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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 **10 June 2014**

Forty-fifth session
Geneva, 23 June – 2 July 2014
Item 2 (a) of the provisional agenda
Explosives and related matters: tests and criteria for flash compositions

The effectiveness of US and HSL modified plugs for the HSL flash composition test

Transmitted by the expert from the United Kingdom

Introduction

1. At the forty-third session of the Sub-Committee of Experts on the Transport of Dangerous Goods held in Geneva in June 2013, the expert from the United States of America presented a paper(0) which detailed the testing of a modified plug design to improve the performance of the HSL Flash Composition Test, which is described in the UN Recommendation on the Transport of Dangerous Goods - Manual of Tests and Criteria(0). Twelve pyrotechnic compositions were prepared and tested and the results indicated that substantial improvements in reproducibility were achieved. In addition, it was claimed that the modified plug was easier to manipulate and allowed faster turnaround times. The sub-committee agreed that experts from other countries would perform tests on a range of compositions to corroborate the reproducibility claimed. The United Kingdom agreed to coordinate the testing between the participating countries.

2. Work had also been undertaken at the Health and Safety Laboratory (HSL) in the UK to improve the plug design and usability. HSL were therefore tasked with comparing the performance of the HSL modified plug with that of the US version when conducting the above trials. In addition, testing was also to be performed on a limited number of compositions using the current plug design for comparison purposes.

3. The first objective of the research was to generate a short list of approximately twenty compositions to provide a range of rise time for testing. These compositions were then subjected to a programme of tests to establish the level of reproducibility and ease of use of the modified plugs compared to the current plug. As part of the project, the compositions were made available to other testing laboratories around the world to establish confidence in the repeatability of results.

4. The report on the testing that has been performed at HSL on both the US modified plug and the HSL original and modified plugs is included in the Annex to this paper. At the time of submission none of the other participants had completed their testing.

Discussion

5. Table 2 in the Annex summarises the results from testing each composition with each of the plugs. Table 3 provides the results from earlier tests on the same composition but different origins.

6. This work, together with the historical data produced previously, has demonstrated that:

- The US and HSL modified plugs are comparable in their performance and, generally, both designs produce shorter rise times than comparable tests using the current plug design.
- The reduction in minimum rise time when using either of the modified plugs is believed to be due to a more effective seal around the fusehead lead wires as they pass through the plug..
- No significant differences in the standard deviation of the minimum rise time were noted in the results from the modified and current plugs.
- Both modified plugs reduce the occurrence of continuity issues between the fusehead and plug.
- Both modified plugs are much easier to handle when preparing the tests. Consequently, the turn-around time between firings is dramatically reduced thereby improving efficiency.
- Commercially available smokeless powder could be used as a standard for calibrating test apparatus.

Proposal

7. Pending the results from other laboratories, these results support the replacement of the existing design with either the US or HSL modifications (or both) since either the US or HSL modifications would improve the existing UN method by improving reproducibility and usability.

8. A formal proposal to modify Figures A7.1 to A7.8 inclusive be prepared for agreement that includes both the US and HSL design for the plug. The proposal will include instructions on modifying the existing plug to the modified HSL version to limit costs.

9. Views be sought on the use of propellant powder to calibrate test apparatus.

Annex 1

Performance and use of modified plugs for the HSL time/pressure test

Experimental

Sample selection and preparation

1. A number of compositions had been tested using the current plug design during previous research[(0,0)] and the data from those tests were used to select a provisional list of twenty-one pyrotechnic compositions with rise times in the range 0.18 to 8.79 ms. Fourteen of the original twenty-one compositions were selected together with a whistle composition and a smokeless powder (). This selection was planned to give rise times of up to 8m/s. Smokeless powder was included as a non-pyrotechnic composition with a well-defined specification and a fast burn rate that had the potential to be a standard for the test.

2. In order to corroborate that the modifications to the plug design improved reproducibility, it was paramount that all laboratories tested compositions of the same specification and quality. Since the international transport of samples of pyrotechnic powders caused problems, this was achieved by using classified pyrotechnic articles instead. HSL arranged for a Theatrical Pyrotechnic manufacturer in the UK to manufacture pyrotechnic articles so that all articles of one type contained the same batches of composition. The finished articles have been made available to all the participating laboratories by HSE along with the full list of compositions under test. Compositions 12-16 were off-the-shelf products which were generally available internationally as standard products and could be purchased by the participating laboratories within their respective countries.

Table 1: List of agreed compositions

Sample No	Product	Use By Date	LOT No
1	Flash Report	08/16	M1265
2	Airburst	11/16	M3361
3	Super Gun Shot	11/15	M34
4	Starburst	11/16	M273
5	Waterfall	21/10/16	M569
6	White + Tail comet	11/16	M721
7	Red Mine	11/16	M196
9	Silver Mine	11/16	M195
10	Yellow Mine	11/16	M197
8	Theatrical Flash		M758
11	Whistle Composition	11/15	M374
12	Swiss No 1 Black Powder	N/A	090.609
13	FOA Black Powder	N/A	N/A
14	Hodgdon triple severn BP Substitute	N/A	N/A
15	Pyrodex TW	N/A	8-3932
16	Alliant "Bullseye" Smokeless Powder	N/A	N/A

3. Samples 1 to 11 were theatrical pyrotechnic articles that had been broken down to obtain the composition for test. Samples 1, 2, 3, 4, and 8 were powders and required no further preparation. Samples 5, 6, 7, 9, 10 and 11 were cut into smaller pieces, ground and sieved. Product which passed through a 500 micron sieve was used for testing.

4. Samples 12 to 16 were off the shelf products and required no further preparation prior to test.

Plug designs and preparation

Current plug

5. The design of the existing plug and the test procedure for the HSL Flash Composition Test are given in the UN Manual[(0)]. The apparatus is reproduced in Figure 1.

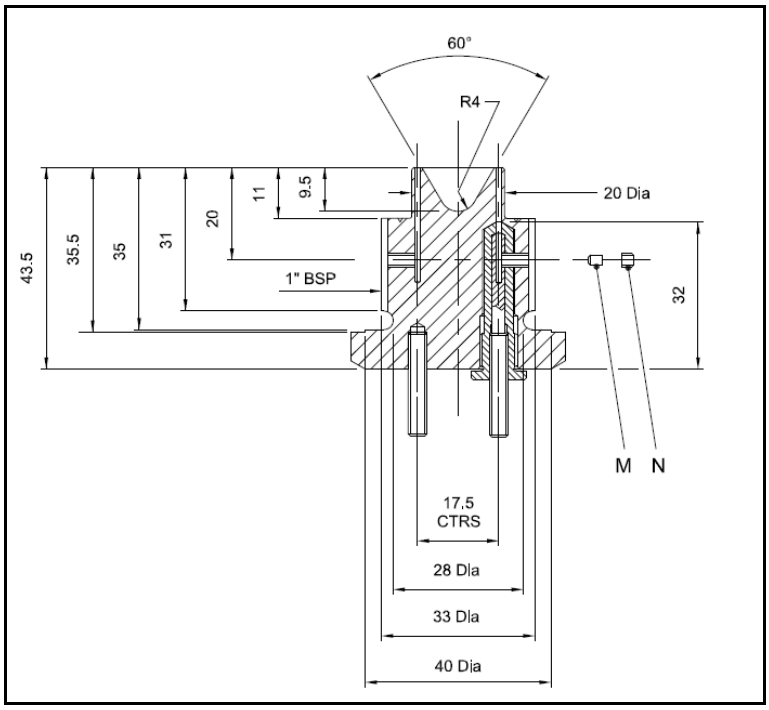


Figure 1: Diagram of existing plug for HSL Flash Composition Test

US plug modification

6. Figure 2 details the US modifications to the plug while Figure 3 shows the separate fabricated components. The plug was prepared as described in the US paper(0) by twisting the leads immediately behind the fusehead, cutting the leads to the required length and then threading them through the holes in the firing plug. The ends of the insulation wire were stripped and kinked. A small dab of silicone grease was applied to each threaded electrode and the fusehead lead wires were slowly drawn to the top of the electrode holes. The electrodes were threaded into the holes until they just pinched and secured the fusehead lead wires. Any excess silicone grease was removed and the lead wire lengths were adjusted so that the fusehead was positioned in place just above the sample cup. Figure 4 summarises the set-up procedure.

7. Once the firing plug preparation was completed, it was checked for electrical continuity before the completed plug assembly was threaded into the body of the test apparatus and tightened securely. Electrical continuity was rechecked before placing the sample into the cone of the plug and performing the test.

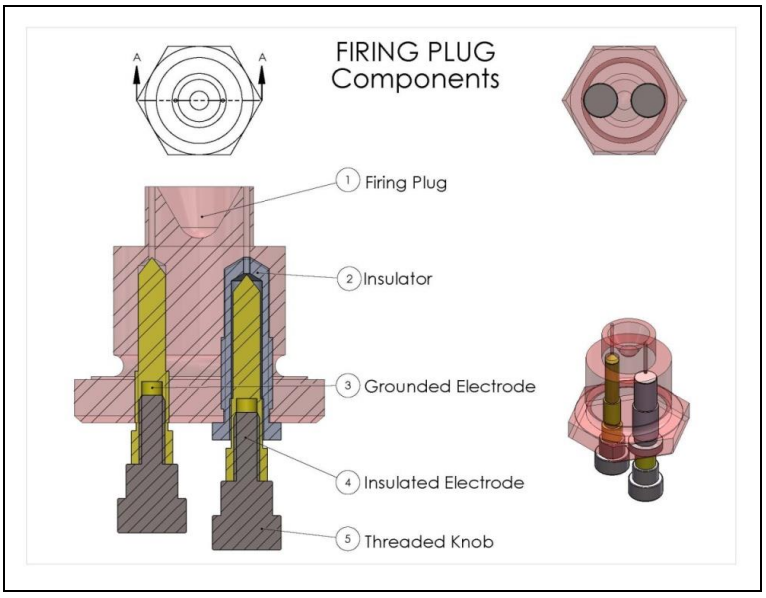


Figure 2: The US Modified HSL Firing Plug Design

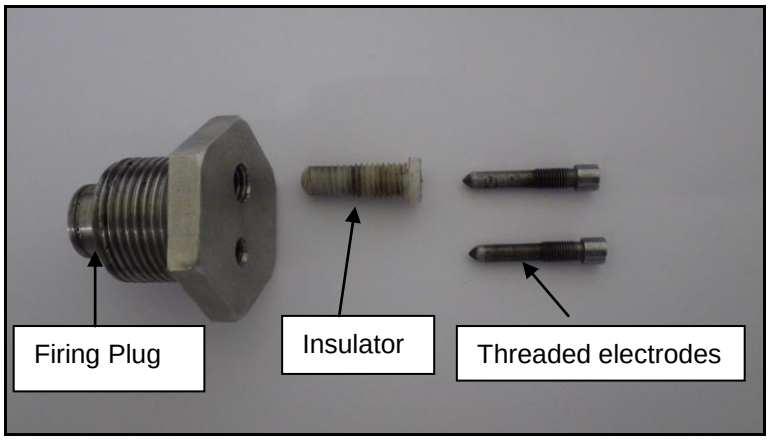


Figure 3: Fabricated US modified plug components

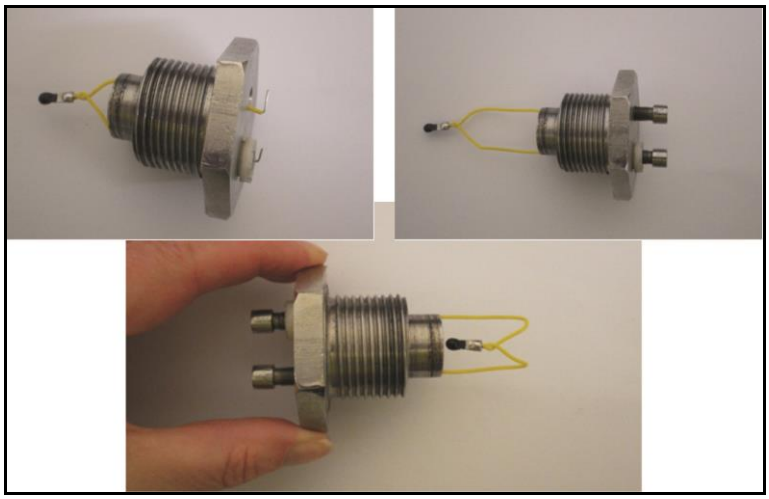


Figure 4: US modified plug set-up

HSL modified plug

8. HSL had reviewed the current test plug (Figure 1), and identified modifications to improve its usability and turnaround time, while attempting to minimise gas leakage and improve reproducibility. The modifications were incorporated into the HSL Modified Plug which consisted of proprietary pressure and vacuum sealed feedthrough assemblies (Figure 5).

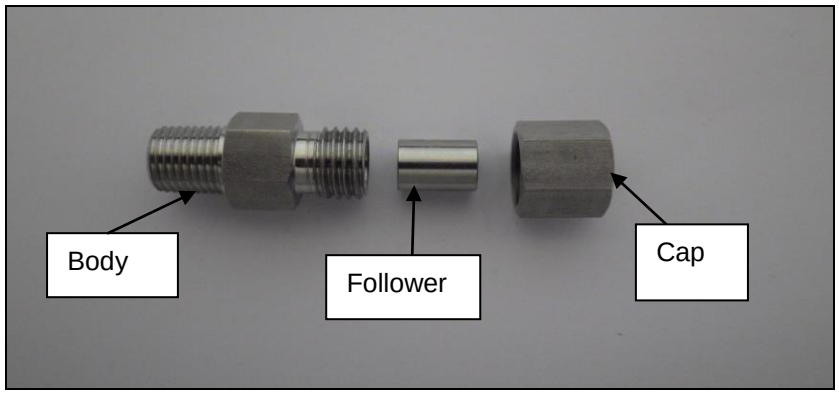


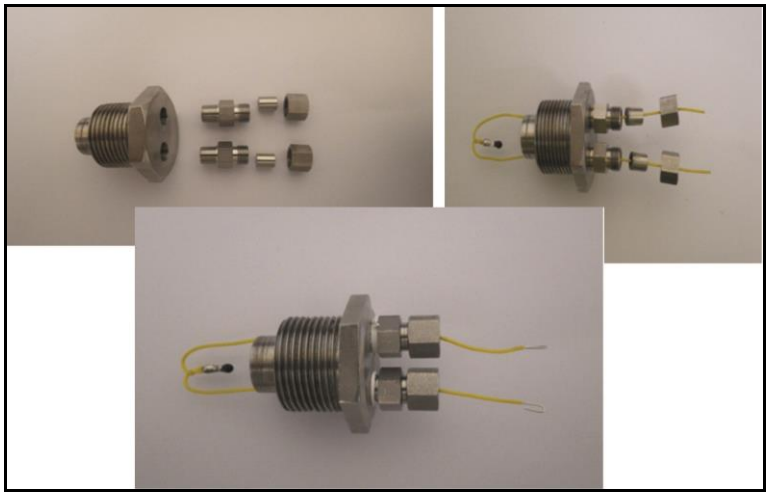
Figure 5: Proprietary pressure and vacuum feedthrough assemblies

9. The modification to the current HSL Flash Composition Test plug required the existing plug to be modified as follows:

- Discarding the nylon insulator assembly completely (electrode and nylon mount).
- Discarding the earth return electrode.
- Drilling completely through the firing plug at the top of the electrode ports to accommodate the size of firing lead fitted to the fusehead.
- Blanking off both grub screw holes with grub screws and sealant/tape.
- Drilling and taping both holes to fit feedthrough assemblies along the centre line of the electrode ports.
- Fitting feedthrough assemblies as detailed in the manufactures procedures.

10. The set-up of the plug was achieved by twisting the leads directly behind the fusehead and inverting as detailed in the current HSL Flash Composition Test(0) so that the fusehead was 10 mm above the top of cone. The fusehead firing leads were then fed through the holes in firing plug, feedthrough assemblies, locking shoulders and securing nuts and tightened as detailed in the manufactures procedures.

11. The plug was checked for electrical continuity and then threaded into the body of the HSL test apparatus before electrical continuity was rechecked. The sample under test was placed into the cone and the test performed. The set-up procedure is shown in **Error! Reference source not found.**.



Test procedure

12. The different firing plug assemblies were prepared as described previously using Vulcan fuses in all cases. The firing plug assembly was then fitted into the body of the apparatus and tightened by hand prior to the test composition (0.5g) being added. The bursting disc was then fitted and the test were fired as detailed in the UN Manual(0).

13. Each series of tests consisted of three individual test firings, as described in the UN manual(0). For each test the time for the pressure to rise from 670kPa to 2070kPa was recorded. Since this research was to corroborate if the modified plugs improved reproducibility, three series of tests (nine individual firings) were performed for each sample. The results from the tests were analysed to calculate the standard Deviation (SD) and to identify the fastest (minimum) rise time. The mean rise time of the nine tests was also calculated for completeness.

14. Due to the longer turnaround times of the current plug set-up only 5 of the compositions were tested using this plug and covered the range of rise times expected across the list of compositions. Samples 8, 9 10 11 and 16 were chosen.

15. While in use the US and HSL modified plugs were assessed to rank their usability. Aspects considered included:

- Ease of assembly,
- Fuse head assembly within the plug,
- Cleaning.

Table 2: Pyrotechnic Composition Test Data from Current and Modified Plugs for the HSL Flash Composition Test

Sample No	Product	Rise time (ms)								
		Current Plug			HSL Modified Plug			US Modified Plug		
		All runs combined*			All runs combined*			All runs combined*		
		Min	Mean	SD	Min	Mean	SD	Min	Mean	SD
1	Flash Report				0.42	0.84	0.20	0.34	0.69	0.21
2	Airburst				0.72	1.25	0.56	0.64	1.87	0.76
3	Super Gun Shot				1.94	3.77	1.11	3.11	4.04	0.88
4	Starburst				1.77	2.49	0.75	0.58	1.32	0.67
5	Waterfall				1.07	2.37	0.76	1.33	1.57	0.25
6	White + Tail comet				1.79	2.02	0.22	1.25	1.71	0.50
7	Red Mine				3.15	4.32	0.94	2.73	3.11	0.55
8	Theatrical Flash	8.51	31.04	14.38	17.92	22.35	6.31	12.73	19.31	7.36
9	Silver Mine	3.70	5.73	1.15	3.99	5.42	0.87	2.49	4.49	1.44
10	Yellow Mine	3.62	5.88	1.47	5.03	5.94	1.23	4.29	4.83	0.43
11	Whistle Composition	1.04	1.20	0.15	0.64	0.98	0.26	0.52	0.72	0.18
12	Swiss No 1 Black Powder				2.04	2.32	0.33	1.36	2.16	0.46
13	FOA Black Powder				3.51	4.40	0.59	3.23	4.27	0.57
14	Hodgdon triple seven BP Substitute				2.65	3.41	0.52	2.77	3.52	1.21
15	Pyrodex TW				4.73	5.84	0.58	3.95	6.51	1.84
16	Alliant "Bullseye" Smokeless Powder	3.18	4.74	0.78	2.80	4.14	0.79	2.41	3.71	0.69
* data from 9 firings										

Usability of HSL modified plug

16. The components of the modified HSL plug apparatus were found to be easy to use and the speed of preparing each plug was improved dramatically compared with the current plug because;

- The fuse head assembly could be fitted into the plug simply and easily.
- The fusehead lead wires did not require fixing with either grub screws or grounded electrodes.
- The feedthrough assemblies required no insulating component.
- Electrical continuity of the fusehead assembly in the plug was consistently maintained throughout the process, as the plug required no insulation.
- A plug could be prepared, loaded and fired within 5 minutes, which was a huge improvement on the 15 minutes experienced with the current plug.

17. A possible disadvantage observed when using this plug was a build-up of residue in the holes housing the fusehead lead wires, which caused difficulties feeding the lead wires through the plug causing delays. This problem was easily resolved by cleaning the holes on a regular basis.

Discussion

18. From the minimum rise time data there does not appear to be a distinct difference between the values obtained from the HSL and US modified plugs apart from a slightly faster rise time for the US plug. This may be due to an increased volume in the apparatus when using the HSL modified plug due to some dead volume in the electrode ports. Further work would need to be performed to confirm this. If new plugs were being fabricated these dead volumes could be designed out. Comparison of the minimum rise time data from the modified plugs with that of the current plug was inconclusive. Some rise times being shorter for the current plug others for the modified plugs.

19. When standard deviations are compared between tests performed on the HSL and US modified plugs neither plug performs better than the other. In addition, the SD values from the modified plugs were not a distinct improvement on the current plug.

20. On the basis of these data neither modified plug could be recommended over the current plug. However, historic data is available for some of the compositions using the current plug (Table 3). If these historic data are included with the data generated in the current research it can be seen that there is a clear benefit to using either the US or HSL modified plugs compared to the current plug because the minimum rise times are generally reduced, which provides a more conservative assessment from a safety point of view. Exceptions were compositions 2, 8 and 13 where the historic current plug minimum rise time values were faster than those of the modified plugs and compositions 3 and 12 where the historic current plug values were intermediate between those of the HSL and US plugs. However, there is still no marked consistent improvement on standard deviation.

Table 3: HSL historic data generated using the current plug

Sample No	Product Rise time (ms)	Original Current			
		All runs combined			
		No. Firings	Min	Mean	SD
1	Flash Report	3	0.52	0.56	0.08
2	Airburst	3	0.18	0.20	0.04
3	Super Gun Shot	3	2.68	2.99	0.27
4	Starburst	3	3.35	3.54	0.32
5	Waterfall	3	3.88	5.10	1.28
6	White + Tail comet	3	4.35	4.90	0.51
7	Red Mine	3	5.17	5.91	1.25
8	Theatrical Flash	3	7.07	7.58	0.74
9	Silver Mine	3	8.79	14.64	6.26
10	Yellow Mine	3	5.51	7.86	2.11
11	Whistle Composition				
12	Swiss No 1 Black Powder	6	2.14	2.84	0.47
13	FOA Black Powder	9	3.00	4.86	1.19
14	Hodgdon triple seven BP Substitute	6	3.08	3.62	0.38
15	Pyrodex TW	9	3.46	6.52	1.90
16	Alliant "Bullseye" Smokeless Powder				

21. When all the data is compared the reductions in rise time obtained using the modified plugs are most pronounced for compositions that gave minimum rise time values in excess of 3.5 ms. Over these relatively long rise times it is believed that gas leakage from the test apparatus becomes important and that both modified plugs reduce the opportunity for this leakage leading to faster rise times. For compositions with minimum rise times below 3.5 ms, the results are mixed with no one modified plug apparatus demonstrating a significant difference to the other. This may suggest that, since the gas is produced quickly, the amount that can escape from the apparatus is small and therefore there is no significant effect on rise time. This leads to the conclusion that while sealing the fusehead lead wires into the plug is essential the importance of the method of sealing is not. This suggests that it may not be necessary to stipulate the exact design of this part of the plug in the UN manual but merely to state that any method is suitable as long as the seal can be demonstrated.

22. While gas leakage from the apparatus may be one of the major factors affecting rise time, other sources of variance could be operator set up and the efficiency of the pickup of the ignition, from the fuse head to the sample.

23. It should be noted that while the historical data used compositions of the same name and from the same manufacturer the performance of them may be different. The compositions used for the historical data were supplied direct from the production line whereas for the current research the compositions were recovered from pyrotechnic articles and sometimes had to be processed prior to testing. These differences could significantly affect the behaviour of the compositions and consequently any comparisons between historical and current data should be treated with caution.

24. Overall, the performance of both the US and HSL modified plugs indicates that both modifications tend to reduce minimum rise times compared to the values obtained from all the current plug data (historic and current). However, there is no clear trend with regard to standard deviation. Since there does not appear to be significant differences between the performance of the two modified plug designs, either would be an improvement on the existing system.

25. Both the US and HSL modified plugs were a significant improvement on the current plug apparatus with regard to usability. This was because fitting the fusehead wires into the current plug required considerable dexterity whereas this was not required to the same extent for either of the modified plugs. In addition, the simpler fusehead/plug assembly process significantly reduced problems obtaining electrical continuity. As a result of these changes both modified plug designs had a quicker turnaround time (5 min) compared to that of the current plug (15 min). In summary, the usability of the modified plugs was comparable and both were a significant improvement over the current plug.

References

1. UN ST/SG/AC.10/C.3/20103/23

2. Transport of Dangerous Goods: Manual of Tests and Criteria, 5th revised edition, Appendix 7, pp443 – 450.

3. XS/07/48 Modifications to the Time/Pressure Test for Pyrotechnic Compositions. Testing and validation of the cone design.

4. XS/11/124 HSL Flash Composition - Torque Wrench Comparison. Determining the ideal torque to be used in the HSL Flash Test