

Energetic Properties of Thermites

Final Report

DOT Contract # DTPH5616D00001
Task Order 693JK318F000058 (TO# 0002)
SwRI® Project # 24178

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1.0 EXECUTIVE SUMMARY

Safety Management Services, Inc. (SMS) performed research on thermite formulations and selected eight thermites for large-scale testing in cooperation with Southwest Research Institute (SwRI) and the U.S. Department of Transportation (DOT) Pipeline and Hazardous Materials Safety Administration (PHMSA). Three of the thermites were available commercially and five were mixed by SMS from raw ingredients that were very fine powders (1 - 5 micron). Each of the thermites was a combination of metal and metal oxide powders; thermite test samples were not selected from nano-thermites (sub-micron particles sizes) nor from thermates (thermites that includes an oxidizer, like sodium nitrate).

Title 49 of the Code of Federal Regulations (CFR), Part 173, Section 124(a)(3)(ii) states that Readily Combustible Solids are materials that show a burning rate faster than 2.2 mm per second (mm/sec) when tested in accordance with the UN Manual of Tests and Criteria. Three of the eight large-scale thermites (Large-scale Mix ID's #1, #4, and #5) burned very rapidly (hundreds of mm/sec), exhibiting hazards more similar to that of explosives classed in Division 1.3G. Two of the five large-scale thermite mixes (Large-scale Mix ID's #7 and #8) exploded when ignited as unconfined 5-gram quantities, exhibiting hazards similar to that of unconfined flash powders classed in Division 1.1G. The following are additional observations:

- Large-scale Mix ID #7 produced an explosion result in the UN Series 3 (d) Small-scale burning and UN Series 6 (c) External bonfire tests when unconfined but passed the UN Series 2 (b) Koenen test as prescribed with a 5-minute maximum test time; an explosion result was only achieved after the 5-minute test limit at the following timecodes (mm:ss): 05:20, 11:40 and 09:37.
 - Large-scale Mix ID #8 also produced an explosion result in the UN Series 3 (d) Small-scale burning and UN Series 6 (c) External bonfire tests when unconfined; this thermite nearly passed the UN Series 2 (b) Koenen test as prescribed with a 5-minute maximum test time with an explosion result achieved at the following timecodes: 07:10, 9:40 and 01:15.
 - Large-scale Mix ID #6 burns slowly and steadily when unconfined but produced an explosion result in the third and final trial of the UN Series 2 (b) Koenen test at the timecode 04:40 (the prior trials were ended at the prescribed 5-minute test limit).
- Large-scale Mix ID's #7 and #8 each experienced a mass-explosion result in the Unconfined UN Series 6 (a) Single package test but each passed a confined UN Series 2 (c) Internal ignition test.

SMS performed auto-ignition temperature testing on a total of twenty-three thermite mixtures. Only one of these thermite mixtures had an auto-ignition temperature greater than 1200 °C. Six of these additional fifteen small-scale thermite mixtures also exploded when ignited as unconfined 5-gram quantities with the most violent being Small-scale Mix #24. Based on the limited data set, the explosiveness of these thermites appears strongly related to two parameters. Additional testing is required to further investigate this relationship.

Large-scale Mix ID's #2, #3, and #5 each passed the following standard tests:

- UN Series 3
- UN Series 2
- UN Test N.4 Test method for self-heating substances
- UN Test N.5 Test method for substances which in contact with water emit flammable gases

NOTE: The auto-ignition temperature of traditional explosives is typically around 200 - 300 °C; the average auto-ignition temperature of the twenty-three small-scale thermite test samples was 809 °C with a standard deviation of 240 °C. Therefore, the 5-minute maximum test time of the UN Series 1 or 2 (b) Koenen test may be insufficient to determine whether a specific thermite exhibits a violent effect on heating under confinement.

Large-scale Mix ID's #2 and #3 also passed the standard UN Test N.1 Test method for readily combustible solids with gas torch and hotwire, and could potentially be offered for transport as non-regulated materials in the absence of additional considerations. However, using hotter ignition sources and wider powder train piles than those specified in UN Test N.1, it quickly becomes evident that all three of these thermite mixes exhibit burning rates in excess of the 2.2 mm/sec threshold specified in 49 CFR 173.124(a)(3)(ii).

NOTE: The current UN Test N.1 test methodology could potentially permit these powders to be offered for transport as non-regulated goods since the powder train pile is too narrow to sustain propagation AND/OR if an ignition source is utilized that is around 1000 °C, which could be below the auto-ignition temperature of the thermite, even though in their shipping configuration they would clearly present hazards consistent with that of flammable solids regulated for transport.

The following table contains the proposed classifications based on the test results.

Table 1: Classification Recommendations for Tested Large-Scale Thermites

Thermite Large-scale Mix ID	Considerations	Proposed Classification
1	Division 1.3 or 1.4 based on the UN Series 3 (d) Small-scale burning, Unconfined UN Series 6 (a) Single package, Sub-scale UN Series 6 (c) External fire (bonfire), Sub-scale UN Series 7 (e), UN Test N.1 and the Large-scale UN Test N.1 test results; subsidiary hazard as Division 4.3 PG III based on UN Test N.5 test results. Could pose a potential threat to the structural integrity of an aircraft based on the unconfined, suspended UN Series 6 (a) Single package test results.	Division 1.3 or 1.4 (depending upon the packaging configuration and quantity) with subsidiary Division 4.3 hazard - FORBIDDEN FROM AIRCRAFT

Thermite Large-scale Mix ID	Considerations	Proposed Classification
2	Division 4.1 based on the Large-scale UN Test N.1 test results using an alternative ignition method. Could pose a potential threat to the structural integrity of an aircraft based on the unconfined, suspended UN Series 6 (a) Single package test results.	UN3178, Flammable solid, inorganic, n.o.s., 4.1, PG II - FORBIDDEN FROM AIRCRAFT
3	Division 4.1 based on the Large-scale UN Test N.1 test results using an alternative ignition method. Could pose a potential threat to the structural integrity of an aircraft based on the unconfined, suspended UN Series 6 (a) Single package test results.	UN3178, Flammable solid, inorganic, n.o.s., 4.1, PG II - FORBIDDEN FROM AIRCRAFT
4	Division 1.3 or 1.4 based on the UN Series 3 (d) Small-scale burning, Unconfined UN Series 6 (a) Single package, Sub-scale UN Series 6 (c) External fire (bonfire) and the Large-scale UN Test N.1 test results. Could pose a potential threat to the structural integrity of an aircraft based on the unconfined, suspended UN Series 6 (a) Single package test results.	Division 1.3 or 1.4 (depending upon the packaging configuration and quantity) - FORBIDDEN FROM AIRCRAFT
5	Division 1.3 or 1.4 based on the Unconfined UN Series 6 (a) Single package, UN Test N.1 and the Large-scale UN Test N.1 test results.	Division 1.3 or 1.4 (depending upon the packaging configuration and quantity) - FORBIDDEN FROM AIRCRAFT
6	Class 1 based on the UN Series 2 (b) Koenen test results, otherwise burning rates consistent with Division 4.1 PG II; subsidiary hazard as Division 4.3 PG III based on UN Test N.5 test results.	Division 1.3 or 1.4 (depending upon the packaging configuration and quantity) with subsidiary 4.3 hazard OR UN3178, Flammable solid, inorganic, n.o.s., 4.1, (4.3), PG II - FORBIDDEN FROM AIRCRAFT

Thermite Large-scale Mix ID	Considerations	Proposed Classification
7	Division 1.1G based on UN Series 3 (d) Small-scale burning, Unconfined UN Series 6 (a) Single package, Sub-scale UN Series 6 (c) External fire (bonfire), Sub-scale UN Series 7 (e), UN Test N.1 and the Large-scale UN Test N.1 test results; subsidiary hazard as Division 4.3 PG III based on UN Test N.5 test results.	UN0476, Substances, explosive, n.o.s., 1.1G, (4.3), PG II
8	Division 1.1G based on UN Series 3 (d) Small-scale burning, Unconfined UN Series 6 (a) Single package, Sub-scale UN Series 6 (c) External fire (bonfire), Sub-scale UN Series 7 (e), UN Test N.1 and the Large-scale UN Test N.1 test results; subsidiary hazard as Division 4.3 PG III based on UN Test N.5 test results.	UN0476, Substances, explosive, n.o.s., 1.1G, (4.3), PG II

2.0 OBJECTIVE

The objective of this effort was to 1) identify families of thermite compositions, 2) procure samples of thermite, and 3) conduct testing to determine the hazard presented by thermites in transport. The testing may help form a technical basis for regulation or exemption of certain thermite formulation families.

3.0 SUMMARY AND CONCLUSIONS

Eight thermites were selected for the large-scale testing. Testing was conducted by Safety Management Services, Inc. (SMS) at their Tooele, Utah test site. Tests were witnessed by Jason T. Ford, Kirt Sasser, and/or Troy Gardner and performed in accordance with the United Nations (UN) Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, Sixth revised edition (2015). The test results are summarized in the following tables for each sample.

Table 2: Testing Summary for Large-scale Mix ID #1

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm; limiting diameter less than 2.0 mm.	Pass

Test	Test Name	Conditions and Results	Conclusions
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging it to approximately 2-cm wide. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.2-grams mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small- scale burning	The two 100-gram samples ignited and burned for less than 1 second each; explosion of the test samples did NOT occur.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through a large hole through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned in 1 - 2 seconds; no significant damage to the steel witness plate.	Minimal threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and rapidly burned over approximately 1 second, producing a fireball with a radius of 3 - 4 meters; no mass explosion.	Reaction rate and fireball more consistent with the hazards of Division 1.3

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard consistent with Class 1 when heated under confinement
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Sample ignited; burning time of powder train pile too fast to be measured (less than 0.1 seconds for the entire 200mm length).	Rapid burning consistent more with the hazard of Class 1 explosives
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited; burning time of powder train 0.3 seconds for the entire 200mm length.	Rapid burning consistent more with the hazard of Class 1 explosives
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by gas torch and hot wire with full propagation (250mm) of powder over 0.5 seconds (500 mm/sec). <u>Crucible bowl</u> : Sample ignited by gas torch and pyrogen igniter (remote) with full consumption of powder over 0.5 seconds.	Rapid burning consistent more with the hazard of Class 1 explosives
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 2.2 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)

Table 3: Testing Summary for Large-scale Mix ID #2

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material remaining in pipe.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm; limiting diameter less than 2.0 mm.	Pass
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging it to approximately 6-cm wide. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.1-grams mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned; several holes in the steel witness plate where thermite melted through.	High threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and briefly produced local flame enhancement within the flames of the fire.	Sub-scale quantities appear to present minor hazards

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	There was no reaction of the three samples.	No reaction when heated under confinement
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Combustion of the sample did not propagate in 5 minutes. <i>NOTE: Sample could ignite by employing a thermite starting powder or heating element, with only partial propagation down the powder train pile (100 mm) over 50.7 seconds.</i>	Consistent with exclusion from Division 4.1
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Combustion of the sample did not propagate in 5 minutes. NOTE: Sample ignited with heating element but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate & crucible bowl</u> : Combustion of the sample did not propagate in 5 minutes when ignited by hot wire (plate) or gas torch (both). <i>Alternative ignition method:</i> <u>Flat plate</u> : Sample ignited by oxy-acetylene torch with full propagation (250mm) of powder over 10 seconds (25.0mm/sec). <u>Crucible bowl</u> : Sample ignited by oxy-acetylene torch with full consumption over 2.7 seconds.	Consistent with exclusion from Division 4.1 for standard ignition; alternative ignition results in Division 4.1, packing group II
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	No significant gas evolution over 7-hour test period for 25 grams of thermite.	Pass (NOT Division 4.3)

Table 4: Testing Summary for Large-scale Mix ID #3

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material remaining in pipe.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm; limiting diameter less than 2.0 mm.	Pass
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging it to approximately 4-cm wide. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.03-grams mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small- scale burning	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; several holes in the aluminum witness plate where thermite melted through.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the steel witness plate.	High threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and briefly produced local flame enhancement within the flames of the fire; package was fully engulfed in the flames of the fire, obscuring the package and reaction.	Sub-scale quantities appear to present minor hazards

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	There was no reaction of the three samples.	No reaction when heated under confinement
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Combustion of the sample did not propagate in 5 minutes. <i>NOTE: Supplemental magnesium strip was also unable to ignite the sample.</i>	Consistent with exclusion from Division 4.1
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Combustion of the sample did not propagate in 5 minutes.	Consistent with exclusion from Division 4.1
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate & crucible bowl</u> : Combustion of the sample did not propagate in 5 minutes when ignited by hot wire or gas torch. <i>Alternative ignition method:</i> <u>Flat plate</u> : Sample ignited by oxy-acetylene torch with partial propagation (170mm) of powder over 75 seconds (2.3 mm/sec). Sample ignited by gas torch (aided by a thermite starting powder) with partial propagation (140mm) of powder over 23 seconds (6.1 mm/sec).	Burning rate > 2.2 mm/sec (consistent with Division 4.1, packing group II)
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	No significant gas evolution over 7-hour test period for 25 grams of thermite.	Pass (NOT Division 4.3)

Table 5: Testing Summary for Large-scale Mix ID #4

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm; limiting diameter less than 2.0 mm.	Pass
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging it to approximately 4-cm wide. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.05-grams mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples ignited and burned for approximately 4 - 7 seconds; explosion of the test samples did NOT occur.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned; no significant damage to the steel witness plate but several melt points on underside of the plate.	Moderate threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and rapidly burned over approximately 3 seconds, producing a fireball with a radius of 2 meters; no mass explosion.	Reaction rate and fireball more consistent with the hazards of Division 1.3 or 1.4

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by torch and hot wire with full propagation (250mm) of powder over 1.3 - 2.1 seconds (119 - 192 mm/sec). <u>Crucible bowl</u> : Sample ignited by torch with full consumption of powder over 7.4 seconds.	Rapid burning consistent more with the hazard of Class 1 explosives
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 0.5 L/(kg·hr).	Pass (NOT Division 4.3)

Table 6: Testing Summary for Large-scale Mix ID #5

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm; limiting diameter less than 2.0 mm.	Pass
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials at a drop height of 10 cm.	Pass
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; several holes in the aluminum witness plate where thermite melted through.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned; no significant damage to the steel witness plate but one very small hole in plate.	Minimal threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and briefly produced local flame enhancement within the flames of the fire.	Sub-scale quantities appear to present minor hazards

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
12	UN Test N.1 Test method for readily combustible solids (gas torch)	For Trial 1, the sample ignited with a burning time of approximately 2 seconds for the entire length of the powder train pile. For Trial 2, the sample ignited but the reaction did not propagate down the powder train pile.	Consistent with Division 4.1, packing group II
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by torch and hot wire with full propagation (250mm) of powder over 1.5 - 2.4 seconds (104 - 167mm/sec). <u>Crucible bowl</u> : Sample ignited by torch (aided by a thermite starting powder) with full consumption of powder over 6.4 seconds.	Rapid burning consistent more with the hazard of Class 1 explosives
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 0.2 L/(kg·hr).	Pass (NOT Division 4.3)

Table 7: Testing Summary for Large-scale Mix ID #6

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "F" (tube fragmented into three or more mainly large pieces) at 2.0 mm; limiting diameter greater than 2.0 mm.	FAIL (consistent with Class 1)
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the steel witness plate.	High threat to 3-mm thick mild steel
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite ignited and burned over approximately 17 seconds, producing local flame enhancement within the flames of the fire.	Reaction rate and fireball more consistent with the hazards of Division 1.4S

Test	Test Name	Conditions and Results	Conclusions
11	Sub-scale UN Series 7 (e) EIS External fire	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by torch and hot wire with full propagation (250mm) of powder over 36 - 75 seconds (3.3 - 6.9 mm/sec). <u>Crucible bowl</u> : Sample ignited by torch with full consumption of powder over 10.5 seconds.	Burning rate > 2.2 mm/sec (consistent with Division 4.1, packing group II)
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 1.6 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)

Table 8: Testing Summary for Large-scale Mix ID #7

Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes or witness plates; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "O" (tube unchanged) at 2.0 mm during 5-minute test duration; limiting diameter greater than 2.0 mm. Effect Type "F" (tube fragmented into three or more mainly large pieces) at 2.0 mm after 5 minutes; limiting diameter greater than 2.0 mm.	Pass (NO violent effect on heating under confinement) at 5 minutes; FAIL (consistent with Class 1) after 5 minutes
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire. There was no other damage to the pipe and no damage to either end cap.	Pass (NO transition from deflagration to detonation)
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples exploded upon ignition after 1.0 - 1.5 minutes.	FAIL (12-meter drop test required to determine whether safe for transport; waived based on impact and friction sensitivity test results)

Test	Test Name	Conditions and Results	Conclusions
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center; severe deformation of suspended aluminum witness plate.	Hazards consistent with Division 1.1
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center slightly bowing the witness plate.	Hazards consistent with Division 1.1
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermite exploded upon ignition with nearly instantaneous consumption of the package contents.	Hazards consistent with Division 1.1
11	Sub-scale UN Series 7 (e) EIS External fire	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard consistent with Class 1 when heated under confinement
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives

Test	Test Name	Conditions and Results	Conclusions
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by pyrogen igniter (flame) and hot wire with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity): much faster than 8,000 mm/sec. Plate broken into pieces. <u>Crucible bowl</u> : Sample ignited by pyrogen igniter (flame) with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity). No visible damage to crucible bowl.	Explosion consistent more with the hazard of Division 1.1 explosives
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 1.4 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)

Table 9: Testing Summary for Large-scale Mix ID #8

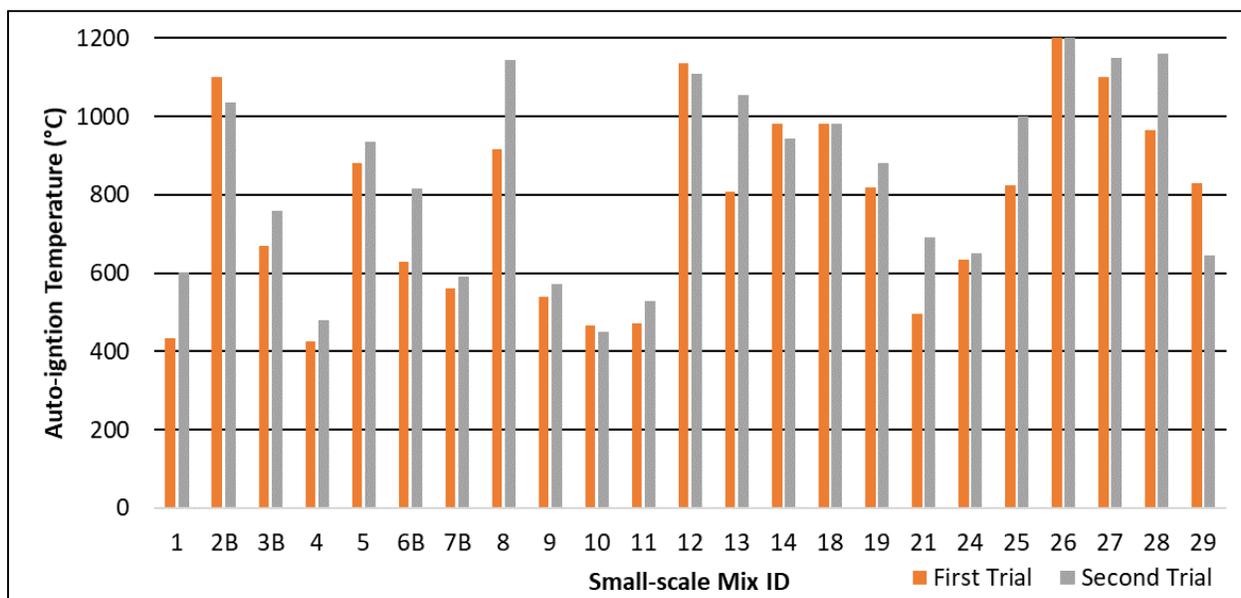
Test	Test Name	Conditions and Results	Conclusions
1	UN Series 2 (a) UN Gap	No damage to tubes and witness plate bent into a "V" shape; material in pipe consumed.	Pass
2	UN Series 2 (b) Koenen	Effect Type "F" (tube fragmented into three or more mainly large pieces) at 2.0 mm; limiting diameter greater than 2.0 mm.	FAIL (consistent with Class 1)
3	UN Series 2 (c) (ii) Internal ignition	For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging it to approximately 4-cm wide. There was no other damage to the pipe and no damage to either end cap.	Pass
4	UN Series 3 (a) BOE impact sensitivity	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	UN Series 3 (b) BAM friction sensitivity	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)

Test	Test Name	Conditions and Results	Conclusions
6	UN Series 3 (c) Thermal stability	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
7	UN Series 3 (d) Small-scale burning	The two 100-gram samples exploded upon ignition with a loud report.	FAIL (12-meter drop test required to determine whether safe for transport; waived based on impact and friction sensitivity test results)
8	Unconfined UN Series 6 (a) Single package on suspended 2-mm thick 1100-0 aluminum witness plate	Package containing 9.1 kg of thermitite exploded upon ignition of pyrogen match in the center.	Hazards consistent with Division 1.1
9	Unconfined UN Series 6 (a) Single package on suspended 3-mm thick mild steel witness plate	Package containing 9.1 kg of thermitite exploded upon ignition of pyrogen match in the center; severe deformation of suspended aluminum witness plate.	Hazards consistent with Division 1.1
10	Sub-scale UN Series 6 (c) External fire (bonfire)	Package containing 9.1 kg of thermitite exploded upon ignition with nearly instantaneous consumption of the package contents.	Hazards consistent with Division 1.1
11	Sub-scale UN Series 7 (e) EIS External fire	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard consistent with Class 1 when heated under confinement

Test	Test Name	Conditions and Results	Conclusions
12	UN Test N.1 Test method for readily combustible solids (gas torch)	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives
13	UN Test N.1 Test method for readily combustible solids (hot wire near 1,000 °C)	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives
14	Large-scale UN Test N.1 Test method for readily combustible solids (gas torch, hot wire near 1,000 °C, severe ignition sources)	<u>Flat plate</u> : Sample ignited by pyrogen igniter (flame) and hot wire with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity): much faster than 8,000 mm/sec. Plate broken into pieces. <u>Crucible bowl</u> : Sample ignited by pyrogen igniter (flame) with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity). No visible damage to crucible bowl.	Explosion consistent more with the hazard of Division 1.1 explosives
15	UN Test N.4 Test method for self-heating substances	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
16	UN Test N.5 Test method for substances which in contact with water emit flammable gases	Maximum gas emission rate of 2.2 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)

The following figure shows a comparison of the auto-ignition temperatures for the various thermite mixtures, organized by Small-scale Mix ID. The average auto-ignition temperature of the twenty-three small-scale thermite test samples was 809 °C with a standard deviation of 240 °C. NOTE: Small-scale Mix ID #26 (Large-scale Mix ID #2) had an auto-ignition temperature greater than 1200 °C.

Figure 1: Comparison of the Auto-ignition Temperatures for the Thermite Mixtures



4.0 BACKGROUND

Historically, industry has offered traditional thermite formulations (containing only metal and metal oxides) for transport in Class 4 as flammable or water-reactive solids. The reaction produces a burst of heat, high temperature, and molten metal for various uses including joining steel railroad ties or copper transmission lines, metal refining, disabling munitions, etc. Traditional thermite formulations pass the UN Manual of Tests and Criteria, Appendix 6 “Screening Procedures” for substances which may have explosive properties as they do not contain any of the chemical groups listed in Table A6.1 that are associated with explosive properties. NOTE: The Appendix 6 Screening Procedures cannot be used for substances manufactured with the view to producing a practical explosive or pyrotechnic effect.

Newer “exotic” thermite formulations include those with additives such as plastics, oxidizers, nano materials, explosives, etc. to confer special compositions or actions upon use. Thermite research is needed to identify families of new and existing thermite compositions and to determine their hazards in transport and to transport vehicles. Forming a technical database can help facilitate PHMSA rulemaking policies and decisions on thermite-based products.

SMS performed research on thermite formulations to arrange the formulations into the following families:

- Metal & metal oxide formulations: Aluminum powder mixed with another metal oxide, such as iron (II or III), copper (I or II), tin (IV), titanium (IV), manganese (III or IV), chromium (III), cobalt (II), silicon (II), nickel (II), vanadium (V), silver (I), molybdenum (IV). Some firework effects powders appear to contain thermite-like mixtures of copper oxide, magnalium, and titanium. Industrial cutting torches contain powder mixtures of aluminum, magnesium, cupric oxide, and molybdenum oxide. Other thermite-like mixtures include dry ice with a metal powder (such as magnesium, aluminium or boron), and Teflon with magnesium or aluminum (used in flare mixtures). A dry ice mixture is impractical for this effort (not thermally stable).
- Nano-thermite, super-thermite, metastable intermolecular composites (MICs): Generally consist of powders with a particle size less than 100 nm. Particle sizes below three micrometers in diameter have rapidly diminishing returns since irregular distribution of binary constituents limits performance. A highly ordered and optimized microstructure is desired for high energy density and high propagation velocity; procurement of this type of optimized sample may be cost prohibitive, impracticable, and of limited applicability.
- Thermate: Metal & metal oxide thermites enriched with a salt-based oxidizer. For example, thermite with barium nitrate and a binder (Dextrin).
- Exotic thermites: Metal & metal oxide thermites enriched with explosives and binders.

A summary of SMS's research on thermites is contained in the document "Summary of Research on Thermite Compositions for U.S. DOT PHMSA", SMS-5216-M1, Rev 0, Safety Management Services, Inc., 26 Feb 2019.

Thermite subject matter experts provided SMS with the following references that list the calculated energy, gas content, and physical product state for various thermite formulations:

- SAND95-2448C, "A Survey of Combustible Metals, Thermites, and Intermetallics for Pyrotechnic Applications", S. H. Fisher and M. C. Grubelich, Sandia National Laboratories, July 1996, Table 1 - Thermite Reactions.
- SAND98-1176C, "Theoretical Energy Release of Thermites, Intermetallics, and Combustible Metals", S. H. Fisher and M. C. Grubelich, Sandia National Laboratories, July 1998, Table 1b - Thermite Reactions 1c - Thermite Reactions (in Descending Order of Heat of Reaction per Unit Mass)

An extracted table from SAND98-1176C is shown below for the highest energy formulations with the following notes:

1. The list is of simple, two component thermite compositions and does not consider the multi-component thermite compositions.
2. The two most common thermites in industry are iron thermite ($\text{Fe}_2\text{O}_3 + 2 \text{Al}$) and copper thermite ($3 \text{CuO} + 2 \text{Al}$) -- highlighted in yellow in the table below.
 - a. Both of these compositions are relatively high in energy (945 - 974 J/gm) and result in liquid-gas products.

- b. The copper thermite has a relatively high gas output (0.54 moles / 100 grams), which may be more prone to fail UN Series 2.
3. Many of the high-energy thermite mixtures contain components that may not be practical to procure or handle for this effort.

Table 1c - Thermite Reactions (in Descending Order of Heat of Reaction per Unit Mass)

reactants		adiabatic reaction temperature (K)		state of products		gas production		heat of reaction	
constituents	ρ_{TMD} , g/cm ³	w/o phase changes	w/ phase changes	state of oxide	state of metal	moles gas per 100 g	g of gas per g	-Q _r , cal/g	-Q _r , cal/cm ³
3Mg + B ₂ O ₃	1.785	6389	3873	l-g	liquid	0.4981	0.2007	2134	1195
3Be + B ₂ O ₃	1.850	3278	2573	liquid	s-l	0.0000	0.0000	1639	3033
2Be + MnO ₂	3.882	6078	2969	liquid	gas	0.9527	0.5234	1586	6158
10Al + 3I ₂ O ₅	4.119	8680	>3253	gas	gas	0.6293	1.0000	1486	6122
4Li + MnO ₂	1.656	3336	2334	liquid	l-g	0.4098	0.2251	1399	2317
6Li + MoO ₃	1.688	4035	2873	l-g	solid	0.2155	0.0644	1342	2265
2Mg + MnO ₂	2.996	5209	3271	liquid	gas	0.7378	0.4053	1322	3961
6Li + B ₂ O ₃	0.891	2254	1843	s-l	solid	0.0000	0.0000	1293	1152
2Al + Ni ₂ O ₃	4.045	5031	3187	liquid	l-g	0.4650	0.2729	1292	5229
3Be + Fe ₂ O ₃	4.163	4244	3135	liquid	l-g	0.1029	0.0568	1281	5332
Be + CuO	5.119	3761	2820	s-l	liquid	0.0000	0.0000	1221	6249
4Be + Fe ₂ O ₃	4.180	4482	3135	liquid	l-g	0.0336	0.0188	1175	4910
4Al + 3MnO ₂	4.014	4829	2918	liquid	gas	0.8136	0.4470	1159	4651
10Y + 3I ₂ O ₅	4.638	12416	>4573	gas	gas	0.4231	1.0000	1144	5308
6Li + Fe ₂ O ₃	1.863	3193	2510	liquid	liquid	0.0000	0.0000	1143	2130
2Li + CuO	2.432	4152	2843	liquid	l-g	0.2248	0.1428	1125	2736
2Al + MoO ₃	3.808	5574	3253	l-g	liquid	0.2425	0.2473	1124	4279
2Y + Ni ₂ O ₃	4.636	7614	3955	liquid	gas	0.5827	0.3420	1120	5194
3Mg + Fe ₂ O ₃	3.224	4703	3135	liquid	l-g	0.2021	0.1129	1110	3579
Mg + CuO	3.934	6502	2843	solid	l-g	0.8186	0.5201	1102	4336
10Al + 3V ₂ O ₅	3.107	3953	3273	l-g	liquid	0.0699	0.0356	1092	3394
8Li + Fe ₃ O ₄	0.517	3076	2412	liquid	liquid	0.0000	0.0000	1053	2036
4Mg + Fe ₃ O ₄	3.274	4446	3135	liquid	l-g	0.1369	0.0764	1033	3383
4Y + 3MnO ₂	4.690	7405	<5731	gas	gas	0.8110	1.0000	1022	4792
8Al + 3Co ₃ O ₄	4.716	3938	3201	liquid	l-g	0.2196	0.1294	1012	4772
2Y + MoO ₃	4.567	8778	>4572	gas	liquid	0.6215	1.0000	1005	4589
2Al + 3CuO	5.109	5718	2843	liquid	l-g	0.5400	0.3431	974.1	4976
10Y + 3V ₂ O ₅	3.970	7243	>3652	l-g	gas	0.2130	0.4181	972.5	3861
2Al + Fe ₂ O ₃	4.175	4382	3135	liquid	l-g	0.1404	0.0784	945.4	3947
2Be + SiO ₂	2.410	2580	2482	solid	liquid	0.0000	0.0000	936.0	2256
2Y + 3CuO	5.404	7668	3124	liquid	l-g	0.7204	0.4577	926.7	5008
3Be + Cr ₂ O ₃	4.089	3107	2820	s-l	liquid	0.0000	0.0000	915.0	3741
2Al + 3AgO	6.085	7503	3253	l-g	gas	0.7519	0.8083	896.7	5457
8Al + 3Fe ₂ O ₃	4.264	4057	3135	liquid	l-g	0.0549	0.0307	878.8	3747
2Be + PbO ₂	7.296	8622	4123	l-g	gas	0.4665	0.8250	875.5	6387
8Y + 3Fe ₂ O ₃	4.803	5791	3135	liquid	l-g	0.3812	0.2129	856.3	4113
10La + 3I ₂ O ₅	5.501	9107	>4472	gas	gas	0.3347	1.0000	849.2	4672
10Nd + 3I ₂ O ₅	5.896	10067	<7580	gas	gas	0.3273	1.0000	840.6	4956
6Li + WO ₃	2.478	3700	2873	l-g	solid	0.0113	0.0034	825.4	2046
2Al + 3CoO	5.077	3392	3201	liquid	l-g	0.0430	0.0254	824.7	4187
2Al + 3NiO	5.214	3968	3187	liquid	l-g	0.0108	0.0063	822.3	4288

In order to enable assessment of multi-component thermite compositions, thermochemical calculations were performed using FactSage 7.2. The following is a summary of that effort.

4.1 Thermochemical Code calculations to Predict output of thermite compositions

4.1.1 Abstract

A need is identified to evaluate a variety of materials for exothermic properties. Testing all materials is cost-prohibitive, so a technical rationale is needed that reduces the number of systems tested. Additionally, high confidence is desired that the full range of energetic performance such as high temperature, thermal energy release, and pressurization rate are sufficiently evaluated, though very few may be considered for testing.

One approach used successfully in prior energetic study efforts¹ used computer simulation to reduce the number of empirical tests. This work follows similar logic, and simulates many systems of interest via computer to guide decisions. In this work, thermochemical calculations using FACTSAGE 7.2 was performed for a few selected thermite systems of interest. Results were compared to reference literature, and upon reasonable agreement, additional chemical composition simulation runs were made that do not appear in literature. The latter is applicable to real world thermite products, which typically use a base simple chemistry modified with additives. This technique using computer simulation allows for efficient screening of both simple and complex thermite systems for consideration in further testing or engineering evaluations.

4.1.2 Approach

Two well published systems having aluminum as the metal fuel, one with iron oxide and the other copper oxide were selected for simulation and comparison with open literature. Upon similar results from FACTSAGE version 7.2 software and work by Fischer and Grubelich², other systems will be simulated and outputs reported. Common simulation initial material conditions of temperature = 298.15 K and pressure = 1 atm shall be used.

4.1.3 Results

For iron oxide and aluminum, the reaction is:



FACTSAGE predicts an adiabatic reaction temperature of 3129K, which is quite similar with Fischer and Grubelich value of adiabatic reaction temperature of 3135 K.

For the copper oxide and aluminum, the reaction is:



¹ Csernica, Mansfield, Boman Studies on Composition and Manufacturing Process for 5.56mm Tracer Ammunition NDIA Presentation, May 2017.

² S.H. Fischer and M.C. Grubelich, Theoretical Energy Release of Thermites, Intermetallics, and Combustible Metals, presented at International Pyrotechnics Seminar, 1998.

FACTSAGE predicts an adiabatic reaction temperature = 2840 K, which is quite similar with Fischer and Grubelich value of adiabatic reaction temperature of 2843 K. With these two common systems verified in simulation, a more complex system not reported in Fischer and Grubelich was selected for evaluation. A thermate mix, known as 'TH3', found in products such as the legacy AN-M14 grenade was selected due to its historical significance and longevity in production articles. US Patent 5,698,812 references³ a formula for TH3 as 68.7% thermite ($\text{Fe}_2\text{O}_3 + 2 \text{Al}$), 29.0% barium nitrate, 2% sulfur, and 0.3% binder. Notably, this patent discloses an improved formula using 66.69% potassium nitrate, 14.96% titanium, 7.78% silicon, 8.67% aluminum, and 1.99% nitrocellulose binder. Table 1 below shows a comparison of temperature, energy, and gas evolved from ideal thermodynamic calculations. The patent notes the reaction temperature is increased due to the addition of oxidizer by promoting increased mass transport and has lower ignition temperature than standard binary thermite. It is interesting that the mixture is an intermetallic base, which as a family have lower energy output on average than the same family with an oxide present.

One shortcoming in the FACTSAGE chemical library available are very few polymers and binders. For sake of ease in simulation, the highest molecular weight hydrocarbon available, naphthalene, was used in place of polymeric binders. The quality of this simplifying assumption was checked with additional runs made for comparison with and without naphthalene faux ingredient. When the binder was omitted, simulation predicts the temperature and energy output were similar, however gas generation per mass reactants varied. Therefore, care should be taken if gas output is an important parameter of study when using polymer/binders in simulation for which accurate thermodynamic input properties are unknown or assumed. Results of these runs and all others performed in this work are shown in Table 2.

³ Song, Thermite Destructive Device US Patent 5,698,812, issued Dec 16, 1997.

Table 10: Two thermate compositions and a thermite compared by computer simulation and literature

System Description	Units	Thermite	Composition TH3	Alternative Composition
Literature citation		Fischer and Grubelich	US 5,698,812	US 5,698,812
	Units			
Composition	wt%		29.0% Ba(NO ₃) ₂	66.7% KNO ₃
	wt%	23.7% Al	45.8% Al	14.96% Ti
	wt%	76.3% Fe ₃ O ₄	22.9% Fe ₃ O ₄	8.67% Al
	wt%		2.0% S	7.78% Si
	wt %		0.3% Binder*	1.99% nitrocellulose**
Adiabatic Rxn Temp	K	3135	3273	2755
Gas Evolved	mol/100g	.0549	-	-
Heat of Reaction	Cal/g	878	795	-
Heat of Reaction	J/g	3674	3326	-
FACTSAGE predictions:				
Adiabatic Rxn Temp	K	3129	2510	3024
Mass, Total	Gram	91.045	55.934	62.463
Solids Mass	Gram	90.341	41.674	30.151
Gas Mass	Gram	0.704	14.260	32.312
Energy per Mass (J/g)	J/gram	-3682	-2613	-3302
Gas Evolved	mol/100g	0.0139	0.439	1.423

*assumed binder is PBAN, simulated as naphthalene

** simulated as naphthalene

Having completed analysis in a military composition, another initial candidate was selected from a commercial application having an inter-metallic reactive system, to compare with Fischer and Grubelich. Indium Corporation discloses a basic formula in the Safety Data Sheet (SDS) for a commercial product called Nanofoil™. The basic thermite family is a metal-metal, primarily Ni-Al system with vanadium and indium in lesser amounts. The SDS was unclear as to the levels of minor additives, so for the sake of simplicity, an ideal mixture of nickel and aluminum was modeled:



Using FACTSAGE, the adiabatic reaction temperature was calculated at 2265 K. Energy output per mass was -141,210 J / 99.166 grams = -1424 J/g = -340 cal/g. The Fischer and Grubelich work cited two other molar ratios:





Using FACTSAGE, the adiabatic reaction temperature for the 1:3 molar ratio scenario was calculated at 1412 K and -138 cal/g, as compared with the Fischer and Grubelich values of 1524 K and -180 cal/g. Similarly, for the 1:1 molar ratio, FACTSAGE calculated adiabatic reaction temperature is 1912 K and -140 cal/g, as compared with the Fischer and Grubelich values of >1910 K and -330 cal/g. There is a much more notable difference between the literature and FACTSAGE calculations in this specific system; additional work is needed to resolve or explain differences in these estimates.

Another system of interest, less common usage, is that of iodine pentoxide and aluminum. Literature search revealed that Dr. Michelle Pantoya has authored/co-authored numerous journal articles involving thermites, including several on the iodine pentoxide-aluminum system. One co-authored work with Oliver Mulamba⁴ involved surface chemistry reaction kinetics work on diiodine pentoxide and aluminum. In this work, Mulamba and Pantoya describe the chemical system as potentially attractive due to its high exothermic energy and the reaction products form I₂, which has biocidal effects. Mulamba and Pantoya notes that the pentoxide dissociates in air in the presence of humidity, forming iodic acid, HIO₃, which is reversibly treated with heat to the pentoxide form at around 470 K.

Though desirable to simulate, the standard ingredient library in FACTSAGE does not include iodine pentoxide. This is identified as an area of potential future investigation to either locate literature values or make estimates of the property inputs required by the software. In lieu of simulation, a short review using the Mulamba and Pantoya work is provided below as it relates to the system energy and kinetic rates.

Upon close inspection of the calorimetry in Mulamba and Pantoya's article, an interpretation is proposed here that the reaction rate is slower than typical Class 1 materials given a thermal stimulus. In the Mulamba and Pantoya article, Figure 2 for the pentoxide with aluminum reaction given a heating rate of 25°C per minute by differential scanning calorimetry (DSC), the onset temperature was reported near 300°C (573 K) with peak exotherms at over 400°C (673 K) or about 100 K difference (equating to 4 minutes of heating). As compared with 1, 3, 5- trinitro-1, 3, 5-triazinane (RDX)⁵, the difference between onset temperature and peak exotherm is much less at 14 K (less than 45 seconds duration of heating). In this RDX versus iodine pentoxide system example, the temporal ratio is about 5X slower to reach peak exotherm. Therefore, it is conceivable that the reaction rate of iodine pentoxide with aluminum is much slower than typical Class 1 material detonations more broadly. In explosive applications where fast velocity profile is desirable, pentoxide materials may be less versatile, as the reactions cited

⁴ Oliver Mulamba, Dr. Michelle Pantoya, [Exothermic surface reactions in alumina-aluminum shell-core nanoparticles with iodine oxide decomposition fragments](#) Journal of Nanoparticle Research, 2014.

⁵ US Patent 6,406,918 B1 June 18, 2002, Figure 13 shows onset temperature at approximately 225°C and exothermic maximum at 239°C at a heating rate of 20°C per minute.

going through a number of oxidation states may be more susceptible to mass-transport limits and therefore be somewhat slower, even though the iodine is a reactive species. Correlation between pressure wave velocity (and detonation) with DSC may be a safer and simpler way to test more materials with lesser quantities of materials. Iodine pentoxide decomposition and kinetics, through modeling and/or empirical testing is an area recommended for consideration in future studies.

In order to help summarize all runs performed in this work, a summary table of the key outputs from the FACTSAGE simulations is shown below as Table 3. Not discussed previously was a variant of the TH3 replacement formula labeled 'XM89 Grenade' in the table below, which adds a new element, carbon into the composition, which has five different metals (complex intermetallic thermite) and one strong oxidizer, making a heavily nitrated thermate. This composition has the highest gas generation rate of any simulated, which at face value may be a reason why XM89 grenade improvements were pursued.

Table 11: Summary of all FACTSAGE Simulations performed in this work

Name:	Fe Std	Cu Std	TH3	TH3 no bind	TH3 Alt	TH3 Alt no bind	XM89 grenade	2 Ni 3 Al	1 Ni 3 Al	1 Ni 1 Al
Input Composition:										
(wt%)										
Al	23.7	18.4	45.8	45.9	8.67	8.84	8.2	40.8	58.0	31.5
Ti					15.0	15.2	11.2			
Si					7.78	7.93	6.1			
Ni								59.2	42.0	68.5
C							5.1			
S			2.0	2.0			2.0			
Fe ₃ O ₄	76.3		22.9	23.0						
CuO		81.6								
Ba(NO ₃) ₂			29.0	29.1						
KNO ₃					66.7	68.0	67.4			
C ₁₀ H ₈ (Naph)					1.99					
Outputs:										
Adiabatic T (K)	3129	2840	2510	2518	3024	3026	2901	2265	1412	1912
Mass, Total (g)	91.0	292.6	55.9	99.7	62.5	100.0	100	198.3	139.6	85.7
Solids Mass (g)	90.3	193.4	41.7	61.5	30.2	48.8	39.5	198.3	139.4	85.7
Gas Mass (g)	0.704	99.2	14.3	38.2	32.3	51.2	61.5	0	0	0
Energy (J/g)	-3682	-1600	-2613	-2213	-3302	-3313	-3281	-1424	-577	-586
Gas (mol/100g)	0.0139	.520	0.439	.633	1.42	1.41	1.65	0	0	0

4.1.4 Conclusions

This work provides some example thermite reaction systems evaluated by computer simulation. For the iron oxide and copper oxide systems with aluminum, using the computer code FACTSAGE agrees very well with literature values. Therefore, the techniques were used to evaluate two thermite systems with additives, one a common thermite and an alternative formula with entirely different family chemistry (Ti-Al-Si-KNO₃). Both of these formula simulations predict noteworthy performance aspects. In the case of both formulas, there is a much higher gas evolution mass than the standard thermite base, due to the addition of oxygen and nitrogen in the system via oxidizer. Also noteworthy is that the alternative formula based

on Ti-Al-Si intermetallic predicts approximately a 500 K increase in temperature as compared with TH3 formula. It also has a much greater gas evolution potential than either the thermite base or the legacy TH3 formula.

A relevant intermetallic thermite system with commercial product examples was selected for simulation and comparison with literature, having Ni-Al composition. Preliminary efforts revealed some differences that could not be explained within the scope of the current effort. Additional work would be needed to resolve or explain differences in these estimates through a more thorough review of the literature and underlying assumptions inherent to the computing routines.

4.2 Test Sample and Test Selection - Large-Scale Thermite Mixes

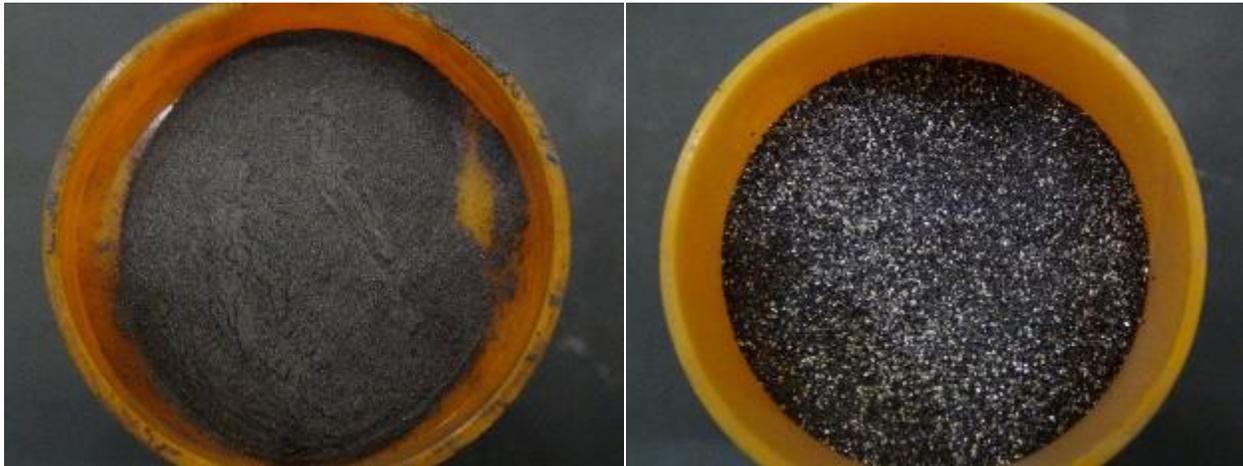
Based on the research, the following were the proposed test samples for PHMSA's consideration and approval.

Table 12: Potential Large-scale Thermite Test Samples

ID	Sample Description	Packaging Configuration	Potential Suppliers & Notes
1	[not disclosed]	[not disclosed]	[not disclosed]
2	[not disclosed]	[not disclosed]	[not disclosed]
3	[not disclosed]	[not disclosed]	[not disclosed]
4	[not disclosed]	[not disclosed]	[not disclosed]
5	[not disclosed]	[not disclosed]	[not disclosed]
6	[not disclosed]	[not disclosed]	[not disclosed]
7	[not disclosed]	[not disclosed]	[not disclosed]
8	[not disclosed]	[not disclosed]	[not disclosed]

The photo below shows examples of thermites with fine particles (left) and coarse particles (right).

Photo 1: Examples of Thermite Fine Powder (left) and Course Powder (right)



One of the families of thermites is Magnesium/Teflon/Viton thermite mixtures. These have common application in aircraft flares (pyrotechnic effect) and are generally included in Class 1 by definition; therefore, one of these formulations is not currently included in the proposed test matrix (this can be included if desired by PHMSA).

One of the thermite Subject Matter Experts informed us of the following:

1. From SMS's potential thermite report in March, they have never seen two of the listed thermites but have personally used two of the thermites in their research at the university. Based on this feedback, SMS adjusted selected test samples.
2. Listed oxides that are common in thermites and nanothermites.

Based on SMS's inquiries to thermite manufacturers, packaging of thermite varies but appears to contain between 1 - 25 lbs of thermite.

The following were the list of proposed tests for classification of thermites:

1. UN Series 3 (a) Impact sensitivity
2. UN Series 3 (b) Friction sensitivity
3. UN Series 3 (c) Thermal stability
4. UN Series 3 (d) Small-scale burning
5. Differential Scanning Calorimeter (DSC) for heat capacity with maximum furnace temperature of 700
6. UN Series 2 (a) UN Gap Test
7. UN Series 2 (b) Koenen Test
8. UN Series 2 (c) (ii) Internal Ignition Test
9. UN Series 6 (d) Unconfined package on suspended witness plate (steel) to represent a threat to the structural integrity of aircraft
10. UN Series 6 (d) Unconfined package on suspended witness plate (aluminum) to represent a threat to the structural integrity of normal ground transport vehicles
11. UN Series 6 (c) External fire (bonfire) test

12. Consider an extreme confinement and heating test (no venting) to quantify maximum violence of reaction: One UN Series 6 (c) External fire (bonfire) using liquid fuel (800 °C) with each thermit sample confined in UN Series 7 (f) capped steel pipes, three pipes of each sample on the bonfire. Pipes could explode, spraying molten metal; melt, releasing molten metal into the liquid fuel (in which case SMS would add a layer of water below the liquid fuel to cool any molten metal; or fully contain the reaction).
13. UN Test N.4 Test method for self-heating substances
14. UN Test N.5 Test method for substances which in contact with water emit flammable gases
15. UN Test N.1 Test method for readily combustible solids using gas burner: Preliminary screening test and Burning rate test*
16. UN Test N.1 Test method for readily combustible solids using hot wire near 1,000 °C, Preliminary screening test and Burning rate test*
17. Large-scale UN Test N.1 Test method for readily combustible solids, three trials with gas burner and three trials with hot wire near 1,000 °C.

*This preliminary screening test requires a hot flame (minimum temperature 1,000 °C) from a gas burner (minimum diameter 5 mm) to be applied to one end of the powder train; the Burning rate test uses any suitable ignition source such as a small flame or a hot wire of minimum temperature 1,000 °C to ignite the pile at one end. Some Competent Authorities and manufacturers have claimed that thermit is not regulated as a flammable solid since if it does not ignite at 1,000 °C for five minutes. SMS has used a MAP-Pro (99.5 - 100% propylene, 0 - 0.5% propane) torch with a flame temperature of 3,600 °F (1980 °C) to ignite thermit samples for UN Test N.1. It was proposed that perhaps a test should be performed using a flame near 1,000 °C and another test with a hotter flame (like that from MAP-Pro gas).

Additionally, some thermites may be capable of passing the preliminary screening test since they can experience sporadic / intermittent reactions (not a continuous burn). For example, consider the photos from the following case. The burn time for each trial was around 1 - 3 seconds (very rapid burn). Only one trial is required in the preliminary screening trial; Trial 1 passed the test criteria since the reaction did not propagate along 200mm of the train within 20 minutes for metal powders, even though it consumed half the sample in less than three seconds. Since the reaction was so rapid, SMS performed additional trials even though the thermit sample technically passed the preliminary screening test. Only two of the six trials resulted in reaction of greater than 165mm of the powder train (passing results without regulation), even though the reactions were extremely rapid. It was proposed that perhaps a larger scale linear burn rate test could be appropriate for thermites, giving more mass for the reaction to propagate (custom test, filling the entire plate with thermit).

Photo 2: UN Test N.1 - Setup



Photo 3: UN Test N.1 - Trial 1 (partial consumption, <180mm)



Photo 4: UN Test N.1 - Trial 4 (full reaction)



Photo 5: UN Test N.1 - Trial 5 (partial consumption, <180mm)



4.3 Test Sample and Test Selection - Small-Scale Thermite Mixes

[Not disclosed]

5.0 DESCRIPTION OF THERMITE TEST SAMPLES

5.1 Thermite Compositions Utilized in Main Test Matrix (Large-scale Samples)

5.1.1 *Large-scale Mix ID #1*

This thermite was a fine, red powder.

Photo 6: Large-scale Mix ID #1



5.1.2 *Large-scale Mix ID #2*

This thermite was a coarse powder that was a mixture of black and light grey particles.

Photo 7: Large-scale Mix ID #2



5.1.3 *Large-scale Mix ID #3*

This thermite was a coarse powder that was a mixture of black and light grey particles with occasional pieces of steel punching.

Photo 8: Large-scale Mix ID #3



5.1.4 *Large-scale Mix ID #4*

This thermite was a very fine, dark grey powder.

Photo 9: Large-scale Mix ID #4



5.1.5 *Large-scale Mix ID #5*

This thermite was grey and silver speckled powder with medium-sized particles.

Photo 10: Large-scale Mix ID #5



5.1.6 *Large-scale Mix ID #6*

This thermite was very fine, light grey powder.

Photo 11: Large-scale Mix ID #6



5.1.7 *Large-scale Mix ID #7*

This thermite was a very fine, dark grey powder.

Photo 12: Large-scale Mix ID #7



5.1.8 *Large-scale Mix ID #8*

This thermite was a very fine, grey powder.

Photo 13: Large-scale Mix ID #8



5.2 Additional Thermite Compositions Utilized in Auto-Ignition Temperature Testing (Small-scale Samples)

5.2.1 Small-Scale Mix ID #1

This thermite was a very fine, grey powder.

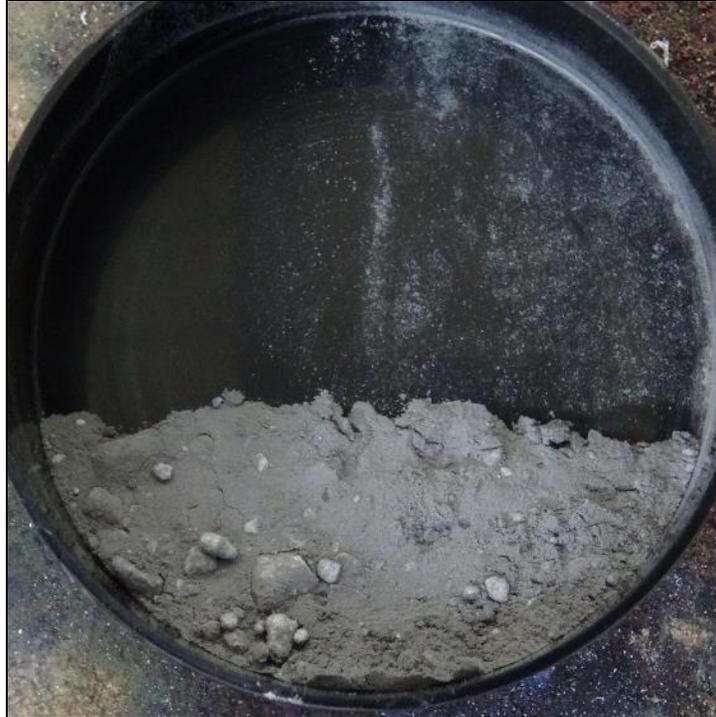
Photo 14: Small-Scale Mix ID #1



5.2.2 *Small-Scale Mix ID #2B*

This thermite was a fine, light grey powder.

Photo 15: Small-Scale Mix ID #2B



5.2.3 *Small-Scale Mix ID #3B*

This thermite was a very fine, light grey powder.

Photo 16: Small-Scale Mix ID #3B



5.2.4 *Small-Scale Mix ID #4*

This thermite was a very fine, grey powder.

Photo 17: Small-Scale Mix ID #4



5.2.5 *Small-Scale Mix ID #6B*

This thermite was a fine, red powder.

Photo 18: Small-Scale Mix ID #6B



5.2.6 *Small-Scale Mix ID #7B*

This thermite was a very fine, dark grey powder.

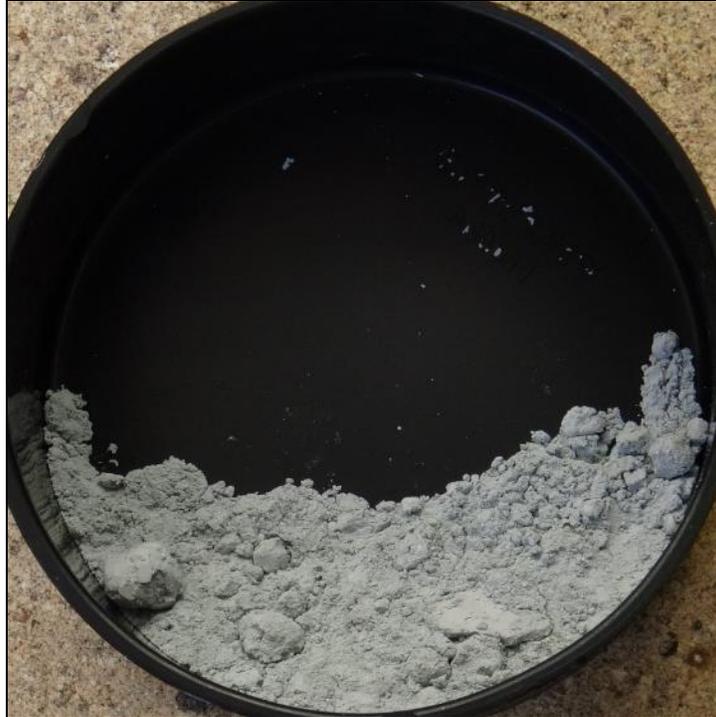
Photo 19: Small-Scale Mix ID #7B



5.2.7 *Small-Scale Mix ID #9*

This thermite was a very fine, off-white powder.

Photo 20: Small-Scale Mix ID #9



5.2.8 *Small-Scale Mix ID #10*

This thermite was a very fine, light red powder.

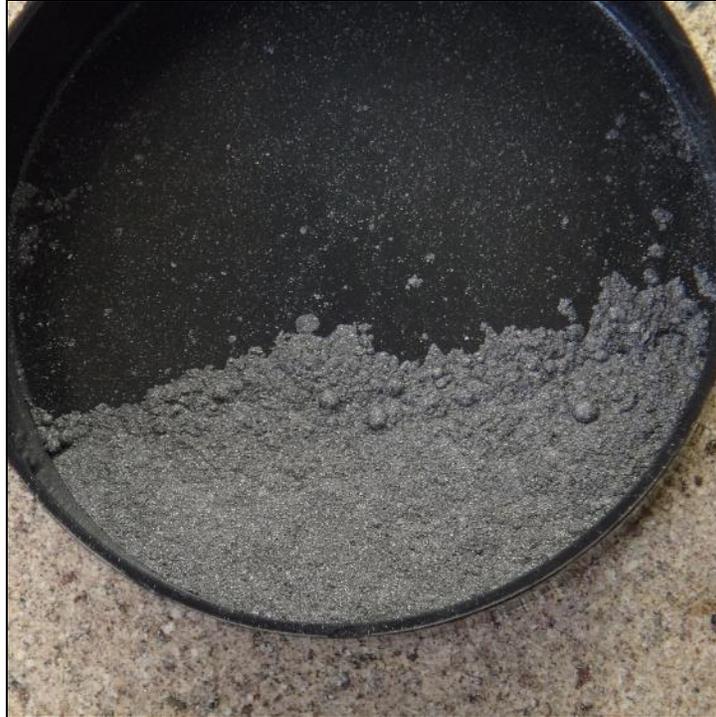
Photo 21: Small-Scale Mix ID #10



5.2.9 *Small-Scale Mix ID #11*

This thermite was a very fine, grey powder.

Photo 22: Small-Scale Mix ID #11



5.2.10 *Small-Scale Mix ID #12*

This thermite was a very fine, dark grey powder.

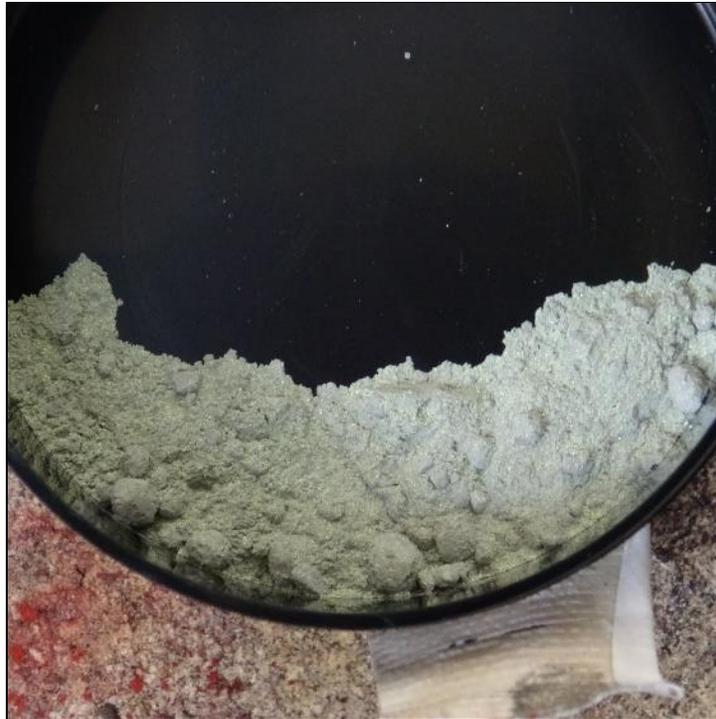
Photo 23: Small-Scale Mix ID #12



5.2.11 *Small-Scale Mix ID #18*

This thermite was a very fine, off-white powder.

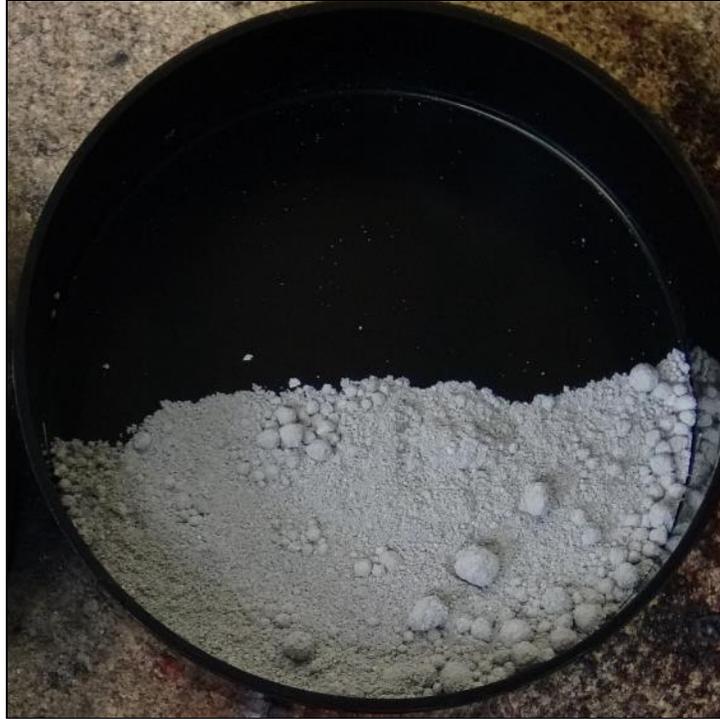
Photo 24: Small-Scale Mix ID #18



5.2.12 *Small-Scale Mix ID #19*

This thermite was a very fine, off-white powder.

Photo 25: Small-Scale Mix ID #19



5.2.13 *Small-Scale Mix ID #21*

This thermite was a fine, light green powder.

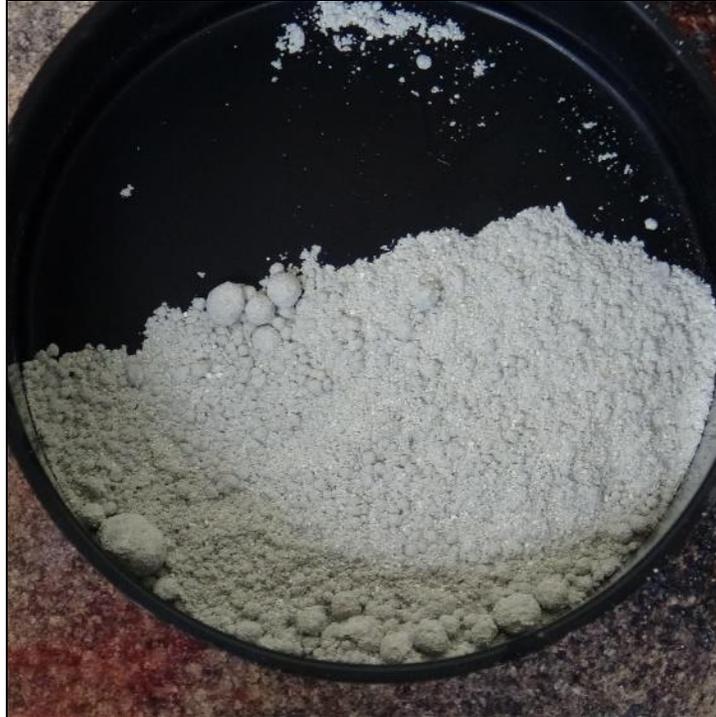
Photo 26: Small-Scale Mix ID #21



5.2.14 *Small-Scale Mix ID #24*

This thermite was a very fine, off-white powder.

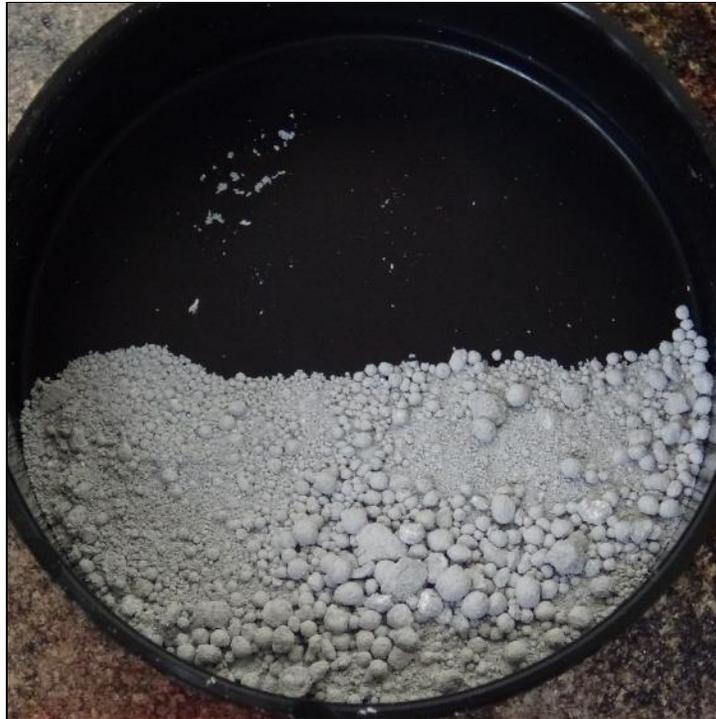
Photo 27: Small-Scale Mix ID #24



5.2.15 *Small-Scale Mix ID #25*

This thermite was a fine, off-white powder.

Photo 28: Small-Scale Mix ID #25



6.0 PACKAGING

Packaging typical for thermites consist of inner packagings of plastic bags and outer packagings of fiberboard boxes, as shown in the following photo, or plastic pails.

Photo 29: Packaging for Thermite (typical)



7.0 TEST DESCRIPTIONS AND RESULTS

7.1 UN Series 2 (a) UN Gap test

7.1.1 *Test Description*

This test is used to measure the ability of a substance under confinement to propagate a detonation by subjecting it to a shock impulse from a booster charge. The sample is loaded to the top of a cold-drawn, seamless, carbon steel tube (48.0 ± 2 mm outer diameter, 4 ± 1 mm wall, and 400 ± 5 mm long). Solid samples are loaded to the density attained by tapping the tube until further settling becomes imperceptible. The sample mass is determined and, if solid, the apparent density calculated.

A 160-gram RDX/wax (95/5) or PETN/TNT (50/50) booster of 50 mm diameter and approximately 50mm length is used to provide a known shock source and separated by a polymethyl methacrylate (PMMA) spacer that is 50 ± 1 mm (1.96 ± 0.04 in) in length and diameter (2-inch Gap). The tube is placed over a 150 ± 10 mm (6-in) square, 3.2 ± 0.2 mm (1/8-in) thick steel witness plate and separated from it by 1.6 ± 0.2 mm (1/16-in) thick spacers. An air gap of at least 50 mm (2 in) is provided between the witness plate and the ground. The booster is initiated by a standard detonator.

The test is performed two times unless a positive result is observed earlier. The test result is considered positive and the substance sensitive to shock if the tube is fragmented completely or the witness plate is holed. Any other result is considered negative.

The result is considered positive and the substance sensitive to shock for the UN Series 2 (a) test if the tube is fragmented completely or the witness plate is holed. Any other result is considered negative and the substance not sensitive to detonative shock for the UN Series 2 (a) test.

7.1.2 *Test Configuration*

A PETN/TNT (50/50) booster was utilized for these tests. The dimensions of the steel tubes were 48 mm outer diameter, 4 mm wall (36 mm ID), and 400 mm long. The quantity of material required to fill each tube is listed in the following table; the apparent density was calculated using a calculated internal volume of 503 cm^3 .

Table 13: Apparent Density for the UN Gap Test Samples

Item	Sample	Trial	Weight (g)	Density (g/cm ³)
1	Large-scale Mix ID #1	1	459	0.91
		2	455	0.90
2	Large-scale Mix ID #2	1	1009	2.01
		2	1017	2.02
3	Large-scale Mix ID #3	1	1221	2.43
		2	1245	2.48
4	Large-scale Mix ID #4	1	787	1.56
		2	768	1.53
5	Large-scale Mix ID #5	1	1168	2.32
		2	1175	2.34
6	Large-scale Mix ID #6	1	782	1.55
		2	750	1.49
7	Large-scale Mix ID #7	1	686	1.36
		2	692	1.38
8	Large-scale Mix ID #8	1	680	1.35
		2	683	1.36

The filled tube was centered over the witness plate, as shown in the following photo.

Photo 30: UN 2-inch Gap Setup (typical)



7.1.3 Test Results

The test results are summarized in the following table.

Table 14: Summary of UN Series 2 (a) UN Gap Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material in pipe consumed. - Trial 2: No damage to tube or witness plate; material in pipe consumed. 	Negative (NOT sensitive to detonative shock)
2	Large-scale Mix ID #2	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material remaining in pipe. - Trial 2: No damage to tube or witness plate; material remaining in pipe. 	Negative (NOT sensitive to detonative shock)
3	Large-scale Mix ID #3	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material remaining in pipe. - Trial 2: No damage to tube or witness plate; material remaining in pipe. 	Negative (NOT sensitive to detonative shock)
4	Large-scale Mix ID #4	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material in pipe consumed. - Trial 2: No damage to tube or witness plate; material in pipe consumed. 	Negative (NOT sensitive to detonative shock)
5	Large-scale Mix ID #5	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material in pipe consumed. - Trial 2: No damage to tube or witness plate; material in pipe consumed. 	Negative (NOT sensitive to detonative shock)
6	Large-scale Mix ID #6	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material in pipe consumed. - Trial 2: No damage to tube or witness plate; material in pipe consumed. 	Negative (NOT sensitive to detonative shock)
7	Large-scale Mix ID #7	2-in Gap: <ul style="list-style-type: none"> - Trial 1: No damage to tube or witness plate; material in pipe consumed. - Trial 2: No damage to tube or witness plate; material in pipe consumed. 	Negative (NOT sensitive to detonative shock)

Item	Sample	Conditions and Results	Assessment
8	Large-scale Mix ID #8	2-in Gap: - Trial 1: No damage to tube and witness plate bent into a "V" shape; material in pipe consumed. - Trial 2: No damage to tube and witness plate bent into a "V" shape; material in pipe consumed.	Negative (NOT sensitive to detonative shock)

The test results are shown in the following photos; pieces of the tube were included in the photo if found.

Photo 31: UN 2-in Gap Test Results for Large-scale Mix ID #1



Photo 32: UN 2-in Gap Test Results for Large-scale Mix ID #2



Photo 33: UN 2-in Gap Test Results for Large-scale Mix ID #3



Photo 34: UN 2-in Gap Test Results for Large-scale Mix ID #4



Photo 35: UN 2-in Gap Test Results for Large-scale Mix ID #5



Photo 36: UN 2-in Gap Test Results for Large-scale Mix ID #6



Photo 37: UN 2-in Gap Test Results for Large-scale Mix ID #7



Photo 38: UN 2-in Gap Test Results for Large-scale Mix ID #8



7.1.4 Assessment of Test Results

Based on the test results, all samples PASSED the UN Series 2 (a) UN 2-inch Gap test (NOT sensitive to detonative shock).

7.2 UN Series 2 (b) Koenen test

7.2.1 Test Description

The Koenen test is used to determine the sensitiveness of solid and liquid substances to intense heat under high confinement. This test utilizes a steel tube to hold the sample. The tube is deep drawn from DC04, A620, or SPCEN sheet steel; 26.5 ± 1.5 grams mass, $75 \text{ }^\circ\text{C}$ 0.5mm length, $0.5 \pm 0.05\text{mm}$ wall, and 30 ± 3 MPa quasi-static bursting pressure. The sample is loaded into the tubes in three equal increments with each increment tamped with an 80 N force applied to the total cross-section of the tube until the tube is filled to 60mm. Liquids are loaded into the tube to a height of 60mm. The tube is assembled into a reusable closing device (threaded nut and collar) and an orifice plate installed on the open end of the tube. Varying the orifice plate over the top of the sample tube changes the degree of confinement of the sample.

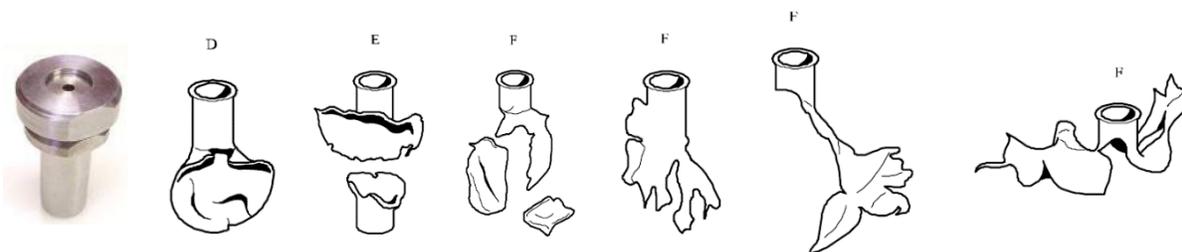
Photo 39: Koenen Tube, Orifice Plates, and Reusable Closing Device



The tube is placed in the heating and protective device, the test area vacated, burners remotely lit, providing a calibrated heating rate to the sample by propane burners located at four locations around the tube for at least 5 minutes or until the tube ruptures. NOTE: The heating rate is calibrated to $3.3 \pm 0.3^\circ\text{K}/\text{sec}$ using the time for 27 cm^3 of dibutyl phthalate to rise from 135°C to 285°C as measured by a 1mm thermocouple placed 43mm below the rim of the tube through a 1.5mm orifice plate (the rise time must be between 41.7 - 50.0 seconds).

After each trial the fragments of the tube, if any, are collected and weighed. The following effects are differentiated:

- "O": Tube unchanged;
- "A": Bottom of tube bulged out;
- "B": Bottom and wall of the tube bulged out;
- "C": Bottom of tube split;
- "D": Wall of tube split;
- "E": Tube split into two fragments;
- "F": Tube fragmented into three or more mainly large pieces which in some cases may be connected with each other by a narrow strip;
- "G": Tube fragmented into many mainly small pieces, closing device undamaged; and
- "H": Tube fragmented into many very small pieces, closing device bulged out or fragmented.



If a trial results in any of the effects "O" to "E", the result is regarded as "no explosion". If a trial gives the effect "F", "G" or "H", the result is evaluated as "explosion". The test is performed three times at the lowest orifice size that the result "no explosion" is observed. The limiting diameter (LD) is the largest orifice diameter at which the result "explosion" is obtained. The orifice sizes are reduced until an explosion effect occurs or the substance passes the test with the smallest orifice (1.0mm).

The result is considered positive for the UN Series 2 (b) test and the substance to show a violent effect on heating under confinement if the LD is 2.0mm or more. The result is considered negative for the UN Series 2 (b) test and the substance to show no violent effect on heating under confinement if the LD is less than 2.0mm.

7.2.2 Test Configuration

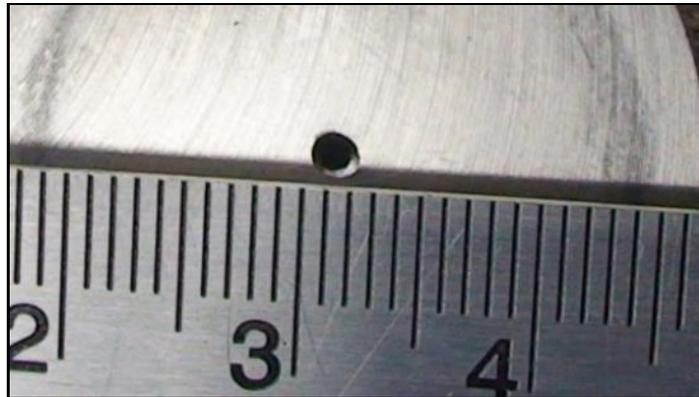
The quantity of sample in each tube, filled in three equal increments with 80 N tamping to a depth of 60mm, is listed in the following table.

Table 15: Sample Quantity Required to Fill Koenen Tubes to a Depth of 60mm

Item	Sample	Trial 1 Mass (g)	Trial 2 Mass (g)	Trial 3 Mass (g)
1	Large-scale Mix ID #1	36.1	36.4	34.1
2	Large-scale Mix ID #2	57.0	58.1	57.0
3	Large-scale Mix ID #3	66.4	65.2	61.9
4	Large-scale Mix ID #4	51.2	45.4	48.7
5	Large-scale Mix ID #5	61.9	62.3	65.6
6	Large-scale Mix ID #6	54.3	56.5	57.0
7	Large-scale Mix ID #7	49.0	51.4	53.4
8	Large-scale Mix ID #8	43.4	46.7	44.1

The orifice plates for UN Series 2 (b) had a 2.0mm diameter hole as shown in the following photo.

Photo 40: Orifice Plate with 2.0mm diameter hole



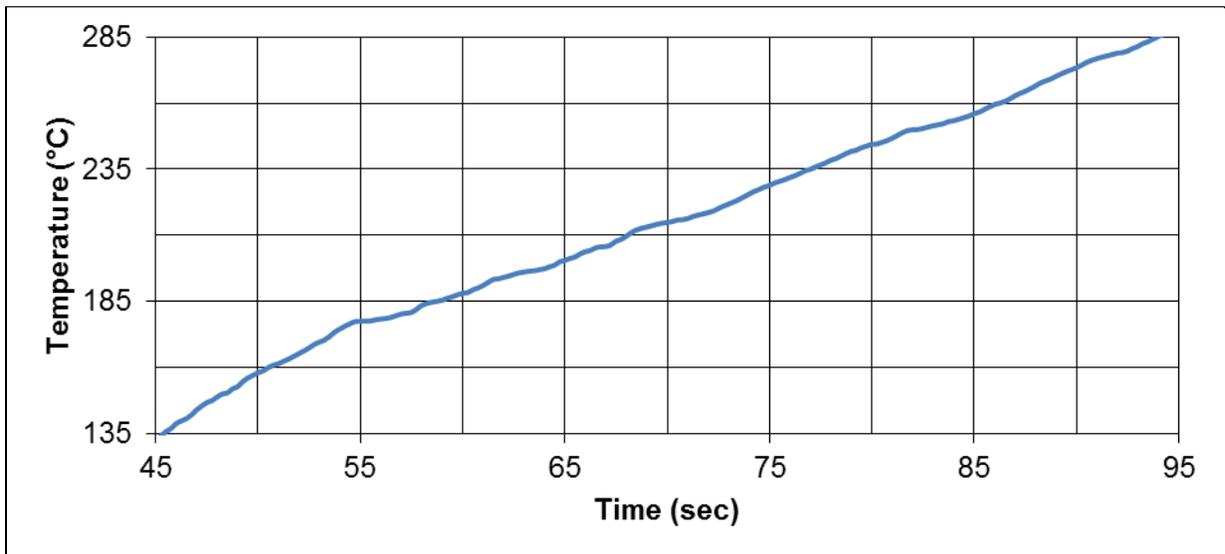
The test tube was centered in the heating and protective device as shown in the following photos.

Photo 41: Koenen Tube Suspended in Heating and Protective Device



For the calibration trial, the time for 27 cm³ of dibutyl phthalate to rise from 135°C to 285°C measured by a 1mm thermocouple placed 43mm below the rim of the tube through a 1.5mm orifice plate was 48.5 seconds (within calibration).

Figure 2: Calibration of Koenen Heating Rate with Dibutyl Phthalate



7.2.3 Test Results

The test results are summarized in the following table.

Table 16: Summary of UN Series 2 (b) Koenen Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "O", tube unchanged (05:00) All trials resulted in "no explosion"; LD less than 2.0mm.	Negative (NO violent effect on heating under confinement)
2	Large-scale Mix ID #2	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "O", tube unchanged (05:00) All trials resulted in "no explosion"; LD less than 2.0mm.	Negative (NO violent effect on heating under confinement)
3	Large-scale Mix ID #3	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "O", tube unchanged (05:00) All trials resulted in "no explosion"; LD less than 2.0mm.	Negative (NO violent effect on heating under confinement)
4	Large-scale Mix ID #4	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "O", tube unchanged (05:00) All trials resulted in "no explosion"; LD less than 2.0mm.	Negative (NO violent effect on heating under confinement)
5	Large-scale Mix ID #5	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "O", tube unchanged (05:00) All trials resulted in "no explosion"; LD less than 2.0mm.	Negative (NO violent effect on heating under confinement)
6	Large-scale Mix ID #6	2.0-mm orifice: <ul style="list-style-type: none"> - Trial 1: Effect type "O", tube unchanged (05:00) - Trial 2: Effect type "O", tube unchanged (05:00) - Trial 3: Effect type "F", tube fragmented into three or more mainly large pieces (04:40). One trial resulted in "explosion"; LD 2.0mm or more.	Positive (violent effect on heating under confinement)

Item	Sample	Conditions and Results	Assessment
7	Large-scale Mix ID #7	2.0-mm orifice: - Trial 1: Effect type "O", tube unchanged (05:00). NOTE: Later, effect type "F", tube fragmented into three or more mainly large pieces (05:20). - Trial 2: Effect type "O", tube unchanged (05:00). NOTE: Later, effect type "F", tube fragmented into three or more mainly large pieces (11:40). - Trial 3: Effect type "O", tube unchanged (05:00). NOTE: Effect type "F", tube fragmented into three or more mainly large pieces (09:37). One trial resulted in "explosion"; LD 2.0mm or more.	Negative (NO violent effect on heating under confinement) at 5 minutes; Positive (violent effect on heating under confinement) after 5 minutes
8	Large-scale Mix ID #8	2.0-mm orifice: - Trial 1: Effect type "O", tube unchanged (05:00). NOTE: Later, effect type "F", tube fragmented into three or more mainly large pieces (07:10). - Trial 2: Effect type "O", tube unchanged (05:00). NOTE: Later, effect type "F", tube fragmented into three or more mainly large pieces (09:40). - Trial 3: Effect type "F", tube fragmented into three or more mainly large pieces (01:15). One trial resulted in "explosion"; LD 2.0mm or more.	Positive (violent effect on heating under confinement)

The test results are shown in the following photos.

Photo 42: Koenen Test Results for Large-scale Mix ID #1



Photo 43: Koenen Test Results for Large-scale Mix ID #2



Photo 44: Koenen Test Results for Large-scale Mix ID #3



Photo 45: Koenen Test Results for Large-scale Mix ID #4



Photo 46: Koenen Test Results for Large-scale Mix ID #5



Photo 47: Koenen Test Results for Large-scale Mix ID #6



Photo 48: Koenen Test Results for Large-scale Mix ID #7



Photo 49: Koenen Test Results for Large-scale Mix ID #8



7.2.4 Assessment of Test Results

Based on the test results and UN assessment criteria, both the Large-scale Mix ID #6 and Large-scale Mix ID #8 samples exploded and FAILED the UN Series 2 (b) Koenen test (violent effect on heating under confinement); the Large-scale Mix ID #7 also exploded and would fail the test if the 5-minute maximum test time were extended.

NOTE: The auto-ignition temperature of traditional explosives is typically around 200 - 300 °C; the average auto-ignition temperature of the twenty-three small-scale thermite test samples was 809 °C with a standard deviation of 240 °C. Therefore, the 5-minute maximum test time of the UN Series 1 or 2 (b) Koenen test may be insufficient to determine whether a specific thermite exhibits a violent effect on heating under confinement.

7.3 UN Series 2 (c) (ii) Internal ignition test

7.3.1 Test Description

This test is used to determine the tendency of a substance to undergo transition from deflagration to detonation. The sample is loaded into a 45.7-cm long 3-inch schedule 80 carbon (A53 Grade B) steel pipe with 3000-pound forged steel end caps. An igniter consisting of black powder (100% passed through a No. 20 sieve, 100% retained by a No. 50 sieve) is located at the center of the sample vessel; the insulated lead wires are fed through small holes in the wall of the pipe and are sealed with epoxy resin. The tube is placed in a vertical position and the igniter fired.

The test result is considered positive if either the pipe or at least one of the end caps is fragmented into at least two distinct pieces. The test result is considered negative if the pipe is merely split or laid open, or if the end caps are blown off. Three trials are performed unless a positive result occurs earlier.

7.3.2 Test Configuration

Each thermite was ignited using a 1.5-gram pyrogen igniter. If the igniter alone was insufficient to ignite the thermite, a bag igniter was added that was comprised of a pyrogen igniter and 10 grams of Mix ID #4 thermite (which is readily ignited by the pyrogen match). The lead wires of the igniters were sealed with epoxy resin and allowed to cure before filling the pipe with sample. Each sample was poured into the steel tubes to a height within one inch of the top of the pipe (to prevent overfilling and contamination of the pipe threads). A typical test setup is shown in the photo below.

Photo 50: Internal Ignition Test Setup (typical)



7.3.3 Test Results

The test results are summarized in the following table. For each trial, the thermite ignited and vented out the hole for the ignition wire, enlarging the hole.

Table 17: Summary of UN Series 2 (c) (ii) Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Pyrogen igniter: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps. NOTE: Hole expanded to 2-cm wide.	Negative (NO transition from deflagration to detonation)
2	Large-scale Mix ID #2	Pyrogen igniter with 10 grams of Mix ID #4: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps. NOTE: Hole expanded to 6-cm wide.	Negative (NO transition from deflagration to detonation)

Item	Sample	Conditions and Results	Assessment
3	Large-scale Mix ID #3	Pyrogen igniter with 10 grams of Mix ID #4: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps. NOTE: Hole expanded to 4-cm wide.	Negative (NO transition from deflagration to detonation)
4	Large-scale Mix ID #4	Pyrogen igniter: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps. NOTE: Hole expanded to 4-cm wide.	Negative (NO transition from deflagration to detonation)
5	Large-scale Mix ID #5	Pyrogen igniter with 10 grams of Mix ID #4: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps.	Negative (NO transition from deflagration to detonation)
6	Large-scale Mix ID #6	Pyrogen igniter: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps.	Negative (NO transition from deflagration to detonation)
7	Large-scale Mix ID #7	Pyrogen igniter: - Trial 1: Full consumption of thermite; no damage to pipe or end caps. - Trial 2: Full consumption of thermite; no damage to pipe or end caps. - Trial 3: Full consumption of thermite; no damage to pipe or end caps.	Negative (NO transition from deflagration to detonation)

Item	Sample	Conditions and Results	Assessment
8	Large-scale Mix ID #8	Pyrogen igniter: - Trial 1: Full consumption of thermitite; no damage to pipe or end caps. - Trial 2: Full consumption of thermitite; no damage to pipe or end caps. - Trial 3: Full consumption of thermitite; no damage to pipe or end caps. NOTE: Hole expanded to 4-cm wide.	Negative (NO transition from deflagration to detonation)

The test results are shown in the following photos.

Photo 51: Internal Ignition Test Results for Large-scale Mix ID #1



Photo 52: Internal Ignition Test Results for Large-scale Mix ID #2 - typical



Photo 53: Internal Ignition Test Results for Large-scale Mix ID #3 - typical



Photo 54: Internal Ignition Test Results for Large-scale Mix ID #4



Photo 55: Internal Ignition Test Results for Large-scale Mix ID #5



Photo 56: Internal Ignition Test Results for Large-scale Mix ID #6



Photo 57: Internal Ignition Test Results for Large-scale Mix ID #7



Photo 58: Internal Ignition Test Results for Large-scale Mix ID #8



7.3.4 Assessment of Test Results

Based on the test results, all samples PASSED the UN Series 2 (c) (ii) Internal ignition test (NO transition from deflagration to detonation).

7.4 UN Series 3 (a) (i) Bureau of Explosives (BOE) impact sensitivity test

7.4.1 Test Description

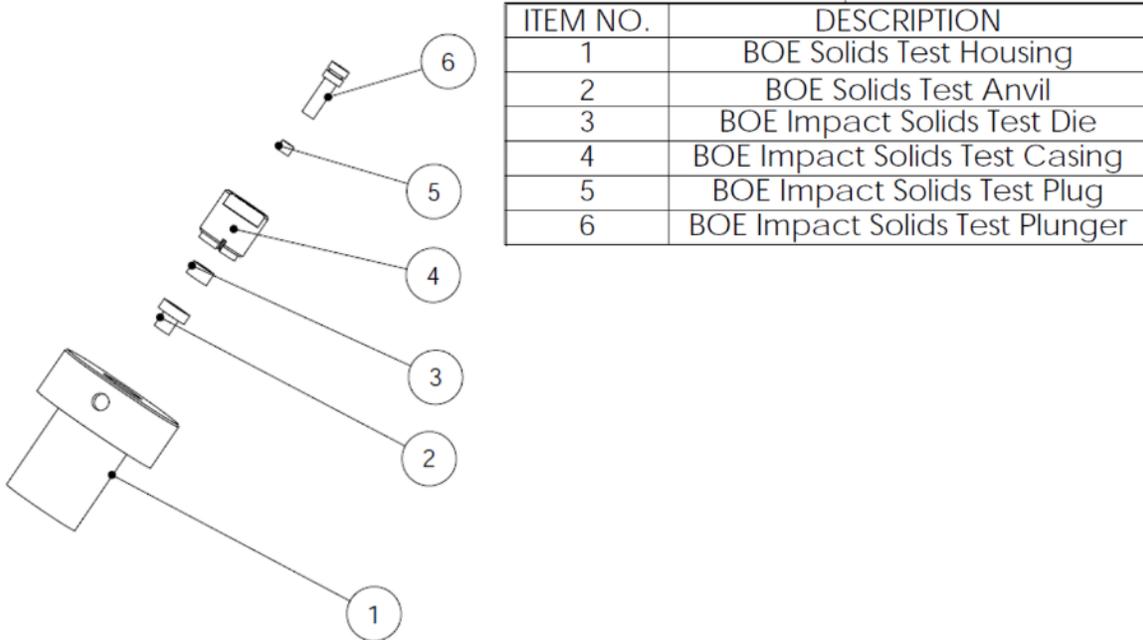
The BOE Impact Test is used to measure the sensitiveness of a substance to drop-weight impact and to determine if the substance is too dangerous to transport in the form tested.

For solids, a 10-mg sample is loaded onto a die. The anvil and die are placed in the sample housing and the casing screwed down over them. The plug and plunger are then inserted on top of the sample. The 3.63-kg drop weight is raised to a drop height of 10.0 cm and released. Observations are made on whether an "explosion" occurs as evidenced by flame or audible report (smoke excluded). Up to ten trials are performed for each sample. The material is considered too dangerous for transport in the form in which it was tested if "explosion" occurs in at least five out of the ten trials.

Photo 59: BOE Impact Apparatus



Figure 3: Components of the BOE Impact Apparatus



7.4.2 Test Results

The test results are summarized in the following table.

Table 18: Summary of UN Series 3 (a) (i) BOE Impact Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	No explosion in 6 trials.	Pass (not too dangerous in tested form)
2	Large-scale Mix ID #2	No explosion in 6 trials.	Pass (not too dangerous in tested form)
3	Large-scale Mix ID #3	No explosion in 6 trials.	Pass (not too dangerous in tested form)
4	Large-scale Mix ID #4	No explosion in 6 trials.	Pass (not too dangerous in tested form)
5	Large-scale Mix ID #5	No explosion in 6 trials.	Pass (not too dangerous in tested form)
6	Large-scale Mix ID #6	No explosion in 6 trials.	Pass (not too dangerous in tested form)
7	Large-scale Mix ID #7	No explosion in 6 trials.	Pass (not too dangerous in tested form)
8	Large-scale Mix ID #8	No explosion in 6 trials.	Pass (not too dangerous in tested form)

7.4.3 Assessment of Test Results

Based on the test results, all samples PASSED the UN Series 3 (a) (i) BOE impact test (not too dangerous in tested form).

7.5 UN Series 3 (b) (i) BAM friction sensitivity test

7.5.1 Test Description

This test is used to measure the sensitiveness of the substance to frictional stimuli and to determine if the substance is too dangerous to transport in the form tested.

A porcelain plate is fixed on the carriage of the friction apparatus so that its sponge-mark grooves run transversely to the direction of movement. Approximately 10 mm³ of the sample is poured into a pile on the porcelain plate. The loading arm is lowered, resting a stationary porcelain peg onto the sample; care is taken so that enough of the substance will come under the peg when the porcelain plate moves forward. The load applied on the pin is provided by varying the actual applied mass and the distance along the loading arm where the mass is placed. The plate is motor driven through a one cycle pass beneath the pin, thereby subjecting the sample to friction created by the rubbing of the pin against the plate.

Observations are made on whether an "explosion" occurs as evidenced by an audible report, crackling, sparking, or flame. Up to six trials are performed for each sample. The material is considered too dangerous for transport in the form in which it was tested if the limiting load (i.e. the lowest friction load at which one "explosion" occurs in six trials) is less than 80 N.

7.5.2 Test Configuration

Each thermite mixture was tested as prepared or received; samples were not sieved through a 0.5-mm mesh sieve.

7.5.3 Test Results

The test results are summarized in the following table.

Table 19: Summary of UN Series 3 (b) (i) BAM Friction Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
2	Large-scale Mix ID #2	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
3	Large-scale Mix ID #3	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
4	Large-scale Mix ID #4	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
5	Large-scale Mix ID #5	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
6	Large-scale Mix ID #6	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
7	Large-scale Mix ID #7	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)
8	Large-scale Mix ID #8	No explosion in 6 trials at a friction load of 80 N (limiting load >80 N).	Pass (not too dangerous in tested form)

7.5.4 Assessment of Test Results

Based on the test results, all samples PASSED the UN Series 3 (b) (i) BAM friction test (not too dangerous in tested form).

7.6 UN Series 3 (c) (i) Thermal stability test - Uninstrumented

7.6.1 Test Description

This test is used to measure the stability of the substance when subjected to elevated thermal conditions to determine if the substance is too dangerous to transport.

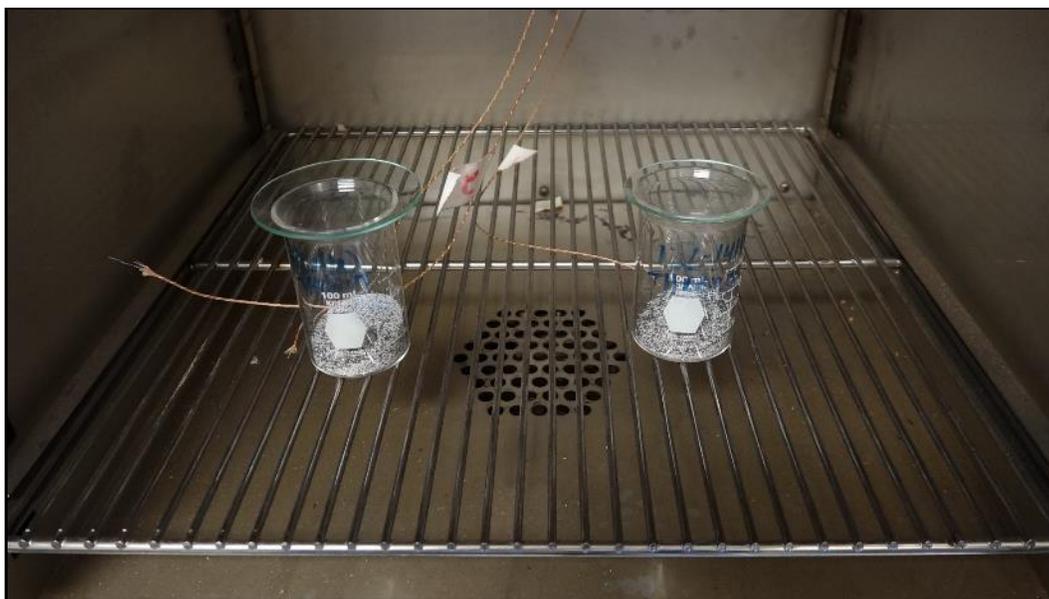
A 50-gram sample is weighed into a beaker, covered with a watch glass and placed in a constant temperature, explosion-proof oven at 75 °C for 48 hours. The temperature is continuously monitored and recorded. At the completion of the test, the sample is examined for evidence of ignition, explosion, or decomposition/self-heating (discoloration, weight loss, dimensional change, etc.).

The result from the uninstrumented test is considered positive if ignition or explosion occurs. Further testing is required if there is evidence that some self-heating has occurred (e.g. fuming or decomposition); otherwise the substance is regarded as thermally stable.

7.6.2 Test Configuration

The following photo shows a representative test setup with 50 grams of sample in a glass beaker, covered with a watch glass, and placed into the heating oven.

Photo 60: Thermal Stability Test Setup (typical)



7.6.3 Test Results

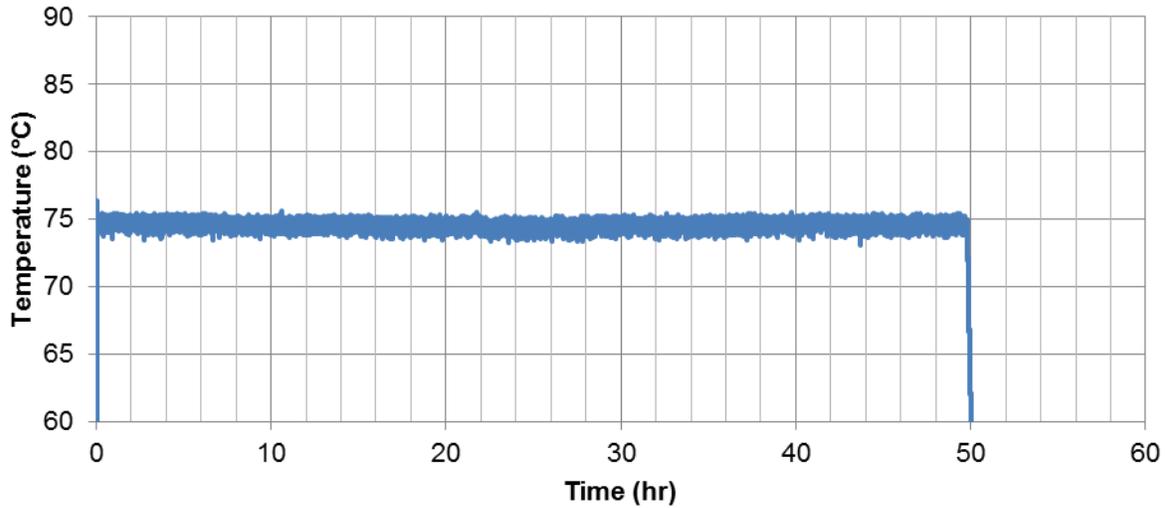
The test results are summarized in the following table.

Table 20: Summary of UN Series 3 (c) (i) Thermal Stability Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.2-grams mass loss.	Pass (not forbidden)
2	Large-scale Mix ID #2	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.1-grams mass loss.	Pass (not forbidden)
3	Large-scale Mix ID #3	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.03-grams mass loss.	Pass (not forbidden)
4	Large-scale Mix ID #4	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); 0.05-grams mass loss.	Pass (not forbidden)
5	Large-scale Mix ID #5	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
6	Large-scale Mix ID #6	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
7	Large-scale Mix ID #7	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)
8	Large-scale Mix ID #8	Ignition or explosion did NOT occur, and no evidence of thermal instability (fuming or decomposition); no mass loss.	Pass (not forbidden)

The following figure is a typical record of the oven temperature for the test.

Figure 4: Oven Temperature Record for the Thermal Stability Test (typical)



7.6.4 Assessment of Test Results

Based on the test results, all samples PASSED the uninstrumented UN Series 3 (c) (i) Thermal stability test (not forbidden).

7.7 UN Series 3 (d) Small-scale burning test

7.7.1 Test Description

This test is used to determine the response of an unconfined substance to fire. A sample in a thin-walled plastic beaker is placed on a bed of sawdust soaked with kerosene or diesel fuel approximately 30-cm square and 1.3-cm thick. The sawdust is ignited with an electric match or similar device and the burn time is measured. The test is performed two times unless an explosion or detonation occurs. The test is normally performed twice with 10 grams of sample and twice with 100 grams.

7.7.2 Test Configuration

The test was performed twice with 100 grams of sample in paper cups. The following photo shows a representative test setup.

Photo 61: Small-scale Burning Test Setup (typical)



7.7.3 Test Results

The test results are summarized in the following table.

Table 21: Summary of UN Series 3 (d) Small-scale Burning Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	The two 100-gram samples ignited and burned for 0.6 - 0.8 seconds each after 1 - 2 minutes; explosion of the test samples did NOT occur.	Pass (not too dangerous in tested form)
2	Large-scale Mix ID #2	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
3	Large-scale Mix ID #3	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
4	Large-scale Mix ID #4	The two 100-gram samples ignited and burned for approximately 3 - 4 seconds after 7 - 11 minutes; explosion of the test samples did NOT occur.	Pass (not too dangerous in tested form)
5	Large-scale Mix ID #5	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
6	Large-scale Mix ID #6	The two 100-gram samples did NOT ignite.	Pass (not too dangerous in tested form)
7	Large-scale Mix ID #7	The two 100-gram samples exploded upon ignition after 1.0 - 1.5 minutes.	FAIL (12-meter drop test required to determine whether safe for transport; waived based on impact and friction sensitivity test results)

Item	Sample	Conditions and Results	Assessment
8	Large-scale Mix ID #8	The two 100-gram samples exploded upon ignition after 37 - 44 seconds with a loud report, clearing the witness plate of sawdust.	FAIL (12-meter drop test required to determine whether safe for transport; waived based on impact and friction sensitivity test results)

The test results are shown in the following photos.

Photo 62: Small-scale Burning Test Results for Large-scale Mix ID #1



Photo 63: Small-scale Burning Test Results for Large-scale Mix ID #2

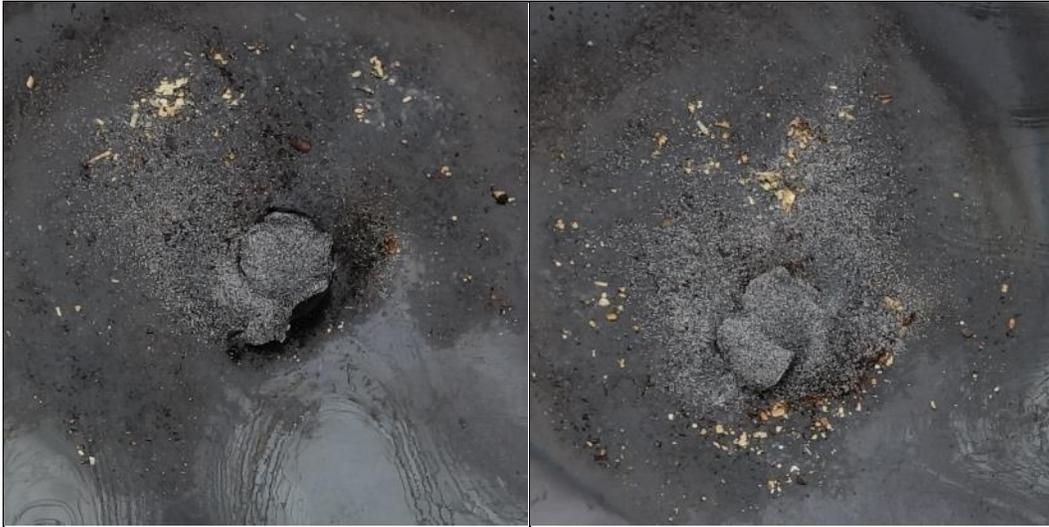


Photo 64: Small-scale Burning Test Results for Large-scale Mix ID #3



Photo 65: Small-scale Burning Test Results for Large-scale Mix ID #4

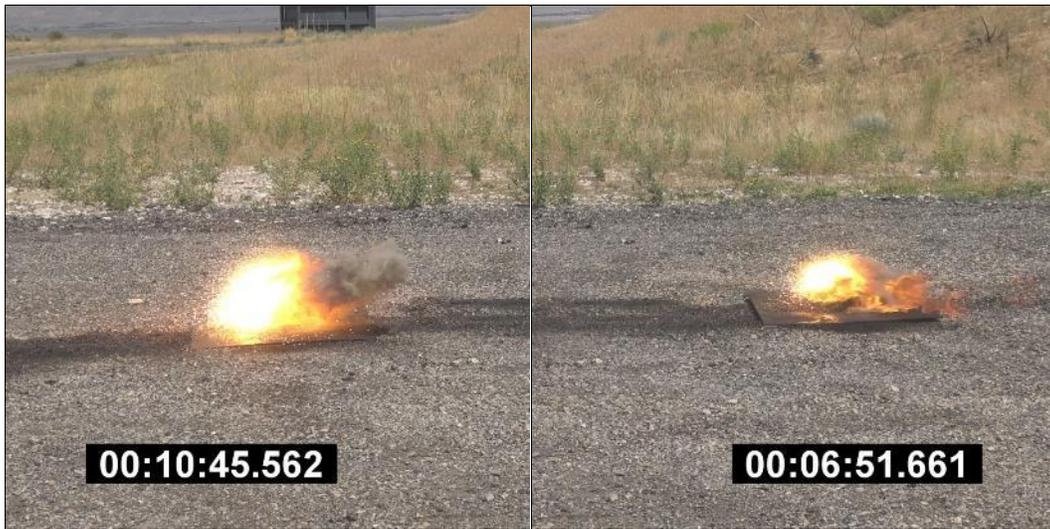


Photo 66: Small-scale Burning Test Results for Large-scale Mix ID #5



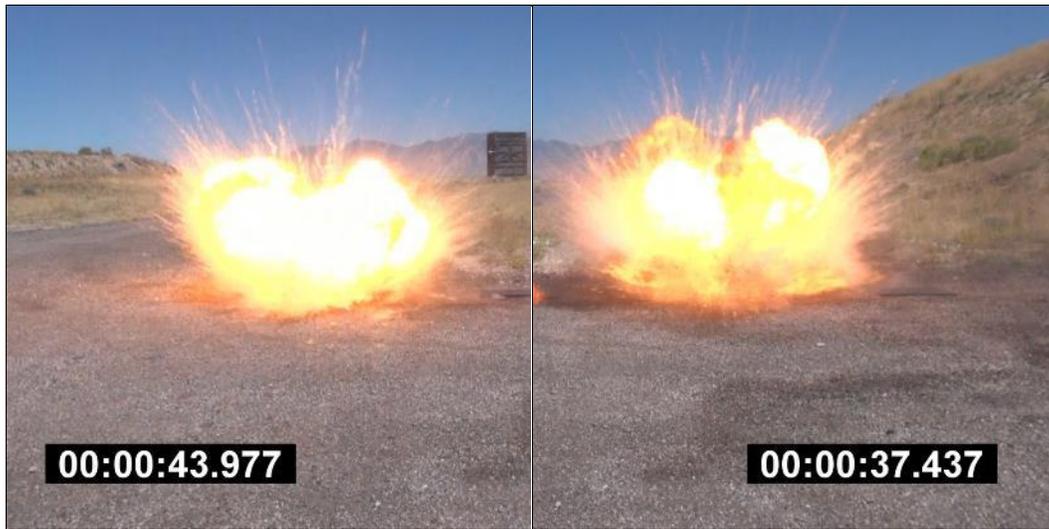
Photo 67: Small-scale Burning Test Results for Large-scale Mix ID #6



Photo 68: Small-scale Burning Test Results for Large-scale Mix ID #7



Photo 69: Small-scale Burning Test Results for Large-scale Mix ID #8



7.7.4 Assessment of Test Results

Based on the test criteria and the test results, both the Large-scale Mix ID #7 and Large-scale Mix ID #8 samples failed the small-scale burning test criteria requiring a 12-meter drop test to determine whether they are safe for transport; the 12-meter drop test is waived based on the passing impact and friction sensitivity test results. All other samples (Large-scale Mix ID's #1, #2, #3, #4, #5 and #6) passed the small-scale burning test criteria.

7.8 Unconfined UN Series 6 (a) Single Package Test on Suspended Witness Plate

7.8.1 Test Description

This test demonstrates the hazardous effects apparent outside of a packaged substance when it is centrally ignited. An igniter is buried in the center of the packaged substance. The package is centered on a suspended witness plate. The igniter is ignited and observations made on the following: evidence of thermal effects, projection effects, detonation, deflagration or explosion of the total contents of the package. Detonation or explosion of the total contents of the package indicates a candidate for Division 1.1. One trial is performed for each configuration (screening test).

7.8.2 Test Configuration

One outer package of each product was placed on a witness plate. Each package contained 9.1 kg of thermite in a plastic bag within a fiberboard box, as shown in the following photo.

Photo 70: Unconfined Single Package Test Setup for Large-scale Mix ID #4 - typical



Each thermite was ignited using a 1.5-gram pyrogen igniter. If the igniter alone was insufficient to ignite the thermite, a bag igniter was added that was comprised of a pyrogen igniter and 10 grams of Mix ID #4 thermite (which is readily ignited by the pyrogen match). Two witness plate types were tested: 2-mm thick 1100-0 aluminum (standard UN Series 6 (c) External bonfire witness screen) and 3-mm thick mild steel (standard UN Series 6 (a) Single package witness plate). A typical test setup is shown in the photo below.

Photo 71: Suspended, Unconfined Single Package Test Setup - typical



7.8.3 Test Results

The test results for the unconfined single package test on suspended 2-mm thick 1100-0 aluminum witness plate (one trial) are summarized in the following table.

Table 22: Summary of Unconfined UN Series 6 (a) Single Package Test Results on a Suspended 2-mm thick 1100-0 Aluminum Witness Plate

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through a large hole through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
2	Large-scale Mix ID #2	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
3	Large-scale Mix ID #3	Package containing 9.1 kg of thermite ignited and burned; several holes in the aluminum witness plate where thermite melted through.	High threat to 2-mm thick 1100-0 aluminum
4	Large-scale Mix ID #4	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
5	Large-scale Mix ID #5	Package containing 9.1 kg of thermite ignited and burned; several holes in the aluminum witness plate where thermite melted through.	High threat to 2-mm thick 1100-0 aluminum
6	Large-scale Mix ID #6	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the aluminum witness plate.	High threat to 2-mm thick 1100-0 aluminum
7	Large-scale Mix ID #7	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center; severe deformation of suspended aluminum witness plate.	Hazards consistent with Division 1.1
8	Large-scale Mix ID #8	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center; severe deformation of suspended aluminum witness plate.	Hazards consistent with Division 1.1

The test results for the unconfined single package test on suspended 3-mm thick mild steel witness plate (one trial) are summarized in the following table.

Table 23: Summary of Unconfined UN Series 6 (a) Single Package Test Results on a Suspended 3-mm thick Mild Steel Witness Plate

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Package containing 9.1 kg of thermite ignited and burned in 1 - 2 seconds; no significant damage to the steel witness plate.	Minimal threat to 3-mm thick mild steel
2	Large-scale Mix ID #2	Package containing 9.1 kg of thermite ignited and burned; several holes in the steel witness plate where thermite melted through.	High threat to 3-mm thick mild steel
3	Large-scale Mix ID #3	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the steel witness plate.	High threat to 3-mm thick mild steel
4	Large-scale Mix ID #4	Package containing 9.1 kg of thermite ignited and burned; no significant damage to the steel witness plate but several melt points on underside of the plate.	Moderate threat to 3-mm thick mild steel
5	Large-scale Mix ID #5	Package containing 9.1 kg of thermite ignited and burned; no significant damage to the steel witness plate but one very small hole in plate.	Minimal threat to 3-mm thick mild steel
6	Large-scale Mix ID #6	Package containing 9.1 kg of thermite ignited and burned; thermite readily melted through the steel witness plate.	High threat to 3-mm thick mild steel
7	Large-scale Mix ID #7	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center slightly bowing the witness plate.	Hazards consistent with Division 1.1
8	Large-scale Mix ID #8	Package containing 9.1 kg of thermite exploded upon ignition of pyrogen match in the center bowing the witness plate.	Hazards consistent with Division 1.1

The test results are shown in the following photos.

Photo 72: Suspended, Unconfined Package Test Results for Large-scale Mix ID #1



Photo 73: Suspended, Unconfined Package Test Results for Large-scale Mix ID #2



Photo 74: Suspended, Unconfined Package Test Results for Large-scale Mix ID #3



Photo 75: Suspended, Unconfined Package Test Results for Large-scale Mix ID #4



Photo 76: Suspended, Unconfined Package Test Results for Large-scale Mix ID #5



Photo 77: Suspended, Unconfined Package Test Results for Large-scale Mix ID #6



Photo 78: Suspended, Unconfined Package Test Results for Large-scale Mix ID #7



Photo 79: Suspended, Unconfined Package Test Results for Large-scale Mix ID #8



7.8.4 Assessment of Test Results

Based on the test results, both the Large-scale Mix ID #7 and Large-scale Mix ID #8 samples explode upon ignition with hazards consistent with that of Division 1.1. Upon ignition, all

samples posed a high threat to compromise 2mm-thick or less aluminum plate/sheet supporting the package; the Large-scale Mix ID #2, Large-scale Mix ID #3, and Large-scale Mix ID #6 samples potentially pose a high threat able to compromise 3mm-thick or less mild steel plate/sheet supporting the package.

7.9 Sub-scale UN Series 6 (c) External fire (bonfire) test

7.9.1 *Test Description*

This test is performed on packages of explosives to determine the hazard when involved in a fire. Normally a stack of packages of an explosive material (0.15 cubic meters volume as packaged for transport) is placed on a non-combustible surface (steel grate) above a fuel source. Three 200-cm × 200-cm × 0.2-cm aluminum witness screens are placed 4 meters from the edge of the stack. The fire is ignited simultaneously on at least two sides and the material is observed for a) evidence of detonation, deflagration, or explosion of the total contents; b) potentially hazardous projections; and c) thermal effects (i.e. rate of burn, size of any fireball, etc.). The test is recorded using regular video with audio from two angles and visual distance marking devices.

7.9.2 *Test Configuration*

One outer package of each product was placed on the steel grate with four outer packages per bonfire (one package in each quadrant of the steel grate). Each package contained 9.1 kg of thermite in a plastic bag within a fiberboard box, as shown in the following photo. Several wood pallets were used as the fuel source and liquid fuel as an accelerant for ignition of the wood.

The environmental conditions at the start of the External bonfire tests were as follows:

- Bonfire #1 for Mix ID's #2, #3, #4, and #5: 3.6 m/s wind, 33°C, 19% relative humidity.
- Bonfire #2 for Mix ID #8: 3.6 m/s wind, 32°C, 20% relative humidity.
- Bonfire #3 for Mix ID #7: 2.2 m/s wind, 12°C, 39% relative humidity.
- Bonfire #4 for Mix ID's #1 and #6: 1.8 m/s wind, 12°C, 39% relative humidity.

Photo 80: Packaging Large-scale Mix ID #4 for Sub-scale External Fire Test (typical)



Photo 81: External Bonfire #1 Test Setup for Mix ID's #2, #3, #4, and #5

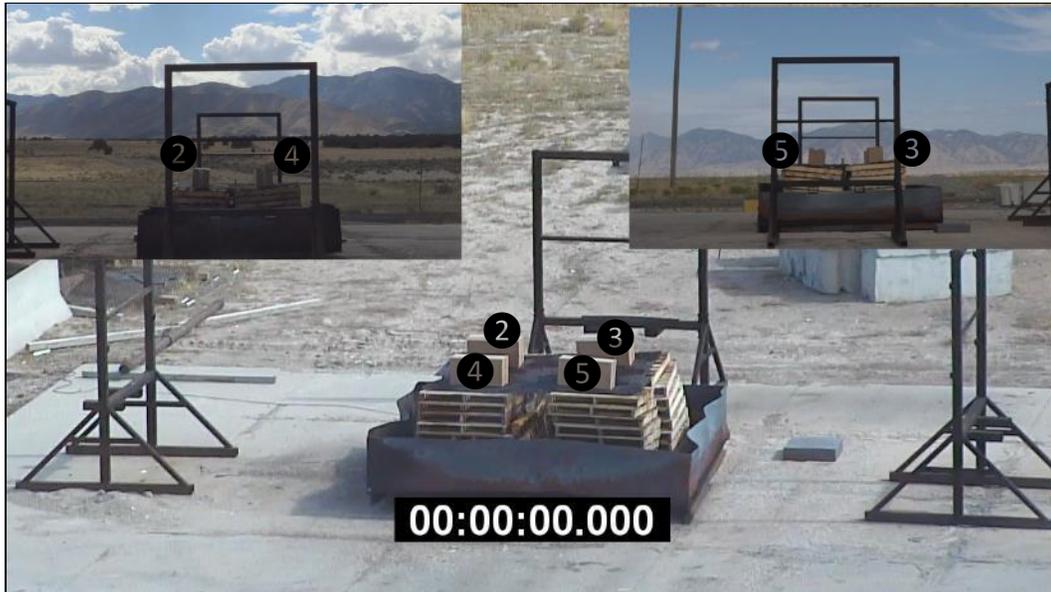


Photo 82: External Bonfire #2 Test Setup for Mix ID #8



Photo 83: External Bonfire #3 Test Setup for Mix ID #7



Photo 84: External Bonfire #4 Test Setup for Mix ID's #1 and #6



7.9.3 Test Results

The test results are summarized in the following table.

Table 24: Summary of Sub-scale UN Series 6 (c) External Bonfire Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Package containing 9.1 kg of thermitite ignited and rapidly burned over approximately 1 second, producing a fireball with a radius of 3 - 4 meters; no mass explosion.	Reaction rate and fireball more consistent with the hazards of Division 1.3
2	Large-scale Mix ID #2	Package containing 9.1 kg of thermitite ignited and briefly produced local flame enhancement within the flames of the fire.	Sub-scale quantities appear to present minor hazards
3	Large-scale Mix ID #3	Package containing 9.1 kg of thermitite ignited and briefly produced local flame enhancement within the flames of the fire; package was fully engulfed in the flames of the fire, obscuring the package and reaction.	Sub-scale quantities appear to present minor hazards
4	Large-scale Mix ID #4	Package containing 9.1 kg of thermitite ignited and rapidly burned over approximately 3 seconds, producing a fireball with a radius of 2 meters; no mass explosion.	Reaction rate and fireball more consistent with the hazards of Division 1.3 or 1.4
5	Large-scale Mix ID #5	Package containing 9.1 kg of thermitite ignited and briefly produced local flame enhancement within the flames of the fire.	Sub-scale quantities appear to present minor hazards
6	Large-scale Mix ID #6	Package containing 9.1 kg of thermitite ignited and burned over approximately 17 seconds, producing local flame enhancement within the flames of the fire.	Reaction rate and fireball more consistent with the hazards of Division 1.4S

Item	Sample	Conditions and Results	Assessment
7	Large-scale Mix ID #7	Package containing 9.1 kg of thermitite exploded upon ignition with nearly instantaneous consumption of the package contents.	Hazards consistent with Division 1.1
8	Large-scale Mix ID #8	Package containing 9.1 kg of thermitite exploded upon ignition with nearly instantaneous consumption of the package contents.	Hazards consistent with Division 1.1

The test results are shown in the following photos.

Photo 85: External Bonfire #1 Test Progression - Typical Flames

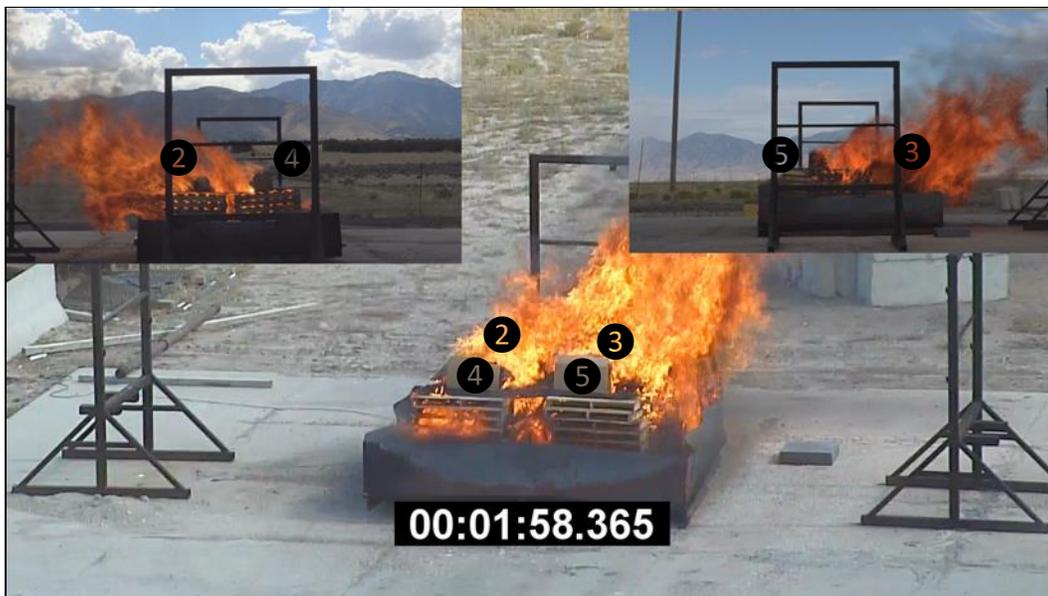


Photo 86: External Bonfire #1 Test Progression - Burning of Large-scale Mix ID #4

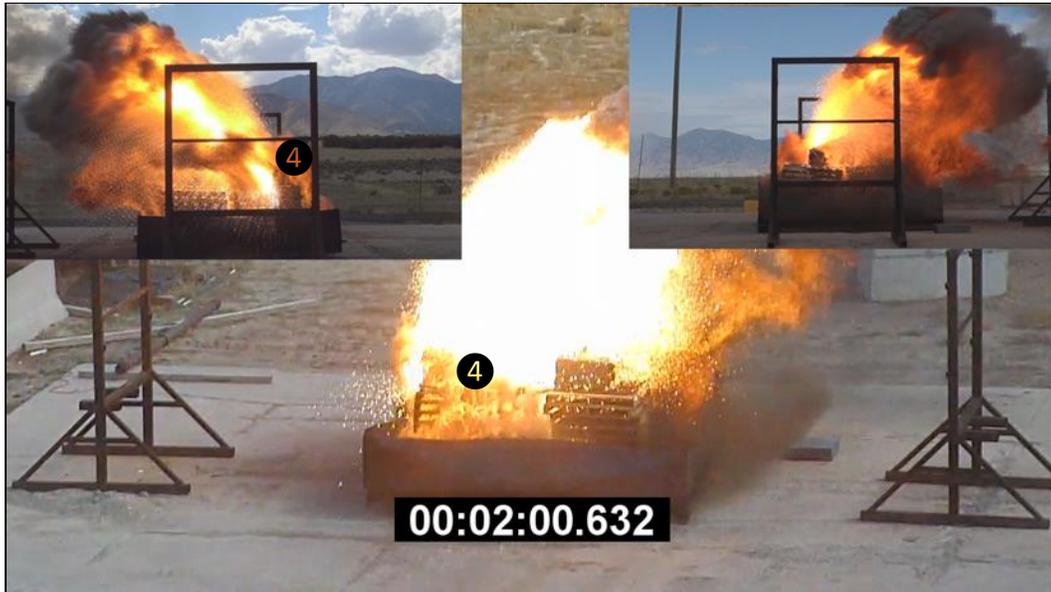


Photo 87: External Bonfire #1 Test Progression - Burning of Large-scale Mix ID #5

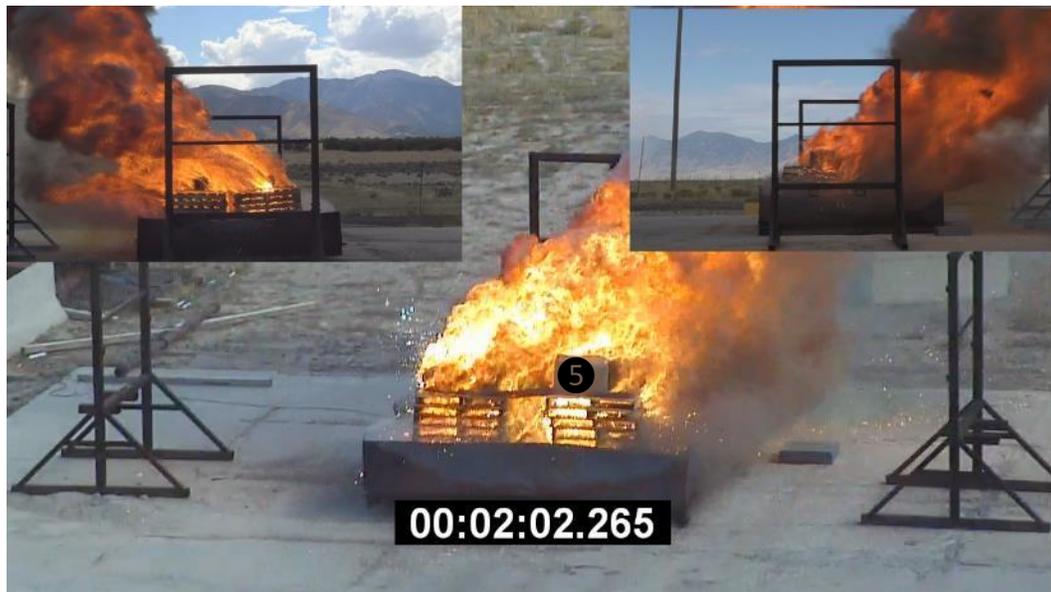


Photo 88: External Bonfire #1 Test Progression - Burning of Large-scale Mix ID #2

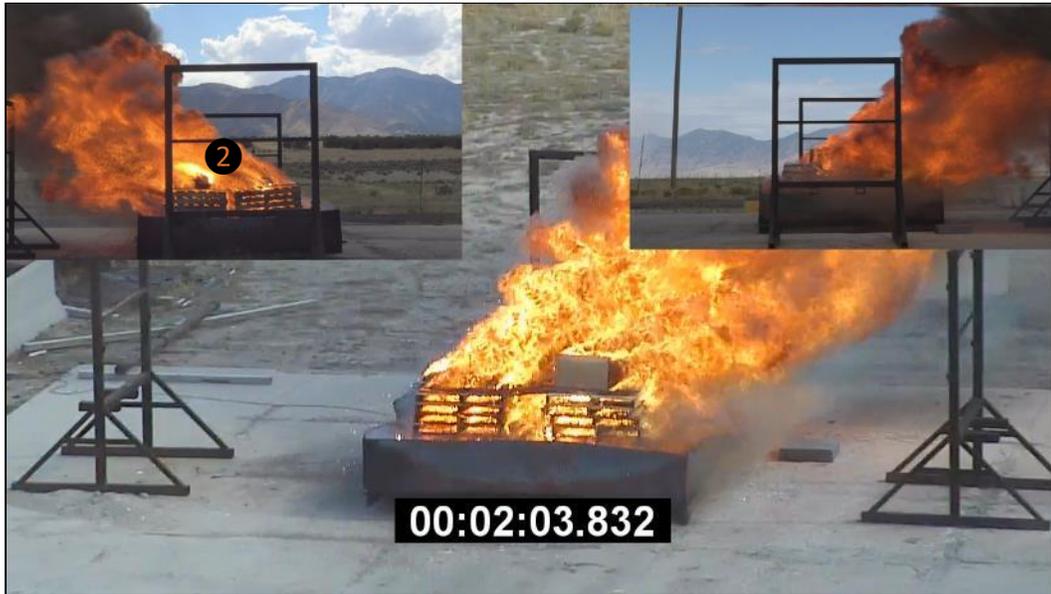


Photo 89: External Bonfire #1 Test Progression - Burning of Large-scale Mix ID #3

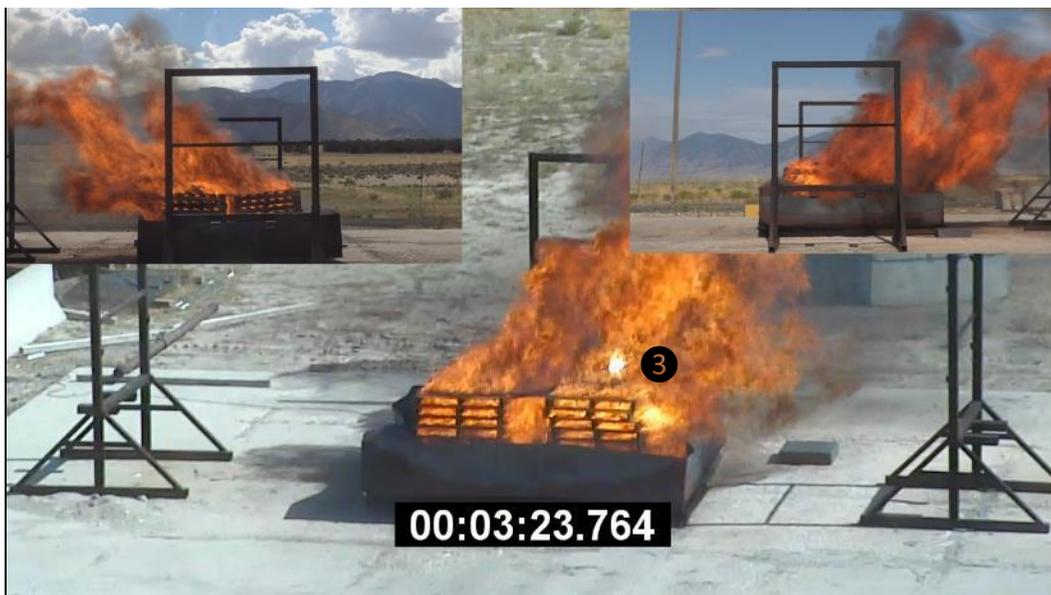


Photo 90: External Bonfire #2 Test Progression - Explosion of Large-scale Mix ID #8



Photo 91: External Bonfire #4 Test Progression - Explosion of Large-scale Mix ID #7

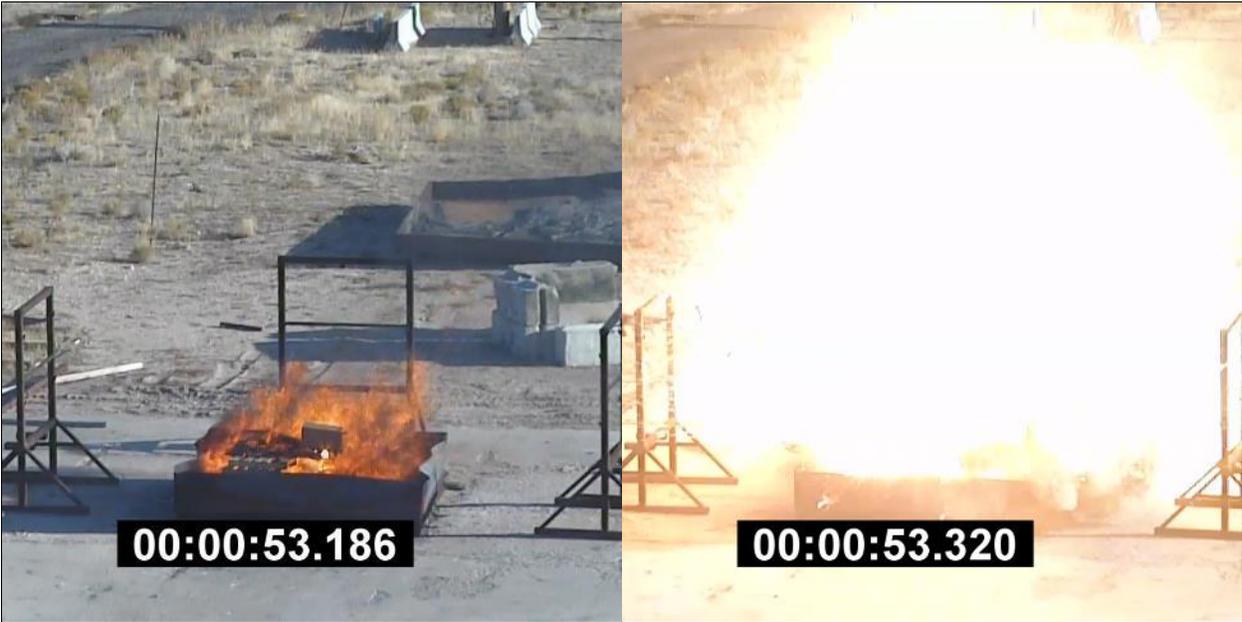
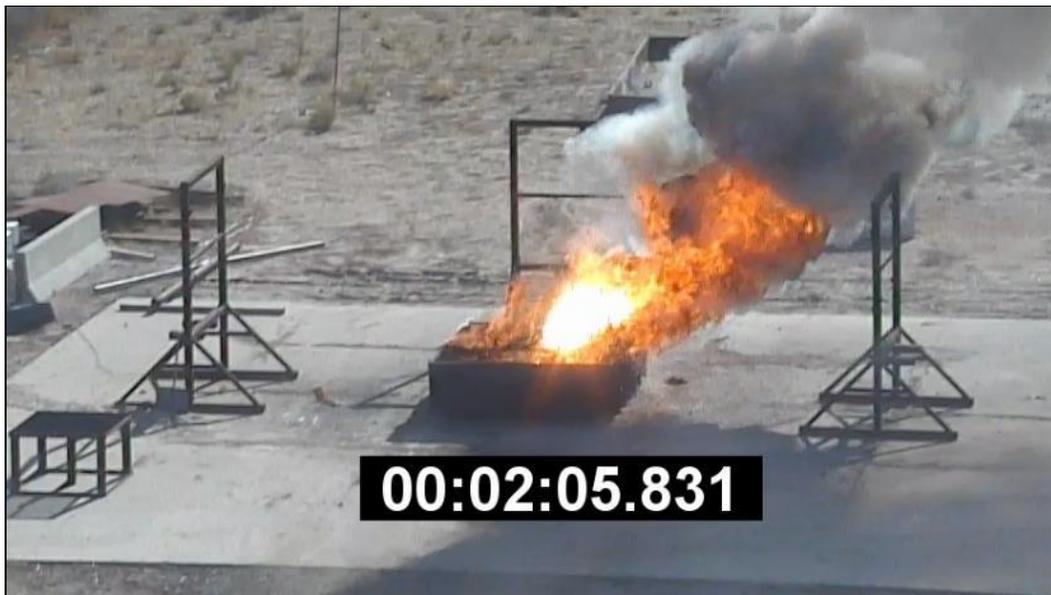


Photo 92: External Bonfire #4 Test Progression - Burning of Large-scale Mix ID #1



Photo 93: External Bonfire #4 Test Progression - Burning of Large-scale Mix ID #6



7.9.4 Assessment of Test Results

Based on the test results, the Large-scale Mix ID #7 and Large-scale Mix ID #8 samples have hazards consistent with that of explosives with a Division 1.1 mass explosion hazard. The Large-scale Mix ID #1 and Large-scale Mix ID #4 samples had reaction rates and fireball sizes more consistent with the hazards of Division 1.3 or 1.4.

7.10 Sub-scale UN Series 7 (e) EIS External fire test

7.10.1 Test Description

This test is used to determine the reaction of a substance to external fire when it is confined. The substance is loaded into seamless steel pipe having an inside diameter of 40.5 - 49.5 mm, a wall thickness of 3.6 - 4.4 mm, and a length of 200 mm. The pipes are closed with steel or cast iron end caps, at least as strong as the tube, torqued to 204 N·m.

The sub-scale test is performed with three confined samples of each product, each secured away from adjacent samples, to a non-combustible surface (steel grate) centered within a fuel basin. The fuel basin is at least 3 feet (1 meter) larger than the stack in every direction with a wall height sufficient to contain a quantity of fuel sufficient to maintain a fully developed fire for a 30-minute fire and not block the flight of projections.

The fire is ignited on at least two sides and the material is observed for a) evidence of detonation, deflagration, or explosion of the total contents; and b) potentially hazardous fragments. The test is recorded using regular video with audio from two angles and visual distance marking devices. A substance shows explosive properties under confinement during fast cook-off which detonates or reacts violently with fragments thrown more than 15 meters.

7.10.2 Test Configuration

The pipe nipples utilized were 1-1/2 inch standard A106B seamless pipe (1.90 inches outer diameter, 0.145 inch wall, 1.61 inch inner diameter) cut to a length of 7-7/8 inches. The end caps were 1-1/2 inch standard threaded caps. Three pipes were loaded for each of the eight products (twenty-four capped pipes total). The capped pipes were secured to the grating immediately before each end cap using baling wire. The following figure and photo illustrate the configuration of the confined samples.

Figure 5: Confined Samples for the EIS External Fire Test

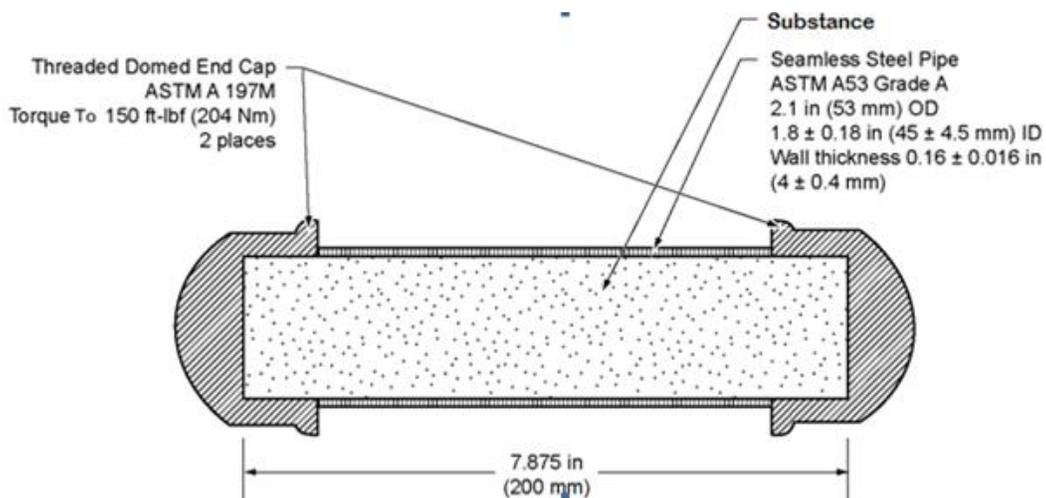


Photo 94: EIS External Fire Test Setup

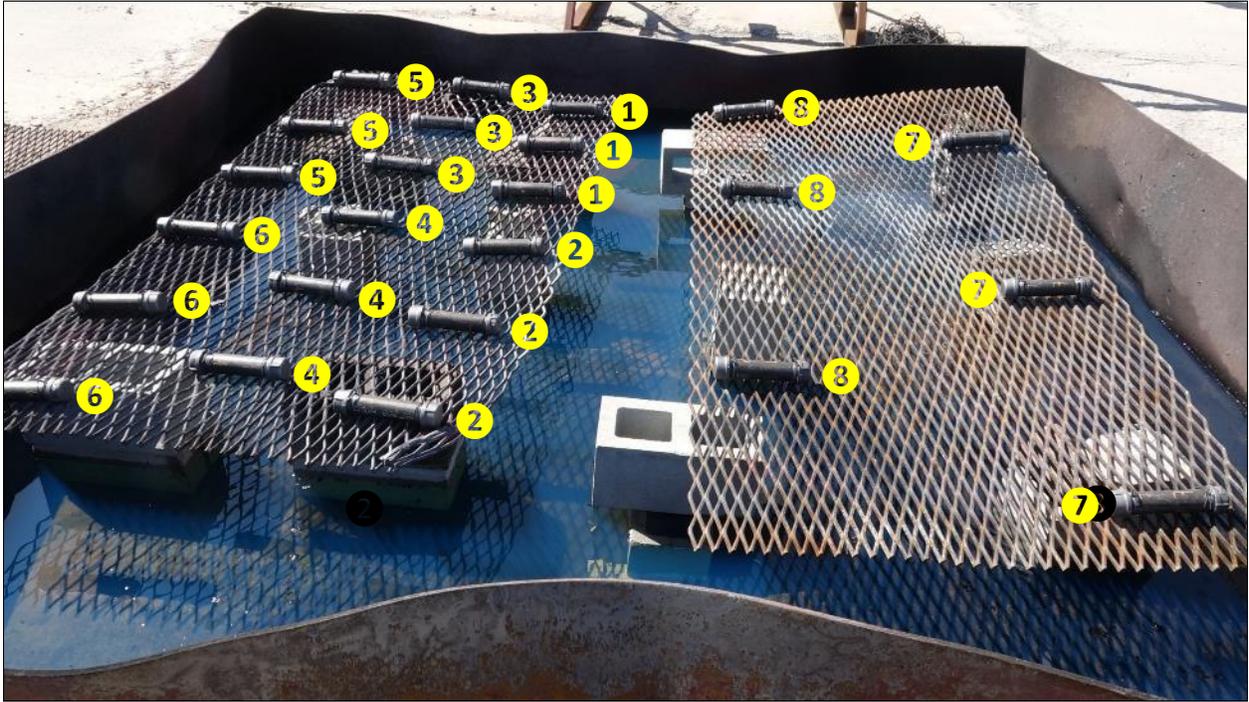
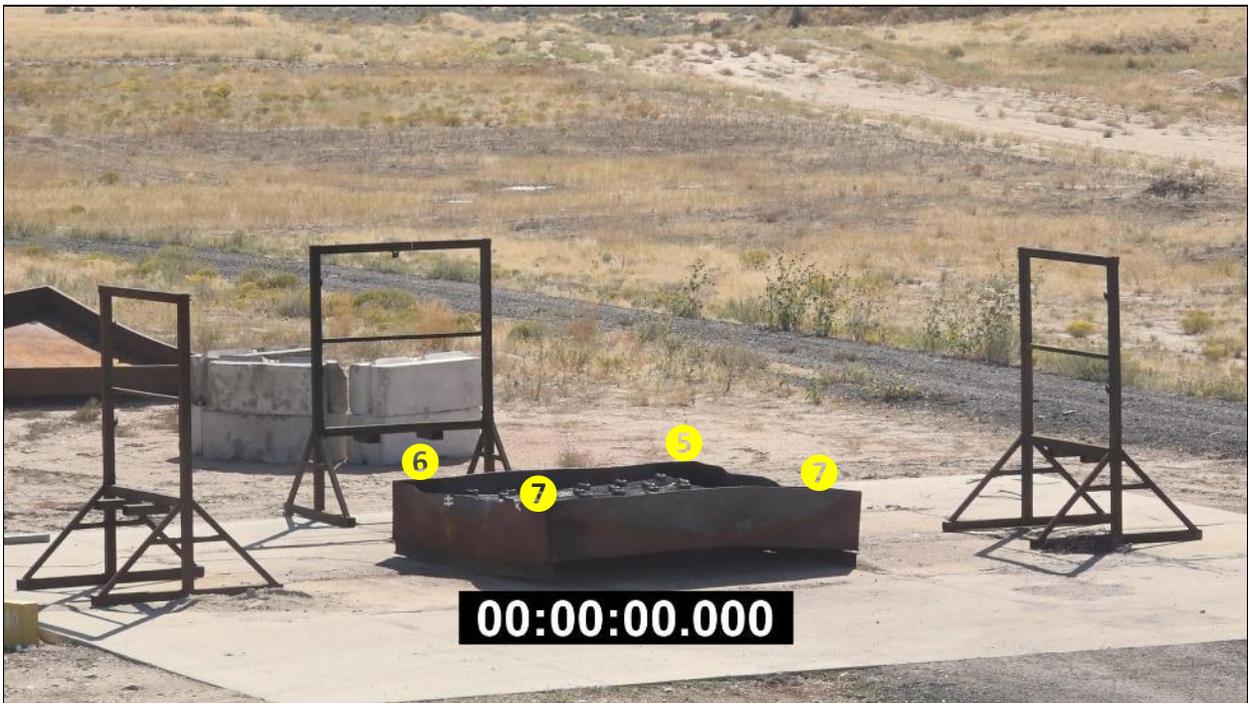


Photo 95: EIS External Fire Test Setup



The fuel basin (12-ft square, 2-ft high walls) was 0.5 meters larger than the stack in every direction. The fuel basin was filled with approximately 600 gallons of Jet-A fuel with 10 inches from the packages to the fuel surface. The environmental conditions at the start of the test were 1.3 m/s wind, 16°C, 32% relative humidity.

7.10.3 Test Results

A total of twenty-four samples were initially secured to the grating. A total of fourteen reactions were observed during the test; a total of four reactions were not detected. The following photo shows the start and time of occurrence of each reaction. The reactions varied in origin, intensity, and severity.

Photo 96: EIS External Fire Test Progression - Flames of Fire (typical)



Photo 97: EIS External Fire Test Progression - Visible Reactions of Samples (typical)







The duration of the bonfire was 65 minutes. Upon post-test inspection, ten samples remained secured to the grating, of which six samples (Mix ID's #2 and #3) were unreacted.

Photo 98: EIS External Fire Test Results - Pipes Still Secured to the Steel Grating

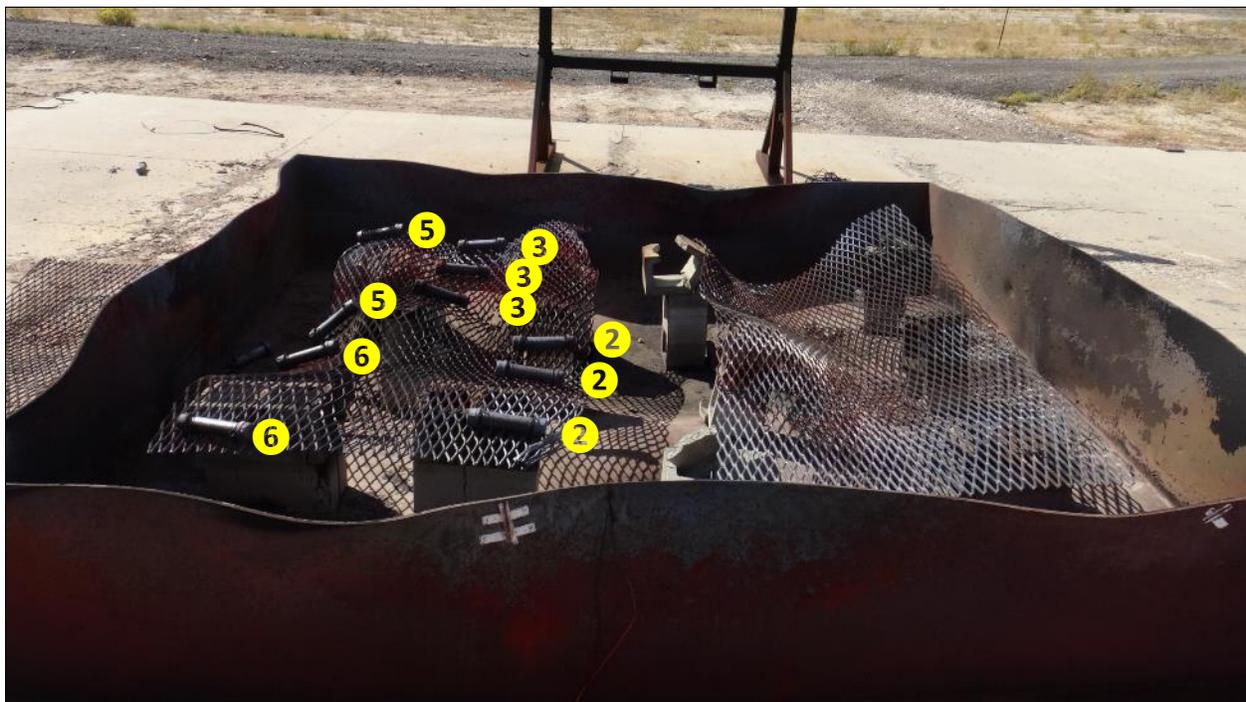


Photo 99: EIS External Fire Test Results - Unreacted Pipes



A total of seventeen reacted pipes were recovered as shown in the following photo; only one pipe was not found. A total of eight of the recovered reacted pipes were missing their end caps. Pipes thrown from the fuel basin and recovered include:

- Mix #1: 31 and 4 m
- Mix #7: 12 m
- Mix #8: 20 and 1 m
- Unknown ID: 19, 9, and 1 m

Photo 100: EIS External Fire Test Results - Reacted Pipes



Table 25: Summary of UN Series 7(e) Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard (Class 1) when heated under confinement
2	Large-scale Mix ID #2	There was no reaction of the three samples.	No reaction when heated under confinement
3	Large-scale Mix ID #3	There was no reaction of the three samples.	No reaction when heated under confinement

Item	Sample	Conditions and Results	Assessment
4	Large-scale Mix ID #4	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
5	Large-scale Mix ID #5	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
6	Large-scale Mix ID #6	Samples ignited; inconclusive whether the reaction resulted in separating end cap from pipe and propelling the pipe beyond 15 meters (Mix ID indiscernible).	Reaction does not appear to be elevated when heated under confinement (inconclusive)
7	Large-scale Mix ID #7	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard (Class 1) when heated under confinement
8	Large-scale Mix ID #8	The three samples ignited, separating end cap from pipe and propelling the pipe beyond 15 meters.	Elevated reaction hazard (Class 1) when heated under confinement

7.10.4 Assessment of Test Results

Three of the thermites failed the UN Series 7 (e) EIS External Fire Tests: Large-scale Mix ID #1 Large-scale Mix ID #4 and the Large-scale Mix ID #8. In each case three samples ignited, separating end cap from pipe and propelled the pipe beyond 15 meters. This the thermites are Class 1 when heated under confinement.

7.11 UN Test N.1 Test method for readily combustible solids

7.11.1 Test Description

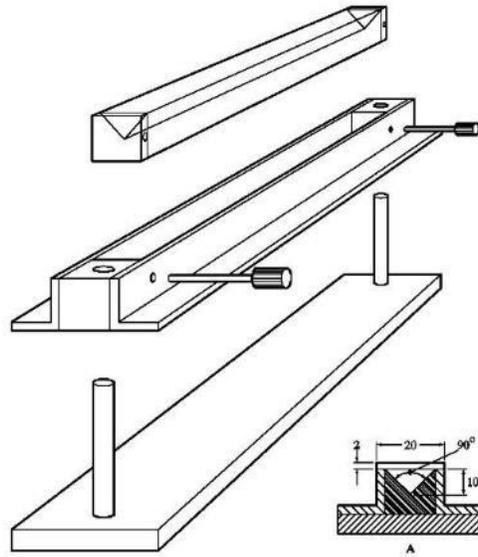
UN Test N.1 is used to determine the ability of a substance to propagate combustion.

For the preliminary screening test, the substance is formed into an unbroken strip or powder train about 250 mm long by 20 mm wide by 10 mm high on a cool, impervious, low heat-conducting base plate. A hot flame (minimum temperature 1000°C) from a gas torch (minimum diameter 5 mm) is applied to one end of the powder train until the powder ignites or for a maximum of 2 minutes (5 minutes for powders of metals or metal-alloys). It is noted whether combustion propagates along 200 mm of the train within the 2-minute test period (or 20 minutes for metal powders). If the substance does not ignite and propagate combustion either by burning with flame or smoldering along 200 mm of the powder train within the 2 minute test period (or 20 minutes), then the substance is not classified as a flammable solid and no further testing is required. If the substance propagates burning of a 200 mm length of the powder train in less than 2 minutes or 20 minutes for metal powders, the full test program is carried out to determine the severity of reaction.

For the burning rate test, the powdered or granular substance, in its commercial form, is loosely filled into the mold. The mold is then dropped three times from a height of 20 mm onto a solid surface. The lateral limitations are then removed and the impervious, non-combustible, low heat-conducting plate is placed on top of the mold, the apparatus inverted and the mold removed. For substances other than metal powders, 1 ml of a wetting solution is added to the pile 30 - 40 mm beyond the 100 mm timing zone; the wetting solution is applied to the ridge drop by drop, ensuring the whole cross-section of the pile is wetted without loss of liquid from the sides (wetting agents free from combustible diluents may be added if necessary). Any suitable ignition source such as a small flame or a hot wire of minimum temperature 1,000 °C is used to ignite the pile at one end. When the pile has burned a distance of 80 mm, the rate of burning is measured over the next 100 mm. For substances other than metal powders, it is noted whether or not the wetted zone stops propagation of the flame for at least 4 minutes. The test is performed six times using a clean cool plate each time, unless a positive result is observed earlier.

Powdered, granular or pasty substances are classified in Division 4.1 when the time of burning of one or more of the test runs is less than 45 seconds or the rate of burning is more than 2.2 mm/s.

Figure 6: Burning Rate Test - Triangular Mold for Forming the Powder Train Pile.



7.11.2 Test Configuration

For the gas torch trials, the hot flame was provided by a MAP-Pro gas torch with a flame temperature of over 2000 °C and a flame diameter of approximately 25 mm. For the hot wire trials, an 18-gauge 80/20 nickel/chromium resistance heating wire (Type A) was supplied with 23 Amps to achieve the test temperature of around 1000 °C. The photo below shows a typical test configuration.

Photo 101: Readily Combustible Solids Test Setup (typical)



7.11.3 Test Results

The test results for ignition by 2000 °C gas torch are summarized in the following table.

Table 26: Summary for UN Test N.1 “Test Method for Readily Combustible Solids” - Ignition by 2000 °C Gas torch

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Sample ignited; burning time of powder train pile too fast to be measured (less than 0.1 seconds for the entire 200mm length).	Rapid burning consistent more with the hazard of Class 1 explosives
2	Large-scale Mix ID #2	Combustion of the sample did not propagate in 5 minutes. <i>NOTE: Sample could ignite by employing a thermite starting powder or heating element, with only partial propagation down the powder train pile (100 mm) over 50.7 seconds.</i>	Consistent with exclusion from Division 4.1
3	Large-scale Mix ID #3	Combustion of the sample did not propagate in 5 minutes. <i>NOTE: Supplemental magnesium strip was also unable to ignite the sample.</i>	Consistent with exclusion from Division 4.1
4	Large-scale Mix ID #4	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
5	Large-scale Mix ID #5	For Trial 1, the sample ignited with a burning time of approximately 2 seconds for the entire length of the powder train pile. For Trial 2, the sample ignited but the reaction did not propagate down the powder train pile.	Consistent with Division 4.1, packing group II
6	Large-scale Mix ID #6	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1

Item	Sample	Conditions and Results	Assessment
7	Large-scale Mix ID #7	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives
8	Large-scale Mix ID #8	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives

The test results are shown in the following photos.

Photo 102: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #1 with Ignition and Rapid Burning

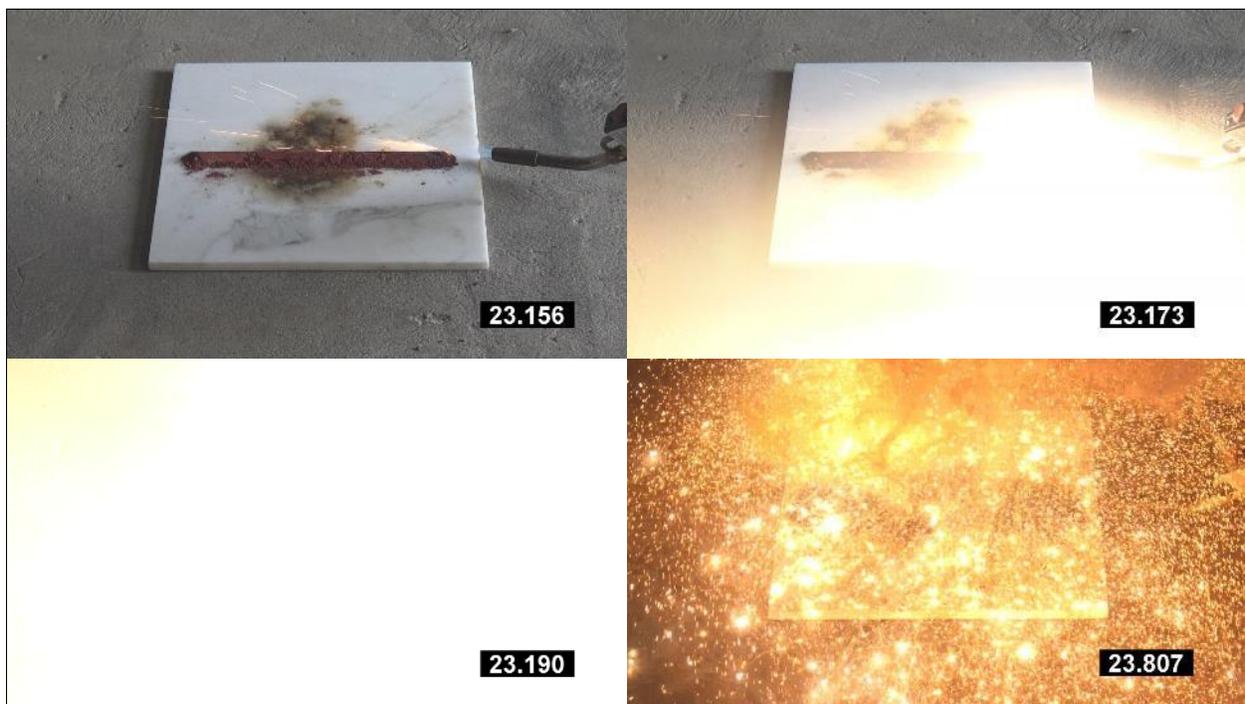


Photo 103: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #2 with No Reaction in 5 minutes



Photo 104: Readily Combustible Test Results - Gas Torch & Magnesium Strip for Large-scale Mix ID #2 with Ignition without Propagation

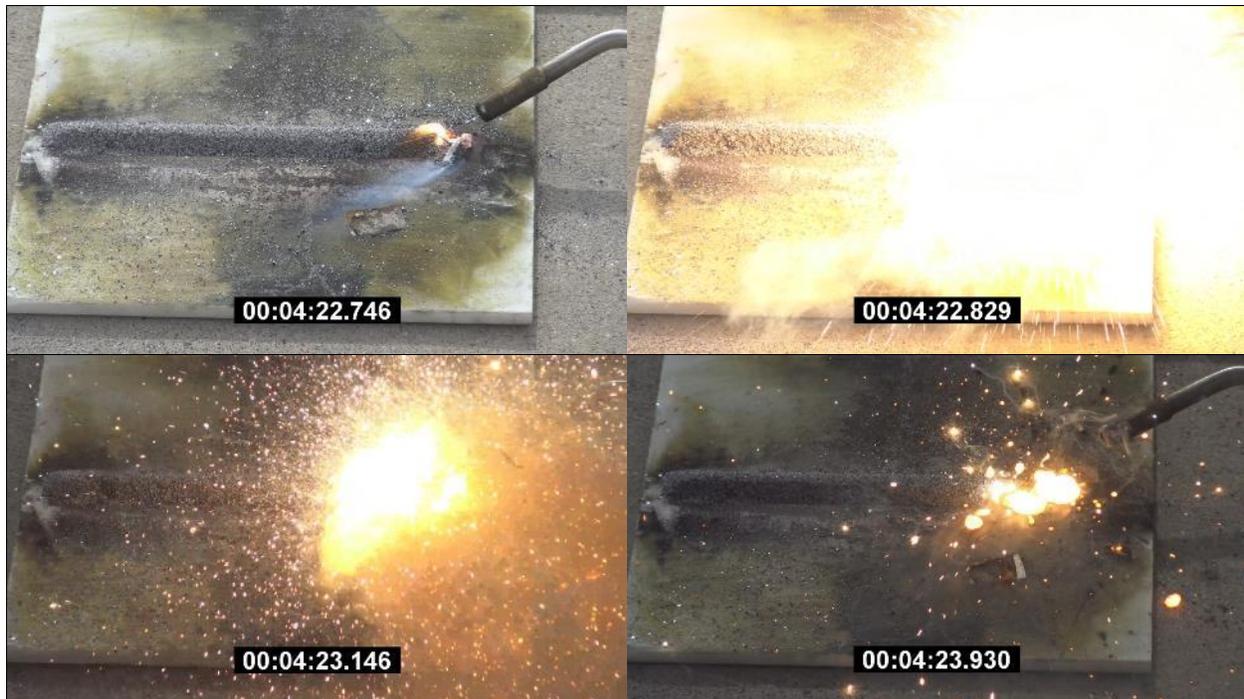


Photo 105: Readily Combustible Test Results - Gas Torch, Sparkler & Thermite Starter Powder for Large-scale Mix ID #2 with Ignition and Partial Propagation over 50.7 seconds

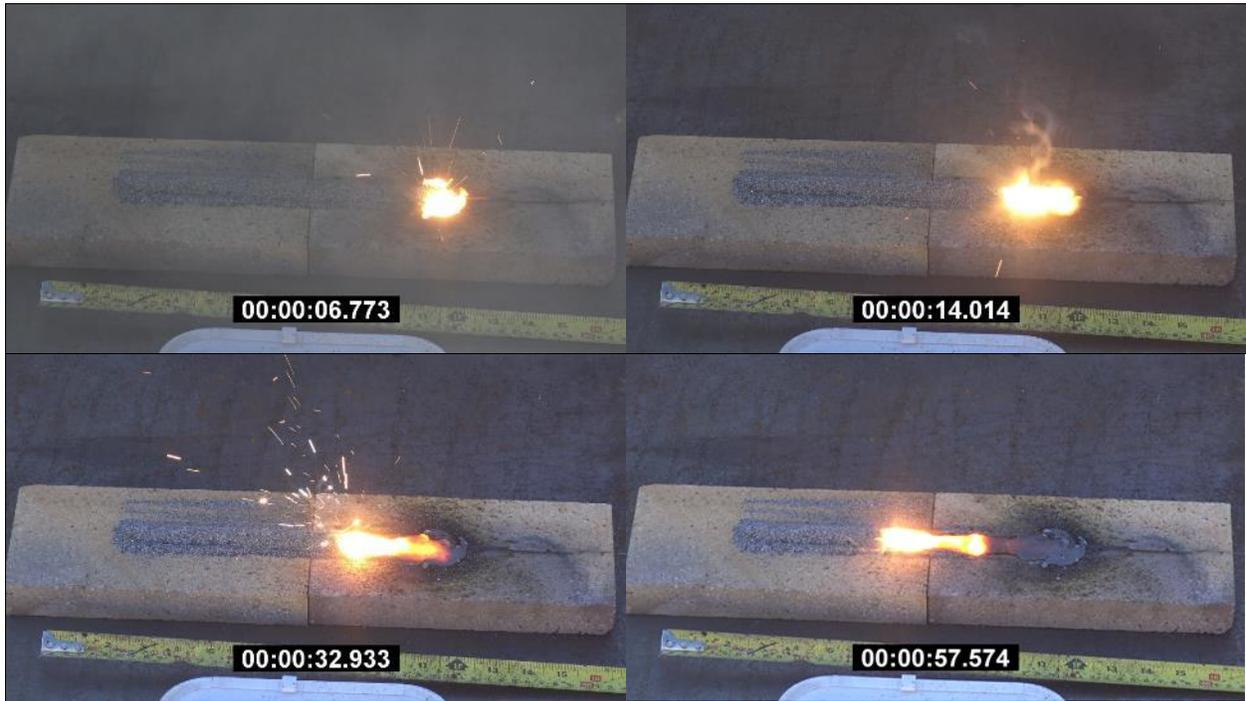


Photo 106: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #3 with No Reaction in 5 minutes

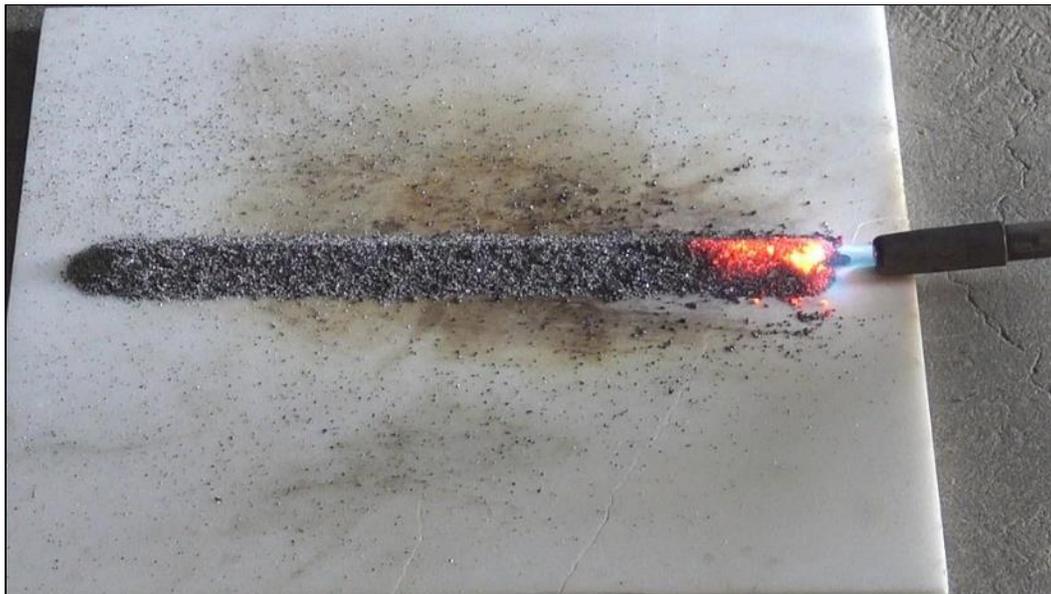


Photo 107: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #4 with Ignition but No Propagation in 5 minutes

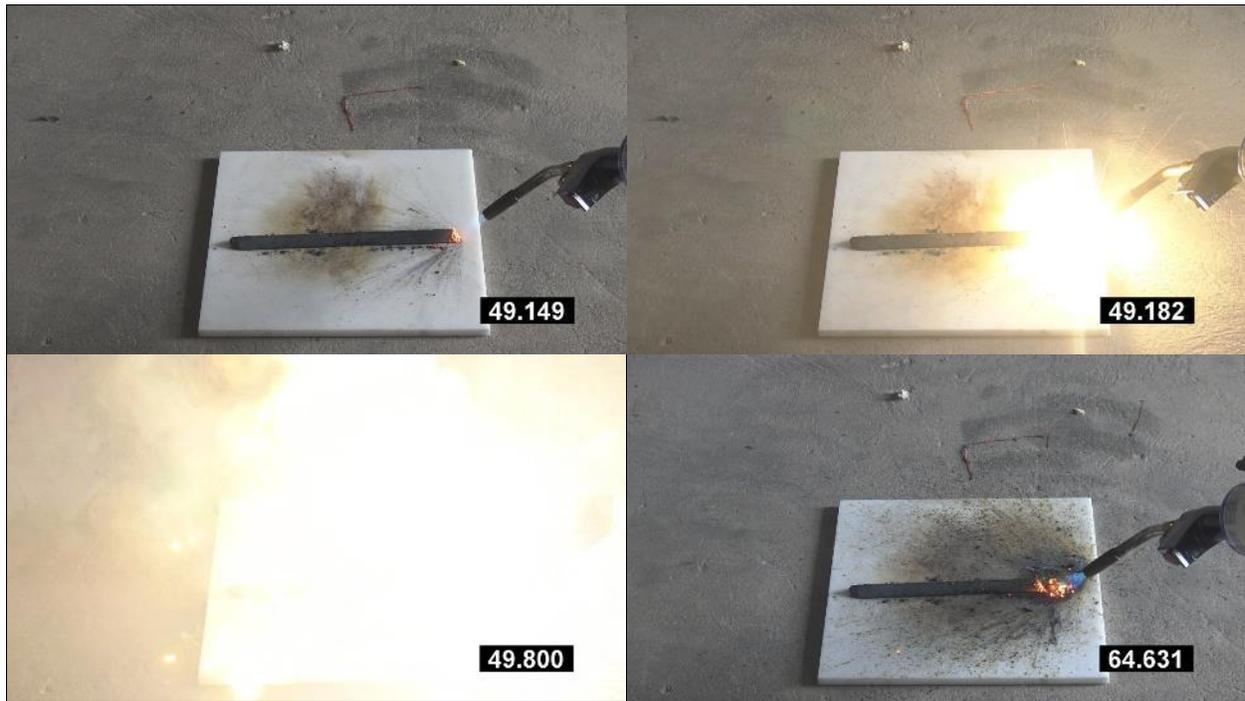


Photo 108: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #5 with Ignition and Propagation (Trial 1)

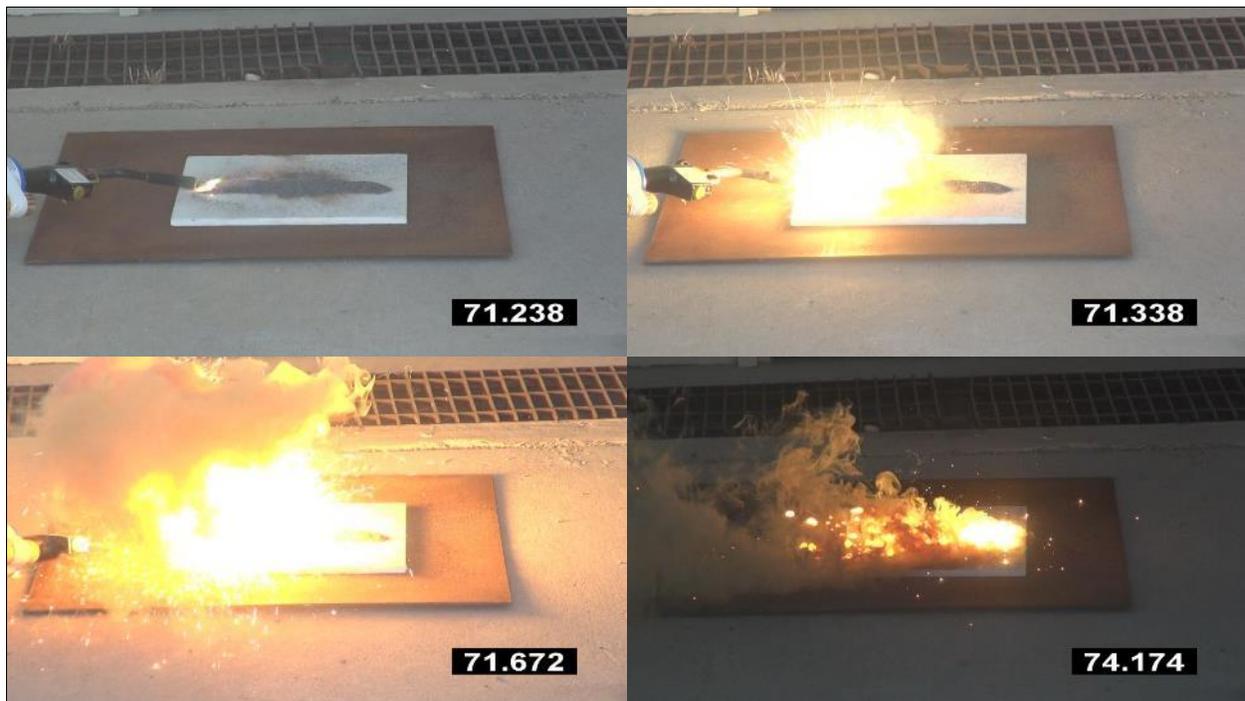


Photo 109: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #5 with Ignition and Propagation (Trial 2)

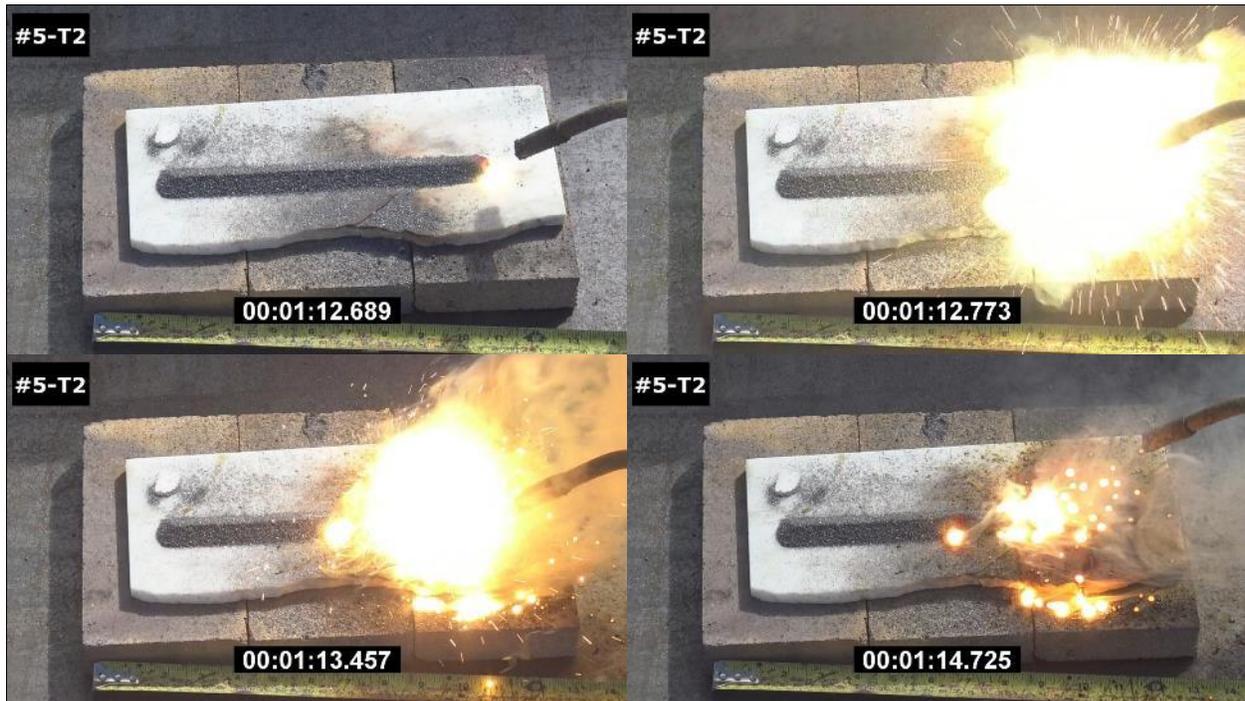


Photo 110: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #6 with Ignition and Partial Propagation over 6.2 seconds

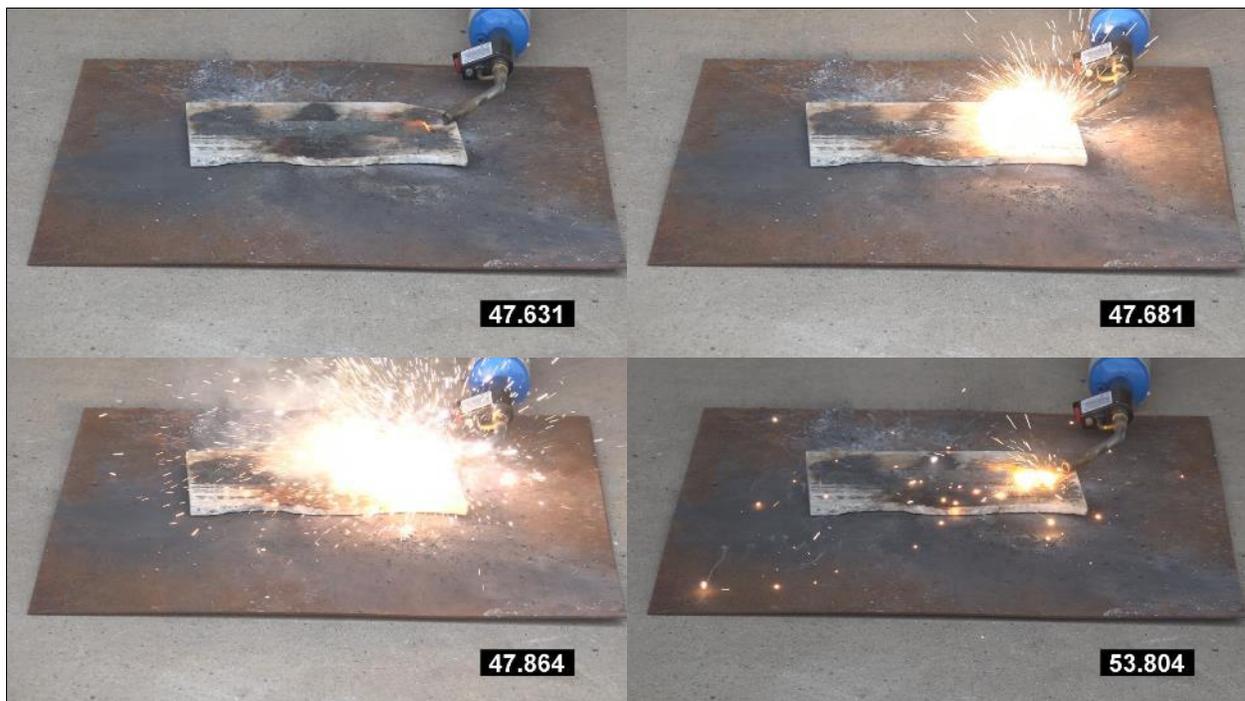


Photo 111: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #7 with Ignition and Explosion

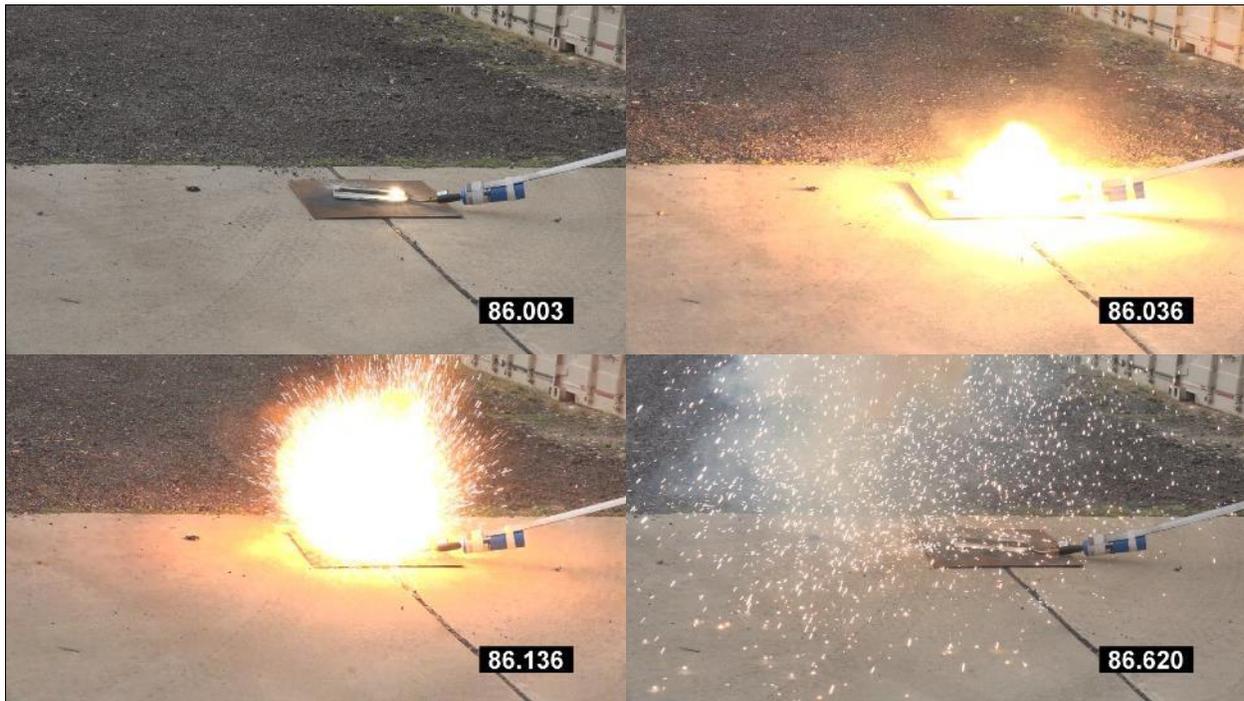
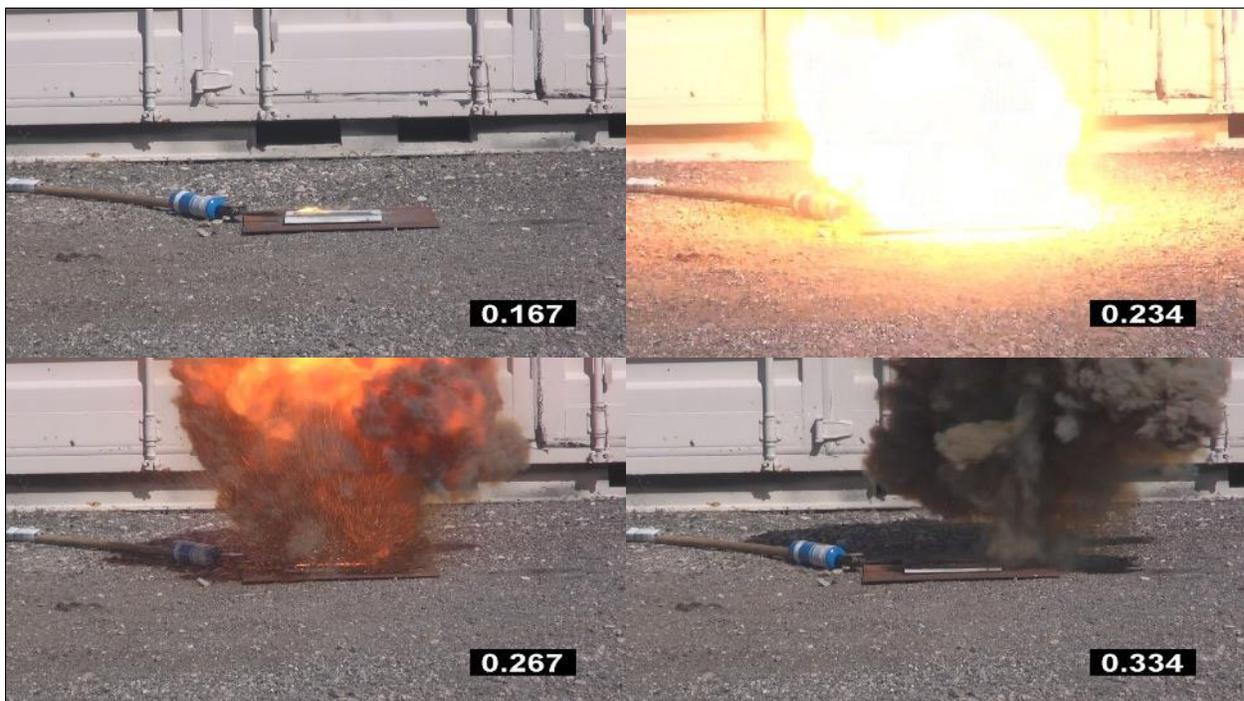


Photo 112: Readily Combustible Test Results - Gas Torch for Large-scale Mix ID #8 with Ignition and Explosion



The test results for ignition by 1000 °C hot wire are summarized in the following table.

Table 27: Summary for UN Test N.1 “Test Method for Readily Combustible Solids” - Ignition by 1000 °C Hot Wire

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Sample ignited; burning time of powder train 0.3 seconds for the entire 200mm length.	Rapid burning consistent more with the hazard of Class 1 explosives
2	Large-scale Mix ID #2	Combustion of the sample did not propagate in 5 minutes. NOTE: Sample ignited with heating element but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
3	Large-scale Mix ID #3	Combustion of the sample did not propagate in 5 minutes.	Consistent with exclusion from Division 4.1
4	Large-scale Mix ID #4	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
5	Large-scale Mix ID #5	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1
6	Large-scale Mix ID #6	Sample ignited but the reaction did not propagate down the powder train pile.	Consistent with exclusion from Division 4.1

Item	Sample	Conditions and Results	Assessment
7	Large-scale Mix ID #7	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives
8	Large-scale Mix ID #8	Sample ignited; nearly instantaneous consumption of the powder train pile (explosion with less than 0.03 seconds for the entire 200mm length).	Explosion consistent more with the hazard of Division 1.1 explosives

The test results are shown in the following photos.

Photo 113: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #1 with Ignition and Full Propagation over 0.3 seconds

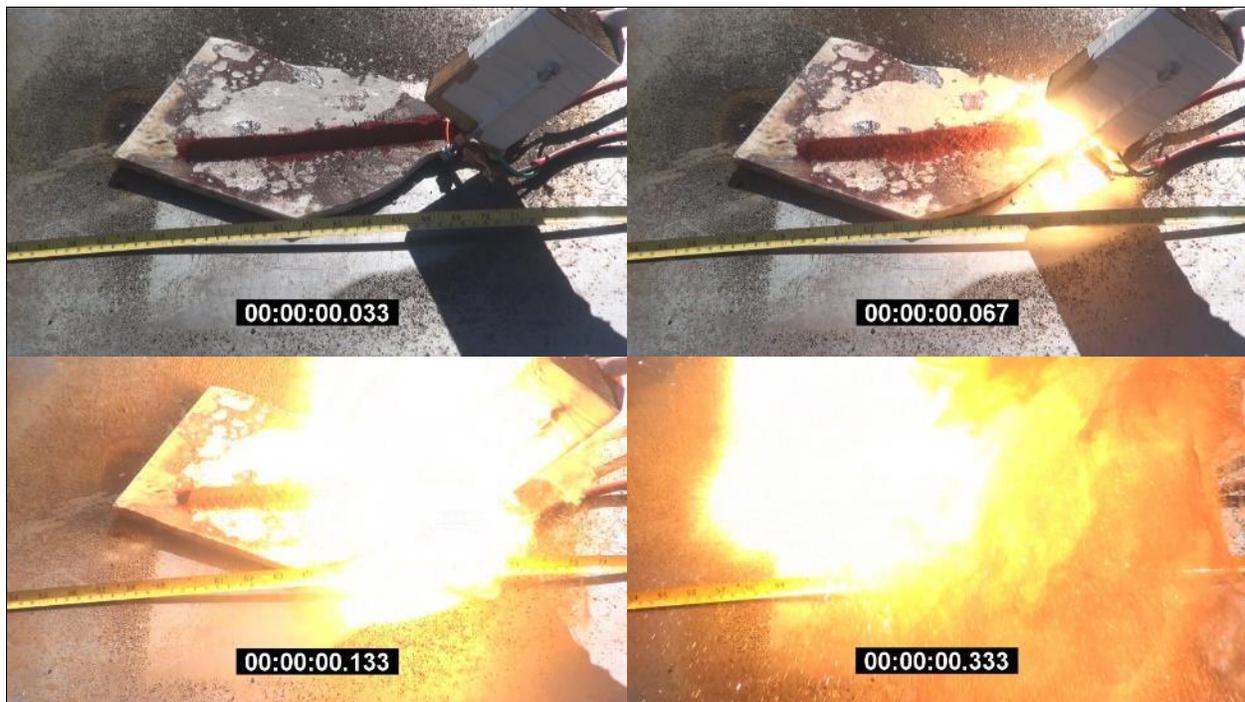


Photo 114: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #2 with No Reaction in 5 minutes



Photo 115: Readily Combustible Test Results - Heating Element for Large-scale Mix ID #2 with Ignition and Partial Propagation over 22 seconds

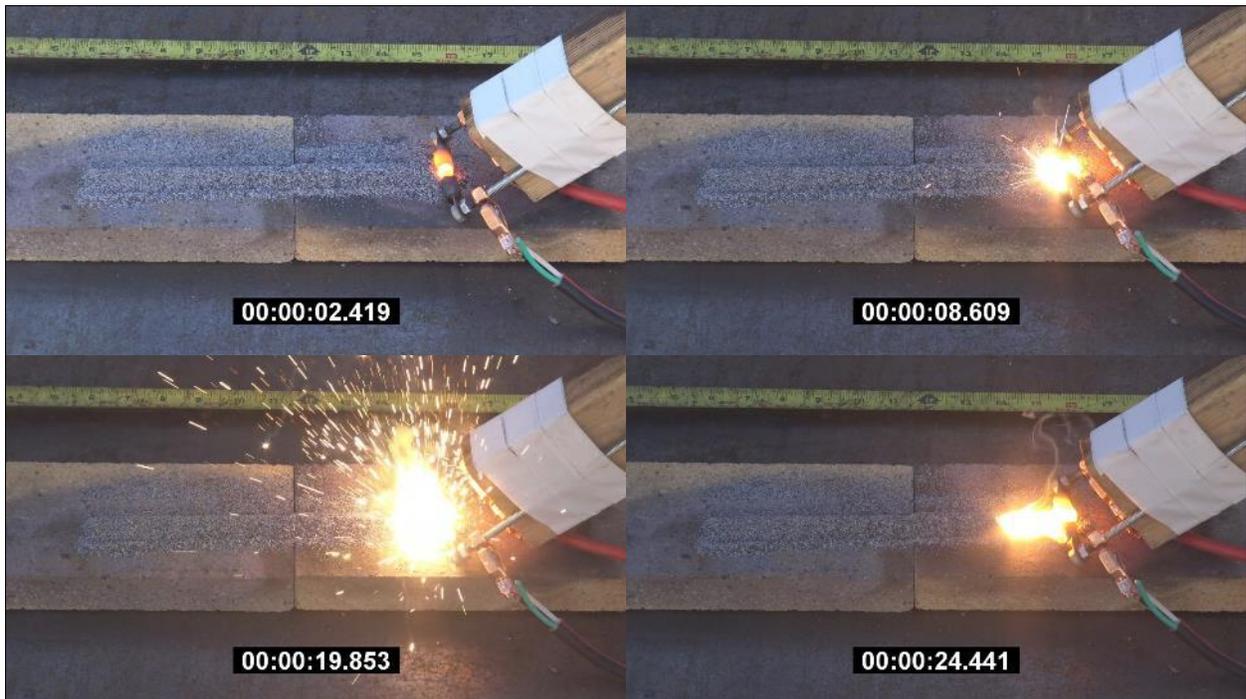


Photo 116: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #3 with No Reaction in 5 minutes



Photo 117: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #4 with Ignition and Partial Propagation over 1.4 seconds

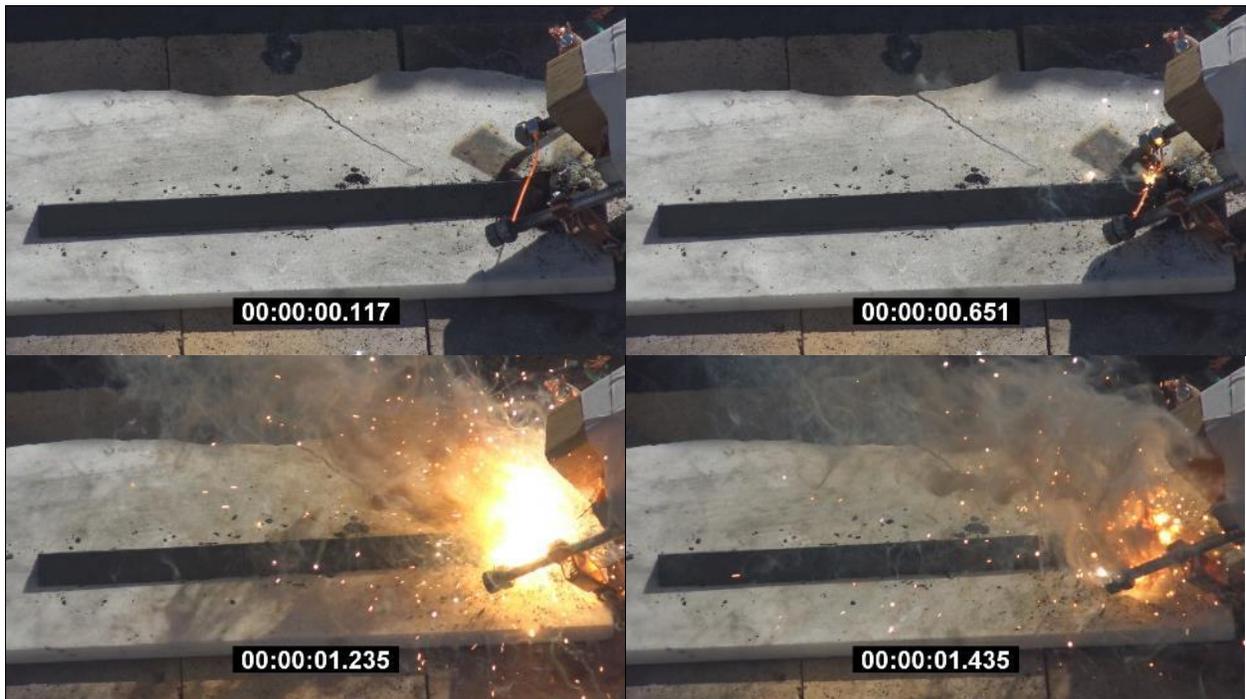


Photo 118: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #5 with Ignition and Partial Propagation over 2.4 seconds



Photo 119: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #6 with Ignition and Partial Propagation over 9.3 seconds

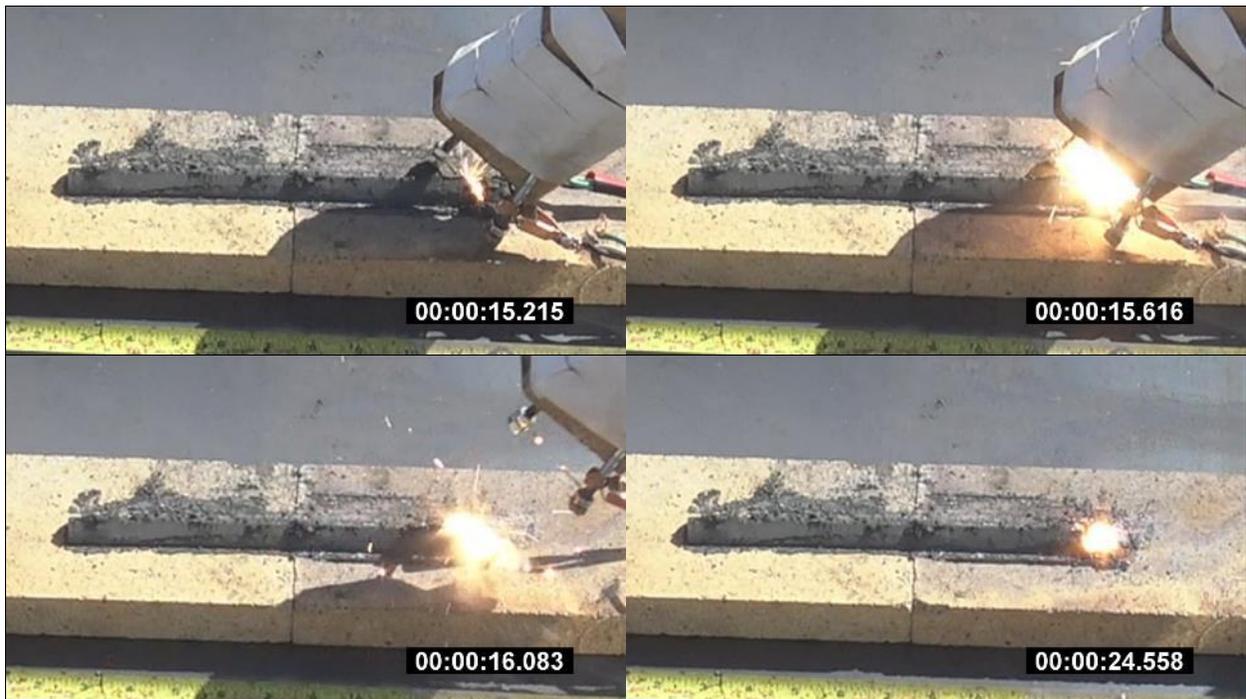


Photo 120: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #7 with Explosion

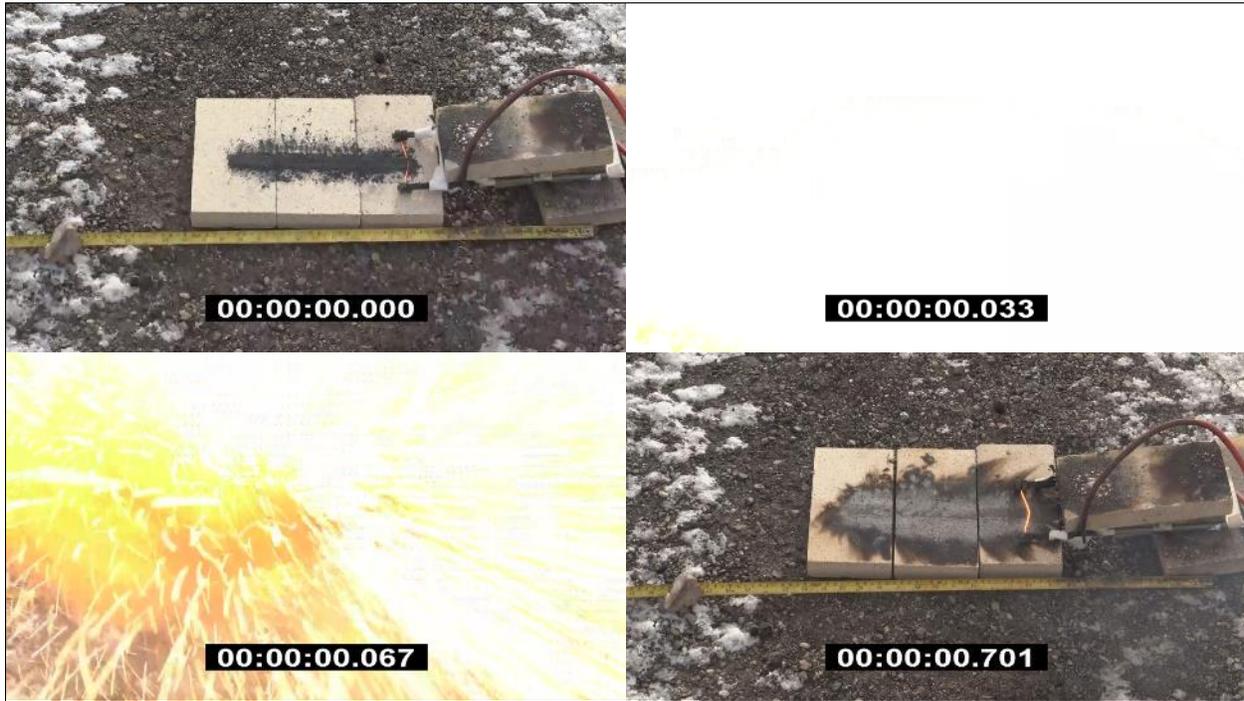


Photo 121: Readily Combustible Test Results - Hot Wire for Large-scale Mix ID #8 with Explosion



7.11.4 Assessment of Test Results

Based on the UN Test N.1 gas torch and hot wire test results, the Large-scale Mix ID #1, Large-scale Mix ID #7 and Large-scale Mix ID #8 samples exhibited burn rates consistent with that of Division 1.1; the ignition and propagation of Mix ID's #2, #3, #4 and #6 were consistent with exclusion from Division 4.1. Burning of the Large-scale Mix ID #5 sample was consistent with assignment into Division 4.1, packing group II.

7.12 Large-scale UN Test N.1 Test method for readily combustible solids

7.12.1 Test Description

The Large-scale UN Test N.1 is used to determine the ability of a bulk substance to propagate combustion.

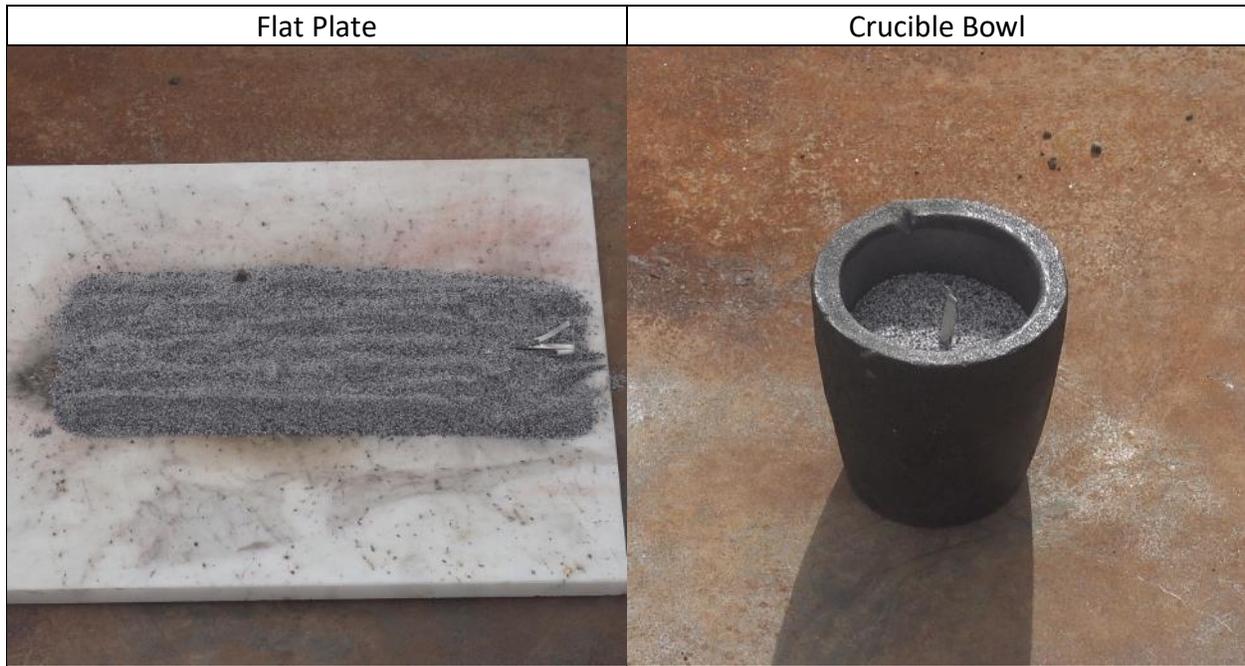
The test may be performed with the powdered or granular substance is formed into an unbroken strip or powder train approximately 250 mm long by 80 mm wide by 10 mm high on a cool, impervious, low heat-conducting base plate. Alternately, the substance is poured into a 60-mm diameter, 80-mm tall, low heat-conducting crucible bowl.

Any suitable ignition source is used to ignite the strip near the center of one end. The test is performed six times using a clean cool plate each time, unless a positive result is observed earlier. Powdered, granular or pasty substances are classified in Division 4.1 when the time of burning of one or more of the test runs is less than 45 seconds or the rate of burning is more than 2.2 mm/s.

7.12.2 Test Configuration

The crucible bowl had an 89-mm outer diameter, 9.5-mm wall thickness, and 89-mm height. For ignition by gas torch, a hot flame was provided by a MAP-Pro gas torch with a flame temperature of over 2000 °C and a flame diameter of approximately 25 mm. For ignition by hot wire, an 18-gauge 80/20 Nickel/Chromium resistance heating wire (Type A) was supplied with 23 Amps to achieve the test temperature of around 1000 °C. For compositions that were difficult to ignite, a hot flame was provided by an oxy-acetylene torch with a flame temperature of around 3500 °C and a flame diameter of approximately 3 mm. The photo below shows the typical test configurations.

Photo 122: Large-scale Readily Combustible Solids Test Setup (typical)



7.12.3 Test Results

The test results are summarized in the following table.

Table 28: Summary for Large-Scale UN Test N.1 “Test Method for Readily Combustible Solids”

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	<p><u>Flat plate:</u> Sample ignited by gas torch and hot wire with full propagation (250mm) of powder over 0.5 seconds (500 mm/sec).</p> <p><u>Crucible bowl:</u> Sample ignited by gas torch and pyrogen igniter (remote) with full consumption of powder over 0.5 seconds.</p>	<p>Rapid burning consistent more with the hazard of Class 1 explosives</p>

Item	Sample	Conditions and Results	Assessment
2	Large-scale Mix ID #2	<p><u>Flat plate & crucible bowl</u>: Combustion of the sample did not propagate in 5 minutes when ignited by hot wire (plate) or gas torch (both).</p> <p><i>Alternative ignition method:</i></p> <p><u>Flat plate</u>: Sample ignited by oxy-acetylene torch with full propagation (250mm) of powder over 10 seconds (25.0mm/sec).</p> <p><u>Crucible bowl</u>: Sample ignited by oxy-acetylene torch with full consumption over 2.7 seconds.</p>	Consistent with exclusion from Division 4.1 for standard ignition; alternative ignition results in Division 4.1, packing group II
3	Large-scale Mix ID #3	<p><u>Flat plate & crucible bowl</u>: Combustion of the sample did not propagate in 5 minutes when ignited by hot wire or gas torch.</p> <p><i>Alternative ignition method:</i></p> <p><u>Flat plate</u>: Sample ignited by oxy-acetylene torch with partial propagation (170mm) of powder over 75 seconds (2.3 mm/sec). Sample ignited by gas torch (aided by a thermite starting powder) with partial propagation (140mm) of powder over 23 seconds (6.1 mm/sec).</p>	Burning rate > 2.2 mm/sec (consistent with Division 4.1, packing group II)
4	Large-scale Mix ID #4	<p><u>Flat plate</u>: Sample ignited by torch and hot wire with full propagation (250mm) of powder over 1.3 - 2.1 seconds (119 - 192 mm/sec).</p> <p><u>Crucible bowl</u>: Sample ignited by torch with full consumption of powder over 7.4 seconds.</p>	Rapid burning consistent more with the hazard of Class 1 explosives
5	Large-scale Mix ID #5	<p><u>Flat plate</u>: Sample ignited by torch and hot wire with full propagation (250mm) of powder over 1.5 - 2.4 seconds (104 - 167mm/sec).</p> <p><u>Crucible bowl</u>: Sample ignited by torch (aided by a thermite starting powder) with full consumption of powder over 6.4 seconds.</p>	Rapid burning consistent more with the hazard of Class 1 explosives

Item	Sample	Conditions and Results	Assessment
6	Large-scale Mix ID #6	<p><u>Flat plate</u>: Sample ignited by torch and hot wire with full propagation (250mm) of powder over 36 - 75 seconds (3.3 - 6.9 mm/sec).</p> <p><u>Crucible bowl</u>: Sample ignited by torch with full consumption of powder over 10.5 seconds.</p>	<p>Burning rate > 2.2 mm/sec (consistent with Division 4.1, packing group II)</p>
7	Large-scale Mix ID #7	<p><u>Flat plate</u>: Sample ignited by pyrogen igniter (flame) and hot wire with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity): much faster than 8,000 mm/sec. Plate broken into pieces.</p> <p><u>Crucible bowl</u>: Sample ignited by pyrogen igniter (flame) with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity). No visible damage to crucible bowl.</p>	<p>Explosion consistent more with the hazard of Division 1.1 explosives</p>
8	Large-scale Mix ID #8	<p><u>Flat plate</u>: Sample ignited by pyrogen igniter (flame) and hot wire with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity): much faster than 8,000 mm/sec. Plate broken into pieces.</p> <p><u>Crucible bowl</u>: Sample ignited by pyrogen igniter (flame) with nearly instantaneous consumption of the powder (explosion with less than 0.03 seconds for the entire quantity). No visible damage to crucible bowl.</p>	<p>Explosion consistent more with the hazard of Division 1.1 explosives</p>

The test results are shown in the following photos.

Photo 123: Large-Scale Readily Combustible Test Results - Gas Torch on Plate for Large-scale Mix ID #1 with Ignition and Full Consumption over 0.5 seconds

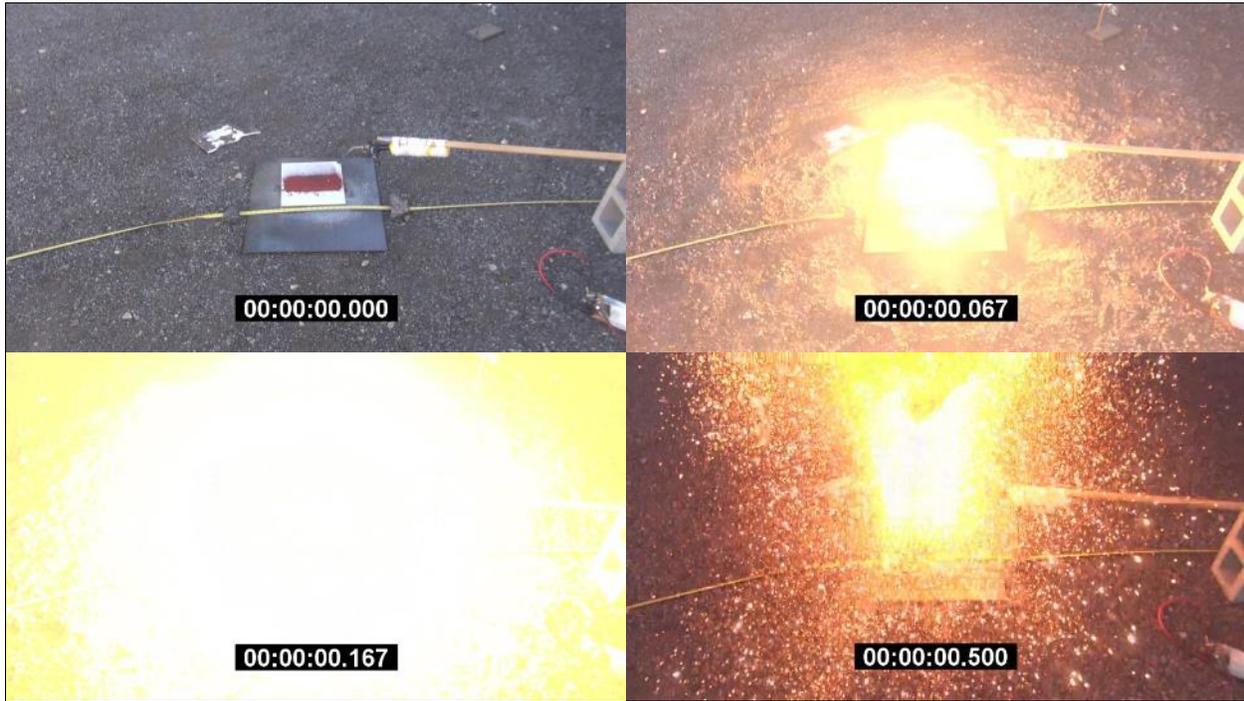


Photo 124: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #1 with Ignition and Full Consumption over 0.5 seconds

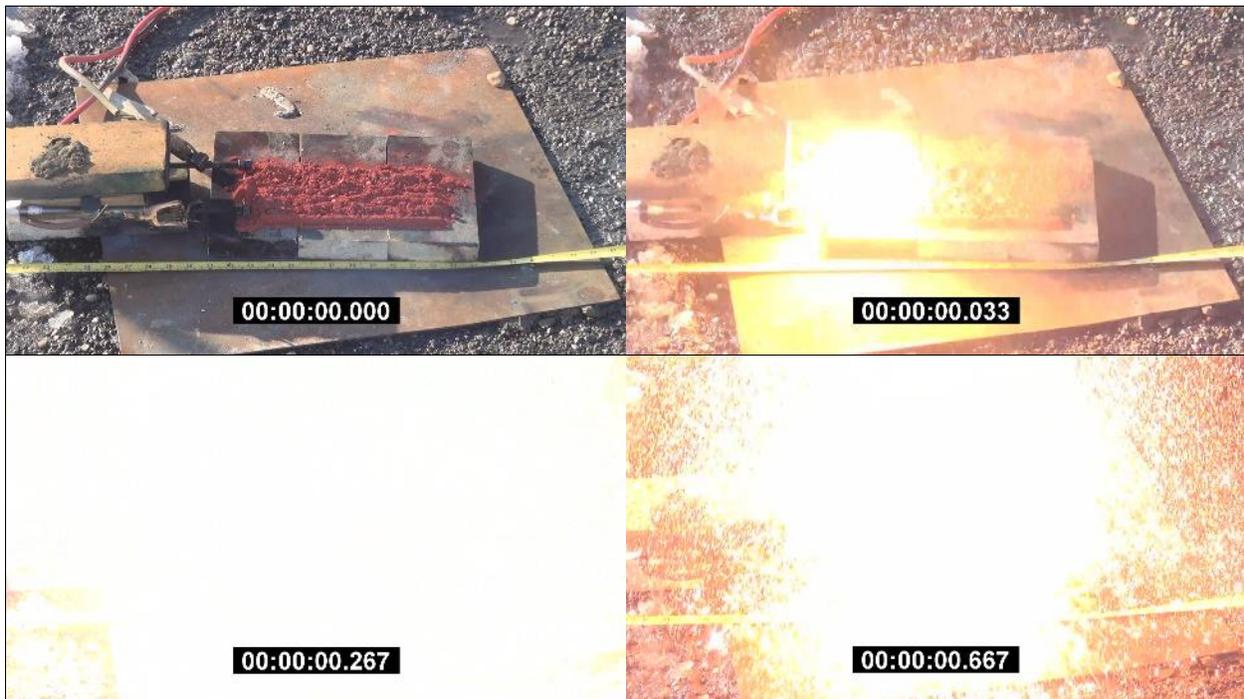


Photo 125: Large-Scale Readily Combustible Test Results - Gas Torch in Crucible for Large-scale Mix ID #1 with Ignition and Full Consumption over 0.5 seconds



Photo 126: Large-Scale Readily Combustible Test Results - Pyrogen Match in Crucible for Large-scale Mix ID #1 with Ignition and Full Consumption over 0.5 seconds



Photo 127: Large-Scale Readily Combustible Test Results - Gas Torch and Hot Wire on Plate / Crucible for Large-scale Mix ID #2 without Ignition

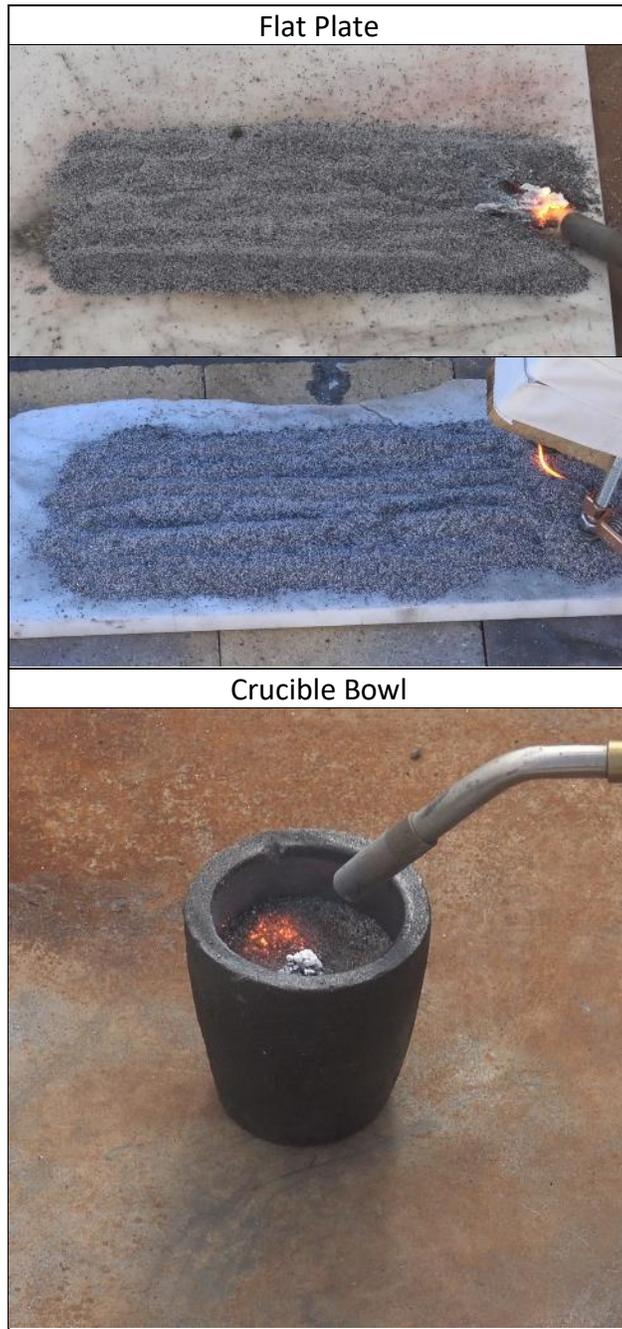


Photo 128: Large-Scale Readily Combustible Test Results - Acetylene Torch on Plate for Large-scale Mix ID #2 with Ignition and Full Consumption over 10 seconds

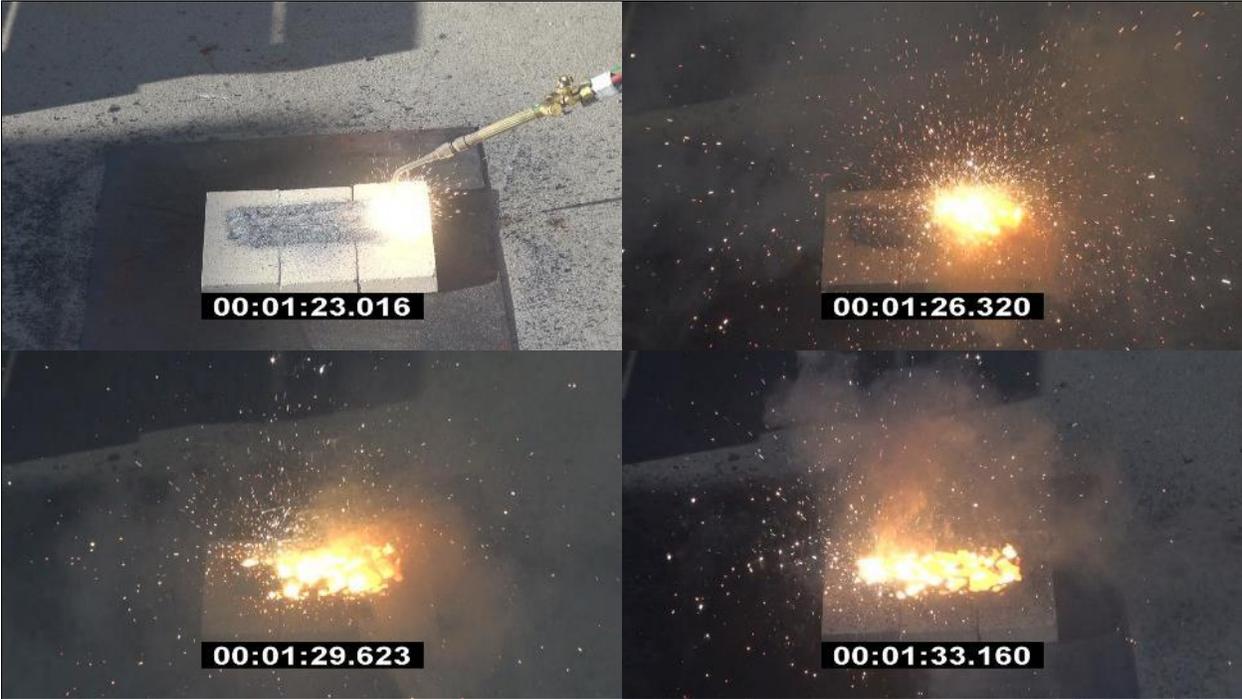


Photo 129: Large-Scale Readily Combustible Test Results - Acetylene Torch in Crucible for Large-scale Mix ID #2 with Ignition and Full Consumption over 2.7 seconds

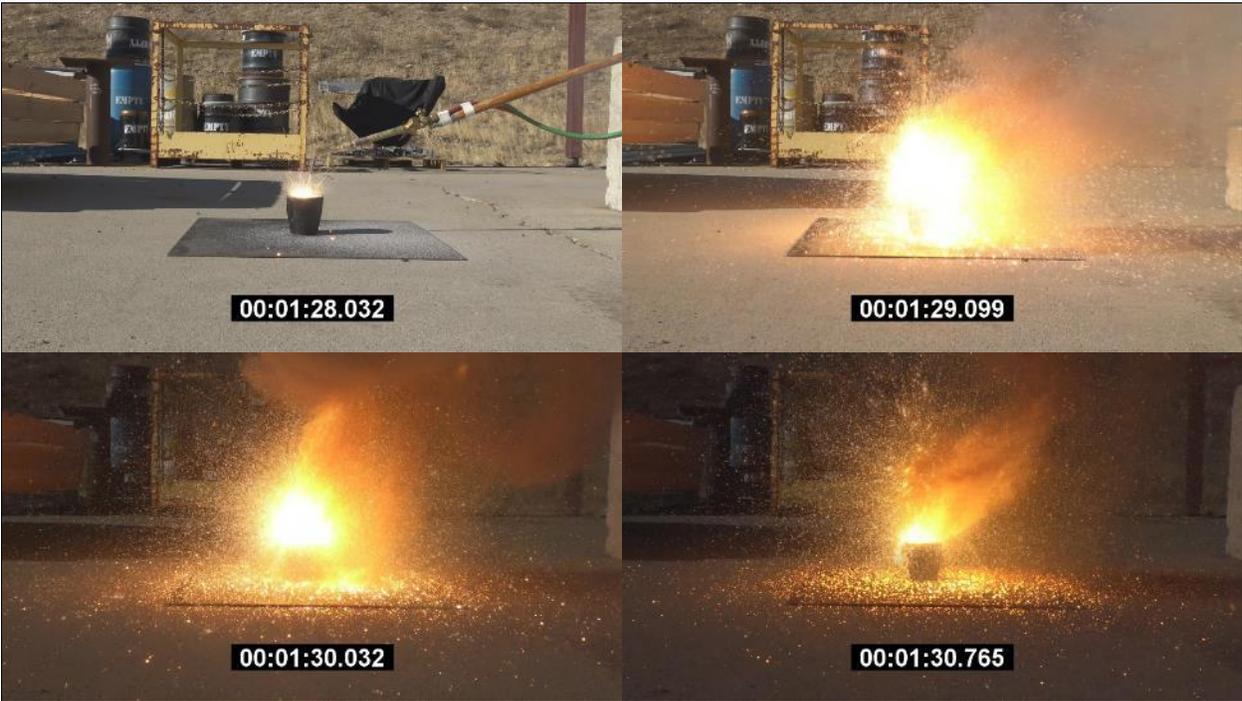


Photo 130: Large-Scale Readily Combustible Test Results - Gas Torch on Plate for Large-scale Mix ID #3 with No Reaction in 5 minutes



Photo 131: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #3 with No Reaction in 5 minutes



Photo 132: Large-Scale Readily Combustible Test Results - Acetylene Torch on Plate for Large-scale Mix ID #3 with Ignition and Partial Consumption over 75 seconds



Photo 133: Large-Scale Readily Combustible Test Results - Gas Torch (aided by a thermite starting powder) on Plate for Large-scale Mix ID #3 with Ignition and Partial Consumption over 23 seconds

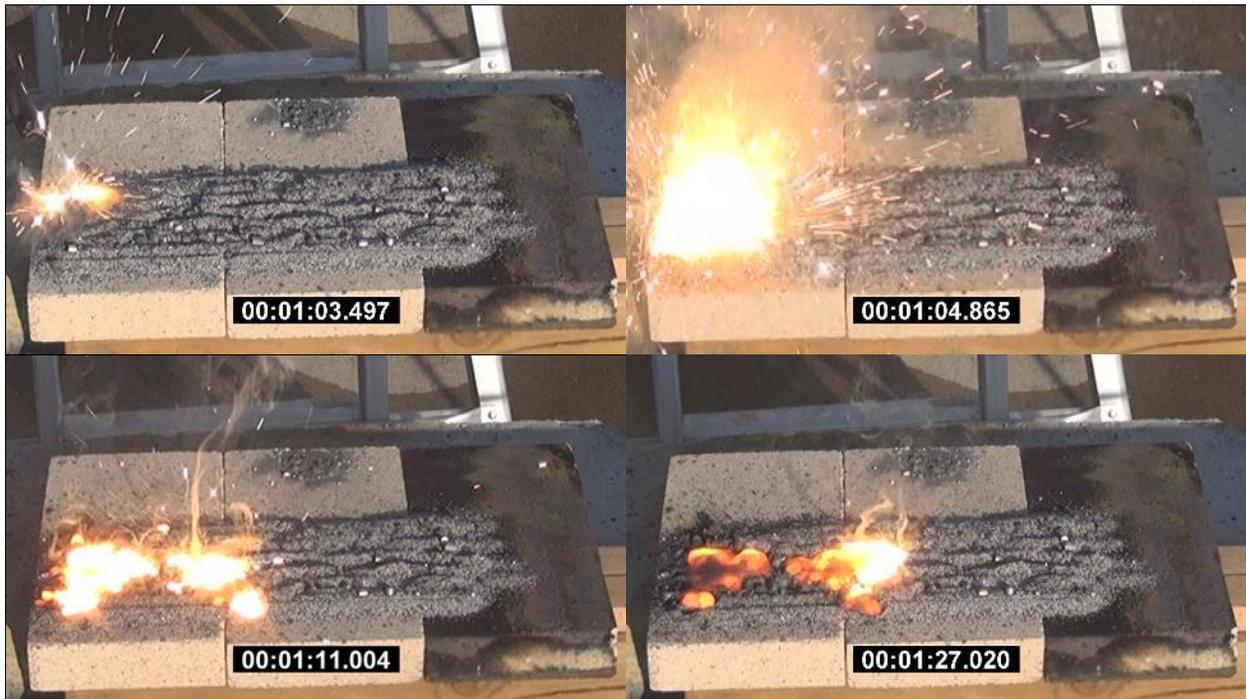


Photo 134: Large-Scale Readily Combustible Test Results - Gas Torch on Plate for Large-scale Mix ID #4 with Ignition and Full Consumption over 2.1 seconds



Photo 135: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #4 with Ignition and Full Consumption over 1.3 seconds



Photo 136: Large-Scale Readily Combustible Test Results - Gas Torch in Crucible for Large-scale Mix ID #4 with Ignition and Full Consumption over 7.4 seconds



Photo 137: Large-Scale Readily Combustible Test Results - Gas Torch on Plate for Large-scale Mix ID #5 with Ignition and Full Propagation over 1.5 seconds



Photo 138: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #5 with Ignition and Full Propagation over 2.4 seconds



Photo 139: Large-Scale Readily Combustible Test Results - Gas Torch (aided by a thermite starting powder) in Crucible for Large-scale Mix ID #5 with Ignition and Full Propagation over 6.4 seconds



Photo 140: Large-Scale Readily Combustible Test Results - Gas Torch on Plate for Large-scale Mix ID #6 with Ignition and Full Propagation over 36 seconds



Photo 141: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #6 with Ignition and Full Propagation over 75 seconds

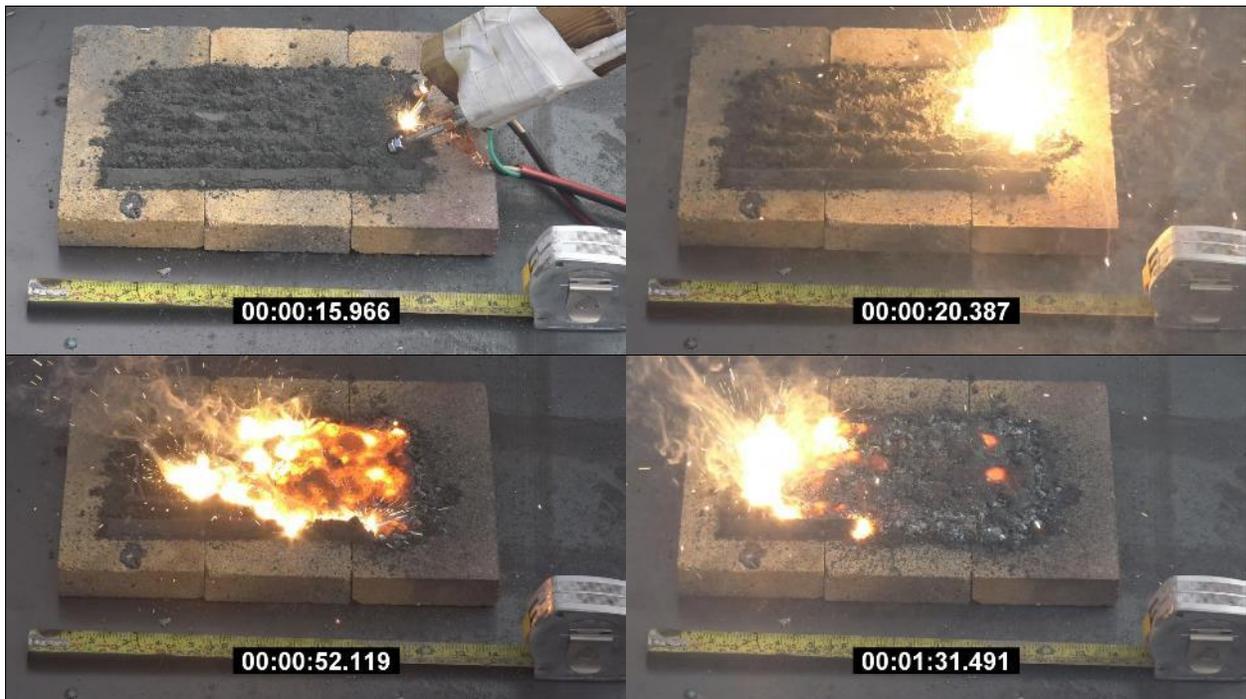


Photo 142: Large-Scale Readily Combustible Test Results - Gas Torch in Crucible for Large-scale Mix ID #6 with Ignition and Full Consumption over 10.5 seconds



Photo 143: Large-Scale Readily Combustible Test Results - Pyrogen Match on Plate for Large-scale Mix ID #7 with Explosion



Photo 144: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #7 with Explosion



Photo 145: Large-Scale Readily Combustible Test Results - Pyrogen Match in Top of Crucible for Large-scale Mix ID #7 with Explosion



Photo 146: Large-Scale Readily Combustible Test Results - Pyrogen Match on Plate for Large-scale Mix ID #8 with Explosion



Photo 147: Large-Scale Readily Combustible Test Results - Hot Wire on Plate for Large-scale Mix ID #8 with Explosion



Photo 148: Large-Scale Readily Combustible Test Results - Pyrogen Match in Top of Crucible for Large-scale Mix ID #8 with Explosion



7.12.4 Assessment of Test Results

An assessment of the results of the large-scale UN Test N.1 indicates the Large-scale Mix ID #1, Large-scale Mix ID #4, and Large-scale Mix ID #5 exhibited rapid burning consistent with the hazard of a Class 1 explosive. The Large-scale Mix ID #2, Large-scale Mix ID #3, and Large-scale Mix ID #6 each had burn rates >2.2 mm/sec which is consistent with Division 4.1, Packing Group II. Both the Large-scale Mix ID #7 and Large-scale Mix ID #8 exploded and are consistent more with the hazard of Division 1.1 explosives.

7.13 UN Test N.4 Test method for self-heating substances screen (one trial using a 100 mm cube at 140 °C)

7.13.1 Test Description

UN Test N.4, “Test method for self-heating substances”, is used to determine the ability of a substance to undergo oxidative self-heating. The sample is placed in a 100 cubic mm, stainless steel mesh sample container. The sample container is placed inside of a slightly larger stainless steel mesh cover box which hangs in the center of a larger stainless steel mesh cage box. The three boxes are placed in the center of an oven and heated at 140 °C for 24 hours. The temperature of the sample and oven are recorded throughout the test.

A substance is considered a self-heating substance if spontaneous ignition occurs or if the temperature of the sample exceeds the oven temperature by 60 °C.

7.13.2 Test Configuration

Each sample was filled to the brim of the 100mm cubic sample container. Thermocouples were inserted into the center of the test samples. The following photo shows a typical test configuration for UN Test N.4 in a 100mm cubic sample container suspended in a slightly larger stainless-steel cover box hanging in the center of a larger stainless-steel mesh cage box.

Photo149: Self-Heating Test Setup for a 100-mm Cube (typical)



7.13.3 Test Results

The test results are summarized in the following table and temperature plots.

Table 29: Summary of UN Series Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
2	Large-scale Mix ID #2	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)

Item	Sample	Conditions and Results	Assessment
3	Large-scale Mix ID #3	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
4	Large-scale Mix ID #4	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
5	Large-scale Mix ID #5	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
6	Large-scale Mix ID #6	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
7	Large-scale Mix ID #7	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)
8	Large-scale Mix ID #8	<u>100mm cube at 140°C</u> : Sample did not experience spontaneous ignition and temperature did NOT exceed that of the oven temperature during the 24-hour test.	Pass (NOT Division 4.2)

Figure 7: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #1

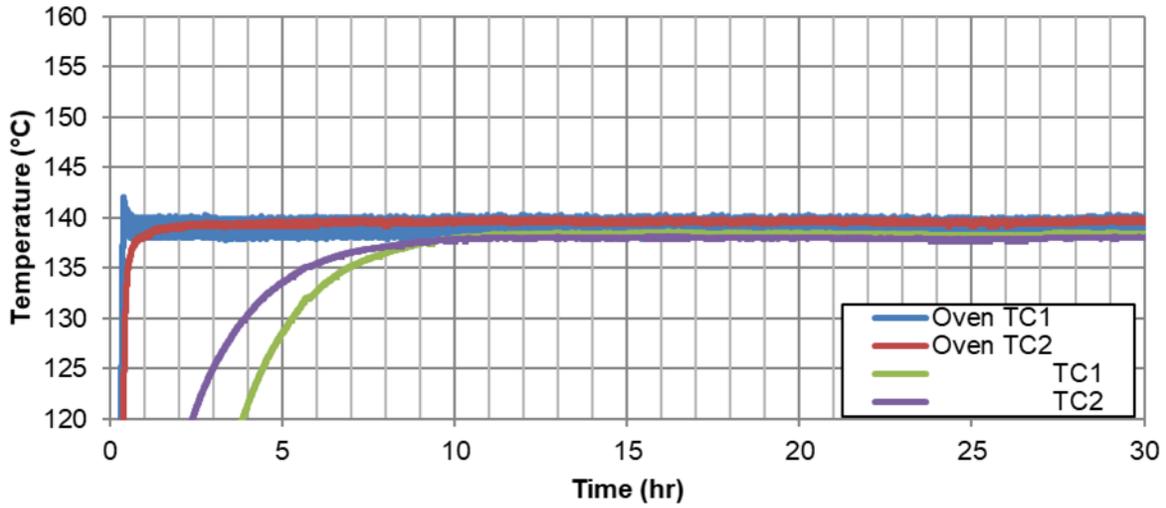


Figure 8: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #2

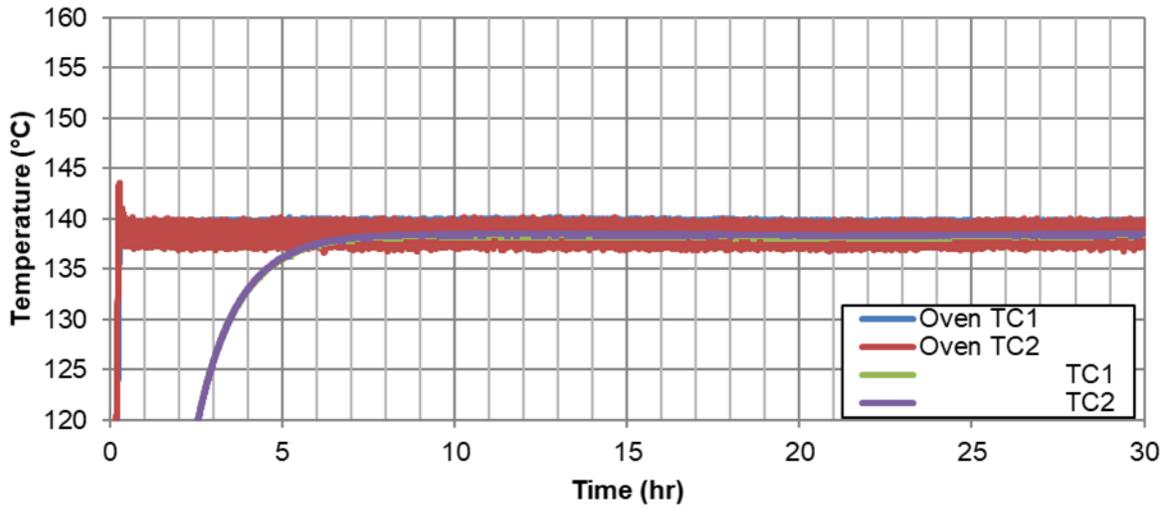


Figure 9: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #3

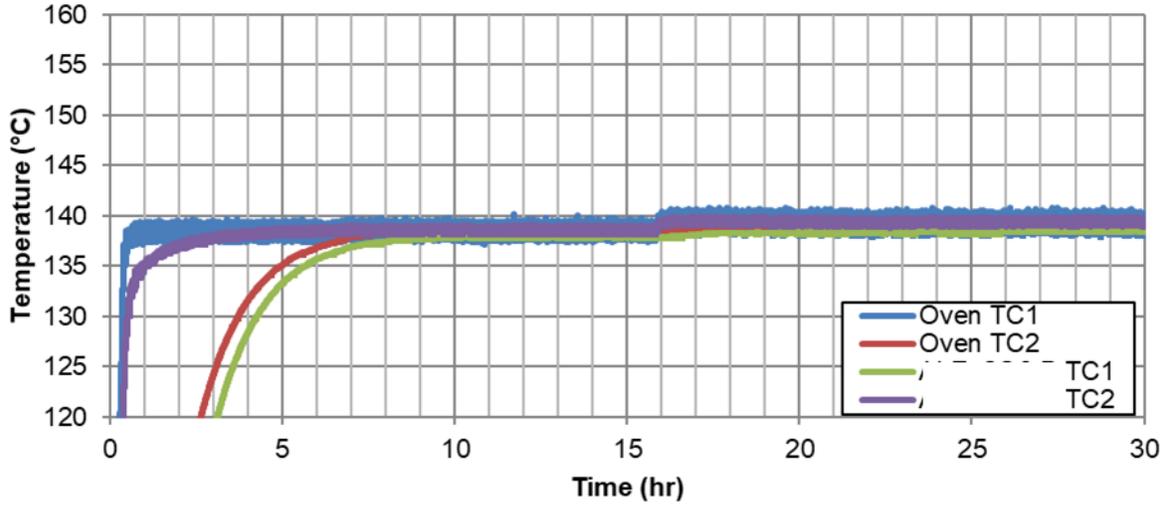


Figure 10: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #4

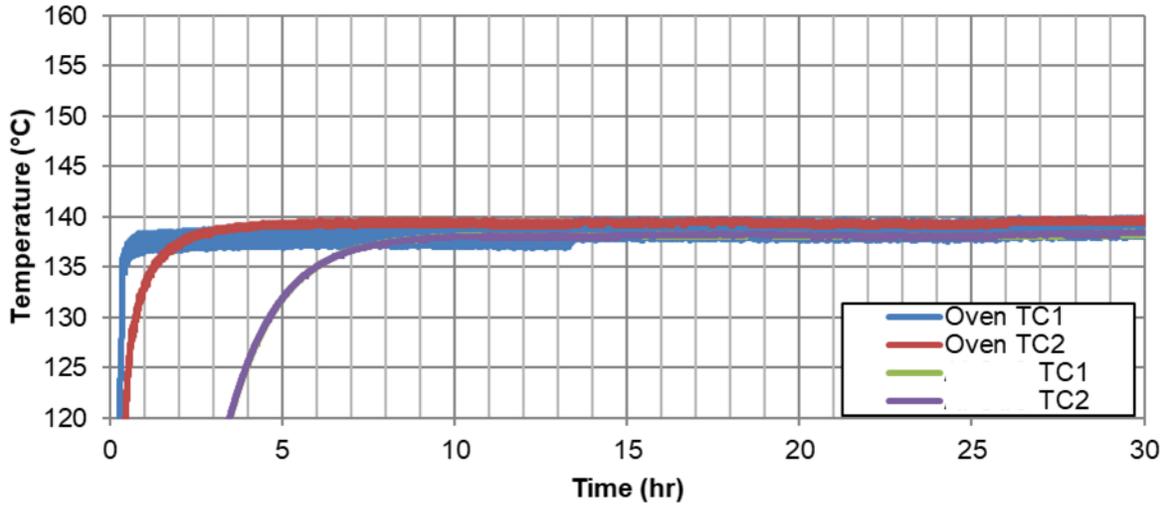


Figure 11: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #5

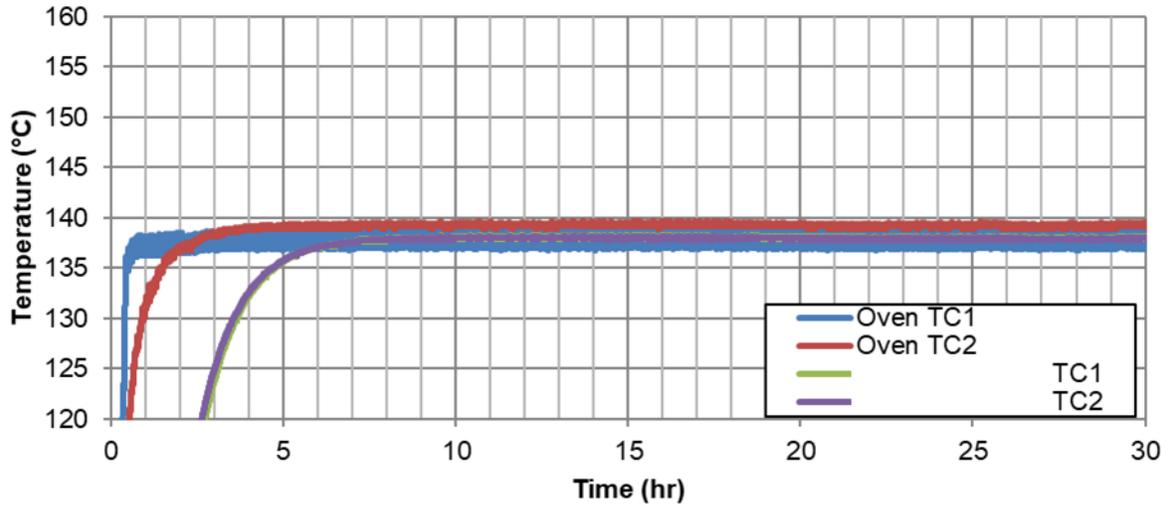


Figure 12: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #6

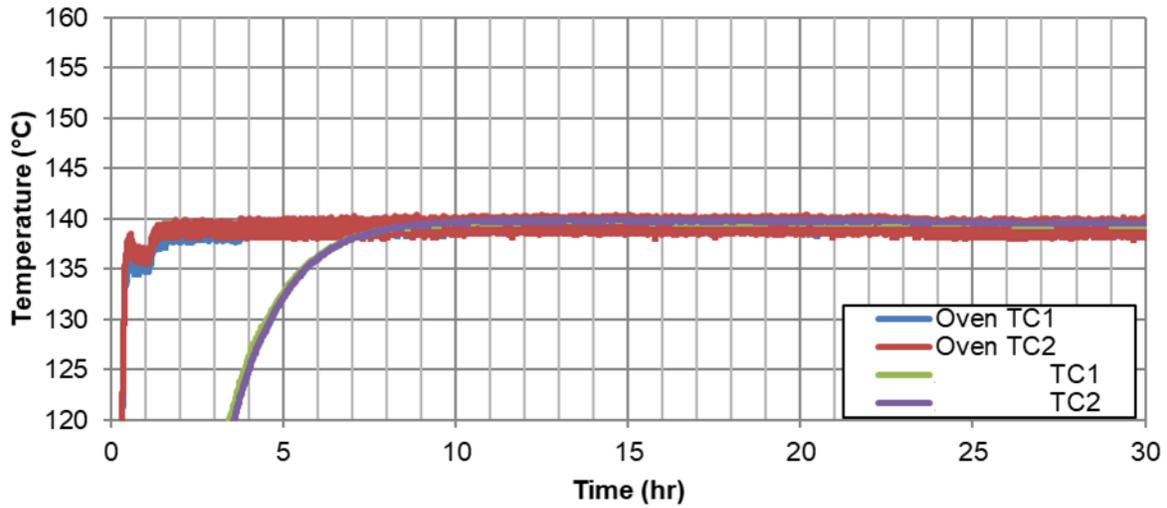


Figure 13: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #7

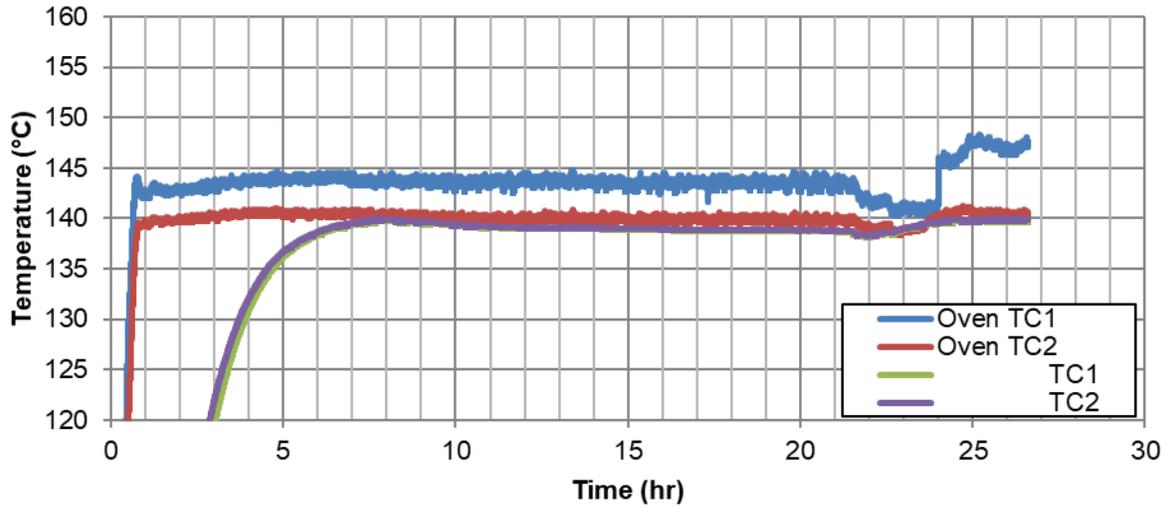
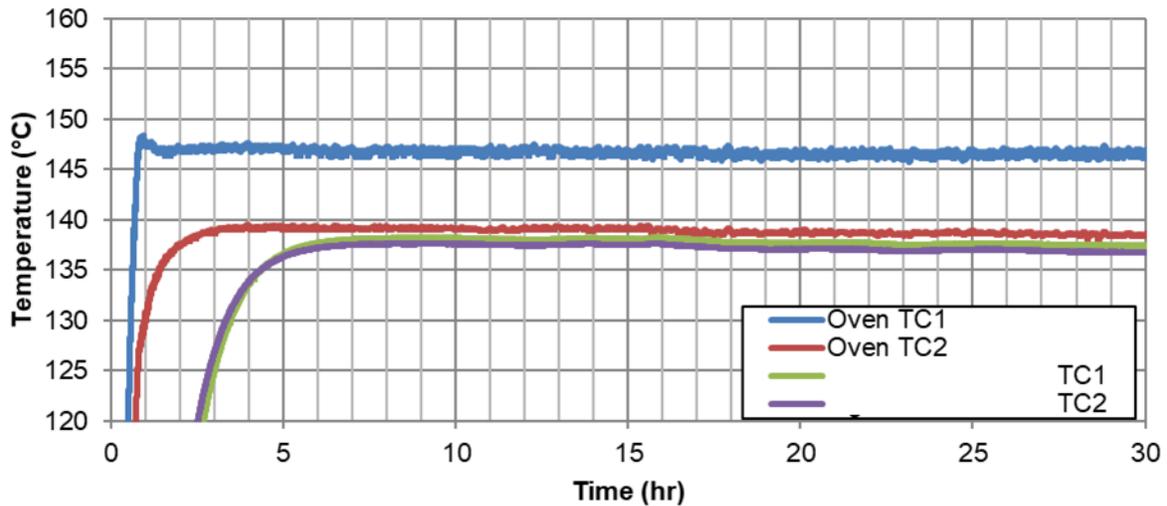


Figure 14: Oven Temperature Record for the UN Test N.4 Self-Heating Test on Large-scale Mix ID #8



7.13.4 Assessment of Test Results

Based on the test results, the samples passed UN Test N.4, “Test method for self-heating substances” and are NOT classified in Division 4.2.

7.14 UN Test N.5 Test method for substances which in contact with water emit flammable gases

7.14.1 Test Description

This test determines whether a substance which in contact with water will emit a significant quantity of flammable gases. The substance is tested by bringing it into contact with water under a variety of conditions provided by a series of safety tests and a quantification test.

The safety tests consist of the following:

- (1) a small quantity (approximately 2 mm diameter) of the test substance should be placed in a trough of distilled water at 20°C;
- (2) a small quantity of the test substance (approximately 2 mm diameter) should be placed on the center of a filter paper which is floated flat on the surface of distilled water at 20 °C in a suitable vessel; and
- (3) the test substance should be made into a pile approximately 20 mm high and 30 mm diameter with a hollow in the top. A few drops of water are added to the hollow.

In each of the tests, it is noted whether any gas is evolved; and if spontaneous ignition of the gas occurs.

Upon completion of the safety tests, the quantification test is performed. For solids, the sample is ground to a powder before testing if the substance is friable (i.e. readily crumbled; brittle) or if any particles of less than 500 µm diameter constitute more than 1% mass of the total weight (accounting for reduction[s] in particle size during handling and transport). Otherwise, as for liquids, the [solid] substance should be tested in its commercial state.

The quantification test should be performed three times at ambient temperature (20°C) and atmospheric pressure. Water is put into a dropping funnel and enough of the substance (up to a maximum mass of 25 g) to produce between 100 ml and 250 ml of gas is weighed and placed in a conical flask. The tap of the dropping funnel is opened to let the water into the conical flask and a stopwatch is started. The volume of gas evolved is measured by any suitable means.

The time taken for all the gas to be evolved is noted and where possible, intermediate readings are taken. The rate of evolution of gas is calculated over 7 hours at 1 hour intervals. If the rate of evolution is erratic or is increasing after 7 hours, the measuring time should be extended to a maximum time of 5 days. The five day test may be stopped if the rate of evolution becomes steady or continually decreases and sufficient data has been established to assign a packing group to the substance or to determine that the substance should not be classified in Division 4.3.

If the chemical identity of the gas is unknown, the gas should be tested for flammability.

NOTE: Per 49 CFR 173.124(c), a material should be classified as a Division 4.3 Dangerous when wet material if it by contact with water, is liable to become spontaneously flammable or to give off flammable or toxic gas at a rate greater than 1 L per kilogram of the material, per hour. Packing group I is assigned if the rate of evolution is 10 L/(kg·hr) or greater over any one minute period or the gas produced ignites spontaneously. Packing group II is assigned if the rate of evolution is 20 L/(kg·hr) or greater. Otherwise, Packing group III is assigned.

7.14.2 Test Configuration

For the safety tests, water was added to a small pile of each sample (~1 gram) contained in a small sample container.

For the quantification test, an Erlenmeyer filter flask, burette, graduated cylinder, and water reservoir were used to measure the volume of gas evolved, as shown in Figure 2. The graduated cylinder is filled with water and placed inverted in a reservoir of water. A 25-gram sample of the test substance is loaded into a 50-mL Erlenmeyer filter flask. Approximately 26-mL of water is added to the substance using a 100-mL glass burette seated in a rubber stopper (results in approximately 0.75-in standing water). Gas evolved by the sample is carried from the side port of the filter flask to the inverted graduated cylinder through a piece of natural rubber tubing. The volume of gas evolved is determined by the amount of water that was displaced out of the inverted graduated cylinder with time. The test configuration is shown in the following figure and photo.

Figure 15: Method for Measuring Volume of Gas Evolved

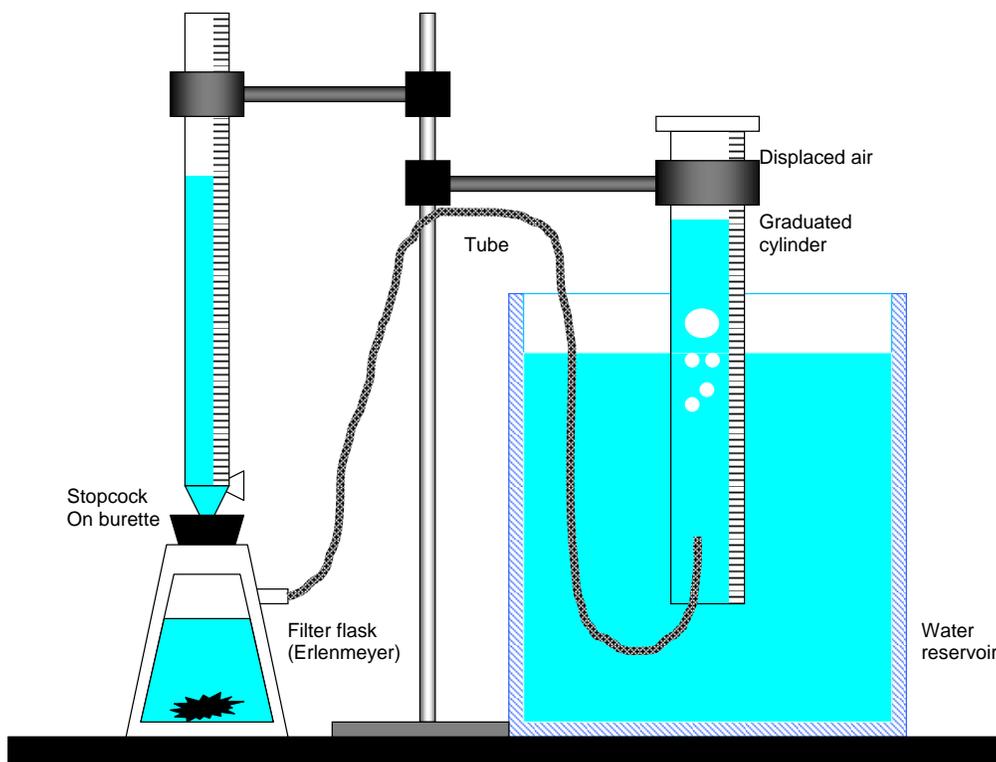


Photo 150: UN Test N.5 Test Setup (typical)



7.14.3 Test Results

Safety Tests

For each of the samples, addition of water to a small pile of each sample (~1 gram) contained in a small sample container did not result in any significant quantity of gas evolution and did not result in spontaneous ignition of the gas. The other safety tests were waived based on the results of this safety test.

Quantification Test

The collected test data is summarized in the following tables.

Table 30: UN Test N.5 Test Data for Large-scale Mix ID #1

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	14.5	 	
1	34	19.5	0.8
2	70 → 0	36	1.4
3	38	38	1.5
4	76	38	1.5
5	120 → 0	44	1.8
6	19	19	0.8
7	72	53	2.1

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	6	 	
1	11	5	1.0
2	25	14	2.8
3	32	7	1.4
4	40	8	1.6
5	50	10	2.0
6	60	10	2.0
7	76	16	3.2

Trial 3 with 25 mL water

Sample: 10 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	50	 	
1	70	20	2.0
2	89	19	1.9
3	96	7	0.7
4	107	11	1.1
5	115	8	0.8
6	124	9	0.9
7	126	2	0.2

Table 31: UN Test N.5 Test Data for Large-scale Mix ID #2

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	19	0	0
1	19	0	0
2	19	0	0
3	19	0	0
4	19	0	0
5	19	0	0
6	19	0	0
7	19	0	0

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	1	0	0
1	1	0	0
2	1	0	0
3	1	0	0
4	1	0	0
5	1	0	0
6	1	0	0
7	1	0	0

Trial 3 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	2	0	0
1	2	0	0
2	2	0	0
3	2	0	0
4	2	0	0
5	2	0	0
6	2	0	0
7	2	0	0

Table 32: UN Test N.5 Test Data for Large-scale Mix ID #3

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	20		
1	20	0	0
2	20	0	0
3	20	0	0
4	20	0	0
5	20	0	0
6	20	0	0
7	20	0	0

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	2		
1	2	0	0
2	2	0	0
3	2	0	0
4	2	0	0
5	2	0	0
6	2	0	0
7	2	0	0

Trial 3 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	20		
1	20	0	0
2	21	1	0
3	22	1	0
4	22	0	0
5	23	1	0
6	24	1	0
7	24	0	0

Table 33: UN Test N.5 Test Data for Large-scale Mix ID #4

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	12.5	 	
1	20	7.5	0.3
2	24	4	0.2
3	27.5	3.5	0.1
4	34	6.5	0.3
5	45	11	0.4
6	49	4	0.2
7	62	13	0.5

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	3	 	
1	4	1	0.2
2	5	1	0.2
3	5	0	0.0
4	7	2	0.4
5	9	2	0.4
6	11	2	0.4
7	13	2	0.4

Trial 3 with 25 mL water

Sample: 10 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	3	 	
1	4	1	0.1
2	5	1	0.1
3	5	0	0.0
4	7	2	0.2
5	9	2	0.2
6	11	2	0.2
7	13	2	0.2

Table 34: UN Test N.5 Test Data for Large-scale Mix ID #5

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	14	0	0
1	14	0	0
2	14	0	0
3	14	0	0
4	14	0	0
5	15	1	0.04
6	15	0	0
7	15	0	0

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	4	0	0.0
1	4	0	0.0
2	4	0	0.0
3	4	0	0.0
4	5	1	0.2
5	5	0	0.0
6	6	1	0.2
7	6	0	0.0

Trial 3 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	25	0	0
1	25	0	0
2	25	0	0
3	25	0	0
4	25	0	0
5	25	0	0
6	25	0	0
7	25	0	0

Table 35: UN Test N.5 Test Data for Large-scale Mix ID #6

Trial 1 with 25 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	27	 	
1	34	7	1.4
2	36	2	0.4
3	39	3	0.6
4	42	3	0.6
5	43	1	0.2
6	44	1	0.2
7	47	3	0.6

Trial 2 with 15 mL water

Sample: 15 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	14	 	
1	34	20	1.3
2	45	11	0.7
3	55	10	0.7
4	60	5	0.3
5	69	9	0.6
6	77	8	0.5
7	90	13	0.9

Trial 3 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	28	 	
1	68	40	1.6
2	90	22	0.9
3	110	20	0.8
4	132	22	0.9
5	145	13	0.5
6	158	13	0.5
7	186	28	1.1

Table 36: UN Test N.5 Test Data for Large-scale Mix ID #7

Trial 1 with 25 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	18	 	
1	25	7	1.4
2	29	4	0.8
3	33	4	0.8
4	38	5	1.0
5	40	2	0.4
6	43	3	0.6
7	49	6	1.2

Trial 2 with 15 mL water

Sample: 15 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	30	 	
1	31	1	0.1
2	31	0	0.0
3	31	0	0.0
4	31	0	0.0
5	31	0	0.0
6	31	0	0.0
7	31	0	0.0

Trial 3 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	33	 	
1	35	2	0.1
2	35	0	0.0
3	35	0	0.0
4	35	0	0.0
5	35	0	0.0
6	35	0	0.0
7	35	0	0.0

Table 37: UN Test N.5 Test Data for Large-scale Mix ID #8

Trial 1 with 25 mL water

Sample: 25 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	45	 	
1	50 → 12	17	0.7
2	16	4	0.2
3	17	1	0.1
4	17	0	0
5	17	0	0
6	17	0	0
7	17	0	0

Trial 2 with 15 mL water

Sample: 5.0 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	5	 	
1	16	11	2.2
2	17	1	0.2
3	18	1	0.2
4	19	1	0.2
5	20	1	0.2
6	20	0	0.0
7	20	0	0.0

Trial 3 with 25 mL water

Sample: 10 gm

Time (hr)	Total (mL)	Delta (mL)	Rate L/(kg·hr)
0	80	 	
1	83	3	0.3
2	88	5	0.5
3	94	6	0.6
4	95	1	0.1
5	96	1	0.1
6	97	1	0.1
7	97	0	0.0

The test results are summarized in the following table.

Table 38: Summary of UN Test N.1 Test Results

Item	Sample	Conditions and Results	Assessment
1	Large-scale Mix ID #1	Maximum gas emission rate of 2.8 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)
2	Large-scale Mix ID #2	No significant gas evolution over 7-hour test period for 25 grams of thermite.	Pass (NOT Division 4.3)
3	Large-scale Mix ID #3	No significant gas evolution over 7-hour test period for 25 grams of thermite.	Pass (NOT Division 4.3)
4	Large-scale Mix ID #4	Maximum gas emission rate of 0.5 L/(kg·hr) without spontaneous ignition.	Pass (NOT Division 4.3)
5	Large-scale Mix ID #5	Maximum gas emission rate of 0.2 L/(kg·hr) without spontaneous ignition.	Pass (NOT Division 4.3)
6	Large-scale Mix ID #6	Maximum gas emission rate of 1.6 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)
7	Large-scale Mix ID #7	Maximum gas emission rate of 1.4 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)
8	Large-scale Mix ID #8	Maximum gas emission rate of 2.2 L/(kg·hr) without spontaneous ignition.	FAIL (consistent with Division 4.3 PG III)

Gas Analysis

Based on the known composition of each sample, the evolved gas is expected to be spontaneously flammable (hydrogen); further chemical analysis was not performed.

The test results are shown in the following photos.

Photo 151: UN Test N.5 Test Results for Large-scale Mix ID #1



Photo 152: UN Test N.5 Test Results for Large-scale Mix ID #2

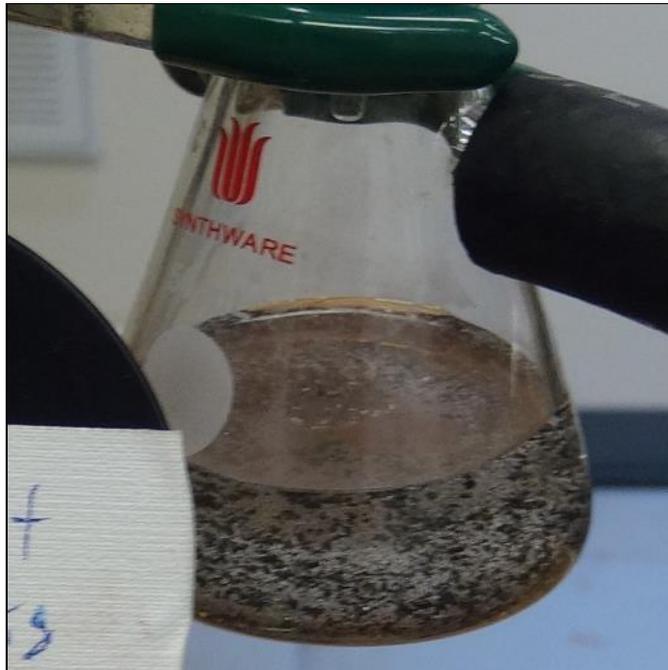


Photo 153: UN Test N.5 Test Results for Large-scale Mix ID #3

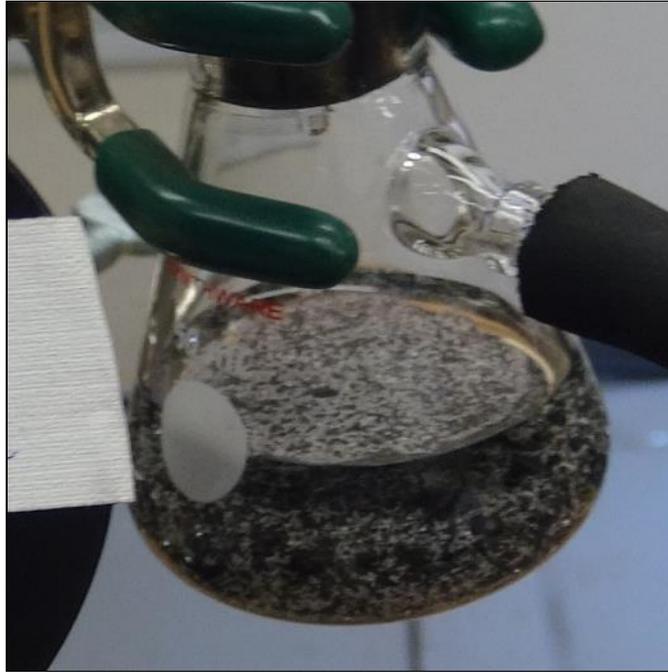


Photo 154: UN Test N.5 Test Results for Large-scale Mix ID #4

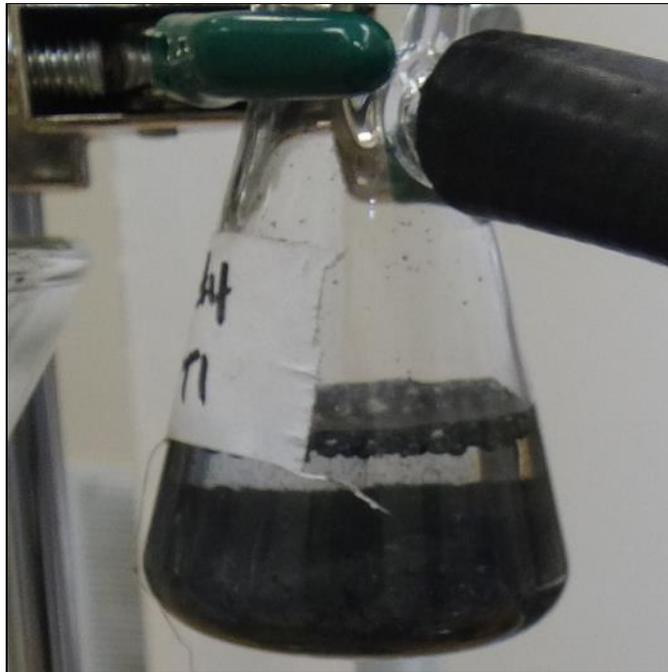


Photo 155: UN Test N.5 Test Results for Large-scale Mix ID #5



Photo 156: UN Test N.5 Test Results for Large-scale Mix ID #6



Photo 157: UN Test N.5 Test Results for Large-scale Mix ID #7

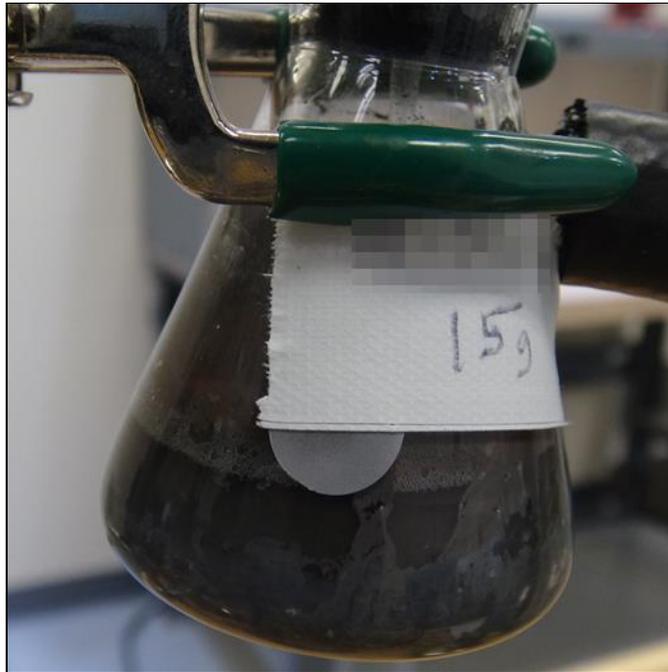


Photo 158: UN Test N.5 Test Results for Large-scale Mix ID #8



7.14.4 Assessment of Test Results

A substance is classified in Division 4.3 if either (a) spontaneous ignition takes place in any step of the test procedure; or (b) there is an evolution of a flammable gas at a rate greater than 1 liter per kilogram of the substance per hour.

For each of the test samples, spontaneous ignition did not take place. There was no evolution of a gas at a rate equal to or greater than 20 liters per kilogram of the substance per hour for Thermite Large-scale Mix ID's #1, #6, #7, and #8 and greater than 1 liter per kilogram of the substance per hour for Thermite Large-scale Mix ID's #2, #3, #4, and #5. Based on these test results, Thermite Large-scale Mix ID's #1, #6, #7, and #8 present a hazard consistent with Division 4.3 PG III; Thermite Large-scale Mix ID's #2, #3, #4, and #5 passed UN Test N.5, "Test Method for Substances which in Contact with Water Emit Flammable Gases" and are NOT classified in Division 4.3.

7.15 Hotwire auto-ignition temperature screening test

7.15.1 Test Description

This screening test determines the approximate temperature at which a substance is ignited by a hotwire. The substance is placed on an impervious, non-combustible, low heat-conducting surface over a heating resistance wire instrumented with thermocouples. The temperature of a heating resistance wire is increased until the substance ignites or until the maximum test temperature is reached. Normally two trials are performed.

7.15.2 Test Configuration

Each end of a 105-mm long, 18-gauge 80/20 nickel/chromium (ni-chrome) resistance heating wire (Type A) was attached to electrodes suspended just above an insulating clay-based firebrick. Two quick-responding thermocouples (30 gauge) were wrapped over the heating wire; approximately 5 grams of the test sample was poured over the heating wire and thermocouples, as shown in the following photo. The resistance heating wire could achieve a maximum test temperature of 1200°C. Generally, each substance was tested with a fast heating rate (increasing the amperage approximately 1 amp every 10 seconds) and a slow heating rate (increasing the amperage approximately 1 amp every 30 seconds). NOTE: Type K thermocouples were normally utilized; when Type B thermocouples were utilized, the actual temperature of the sample was not measured until the temperature was above 100°C (minimum temperature measurement limit).

Photo 159: Hotwire Auto-ignition Temperature Apparatus with Dual Thermocouples

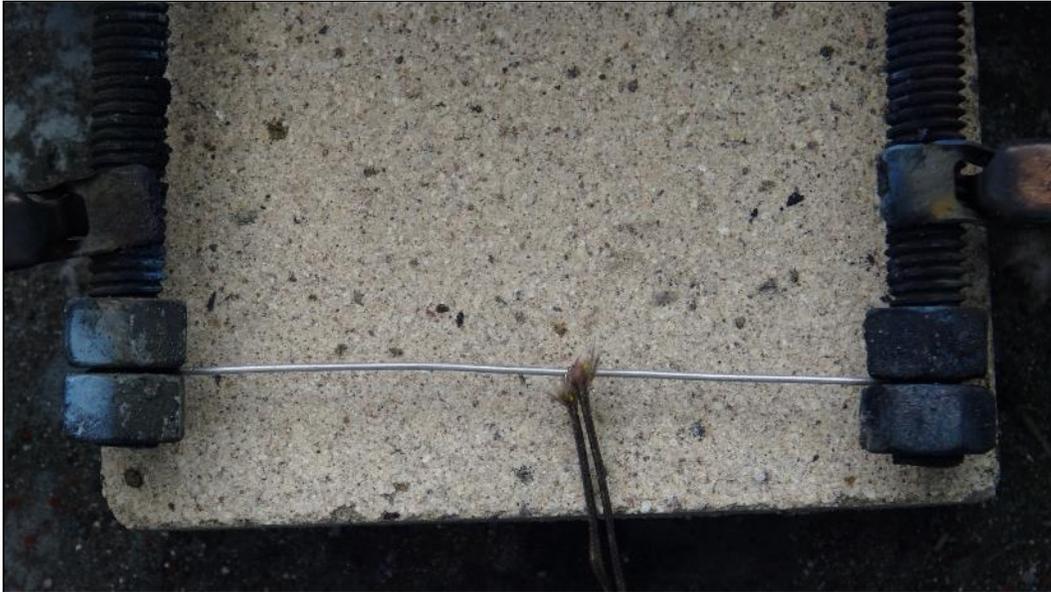


Photo 160: Test Sample Loaded onto Hotwire Auto-ignition Temperature Apparatus



7.15.3 Test Results

The test results are summarized in the following table. NOTE: The auto-ignition temperature of Small-scale Mix #26 (Large-scale Mix #2) exceeded that of the ni-chrome heating wire (>1200 °C).

NOTE: Originally, high-energy thermites utilizing beryllium as their fuel were to be tested for auto-ignition temperature. However, titanium was substituted for beryllium due to the

potential health hazard of beryllium to test personnel in very small quantities (toxic by inhalation).

Table 39: Summary of Auto-Ignition Temperature Test Results for Small-Scale Mixes

Small-Scale Mix ID	Ignition 1st Trial (°C)	Ignition 2nd Trial (°C)	Reaction Type
1	440 [TC1] 425 [TC2]	595 [TC1] 610 [TC2]	Burned for 2 seconds
2B	1100 [TC1]	1030 [TC1] 1040 [TC2]	Very slow, subtle burn
3B	670 [TC1]	780 [TC1] 740 [TC2]	Exploded
4	425 [TC1]	480 [TC1]	Exploded
6B	630 [TC1]	790 [TC1] 840 [TC2]	Burned for 1 - 2 seconds
7B	560 [TC1]	640 [TC1] 540 [TC2]	Low-order explosion (burned <0.1 seconds)
9	540 [TC1] 540 [TC2]	550 [TC1] 595 [TC2]	Exploded
10	510 [TC1] 420 [TC2]	470 [TC1] 430 [TC2]	Burned for 1 - 2 seconds
11	460 [TC1] 485 [TC2]	575 [TC1] 480 [TC2]	Burned for 1 second
12	1210 [TC1] 1060 [TC2]	1130 [TC1] 1090 [TC2]	Burned for 2 - 3 seconds
18	980 [TC1] --- [TC2]	--- [TC1] ~980 [TC2]	Sporadic thermal behavior; burned for 3 seconds
19	775 [TC1] 860 [TC2]	900 [TC1] 860 [TC2]	Exploded
21	~400 [TC1]	~590 [TC1] ~690 [TC2]	Endotherm preceding exotherm; slow reaction
24	560 [TC1]	710 [TC1] 650 [TC2]	Exploded
25	800 [TC1] 850 [TC2]	1010 [TC1] 990 [TC2]	Burned for 20 seconds

Table 40: Summary of Auto-Ignition Temperature Test Results for Large-Scale Mixes

Large-Scale Mix ID	Small-Scale Mix ID	Ignition Trial 1 (°C)	Ignition Trial 2 (°C)	Reaction Type
1	14	980 [TC1] --- [TC2]	965 [TC1] 920 [TC2]	Low-order explosion (burned <0.1 seconds)

Large-Scale Mix ID	Small-Scale Mix ID	Ignition Trial 1 (°C)	Ignition Trial 2 (°C)	Reaction Type
2	26	>1200 [TC1]	n/a	Unable to ignite using 18-gauge ni-chrome wire
3	27	1100 [TC1]	>1050 [TC1] >1050 [TC2]	Burned for 2 seconds
4	13	815 [TC1] 800 [TC2]	970 [TC1] 1140 [TC2]	Burned for 3 seconds
5	28	~750 [TC1]	1180 [TC1] 1160 [TC2]	Burned for 1 - 2 seconds
6	5	~880 [TC1]	915 [TC1] 955 [TC2]	Burned for 15 seconds
7	8	915 [TC1] 915 [TC2]	1140 [TC1] 1150 [TC2]	Exploded
8	29	860 [TC1] 800 [TC2]	600 [TC1] 690 [TC2]	Exploded

Figure 16: Auto-ignition Temperature Plots for Small-Scale Mix ID #1

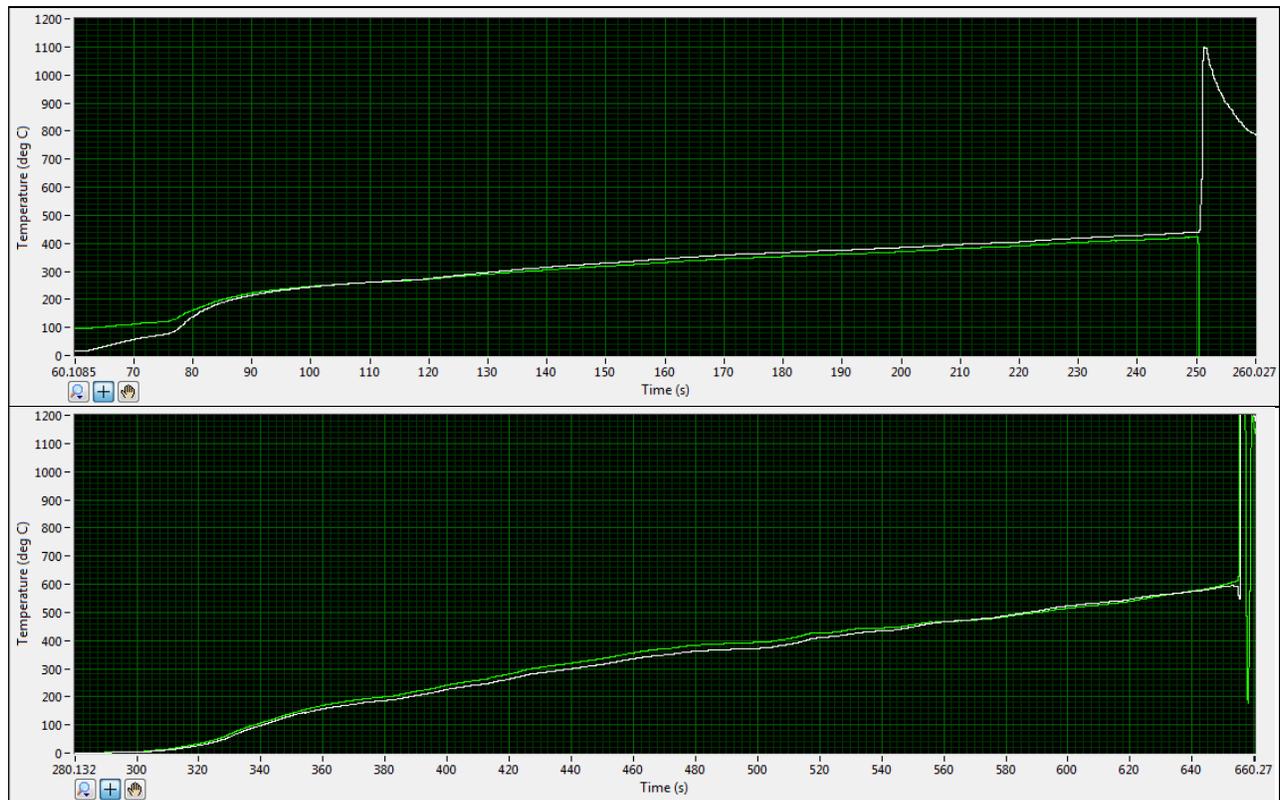


Figure 17: Auto-ignition Temperature Plots for Small-Scale Mix ID #2B

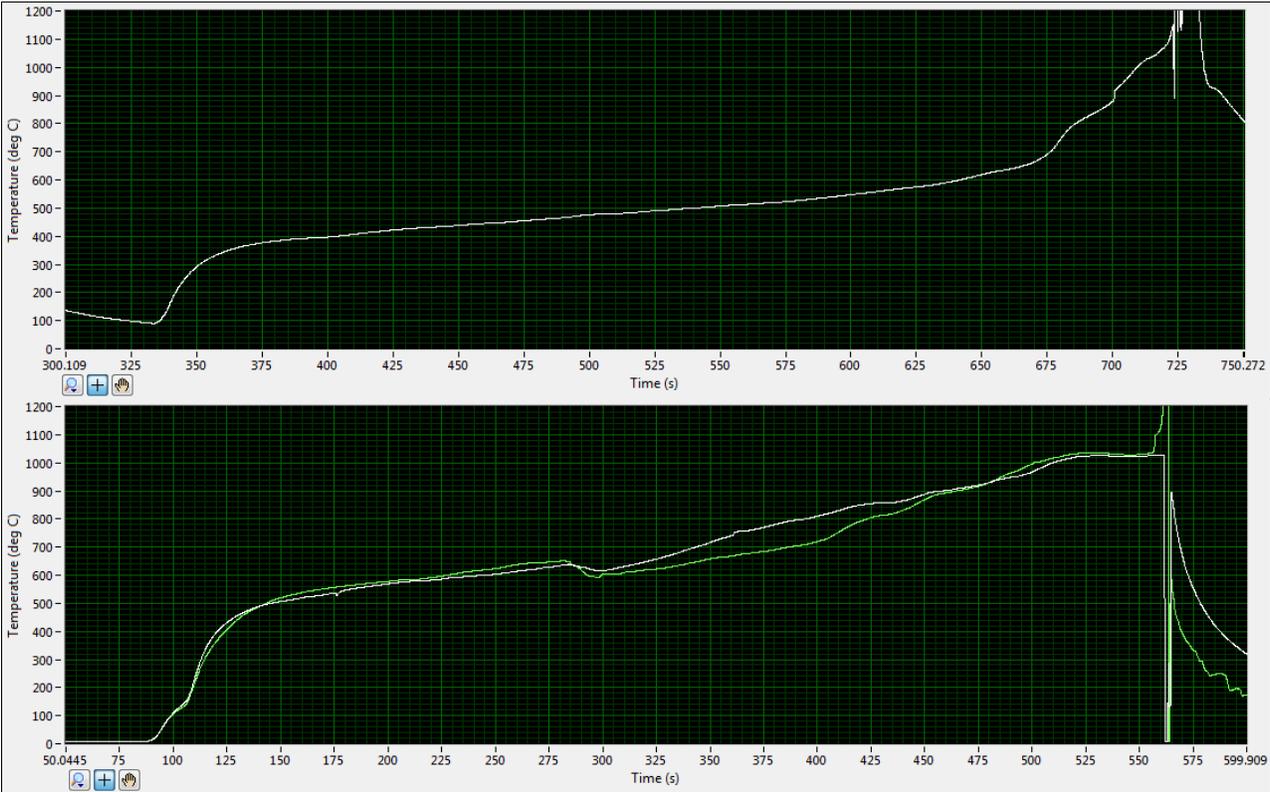
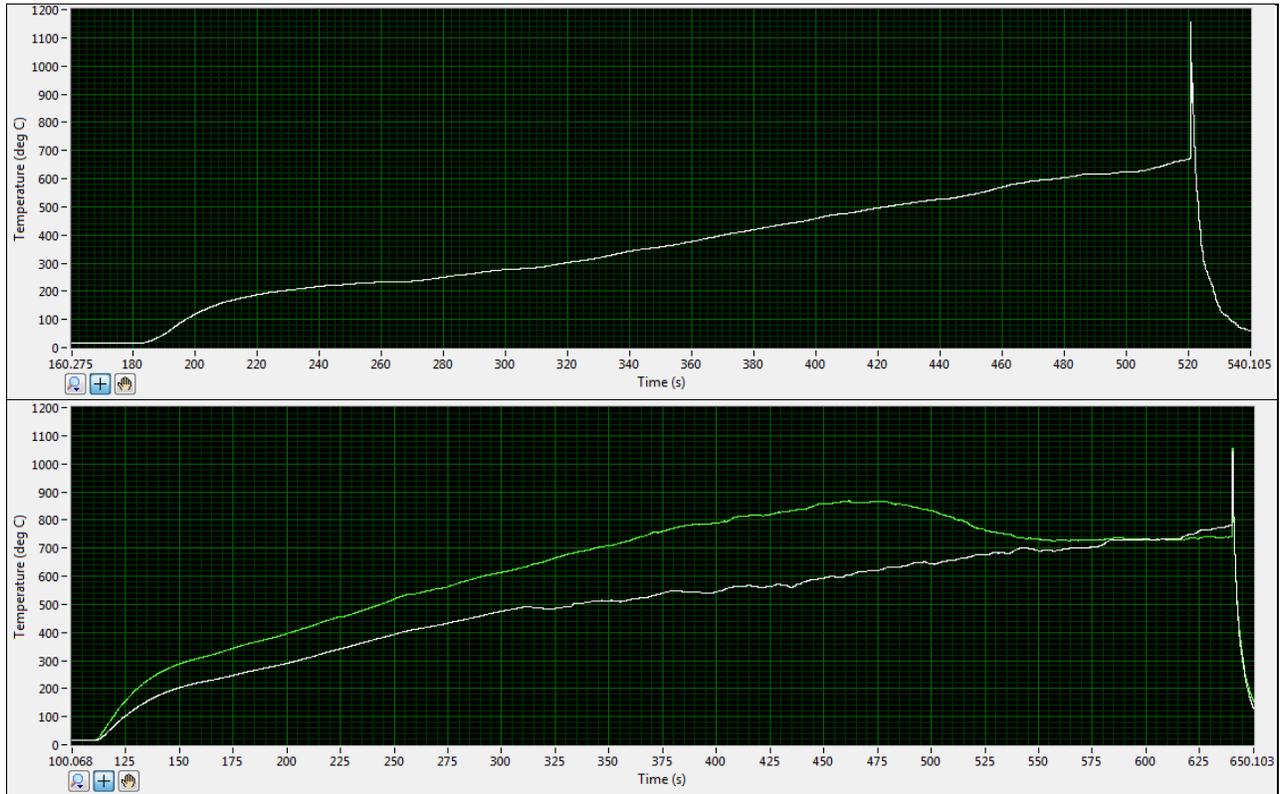


Figure 18: Auto-ignition Temperature Plots for Small-Scale Mix ID #3B



NOTE: For the second trial on this mix, one of the thermocouples unexpectedly departs from the baseline and then returns prior to auto-ignition of the sample; the cause for the departure is unknown.

Figure 19: Auto-ignition Temperature Plots for Small-Scale Mix ID #4

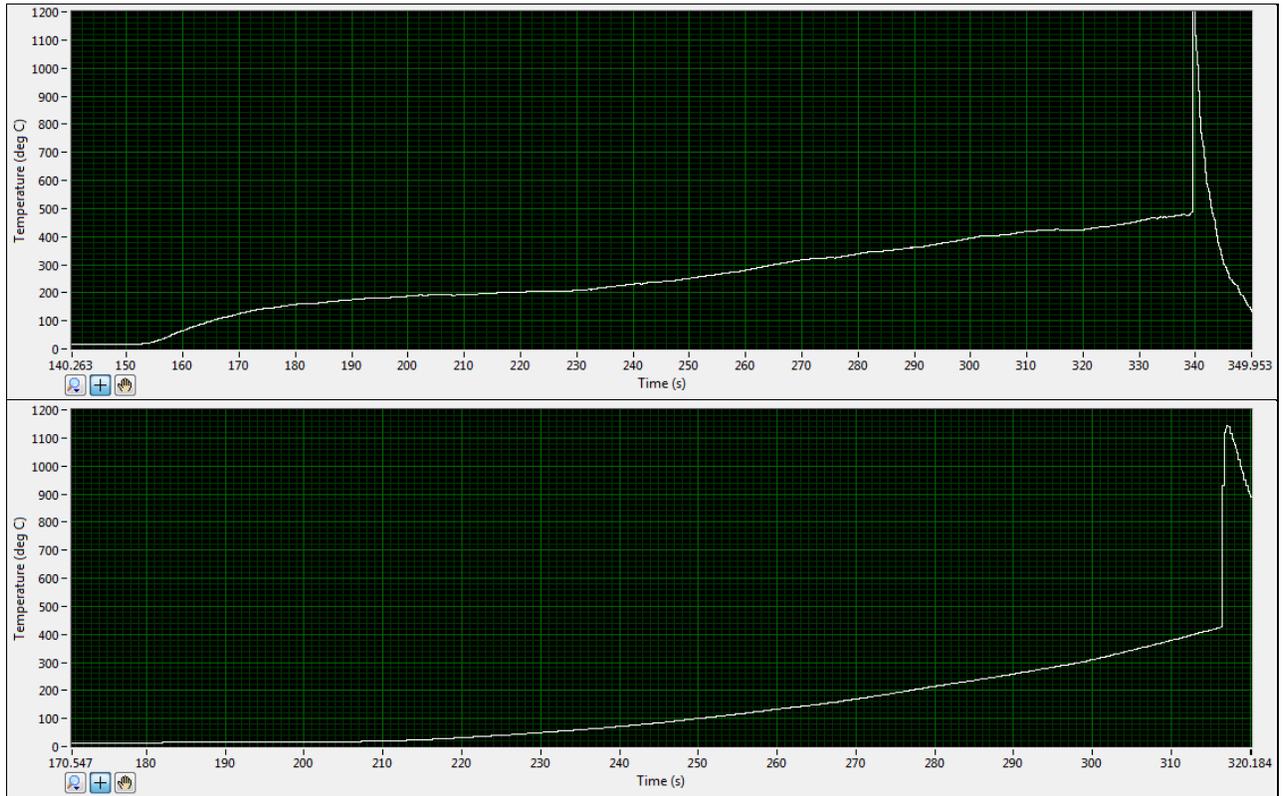
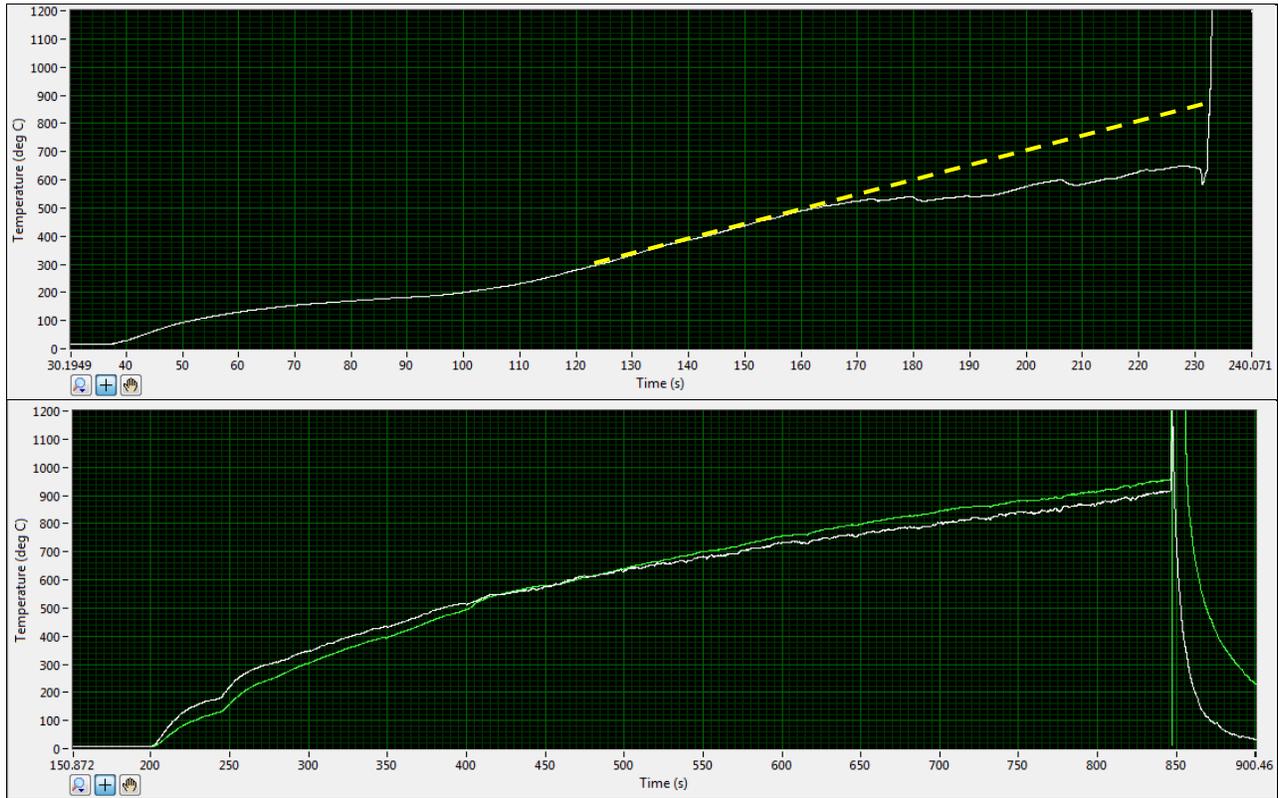


Figure 20: Auto-ignition Temperature Plots for Small-Scale Mix ID #5



NOTE: For this mix, one of the thermocouples unexpectedly departs from the baseline; a dashed line has been added to smooth out this departure. The cause for the departure is unknown but could be due to the thermocouple detaching from the ni-chrome heating wire as it experiences greater elongation and deformation at higher temperatures.

Figure 21: Auto-ignition Temperature Plots for Small-Scale Mix ID #6B

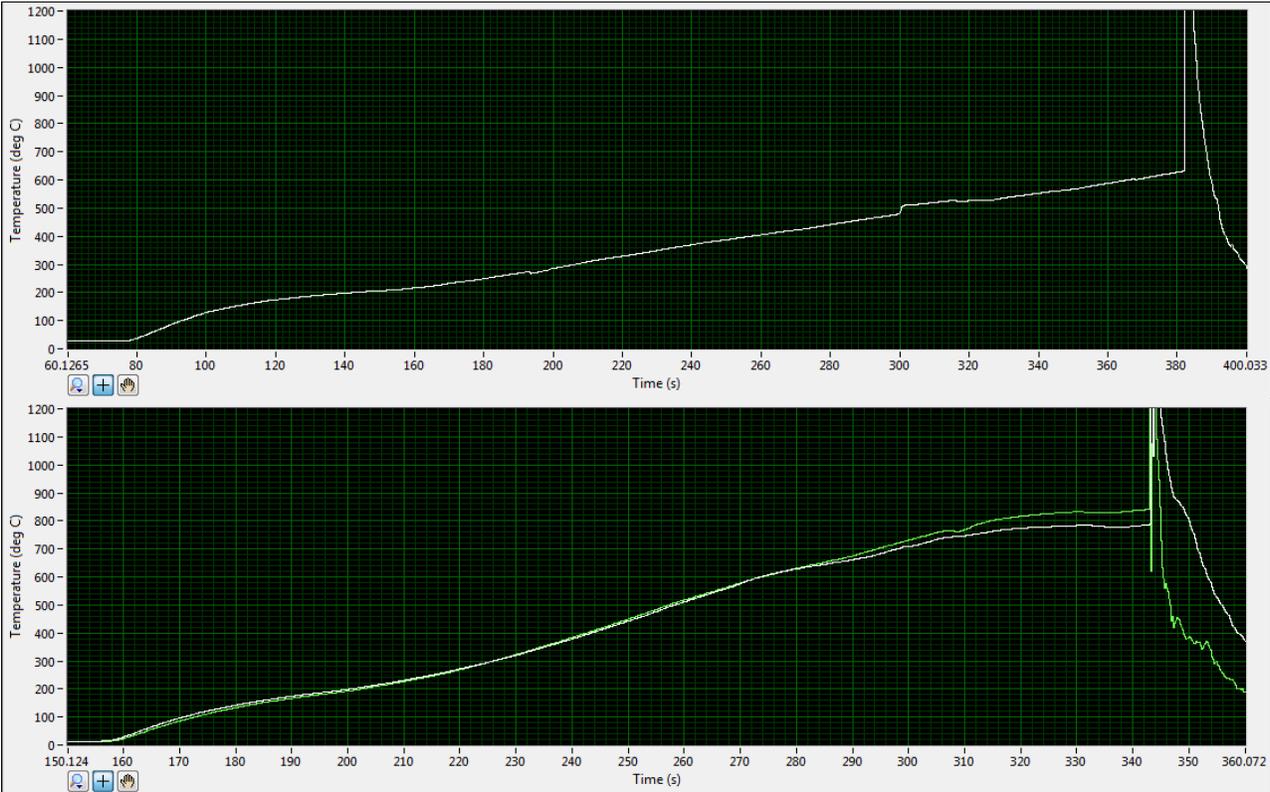


Figure 22: Auto-ignition Temperature Plots for Small-Scale Mix ID #7B

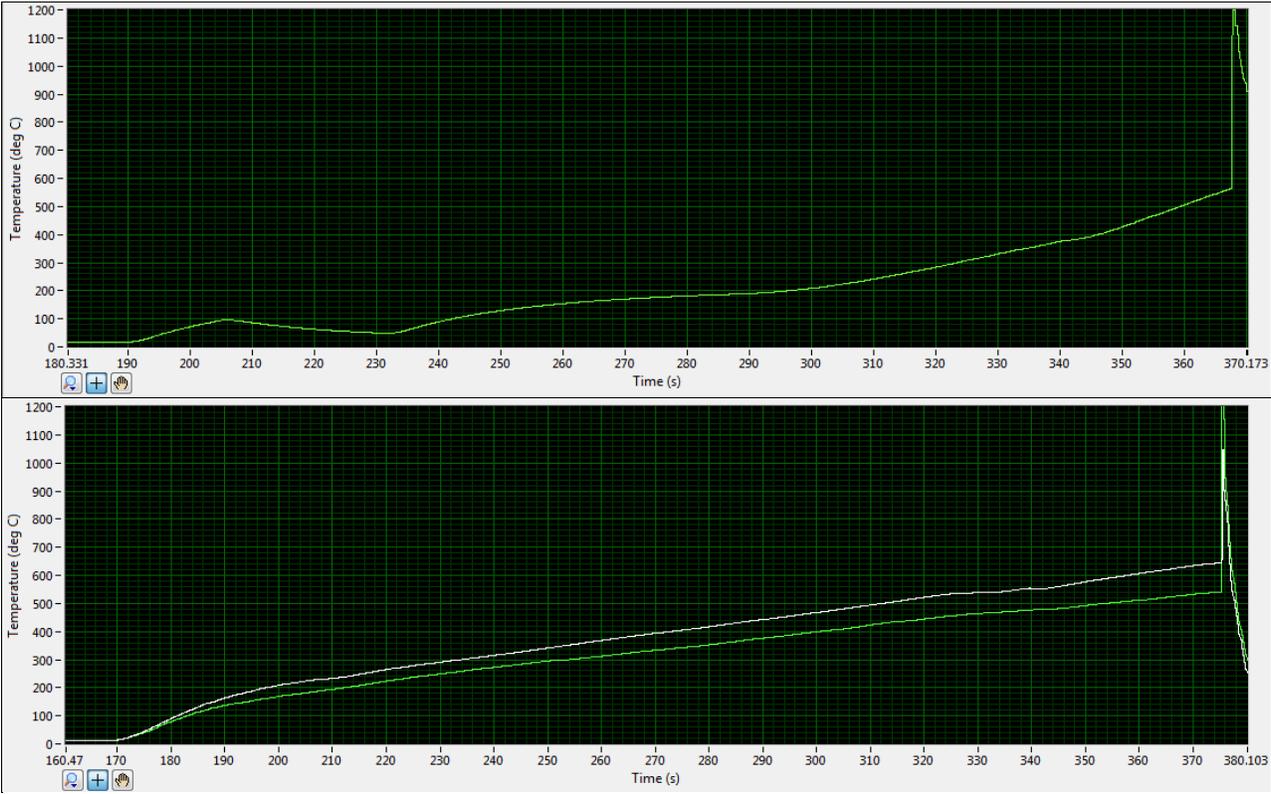


Figure 23: Auto-ignition Temperature Plots for Small-Scale Mix ID #8

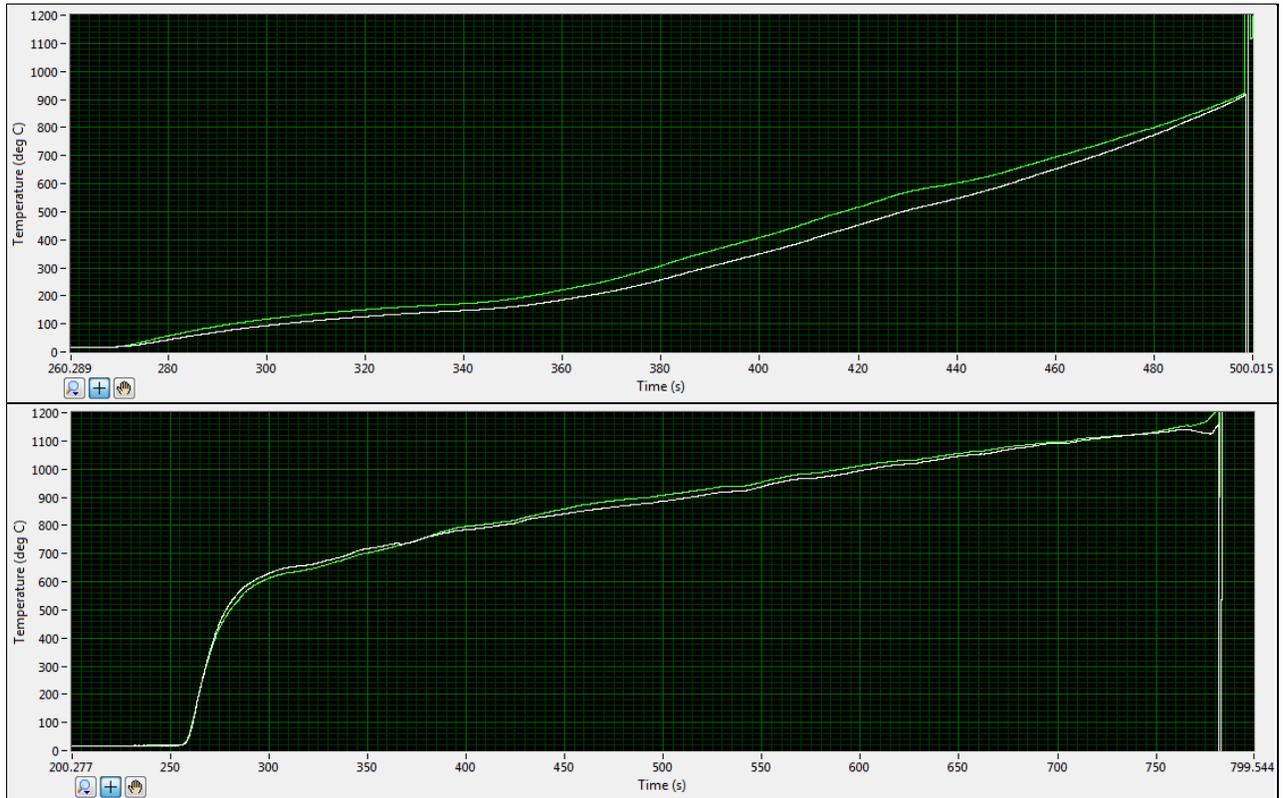


Figure 24: Auto-ignition Temperature Plots for Small-Scale Mix ID #9

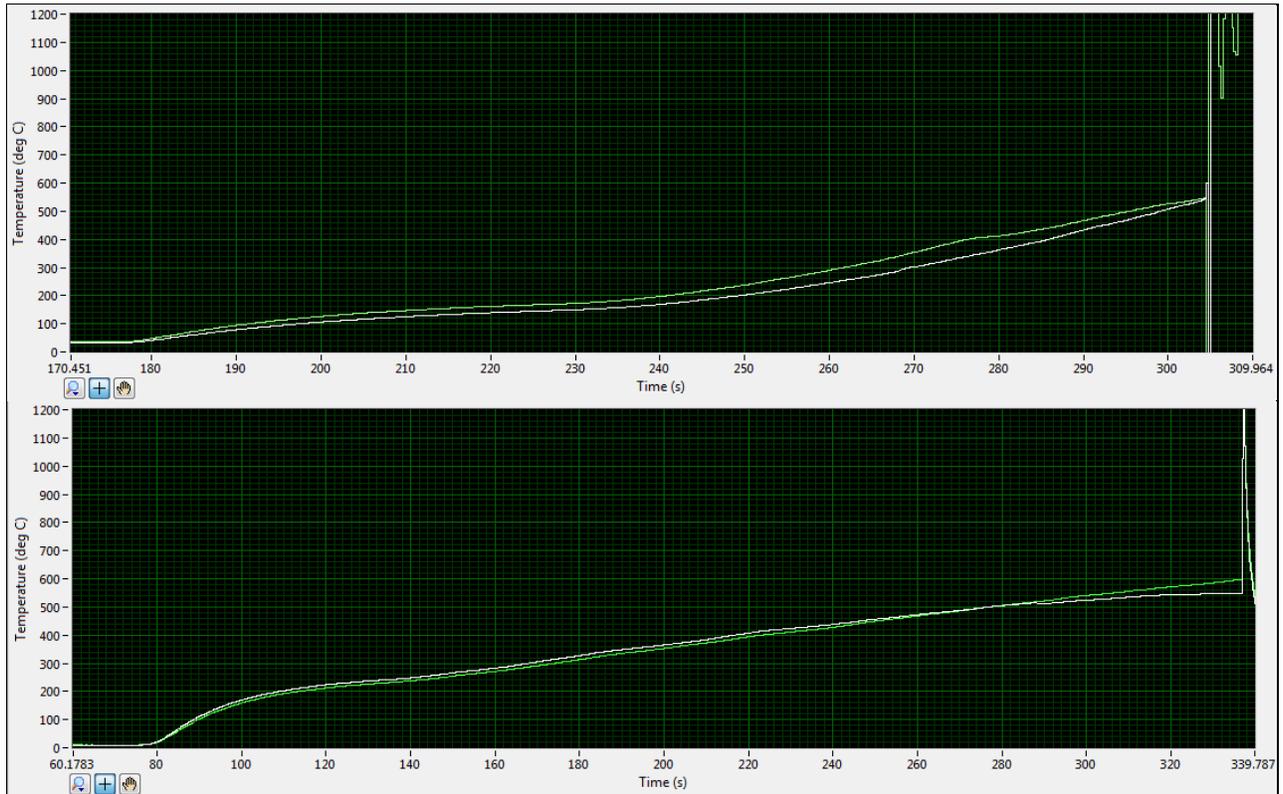


Figure 25: Auto-ignition Temperature Plots for Small-Scale Mix ID #10

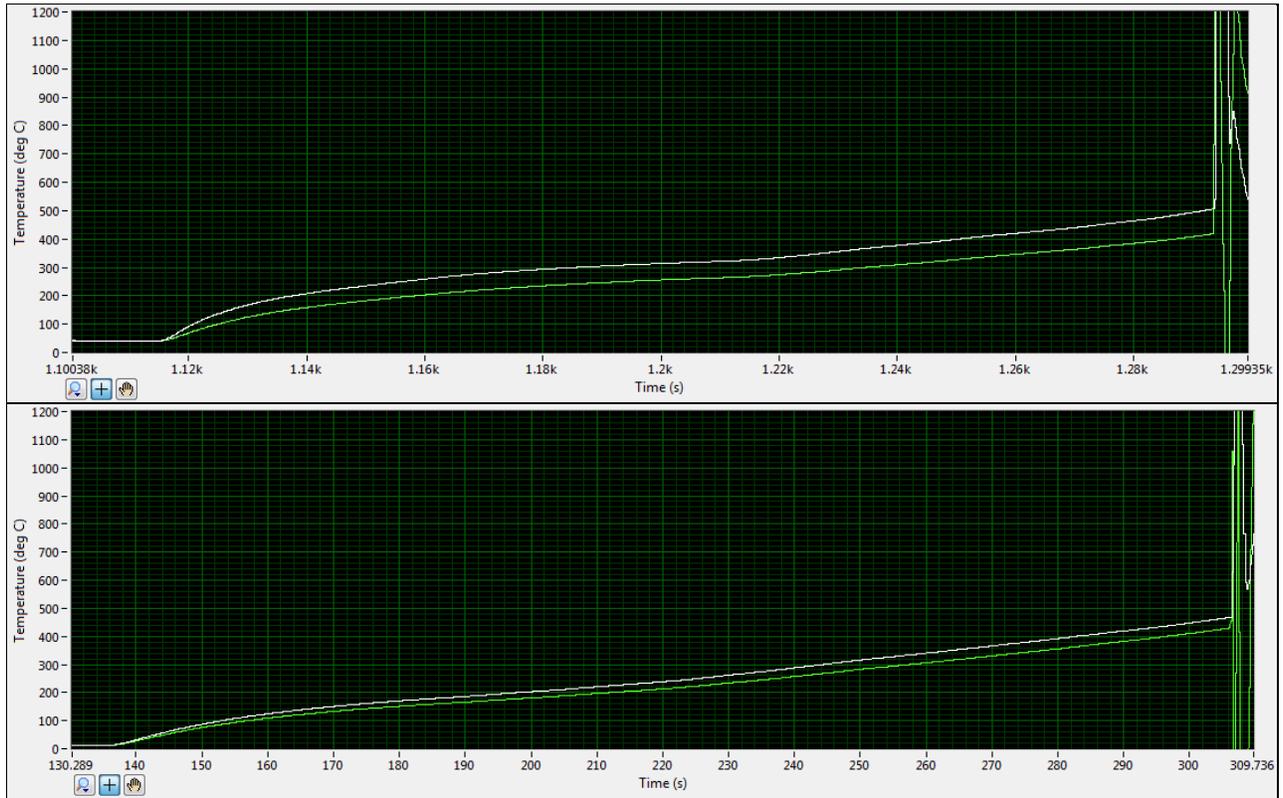


Figure 26: Auto-ignition Temperature Plots for Small-Scale Mix ID #11

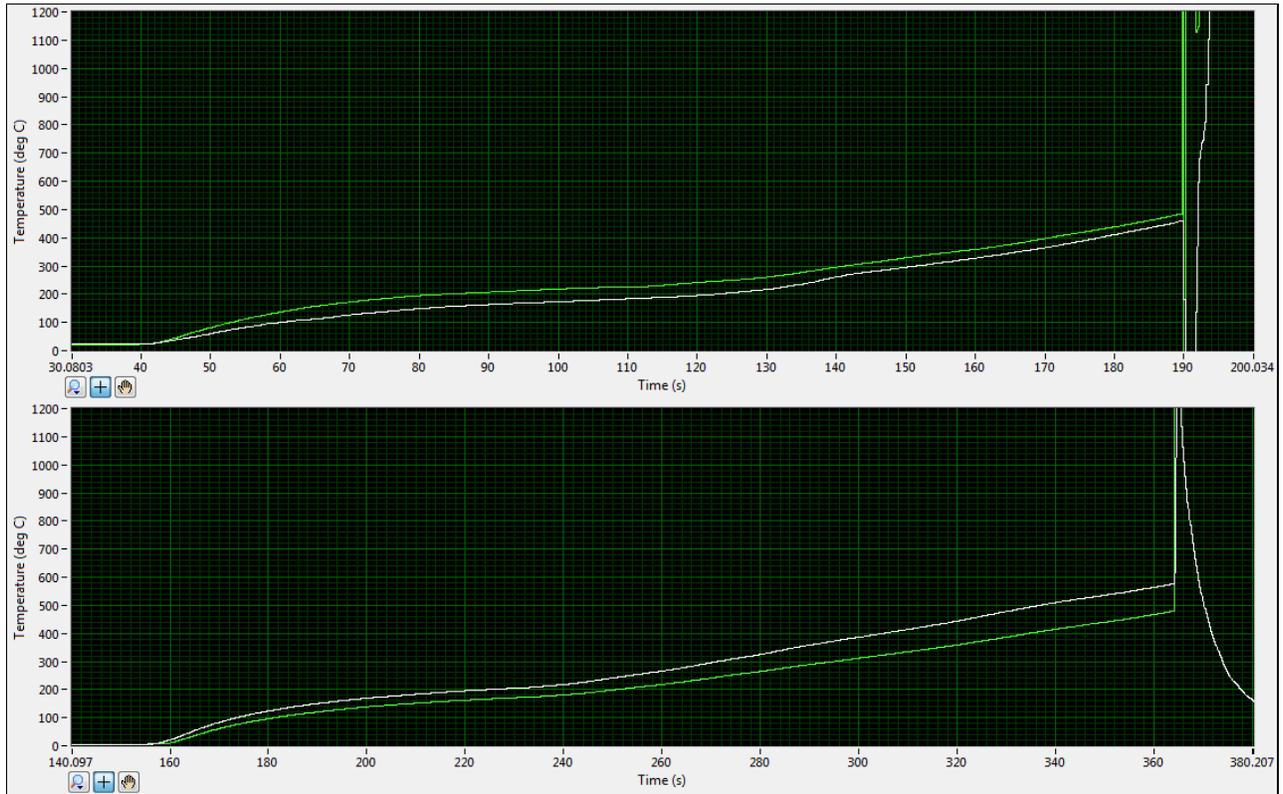


Figure 27: Auto-ignition Temperature Plots for Small-Scale Mix ID #12

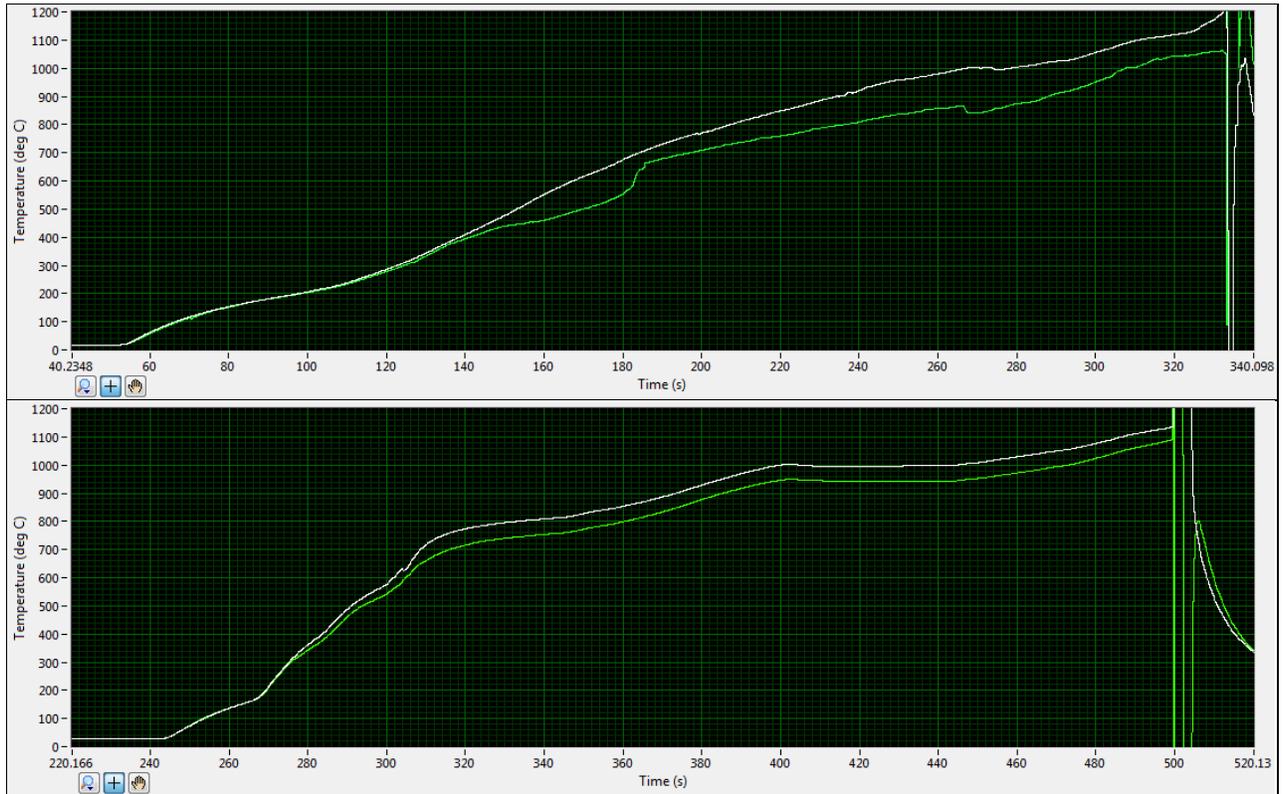
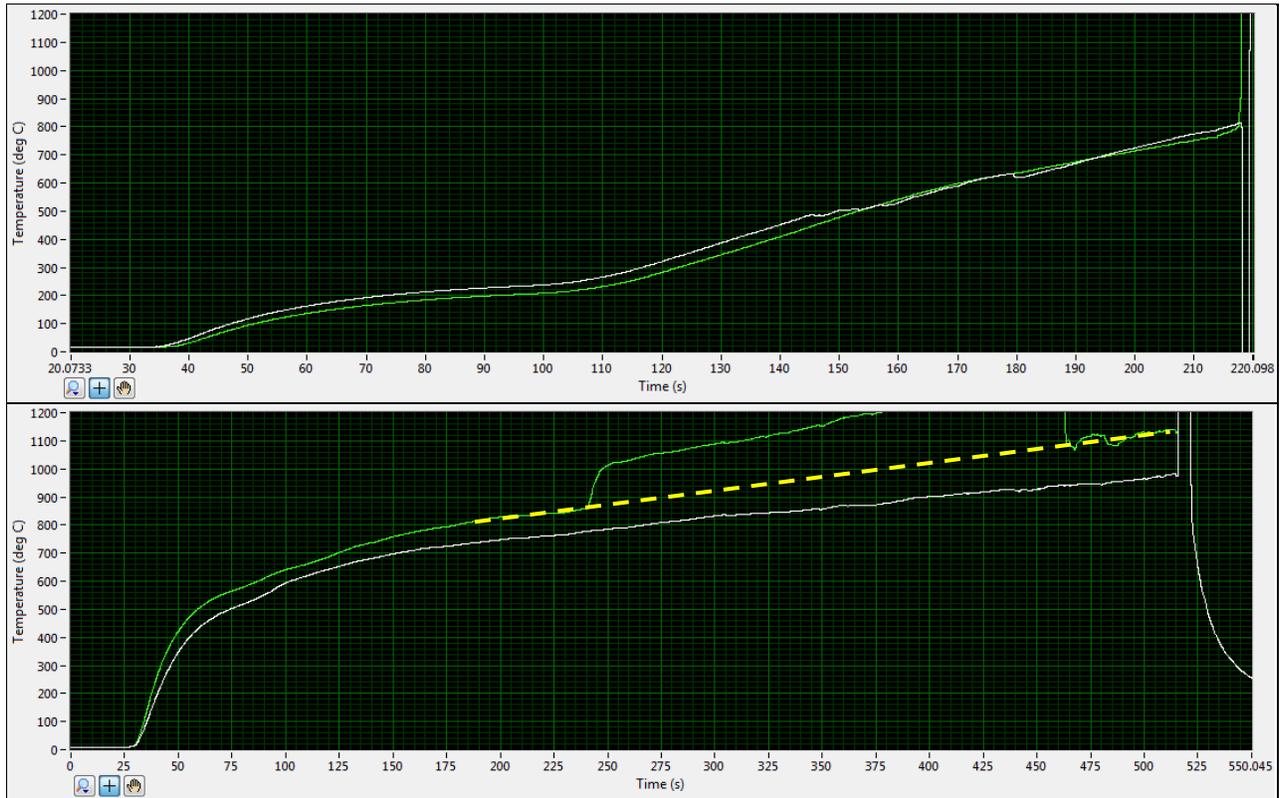


Figure 28: Auto-ignition Temperature Plots for Small-Scale Mix ID #13



NOTE: For this mix, one of the thermocouples unexpectedly departs from the baseline and then returns prior to auto-ignition of the sample (a dashed line has been added to smooth out this departure); the cause for the departure is unknown.

Figure 29: Auto-ignition Temperature Plots for Small-Scale Mix ID #14

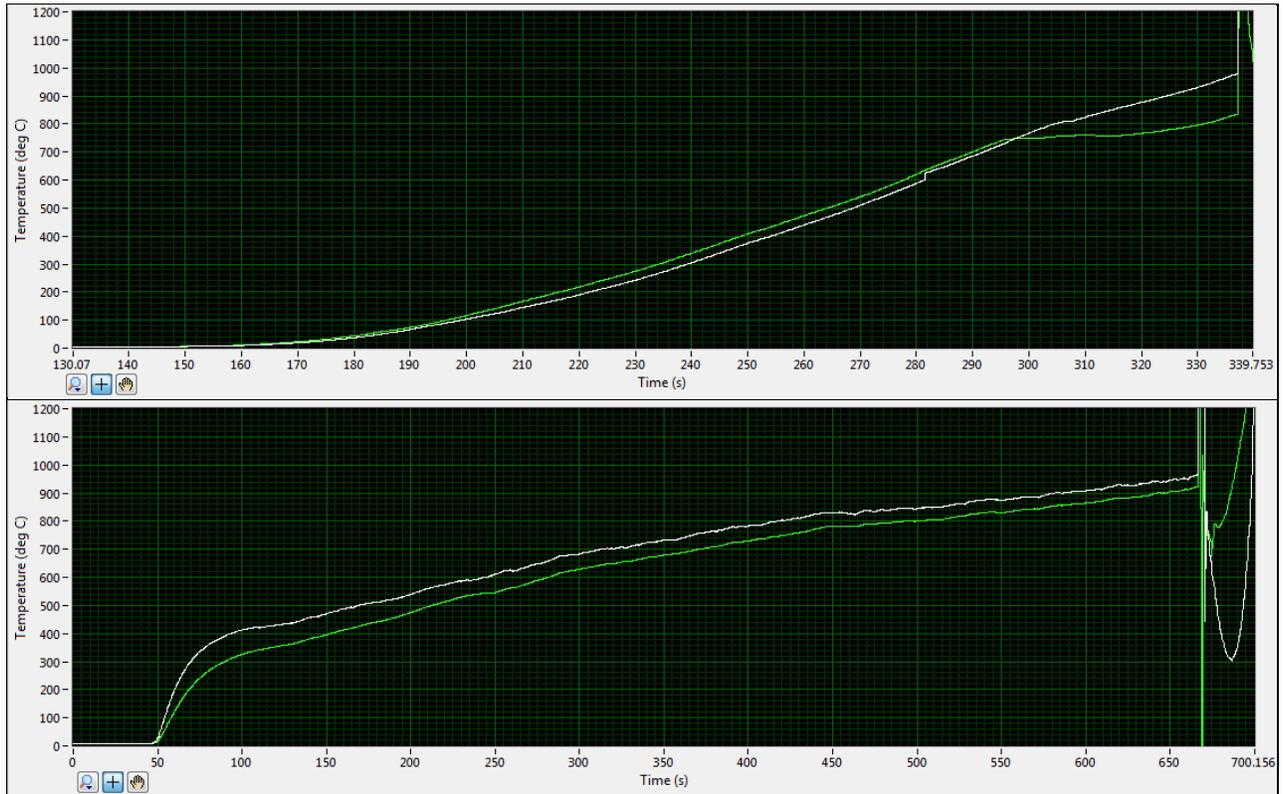
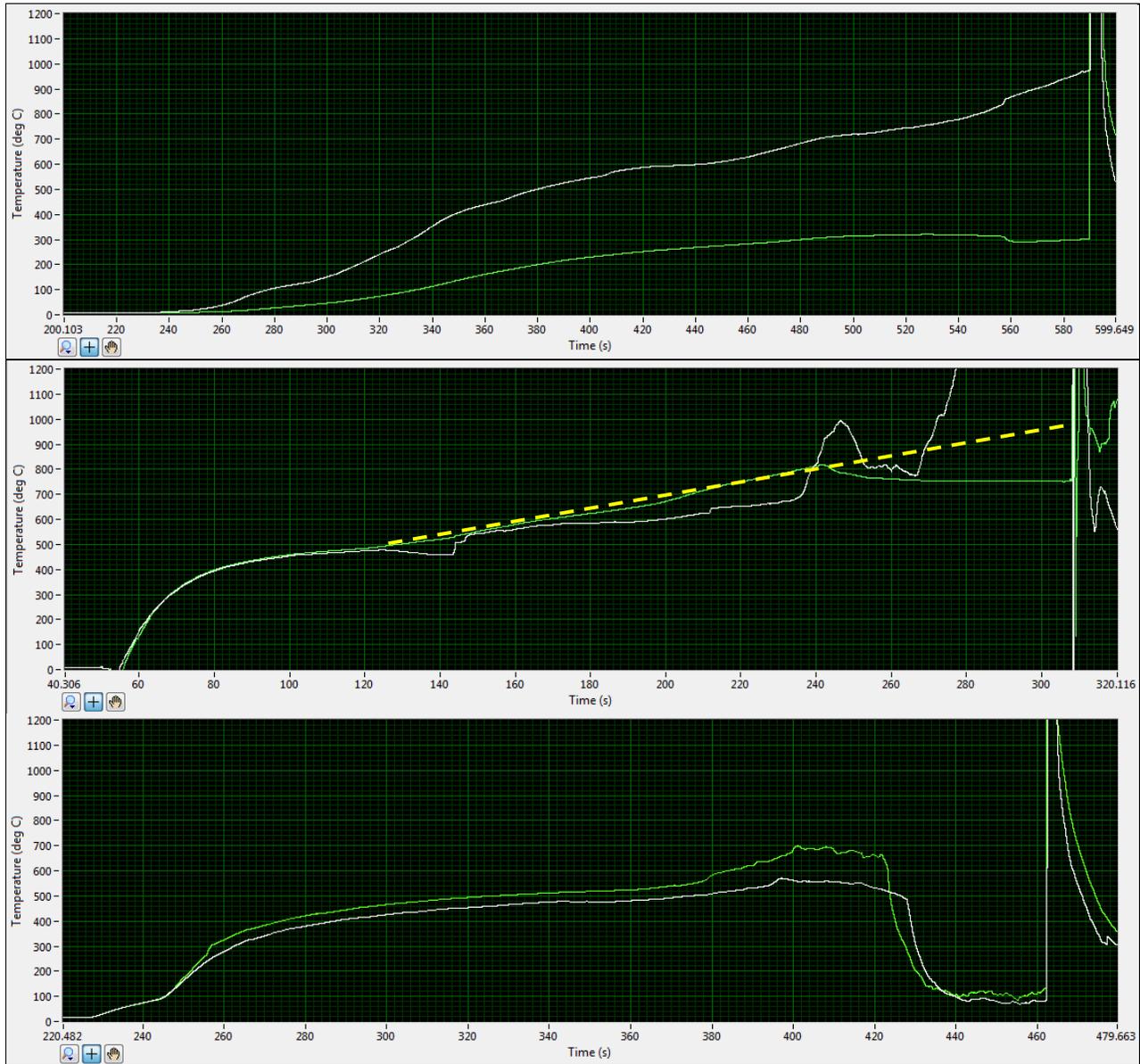


Figure 30: Auto-ignition Temperature Plots for Small-Scale Mix ID #18



NOTE: For this mix, one thermocouple in each of the first two trials unexpectedly departs from the baseline; for the second trial, a dashed line has been added to smooth out its departure. The cause for the departure is unknown but could be due to the thermocouple detaching from the ni-chrome heating wire as it experiences greater elongation and deformation at higher temperatures. Examining the temperature plots for the first two trials, one of the thermocouples appears to be providing very different measurements; a third trial was performed to increase confidence and repeatability but resulted in very different results from either of the first two trials. The cause of the variation between these trials is unknown.

Figure 31: Auto-ignition Temperature Plots for Small-Scale Mix ID #19

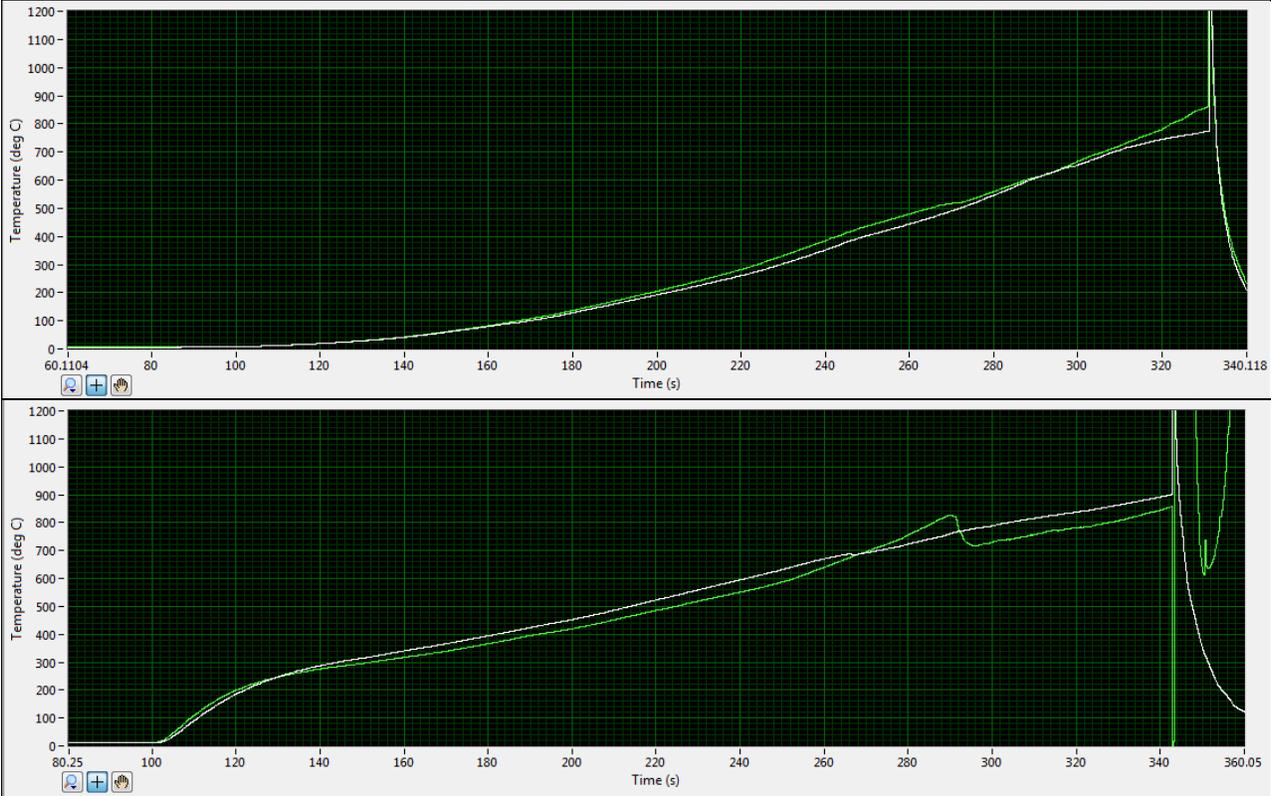
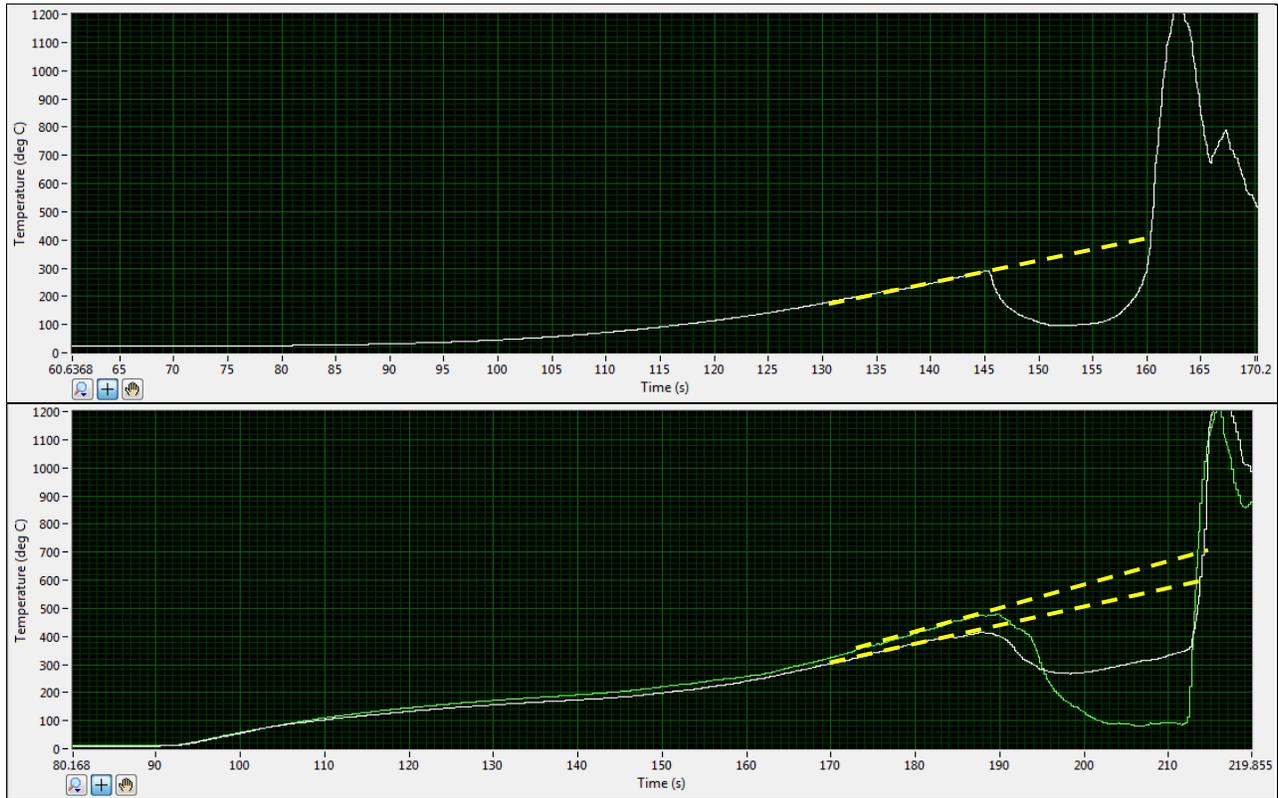


Figure 32: Auto-ignition Temperature Plots for Small-Scale Mix ID #21



NOTE: For this mix, the thermocouples depart from the baseline; a dashed line has been added to estimate the temperature of the hot wire at auto-ignition. The departure appears to be repeatable and may be the result of endothermic activity.

Figure 33: Auto-ignition Temperature Plots for Small-Scale Mix ID #24

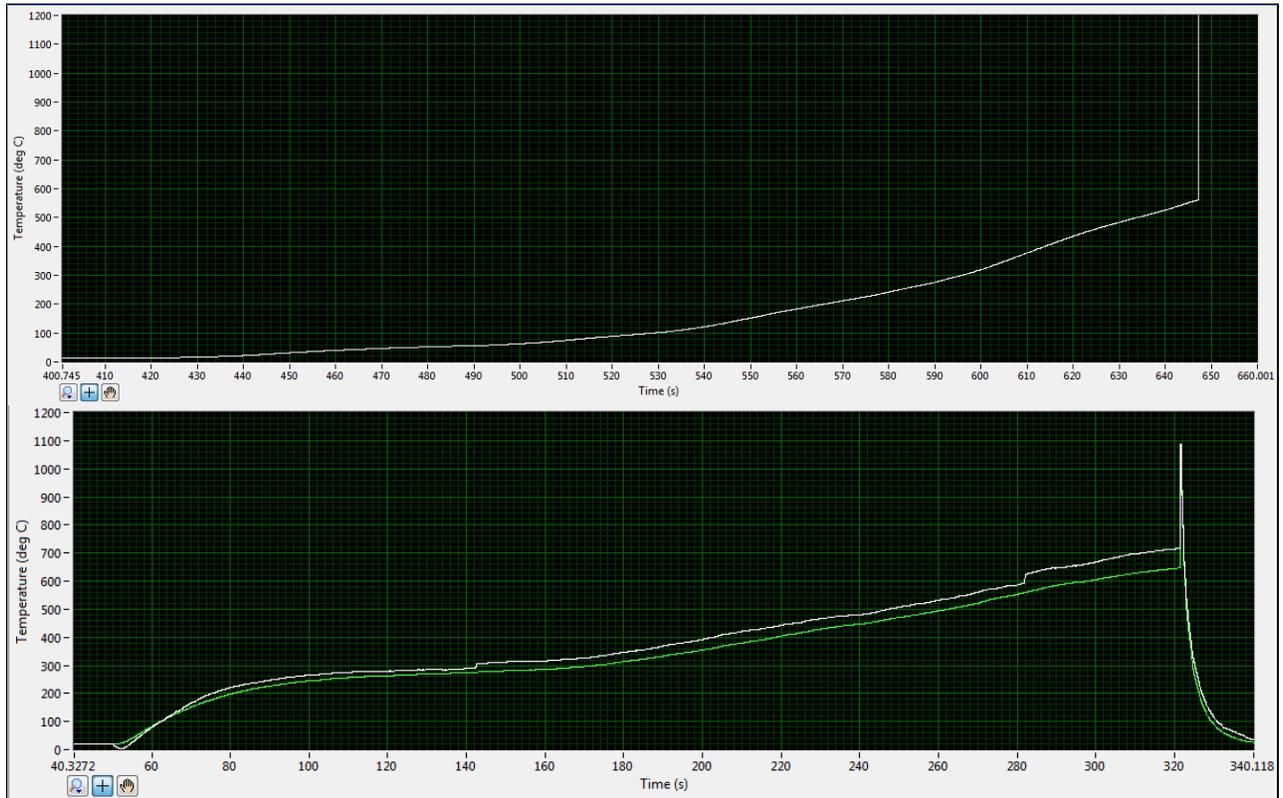
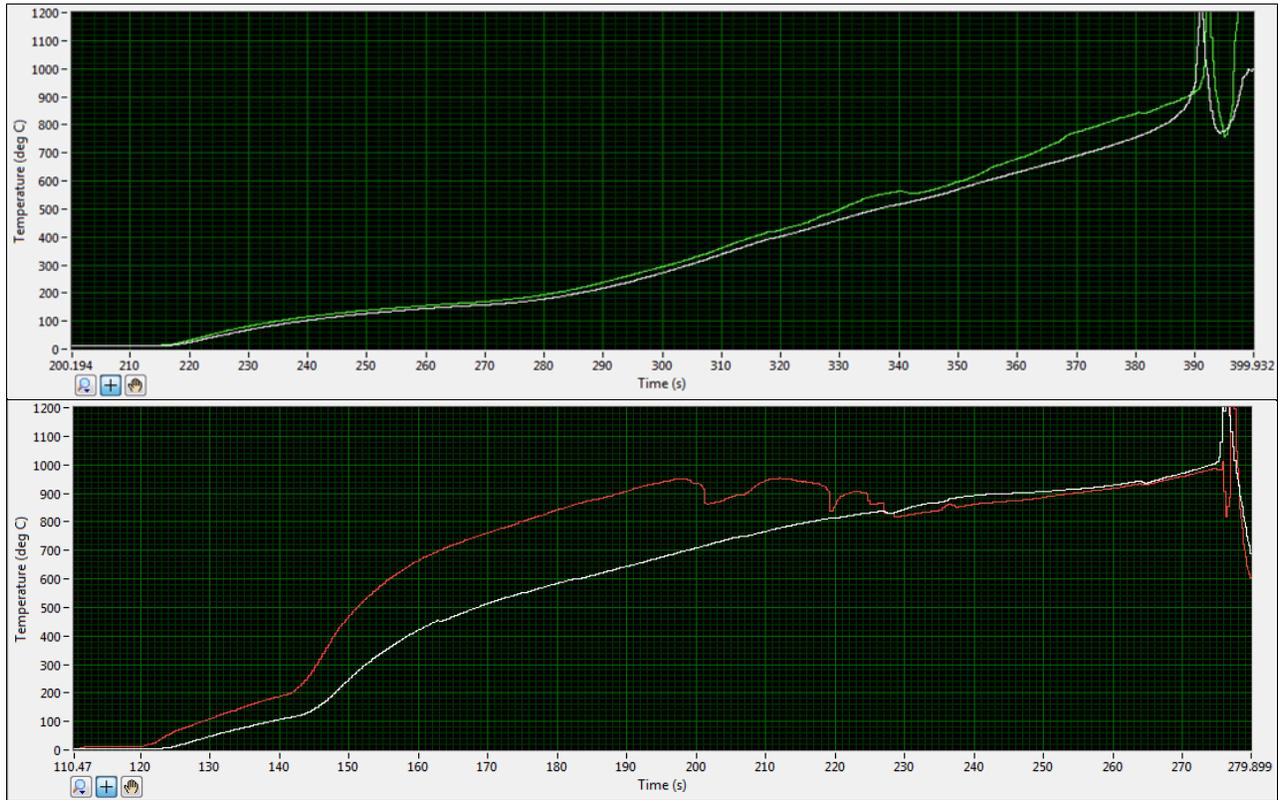


Figure 34: Auto-ignition Temperature Plots for Small-Scale Mix ID #25



NOTE: For the second trial on this mix, one of the thermocouples unexpectedly departs from the baseline and then returns prior to auto-ignition of the sample; the cause for the departure is unknown.

Figure 35: Auto-ignition Temperature Plots for Small-Scale Mix ID #26: Large-scale Mix ID #2

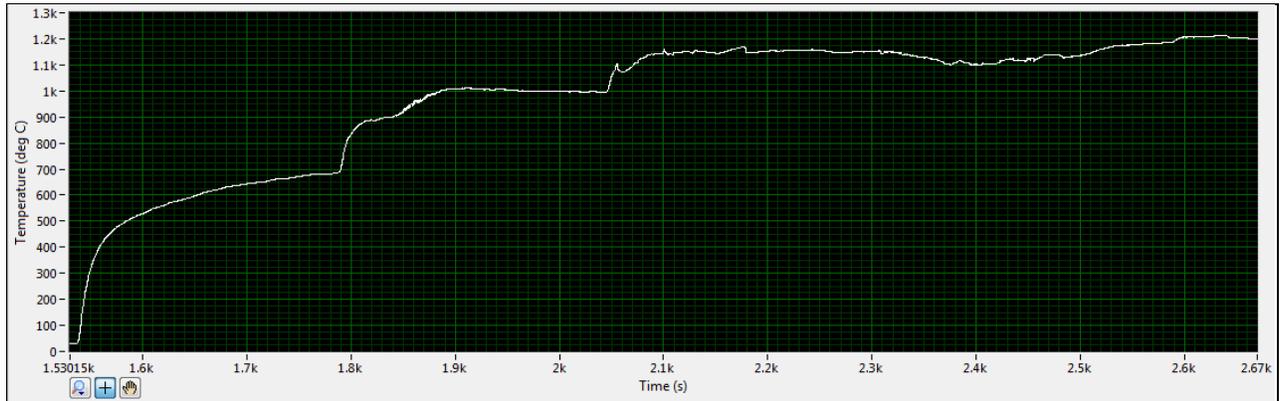


Figure 36: Auto-ignition Temperature Plots for Small-Scale Mix ID #27: Large-scale Mix ID #3

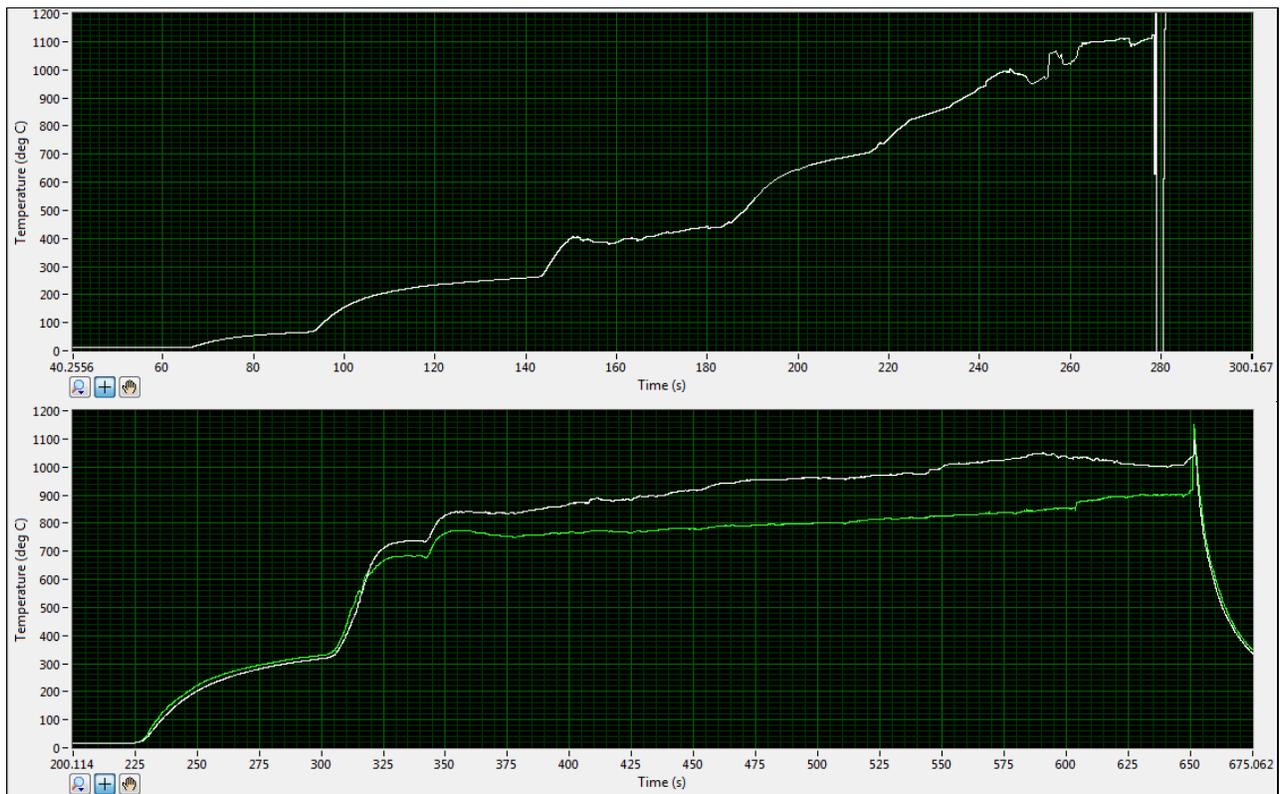
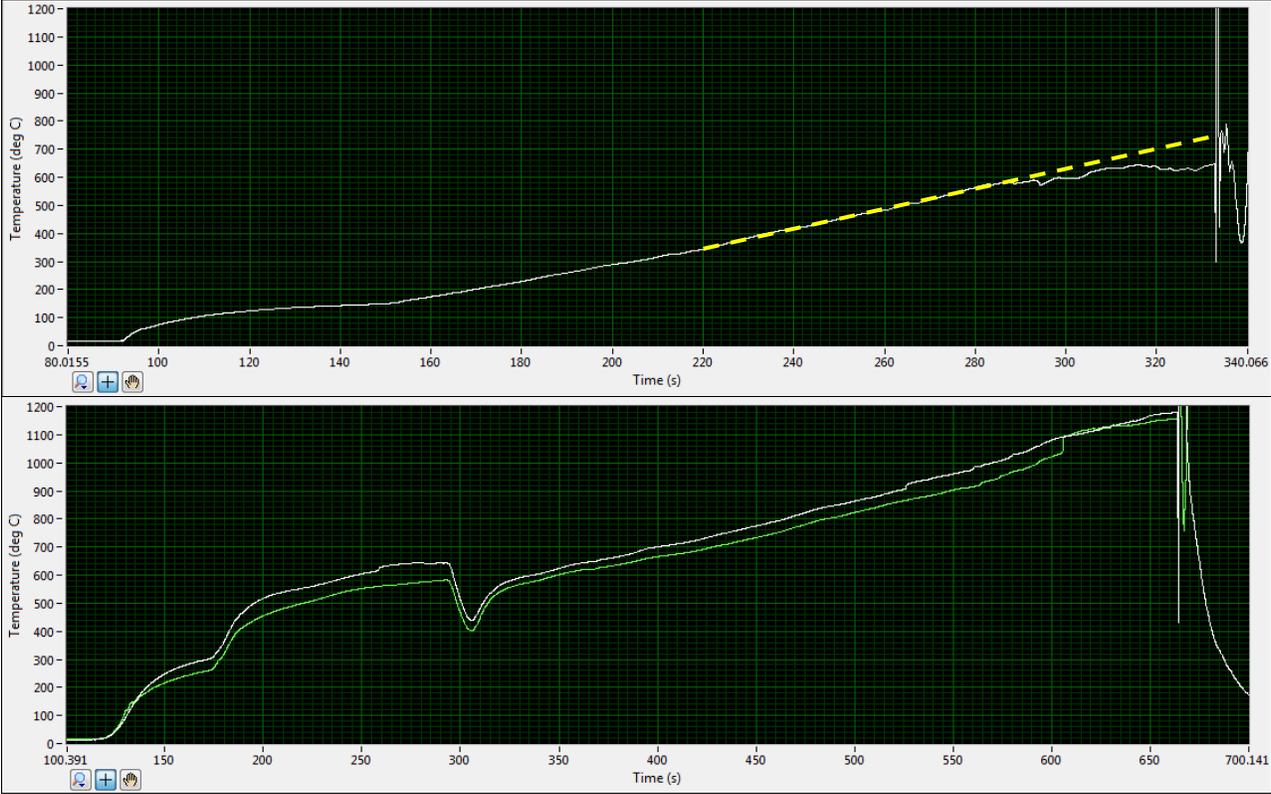
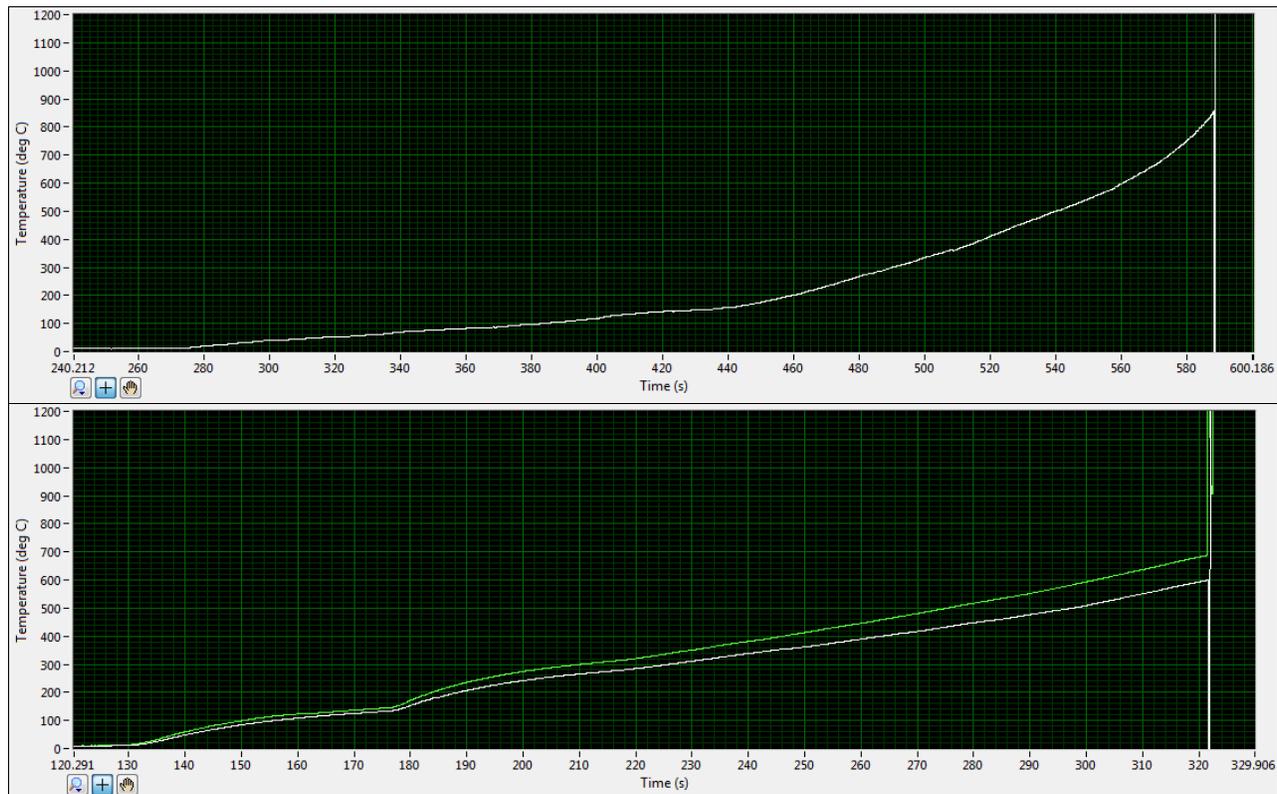


Figure 37: Auto-ignition Temperature Plots for Small-Scale Mix ID #28: Large-scale Mix ID #5



NOTE: For the first trial on this mix, one of the thermocouples unexpectedly departs from the baseline; a dashed line has been added to smooth out this departure). The cause for the departure is unknown but could be due to the thermocouple detaching from the ni-chrome heating wire as it experiences greater elongation and deformation at higher temperatures.

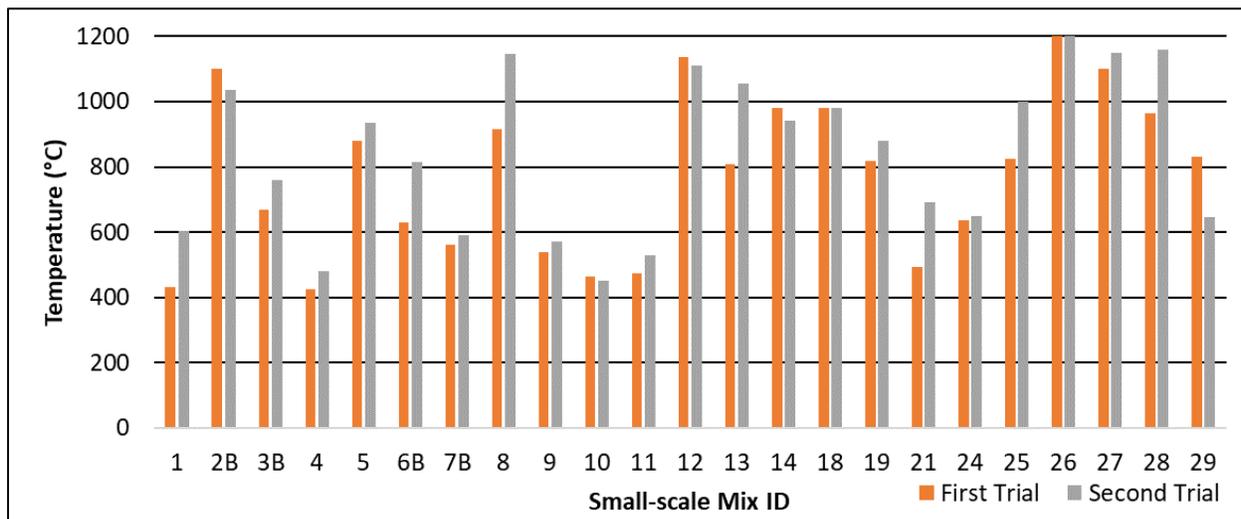
Figure 38: Auto-ignition Temperature Plots for Small-Scale Mix ID #29: Large-scale Mix ID #8



7.15.4 Assessment of Test Results

The follow figure contains a comparison of the auto-ignition temperatures for the various thermite mixtures, organized by Small-scale Mix ID. NOTE: The auto-ignition temperature of Small-scale Mix #26 exceeded that of the ni-chrome heating wire (>1200 °C).

Figure 39: Comparison of the Auto-ignition Temperatures for the Thermite Mixtures



The auto-ignition temperature of traditional explosives is typically around 200 - 300 °C; the average auto-ignition temperature of the twenty-three small-scale thermite test samples was 809 °C with a standard deviation of 240 °C.

8.0 ASSESSMENT AND EXAMINATION OF TEST RESULTS

Two of the five thermites mixed by SMS (Large-scale Mix ID's 7 and 8) exploded when ignited as unconfined 5 gram quantities, exhibiting hazards similar to that of explosives classed in Division 1.1; one of the five thermites mixed by SMS (Large-scale Mix ID 1) burned very rapidly, exhibiting hazards similar to that of explosives classed in Division 1.3.

SMS also mixed an additional fifteen small-scale thermite mixtures for auto-ignition temperature testing on a total of twenty-three thermite mixtures. Only one of these thermite mixtures had an auto-ignition temperature greater than 1200 °C. Six of these additional fifteen thermite mixtures also exploded when ignited as unconfined 5 gram quantities; based on the limited data set, the explosiveness of these thermites appears strongly related to two properties. Additional testing is required to further investigate this relationship.

Large-scale Mix ID's 2, 3, and 5 each passed the following standard tests:

- UN Series 3
- UN Series 2
- UN Test N.4 Test method for self-heating substances

- UN Test N.5 Test method for substances which in contact with water emit flammable gases

Large-scale Mix ID's 2 and 3 also passed the standard UN Test N.1 Test method for readily combustible solids with gas torch and hotwire, and could potentially be offered for transport as non-regulated materials in the absence of additional considerations. However, using hotter ignition sources and wider powder train piles than those specified in UN Test N.1, it quickly becomes evident that all three of these thermite mixes exhibit burning rates in excess of the regulated 2.2 mm/sec threshold.

NOTE: The current UN Test N.1 test methodology could potentially permit these powders to be offered for transport as non-regulated goods since the powder train pile is too narrow to sustain propagation AND the ignition source is specified around 1000 °C, which is below the auto-ignition temperature of some thermites, even though in their shipping configuration they would clearly present hazards consistent with that of flammable solids regulated for transport.

9.0 FURTHER RESEARCH

[Not disclosed]

10.0 PRODUCT CERTIFICATIONS

[Not disclosed]