates exceeds 15 millimetres.

Figure A7.10



8. Proposed amendments to the Model Regulations (Refer to ST/SG/AC.10/C.3/82/Add.1, annex II; changes are shown in bold)

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 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."

Annex [English only]

Discussion of the four questions in paragraph 5

- 1. Expanding on the conceptual idea of additionally measuring plate indentation effects (as well as the puncture or piercing phenomenon) on the steel witness plate in the US Test, answers to the following four questions were sought in this work:
 - (a) Which measurement is better indentation depth or volume displacement?
 - (b) Is it better to use the average of three measurements or the maximum of three measurements?
 - (c) What effects do the properties of the steel witness plate play?
 - (d) How to choose the indentation criteria to maximize comparability to the existing HSL Test?

Experimental I – Depth vs. Volume? Average Values vs. Maximum Values?

2. Data from informal document UN/SCETDG/42/INF.28 previously obtained on indentation depths (average and maximum) for 9 formulations with 1.219 mm (18 Gage) thick steel witness plates and those 5 formulations with 0.912 mm (20 Gage) thick steel witness plates not punctured or pierced (See Figures 1 and 2 below for technique of measurement.). The indentation depth data was expanded for this work to include an additional 14 formulations not previously tested with 0.912 mm thick steel witness plates and the average and maximum indentation depth values are summarized in columns 3, 4, 7 and 8 of Table IA below

Figure 1
Micrometer "Zeroing"

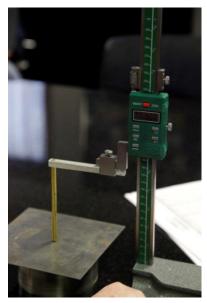


Figure 2
Micrometer Measuring at Deepest Point



3. In addition, each set of three 1.219 mm and 0.912 mm steel witness plates for those same 19 compositions which did not puncture through were measured for average and maximum displacement volumes in milliliters of dry fine sand (see Figures 3 through 5 below for technique of measurement.) For each set of three trials, the average and maximum (of 3) indentation volume data for both the 1.219 mm and 0.912 mm witness plates is summarized in columns 5, 6, 9 and 10 of Table IA below

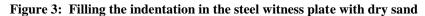




Figure 4: Emptying the Plate



Figure 5: Figure 5. Quantifying Displacement



4. Common beach sand was for the indentation volume displacement measurements with the following particle size analysis: 78.0% Thru 250 micron screen, 16.6% Thru 149 micron screen, 3.9% Thru 105 micron screen, 1.0% Thru 74 micron screen, 0.2% Thru 53 micron screen, 0.06% Thru 44 micron screen and 0.14% Residue On 37 micron screen.

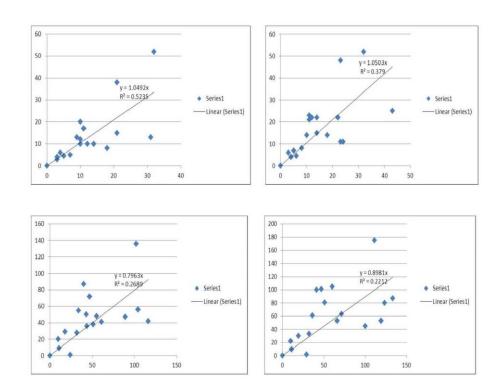
Table IA - Correlation of Steel Witness Plate Indentation Depths & Volumes for Formulations 1-30

| 1.219 mm Witness Plates (18 Gage) 0.912 mm Steel Witness Plates (20 Gage) | | | | | | | | | | | |
|---|-----------|----------|----------|----------|----------|----------|-------------|------------|--|--|--|
| | | | | | | | | | | | |
| Formulation | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent | | | |
| Number | Depth, | | | Volume, | Depth, | Depth | Volume, | Volume, | | | |
| | Ave (3) | Max of 3 | Ave (3) | Max of 3 | Ave. (3) | Max of 3 | Ave. (3) | Max of 3 | | | |
| 1 | 9 mm | 10 mm | 34 cc | 36 cc | 13 mm | 14 mm | 55 cc | 61 cc | | | |
| 2 | 21 mm | 23 mm | 102 cc | 111 cc | 38 mm | 48 mm | 136 cc | 175 cc | | | |
| 3 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 4 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 5 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 6 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 7 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 8 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 9 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Punctures | Punctures | | | |
| 10 | 32 mm (P) | 32mm (P) | Puncture | Puncture | 52mm(P) | 52mm(P) | Puncture(2) | 160 cc (1) | | | |
| 11 | 5 mm | 6 mm | 24 cc | 29 cc | 4.5 mm | 4.5 mm | 1 cc | 2 cc | | | |
| 12 | 18 mm | 24 mm | 89 cc | 119 cc | 8 mm | 11 mm | 47 cc (2) | 53 cc | | | |
| 13 | 31 mm | 43 mm | 116 cc | 133 сс | 13 mm | 25 mm | 42 cc | 87 cc | | | |
| 14 | 14 mm | 23 mm | 61 cc | 100 cc | 10 mm | 11 mm | 41 cc | 45 cc | | | |
| 15 | 12 mm | 14 mm | 51 cc | 66 cc | 10 mm | 15 mm | 38 cc | 53 cc | | | |
| 16 | 10 mm | 12 mm | 43 cc | 47 cc | 12 mm | 22 mm | 50 cc | 101 cc | | | |
| 17 | 11 mm | 14 mm | 47 cc | 60 cc | 17 mm | 22 mm | 72 cc | 105 cc | | | |
| 18 | 7 mm | 8 mm | 32 cc | 32 cc | 5 mm | 8 mm | 28 cc (2) | 33 cc (2) | | | |
| 19 | 21 mm | 22 mm | 104 cc | 123 cc | 15 mm | 22 mm | 56 cc | 80 cc | | | |
| 20 | 10 mm | 11 mm | 44 cc | 51 cc | 10 mm | 23 mm | 36 cc | 81 cc | | | |
| 21 | 14 mm | 18 mm | 55 cc | 71 cc | 10 mm | 14 mm | 48 cc | 63 cc | | | |
| 22 | 0 mm | 0 mm | 0 mm | 0 mm | 0 mm | 0 mm | Осс | O cc | | | |
| 23 | 24 mm | 29 mm | 104 cc | 127 cc | Puncture | Puncture | Punctures | Punctures | | | |
| 24 | 10 mm | 11 mm | 40 cc | 41 cc | 20 mm | 21mm | 87 cc | 100 cc | | | |
| 25 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 26 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 27 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | |
| 28 | 3 mm | 3 mm | 10 cc | 10 cc | 4 mm | 6 mm | 20 cc | 22 cc | | | |
| 29 | 4 mm | 5 mm | 18 cc | 19 cc | 6 mm | 7 mm | 29 cc | 30 cc | | | |
| 30 | 3 mm | 4 mm | 11 cc | 11 cc | 3 mm | 4 mm | 9 cc | 10 cc | | | |
| | | | | | | | | | | | |

- 5. In order to determine which metric was the best possible choice either:
 - A. Indentation Depth Ave. of 3;
 - B. Indentation Depth Max. of 3;
 - C. Indentation Volume Ave. of 3; or
 - D. Indentation Volume Max. of 3.

Graphs of the data for all formulations that were tested with 1.219mm (18 Gage) thick witness plates were compared against data for the same formulation data tested with the 0.912 mm (20 Gage) thick witness plates. A linear correlation was assumed probable between the two differing thickness of steel for the same formulation and each of the four metrics was tested for degree of correlation using least squares (R^2) analysis. The four graphs are shown in Figure 6 below.

Figure 6



6. The average indentation depth metric had the highest correlation between two thicknesses of steel witness plate with an R^2 value of 0.5235. The maximum indentation depth had the second highest correlation between two thickness of steel plate with an R^2 value of 0.379. The indentation volume metrics had significantly lower R^2 values and were not considered further. Therefore, the average (of 3) indentation depth measurement was considered the best metric to standardize the US Flash Composition Method against for measurement of deflagration characteristics.

Experimental II – Variable Steel Properties Other Than Thickness?

- 7. Although all prior US Flash Composition Testing had been performed with US made 18 Gage (1.219 mm thick) and 20 Gage (0.912 mm thick) steel witness plates, in order to explore the role of the steel witness plate sources play in the outcomes of the test, a small sample (60) of true 1.0 mm thick steel witness plates was obtained from Stuttgart, Germany. The reported properties of these German-sourced steel plates given by the supplier were as follows:
 - A "Standard steel" in Germany, called ST37 or Steel S235JR;
 - Density = 7850 Kg/m^2 ;
 - Stretch Limit = 185-355 N/mm²;
 - Ultimate Strength = 340 N/mm²;
 - Break Limit = 26%.

8. Two 1.0 mm steel witness plates were tested with each of five formulations for which 18 Gage and 20 Gage US Steel witness plate data had been previously obtained. These formulations were 2, 11, 14, 16 and 18. The results as shown in Table 1B were as follows:

| 1.219 mm Witness Plates (18 Gage) | | | 0.912 mm Steel Witness Plates (20 Gage) | | | | 1.0 mm Steel Witness Plates | | | | | |
|-----------------------------------|-----------|----------|---|----------|----------|----------|-----------------------------|------------|----------|----------|-----------|----------|
| Formulation | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent | Dent |
| Number | Depth, | Depth | Volume, | Volume, | Depth, | Depth | Volume, | Volume, | Depth, | Depth | Volume, | Volume, |
| | Ave (3) | Max of 3 | Ave (3) | Max of 3 | Ave. (3) | Max of 3 | Ave. (3) | Max of 3 | Ave. (2) | Max of 2 | Ave. of 2 | Max of 2 |
| 1 | 9 mm | 10 mm | 34 cc | 36 cc | 13 mm | 14 mm | 55 cc | 61 cc | | | | |
| 2 | 21 mm | 23 mm | 102 cc | 111 cc | 38 mm | 48 mm | 136 cc | 175 cc | 22 mm | 25 mm | 93 cc | 110 cc |
| 3 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | _ |
| 4 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 5 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 6 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 7 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 8 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 9 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Punctures | Punctures | | | | |
| 10 | 32 mm (P) | 32mm (P) | Puncture | Puncture | 52mm(P) | 52mm(P) | Puncture(2) | 160 cc (1) | | | | |
| 11 | 5 mm | 6 mm | 24 cc | 29 cc | 4.5 mm | 4.5 mm | 1 cc | 2 cc | 1 mm | 1 mm | 5 cc | 5 cc |
| 12 | 18 mm | 24 mm | 89 cc | 119 cc | 8 mm | 11 mm | 47 cc (2) | 53 cc | | | | |
| 13 | 31 mm | 43 mm | 116 cc | 133 сс | 13 mm | 25 mm | 42 cc | 87 cc | | | | |
| 14 | 14 mm | 23 mm | 61 cc | 100 cc | 10 mm | 11 mm | 41 cc | 45 cc | 3 mm | 4 mm | 8 cc | 10 cc |
| 15 | 12 mm | 14 mm | 51 cc | 66 cc | 10 mm | 15 mm | 38 cc | 53 cc | | | | |
| 16 | 10 mm | 12 mm | 43 cc | 47 cc | 12 mm | 22 mm | 50 cc | 101 cc | 6 mm | 7 mm | 19 cc | 25 cc |
| 17 | 11 mm | 14 mm | 47 cc | 60 cc | 17 mm | 22 mm | 72 cc | 105 cc | | | | |
| 18 | 7 mm | 8 mm | 32 cc | 32 cc | 5 mm | 8 mm | 28 cc (2) | 33 cc (2) | 7 mm | 10 mm | 19 cc | 32 cc |
| 19 | 21 mm | 22 mm | 104 cc | 123 cc | 15 mm | 22 mm | 56 cc | 80 cc | | | | |
| 20 | 10 mm | 11 mm | 44 cc | 51 cc | 10 mm | 23 mm | 36 cc | 81 cc | | | | |
| 21 | 14 mm | 18 mm | 55 cc | 71 cc | 10 mm | 14 mm | 48 cc | 63 cc | | | | |
| 22 | 0 mm | 0 mm | 0 mm | 0 mm | 0 mm | 0 mm | 0 сс | Осс | | | | |
| 23 | 24 mm | 29 mm | 104 cc | 127 cc | Puncture | Puncture | Punctures | Punctures | _ | | | |
| 24 | 10 mm | 11 mm | 40 cc | 41 cc | 20 mm | 21mm | 87 cc | 100 cc | | | | |
| 25 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 26 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 27 | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | Puncture | | | | |
| 28 | 3 mm | 3 mm | 10 cc | 10 cc | 4 mm | 6 mm | 20 cc | 22 cc | | | | |
| 29 | 4 mm | 5 mm | 18 cc | 19 cc | 6 mm | 7 mm | 29 cc | 30 cc | | | | |
| 30 | 3 mm | 4 mm | 11 cc | 11 cc | 3 mm | 4 mm | 9 cc | 10 cc | | | | |

Table IB - Correlation of Steel Witness Plate Indentation Depths and Volumes for Formulations 1-30

The results from the 1.0 mm thick steel witness plates did not fall in between the 1.219 mm (18 Gage) and 0.912 mm (20 Gage) US steel witness plate data as expected. Instead they more closely resembled the 1.219 mm (18 Gage) US Steel plates. Therefore it was concluded that for the method to be reproducible, a steel plate source and specification must be included in the procedure.

Experimental III – use deflagration property criteria to estimate comparability between the US and HSL flash test method?

- 9. Assuming that the deflagration properties of a given pyrotechnic mixture is reflected in the steel witness plate indentation results (if no puncture or piercing is evidenced) and subsequently assuming the best metric to assess that deflagration characteristic is the average indentation depth in mm, then by direct comparison between the US and HSL Flash Composition Test data. it should be possible to deduce an average indentation depth value that will give the highest degree of agreement between the two methods.
- 10. Twelve new compositions (five different perchlorate-containing whistle/burst mixtures and seven different perchlorate-containing color star mixtures) were evaluated for Flash Composition properties by the Modified HSL Flash Composition Test (three trials). The minimum pressure rise time (of three) was taken as the determinative result and if it

was less than 6 milliseconds the result was positive (+). These results summarized in Table II.

Table II – 12 New Formulations Evaluated for Flash Composition Properties by Modified HSL Test Apparatus

| Formula Number | Compo sition Type | Formula Compositions (all ingredients mixed as received) | All 3 HSL Test Results, ms. | Low HSL Result, ms | Pres- sure Rise > 6 ms? | Flash Compos ition by HSL? |
|-------------------|------------------------------|--|--------------------------------------|-----------------------------|----------------------------------|-------------------------------------|
| 31 | Whistle /Burst | 75 wt. % Potassium Perchlorate/25 wt.% Potassium Benzoate | 1.8, 2.3, 1.9 | 1.8 | Yes | Yes |
| 32 | " | 56 wt. % Potassium Perchlorate/44 wt.% Potassium Hydrogen Phthalate | 2.2, 3.7, 3.2 | 2.2 | Yes | Yes |
| 33 | " | 60 wt.% Potassium perchlorate/40 wt.% Potassium Hydrogen Phthalate | 4.0, 4.7, 3.5 | 3.5 | Yes | Yes |
| 34 | " | 75 wt.% Potassium Perchlorate/25 wt.% Sodium Salicylate | 2.1, 1.2, 2.4 | 1.2 | Yes | Yes |
| 35 | " | 70 wt.% Potassium Perchlorate/30 wt.% Sodium Salicylate | 1.4, 1.2, 1.0 | 1.0 | Yes | Yes |
| 36 | "Red" Star Mixture | 65 wt. % Potassium Perchlorate/16wt.% Strontium Carbonate/11 wt.% Red Gum/7 wt.% Sodium Oxalate/4 wt.% Dextrin/4 wt.% Charcoal(airfloat) | 7.6, 22.0, 18.4 | 7.6 | No | No |
| 37 | "Blue" Star Mixture | 70 wt.% potassium Perchlorate/ 8.5wt.% Copper Oxide/8.5 wt.% Chlorowax/8.5 wt.% Rosin/4.5 wt.% Dextrin | 29.6, 50.8, 40.0 | 29.6 | No | No |
| 38 | "Green " Star Mixture | 38 wt.% Potassium Perchlorate/37 wt.% Barium Nitrate/12 wt.% Red Gum/5 wt.% Chlorowax/4wt.% Charcoal(airfloat)/4 wt.% Dextrin | 36.4, 56,8, 37.2 | 36.4 | No | No |
| 39 | "Amber Star Mixture | 72 wt.% Potassium Perchlorate/16 wt.% Red Gum/ 7 wt.% Sodium Oxalate/5 wt.% Dextrin | 8.0, 1.7, 18.0 | 1.7 | Yes | Yes |
| 40 | "Silver " Star Mixture | 64 wt.% Potassium Perchlorate/18 wt.% Dark "Pyro" Aluminum powder/10 wt. Bright "Pyro" aluminum powder/6 wt.% Dextrin/2 wt.% Rosin | 2.0, 4.4, 2.4 | 2.0 | Yes | Yes |
| 41 | "White " Star Mixture | 80 wt.% Potassium Perchlorate/6 wt.% Charcoal(airfloat)/6 wt.% Red Gum/4 wt.% Barium Carbonate/ 4 wt.% Dextrin | 40.4, 24.0, 45.2 | 24.0 | No | No |
| 42 | Salmon Star Mixture | 70 wt.% Potassium Perchlorate/ 12 wt.% Calcium Carbonate/ 12 wt.% Rosin/4 wt.% Dextrin/2 wt.% Lampblack | 42.0, 40.8, 22.0 | 22.0 | No | No |

11. The same 12 new compositions were then evaluated for flash composition properties by the modified US Flash Composition Test (three trials) taking both detonation properties (puncture of the 1.0 mm thick steel witness plate) and deflagration properties (indentation depth of the 1.0 mm thick steel witness plate in millimeters) recorded. These results are summarized in Table III. A trial pass/fail average indentation depth (of 3) of 15 mm was selected to see how the results compared to the outcomes of the HSL Flash Composition Test in Table III.

Table III – 12 New Formulations Evaluated for Flash Composition Properties by Modified US Flash
Composition Test Using 1.0 mm Thick Steel Witness Plates from Germany

| For mula No. | Composi -tion Type | Formula Compositions (all ingredients mixed as received) | Plate Punc- tured ? | Measured Indent Depths, mm. | Ave. Indent Depth, mm | Ave. Indent Depth >15mm ? | Flash Compo- sition by US Test? |
|--------------------|------------------------------|--|------------------------------|--------------------------------------|--------------------------------|---------------------------------------|---|
| 31 | Whistle/ Burst | 75 wt. % Potassium Perchlorate/25 wt.% Potassium Benzoate | Yes (3X) | N/A | N/A | N/A | Yes |
| 32 | W | 56 wt. % Potassium Perchlorate/44 wt.% Potassium Hydrogen Phthalate | No (3X) | 35.0, 14.2, 6.6 | 18.6 | Yes | Yes |
| 33 | " | 60 wt.% Potassium perchlorate/40 wt.% Potassium Hydrogen Phthalate | No (3X) | 22.3, 23.6,21.4 | 22.7 | Yes | Yes |
| 34 | n . | 75 wt.% Potassium Perchlorate/25 wt.% Sodium Salicylate | Yes (3X) | N/A | N/A | N/A | Yes |
| 35 | " | 70 wt.% Potassium Perchlorate/30 wt.% Sodium Salicylate | Yes (2X) | 23.3 (1) | 23.3 (1) | Yes | Yes |
| 36 | "Red" Star Mixture | 65 wt. % Potassium Perchlorate/16wt.% Strontium Carbonate/11 wt.% Red Gum/7 wt.% Sodium Oxalate/4 wt.% Dextrin/4 wt.% Charcoal(airfloat) | No (3X) | 2.4, 1.5, 4.4 | 2.8 | No | No |
| 37 | "Blue" Star Mixture | 70 wt.% potassium Perchlorate/ 8.5wt.% Copper Oxide/8.5 wt.% Chlorowax/8.5 wt.% Rosin/4.5 wt.% Dextrin | No (3X) | 4.3, 14.8, 2.4 | 7.2 | No | No |
| 38 | "Green" Star Mixture | 38 wt.% Potassium Perchlorate/37 wt.% Barium Nitrate/12 wt.% Red Gum/5 wt.% Chlorowax/4wt.% Charcoal(airfloat)/4 wt.% Dextrin | No (3X) | 1.6, 2.5, 3.5 | 2.5 | No | No |
| 39 | "Amber" Star Powder | 72 wt.% Potassium Perchlorate/16 wt.% Red Gum/ 7 wt.% Sodium Oxalate/5 wt.% Dextrin | No (3X) | 26.9, 26.1,16.2 | 23.1 | Yes | Yes |
| 40 | "Silver" Star Mixture | 64 wt.% Potassium Perchlorate/18 wt.% Dark "Pyro" Aluminum powder/10 wt. Bright "Pyro" aluminum powder/6 wt.% Dextrin/2 wt.% Rosin | Yes (3X) | N/A | N/A | N/A | Yes |
| 41 | "White" Star Mixture | 80 wt.% Potassium Perchlorate/6 wt.% Charcoal(airfloat)/6 wt.% Red Gum/4 wt.% Barium Carbonate/ 4 wt.% Dextrin | No (3X) | 21.0, 6.0, 18.1 | 15.0 | No | No |
| 42 | "Salmon " Star Mixture | 70 wt.% Potassium Perchlorate/ 12 wt.% Calcium Carbonate/ 12 wt.% Rosin/4 wt.% Dextrin/2 wt.% Lampblack | No (3X) | 11.8, 19.8, 4.3 | 12.0 | No | No |

12. Finally, the results for these 12 new formulations were directly compared for potential agreement between the HSL and the Modified US Flash Composition Test Methods in Table IV. As the outcomes demonstrate, there was perfect agreement between the two test methods when the Modified US Method Pass/Fail Criteria of 15 mm average indentation depth is used.

Table IV – Comparison of 12 New Formulations Evaluated for Flash Composition Properties by Both the Modified HSL and the Modified US Flash Composition Test Using 1.0 mm Thick Steel Witness Plates from Germany

| Form -ula No. | Compo- sition Type | Formula Compositions (all ingredients mixed as received) | Puncture or Average Indent Depth >15mm? | Flash Com- pos- ition by US Test? | HSL Min. Press -ure rise, ms. | Flash Com- position by HSL Test? | Agree ment Be- tween Tests? |
|---------------------|------------------------------|--|--|--|--|--|---|
| 31 | Whistle/ Burst | 75 wt. % Potassium Perchlorate/25 wt.% Potassium Benzoate | Puncture (3X) | Yes | 1.8 | Yes | Yes |
| 32 | " | 56 wt. % Potassium Perchlorate/44 wt.% Potassium Hydrogen Phthalate | 18.6 mm | Yes | 2.2 | Yes | Yes |
| 33 | " | 60 wt.% Potassium perchlorate/40 wt.% Potassium Hydrogen Phthalate | 22.7 mm | Yes | 3.5 | Yes | Yes |
| 34 | " | 75 wt.% Potassium Perchlorate/25 wt.% Sodium Salicylate | Puncture (3X) | Yes | 1.2 | Yes | Yes |
| 35 | W | 70 wt.% Potassium Perchlorate/30 wt.% Sodium Salicylate | Puncture (2X) | Yes | 1.0 | Yes | Yes |
| 36 | "Red" Star Mixture | 65 wt. % Potassium Perchlorate/16wt.% Strontium Carbonate/11 wt.% Red Gum/7 wt.% Sodium Oxalate/4 wt.% Dextrin/4 wt.% Charcoal(airfloat) | 2.8 mm | No | 7.6 | No | Yes |
| 37 | "Blue" Star Mixture | 70 wt.% potassium Perchlorate/ 8.5wt.% Copper Oxide/8.5 wt.% Chlorowax/8.5 wt.% Rosin/4.5 wt.% Dextrin | 7.2 mm | No | 29.6 | No | Yes |
| 38 | "Green" Star Mixture | 38 wt.% Potassium Perchlorate/37 wt.% Barium Nitrate/12 wt.% Red Gum/5 wt.% Chlorowax/4wt.% Charcoal(airfloat)/4 wt.% Dextrin | 2.5 mm | No | 36.4 | No | Yes |
| 39 | "Amber" Star Mixture | 72 wt.% Potassium Perchlorate/16 wt.% Red Gum/ 7 wt.% Sodium Oxalate/5 wt.% Dextrin | 23.1mm | Yes | 1.7 | Yes | Yes |
| 40 | "Silver" Star Mixture | 64 wt.% Potassium Perchlorate/18 wt.% Dark "Pyro" Aluminum powder/10 wt. Bright "Pyro" aluminum powder/6 wt.% Dextrin/2 wt.% Rosin | Puncture (3X) | Yes | 2.0 | Yes | Yes |
| 41 | "White" Star Mixture | 80 wt.% Potassium Perchlorate/6 wt.% Charcoal(airfloat)/6 wt.% Red Gum/4 wt.% Barium Carbonate/ 4 wt.% Dextrin | 15.0 mm | No | 24.0 | No | Yes |
| 42 | "Salmon " Star Mixture | 70 wt.% Potassium Perchlorate/ 12 wt.% Calcium Carbonate/ 12 wt.% Rosin/4 wt.% Dextrin/2 wt.% lampblack | 12.0 mm | No | 22.0 | No | Yes |