

APPENDIX 3

BENCH-SCALE SCREENING TEST

Overview

The UN Test O.1 Test for Oxidizing Solids was modified to include (1) a calibrated, laboratory balance with data output to record the dynamic mass loss of oxidizer-cellulose powder mixtures exposed to a glowing wire, (2) three K-type thermocouples centered above the pile at fixed distances to measure plume temperatures and to estimate peak flame height and (3) a video camera. A test series consisted of five trials each with two or more oxidizer:cellulose powder mixtures (30-g, 1:1, 4:1, 9:1). Each oxidizer was tested with the as-received particle size (i.e., no grinding). The bench-scale screening test does not use a reference oxidizer. The bench-scale screening test criteria are based on the results of intermediate-scale fire exposure testing of oxidizers with a range in burning behavior.

The instrumentation, test methodology and data reduction described below will be the basis of a step-by-step screening test method.

Instrumentation and Test Set-Up

A laboratory balance¹ was positioned and leveled inside a clean, standard laboratory chemical hood. A metal frame, constructed of welded steel, with two pins at the diagonal of the weigh pan was used to center scale protection materials. The metal frame does not contact the laboratory balance.

The weighing surface of the balance (6-in x 6-in) was protected with three layers of ceramic fiberboard. The bottom or base layer was Kaowool HT board² (12-in x 12-in x ½-in, mass 356g). The base layer extended beyond the balance to capture material ejected from the test platform. Two openings in the base layer corresponded to the location of the centering pins of the metal frame. The middle layer was Kaowool HT board (7-in x 7-in x ½-in, mass 124g), and the top layer, or test platform, was Kaowool PM board³ (4 ½-in x 4 ½-in x ¼-in, mass 22g). The base layer of protection remained on the balance after each trial. Due to heat retained from the glowing wire and/or test materials, the two top layers of ceramic board on the base layer were removed after each test. The middle layer of HT board was re-used after cooling to room temperature. A suitable 'barrier' was constructed around the laboratory balance and protection materials to prevent air currents from the operating hood from influencing the balance stability. In this project, the barrier consisted of two, ½-inch thick, compressed, cellulose-based fiber board building material (Homasote®). The barrier extended from the back of the hood to the front of the hood. Figure 1 and Figure 2 show an overview of the test set-up inside a laboratory hood.

¹ A&D Company, Limited, Model GX-2000, 2100 g weighing capacity, 0.01g repeatability. Internal calibration weight

² Kaowool HT board, maximum temperature rating 2600F, thermal conductivity at 1500 F is 1.01 BTU in/hr ft² F.

³ Kaowool PM Board, Lot No. 7332S1, Maximum temperature rating 2300F, thermal conductivity at 1500F is 0.87 BTU in/hr ft² F

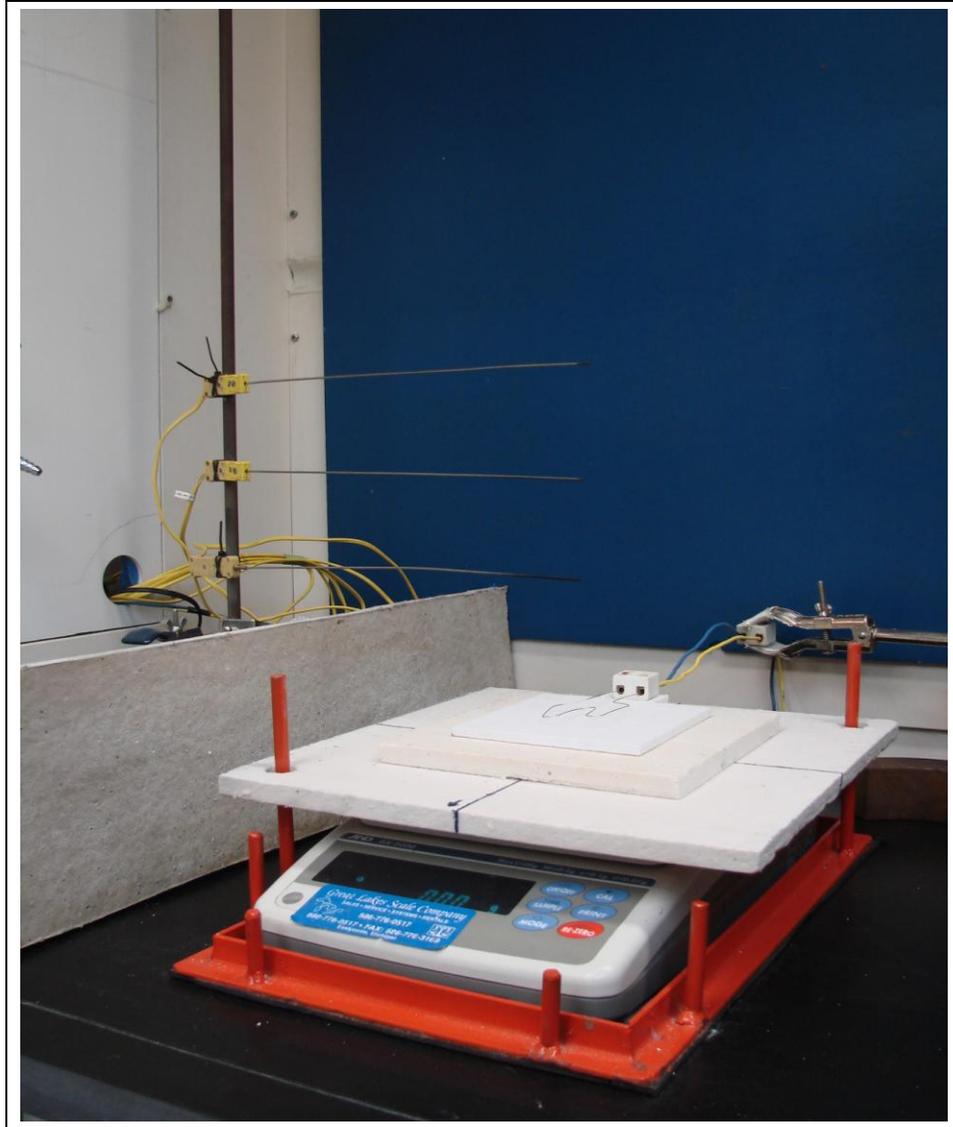


FIGURE 1. Overview showing the protected balance with metal frame and array of thermocouples inside a laboratory hood.

The laboratory balance has programmable settings that were used to optimize the response-time and to establish the data output interval. An RS232 cable connected the balance to a data acquisition system. TalTech WinWedge⁴ software was used to collect and record the mass data from the balance. The WinWedge software parameters were adjusted to receive and timestamp mass data from the balance into an Excel spreadsheet.

The auto-calibration function of the balance was used between tests and was disabled during tests. A steel calibration mass (2kg) was used to calibrate the balance without protection materials; three smaller calibration masses (20g, 10g, and 5g) were periodically used to check the balance output with the protection materials in place (Figure 3).

⁴ WinWedge Version 3.0 Standard Edition

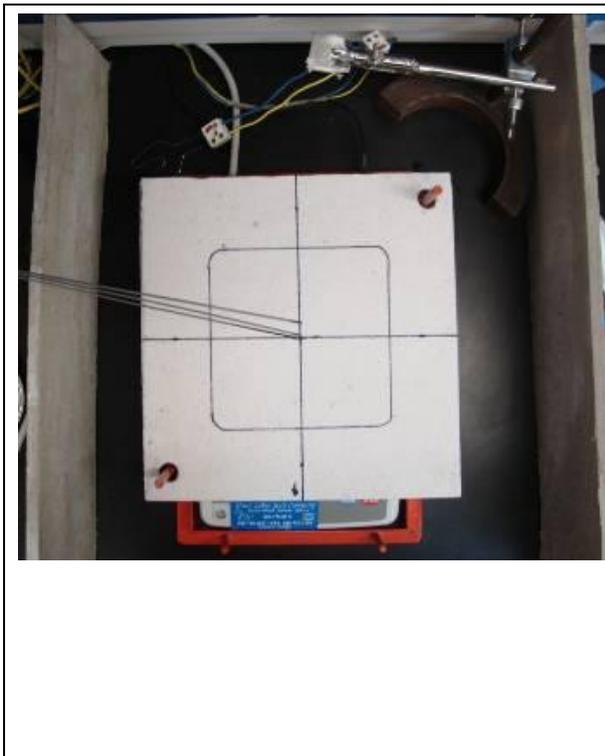


FIGURE 2. Photograph showing the base layer of protection material centered on the balance using the centering pins.

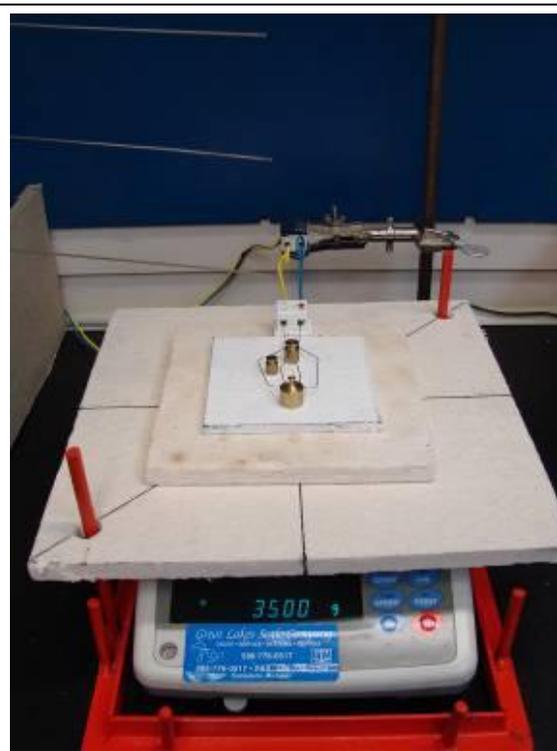


FIGURE 3. Photograph showing the lab balance with three layers of protection materials, the Nichrome wire and calibration masses.

Three, K-type⁵ air thermocouples (TC) were secured to a ring stand to one side of the protected lab balance. The tips of the thermocouples were centered above the test platform. The distance from the surface of the test platform to the thermocouples was 7.6cm (3 inches), 15.2 cm (6 inches), and 22.8cm (9 inches). A fourth K-type thermocouple monitored the ambient temperature. The TCs were attached to the data acquisition system,⁶ which was programmed to record the temperature at the same time interval as the lab balance.

A Nichrome⁷ wire was formed as specified in the UN Test O.1 method⁸. The length of the wire was 30 ± 1 cm (11.8 inches); the wire diameter was 0.6 ± 0.05 mm. A variable power supply⁹ was used to regulate the voltage and current to the wire to achieve 150 ± 7 Watts when energized. Conductors from the power supply output were secured to a ceramic terminal block, which was secured with a clamp at the rear of the lab balance. From the first terminal block, conductors were routed to a second ceramic terminal block. The looped Nichrome wire was inserted in the second terminal block to complete the circuit. A digital multi-meter was used to check the voltage and current across the terminal blocks and in the current path of each terminal block.

⁵ Length 12 inches, OD 1/16th inch Inconel sheath

⁶ Omega DAQ-3000 Data Acquisition and DAQView software.

⁷ Wiretronic, 22 (0.025 in) NiCr A wire PN NiCrA-22, Lot 4668

⁸ UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, Section 34.4.1, 4th Revised Edition, 2003, Geneva and NY.

⁹ TekPower Adjustable DC Regulated Power Supply, Model HY3020E, Output 0-30V@0-20A single channel

The second terminal block with the Nichrome wire was positioned on the middle layer of ceramic board such that the looped portion of the wire was centered over the test platform (Figure 4). When energized, the Nichrome wire glowed bright orange (Figure 5).

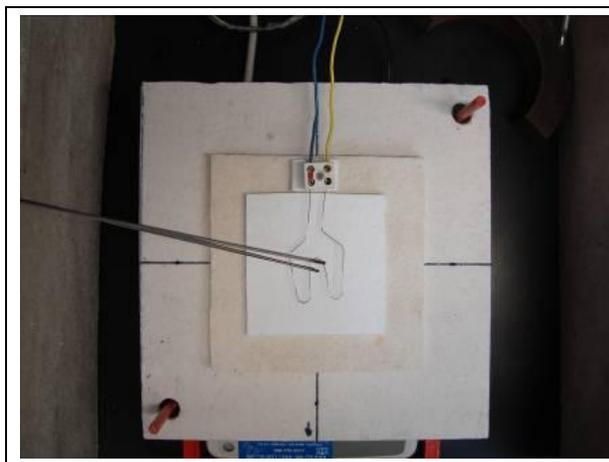


FIGURE 4. Photograph showing the lab balance with protection materials, the Nichrome wire and thermocouples centered over the test platform.

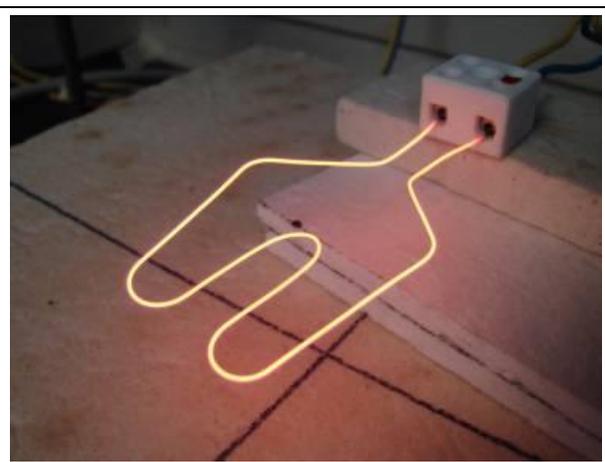


FIGURE 5. Glowing, energized, looped, Nichrome wire ignition source.

Materials Characterization

The oxidizer material safety data sheet was reviewed. The materials composition, specifically the concentration of the oxidizer, CAS number and physical properties were recorded. If formulated, the chemical structure and CAS number of the non-oxidizer additive(s) were recorded. The oxidizer was characterized as granular, crystalline, or powder. The particle size distribution of the oxidizer was determined with calibrated sieves (500, 300, 150, 90 and 45 micron). The test material was not ground or subject to any mechanical attrition or sieving to reduce the particle size. The mass of material retained in each sieve was recorded to determine the mass percent of each particle size fraction. The bulk density of the oxidizer was determined by recording the mass of a known volume of the material using a graduated cylinder.

Test Methodology

Whatman cellulose powder (200±50g) was transferred to a pan (8 in x 8in x 1 ½ in; 20 cm x 20 cm x 3.8 cm) and dried in a laboratory oven¹⁰ at 105±3°C for 4 to 5 hours. After drying, the powder was transferred to a desiccator cabinet with desiccant. When cool, the moisture content of the cellulose powder was measured using an analytical moisture analyzer¹¹ (Figure 6).

For each test, individual 30±1 gram mixtures of the test material and dried, cellulose powder (CF11) were prepared, centered on the protected laboratory balance, and exposed to the Nichrome wire ignition source. The three concentrations of oxidizer-cellulose powder mixtures evaluated were (1) 1:1 by mass mixtures containing 15-g oxidizer and 15-g cellulose powder,

¹⁰ Yamoto Model DVS402 Drying Oven

¹¹ A&D Weighing, Programmable Model MX-50, 0.001g weight resolution.

(2) 4:1 by mass mixtures containing 24-g oxidizer and 6-g cellulose powder, and (3) 9:1 by mass mixtures containing 27-g oxidizer and 3-g cellulose powder.

The oxidizer:cellulose powder mixtures were tested in the following sequence: 1:1, 4:1 then 9:1. If either the 1:1 or 4:1 mixture resulted in vigorous burning with stopwatch burning durations less than 6 seconds and/or the calculated peak mass loss rate was greater than 3 g/s, the 9:1 mixture was not tested.



FIGURE 6. Moisture analyzer used to measure the moisture content of the dried cellulose powder.

Each mixture of oxidizer and cellulose powder was prepared immediately prior to each trial in a test series. A second laboratory balance was used to weigh the required amounts of cellulose powder and oxidizer. The materials were transferred to and combined in a clean, labeled polyethylene container (500 mL) with a screw top lid. Mixing of the contents was done by inverting (not shaking) the container no less than 25 times. After mixing, the contents of the container were transferred into a clean, funnel¹² positioned stem-down in an Erlenmeyer flask. The stem opening at the base of the funnel was sealed with tape. The funnel with the mixture was tamped lightly. Using cardstock paper, the funnel with the mixture was inverted over the looped Nichrome wire ignition source on the test platform.

After transferring the material to the balance, the balance was tared to zero (Figure 7). The sash of the hood was lowered and the hood blower energized. The laboratory temperature and relative humidity were recorded. Balance stability was confirmed, data acquisition was initiated, baseline data was collected, and then the Nichrome wire was energized at the power supply. A stop-watch was activated when the wire was energized. Mass loss and the temperature data were collected until spreading discoloration and/or visible flaming combustion ceased. In the

¹² VWR International LLC, Product No. 89026-148, PTFE, 70 mm dia, 102 mL volume

event of little or no flaming combustion, mass and temperature data were collected for 5 minutes. Figure 8 is a photograph during a test with cellulose powder.

Visual observations were recorded. Observations included the peak flame height; whether the pile ignited readily or was slow to ignite; whether flames readily spread over the pile or were slow to spread; the flame color; the relative volume and color of gaseous products generated; material displaced from the test platform, if any; unusual burning behavior such as swelling or 'boiling'; observable or clear dependence of burning rate on the duration of the energized wire; irregular or intermittent burning; and presence of unburned or uninvolved material remaining at the end of a test. Photographs and/or video recordings were used to document, at a minimum, one representative trial in a test series.

After each trial, the Nichrome wire was released from the terminal block, and the top two layers of balance protection materials with the test residue were removed and set aside in the hood to cool to room temperature. After cooling, the mass of the post-test residue was recorded. The post-test material was transferred to a labeled glass jar for waste disposal or further characterization, if desired. In preparation for the next trial, new and/or cool test platforms were positioned on the laboratory balance, a new, Nichrome wire was inserted into the terminal block, and a new data acquisition file was created.

After each series of tests, the hood, test area, and funnel were thoroughly cleaned.

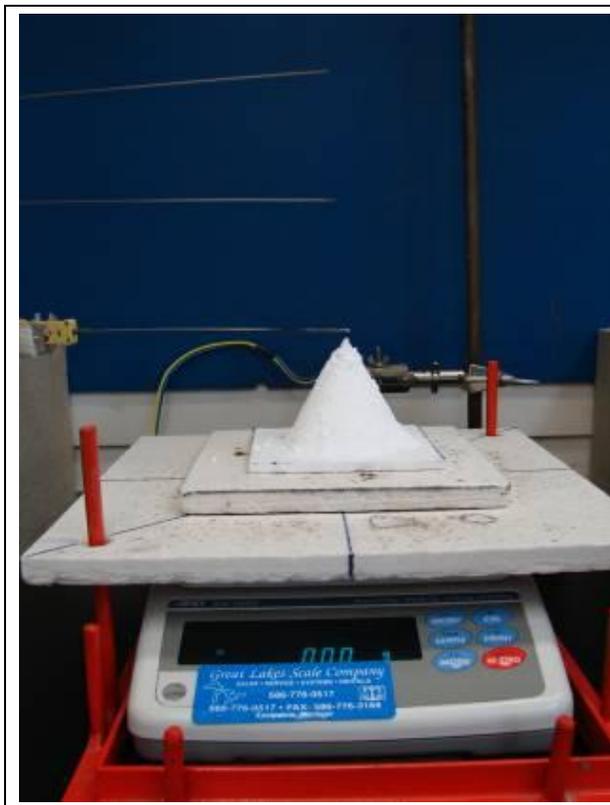


FIGURE 7. Photograph showing 30-g of cellulose powder centered over the Nichrome wire.



FIGURE 8. Photograph during a test with cellulose powder.

Data Reduction

Test data recorded for each trail are summarized in Table A1.

TABLE 1. Summary of Data Recorded and Calculated for Each Trial

TEST DATA	
Concentration of oxidizer (source of information)	%
Test mixture mass ratio with dry CF11 (Lot No.)	1:1, 4:1 or 9:1
Lab temperature	degree C
Lab relative humidity	%
Initial mass of oxidizer or formulated product	g
Initial mass of cellulose powder	g
Data collection interval	s
Power to Nichrome wire	Watts
Time igniter wire opens	s
Time at end of visible flaming combustion or spreading discoloration	s
Mass of post test residue	g
CALCULATED TEST DATA	
Mass loss rate from 20% to 80% consumed	g/s
Active burning time	s
Peak temperature at 6-in	degree C
Percent material consumed	%

After completing a series of bench-scale screening tests with 1:1, 4:1, and 9:1 oxidizer-cellulose powder mixtures, the mass loss profiles were examined for repeatability and/or clear deviation based on the continuity of the wire.

When spreading discoloration and/or visible flaming combustion ceased, the mass loss and temperature data acquisition files were terminated. The percent material consumed was calculated from the final measured mass over the initial mass of the mixture. The final recorded mass (m_f), or amount of mixture consumed, was similarly used to calculate the mass of the test mixture at 20%, 50% and 80% consumed. The software's linear regression function was used to calculate the mass loss rate from 20% to 80% consumed. The active burning time was the time from first measurable mass loss to the time at 80% consumed. A second burning duration was evaluated: the time to 50% of the mixture consumed. Figure 9 is a mass loss profile that illustrates the mass at 20%, 50% and 80% consumed, the mass loss rate from 20% to 80% consumed and the burning times. The mass loss rate and active burning time data, reduced from each trial mixture, were then used to calculate the average and sample standard deviation.

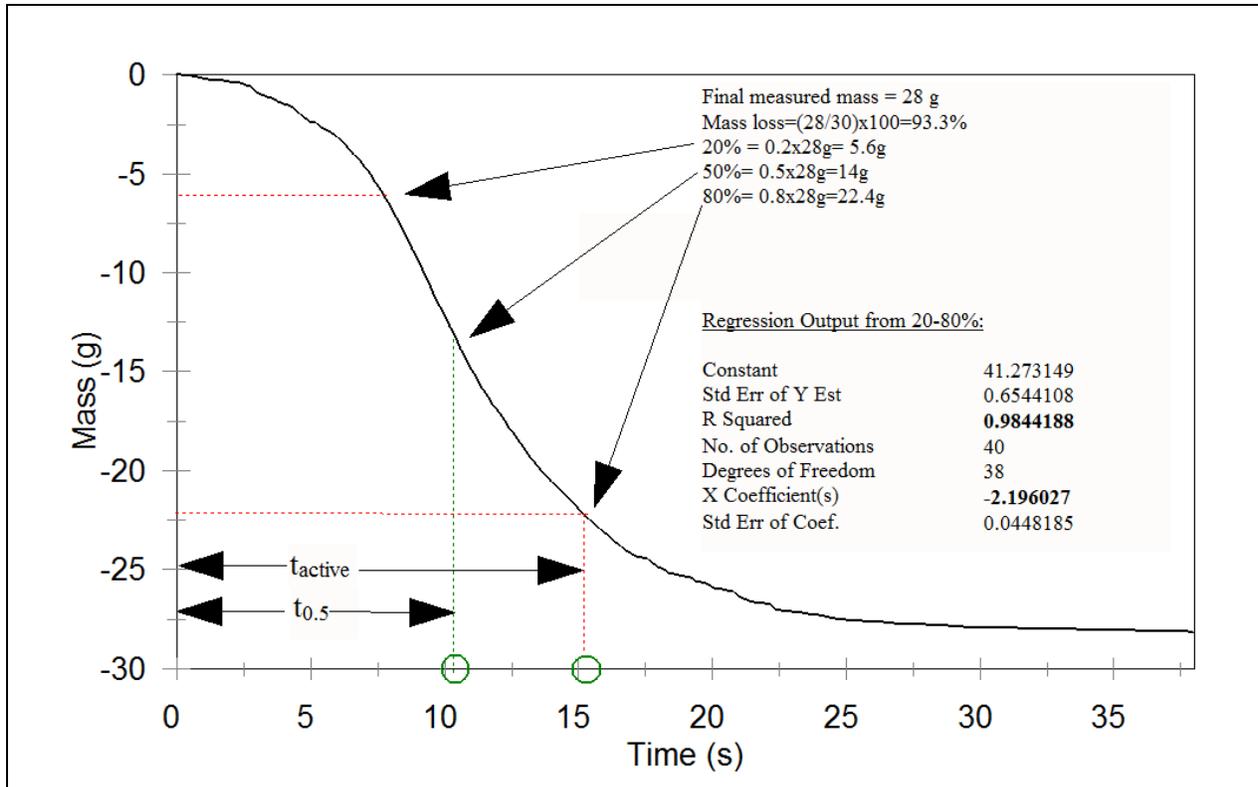


FIGURE 9. Representative mass loss profile illustrating the final mass, mass at 20%, 50% and 80% consumed, the active burning time and the time to 50% consumed.

The temperature profile from each trial should be representative of the test material's reaction to the glowing wire, namely decomposition or combustion. For mixtures that ignite and spread fire, the mass at 20% and 80% consumed should be within the elevated temperature envelope. For each test, the peak temperature at the thermocouple located 6-in above the test platform was recorded and averaged over the five trials. A representative mass loss-temperature profile is shown in Figure 10.

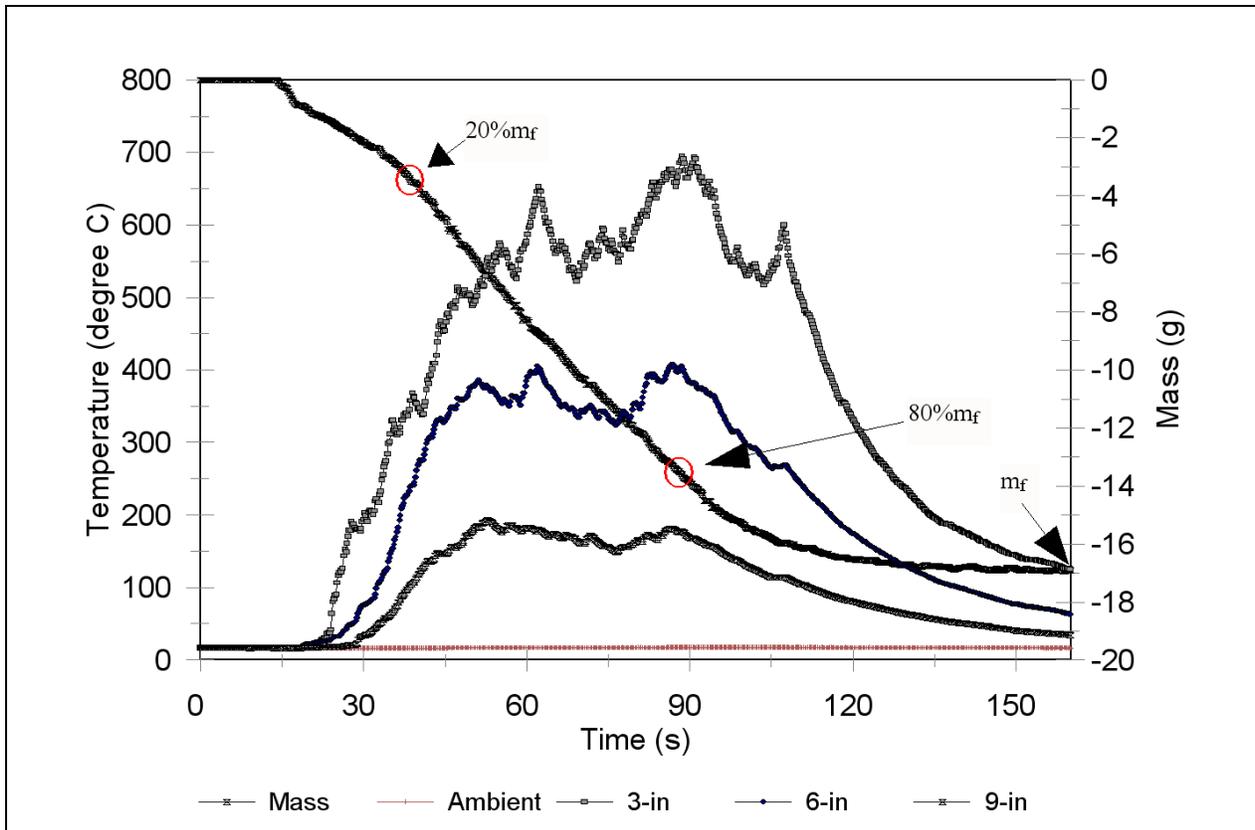


FIGURE 10. Representative mass loss-temperature profile.

The 1:1 lithium hypochlorite (29%):cellulose powder (30g) mixture is used to illustrate the data generated from the screening test with one mixture. Representative photographs during one trial are shown in Figure 11. The mixture was slow to ignite and spread orange flames over the pile; the pile was observed to swell during coombustion. The peak flame height was 9 inches from the test platform. The mass loss profiles from 5 trials are shown in Figure 12. Figure 13 is the mass loss and temperature profile from Trail 1. Table 2 is a compilation of the test data with the average and sample standard deviation shown in the last two columns.

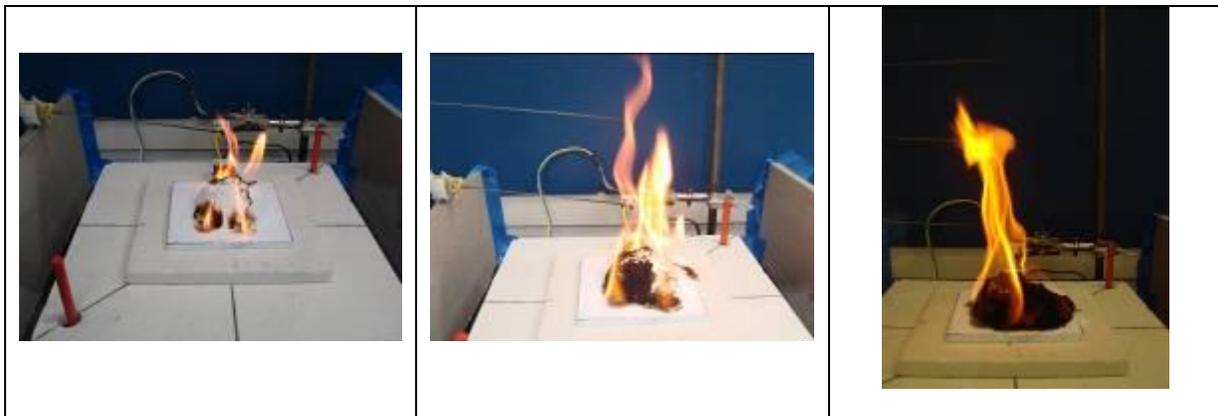


FIGURE 11. Representative photographs during one trial with 1:1 LiOCl(29%):CF11.

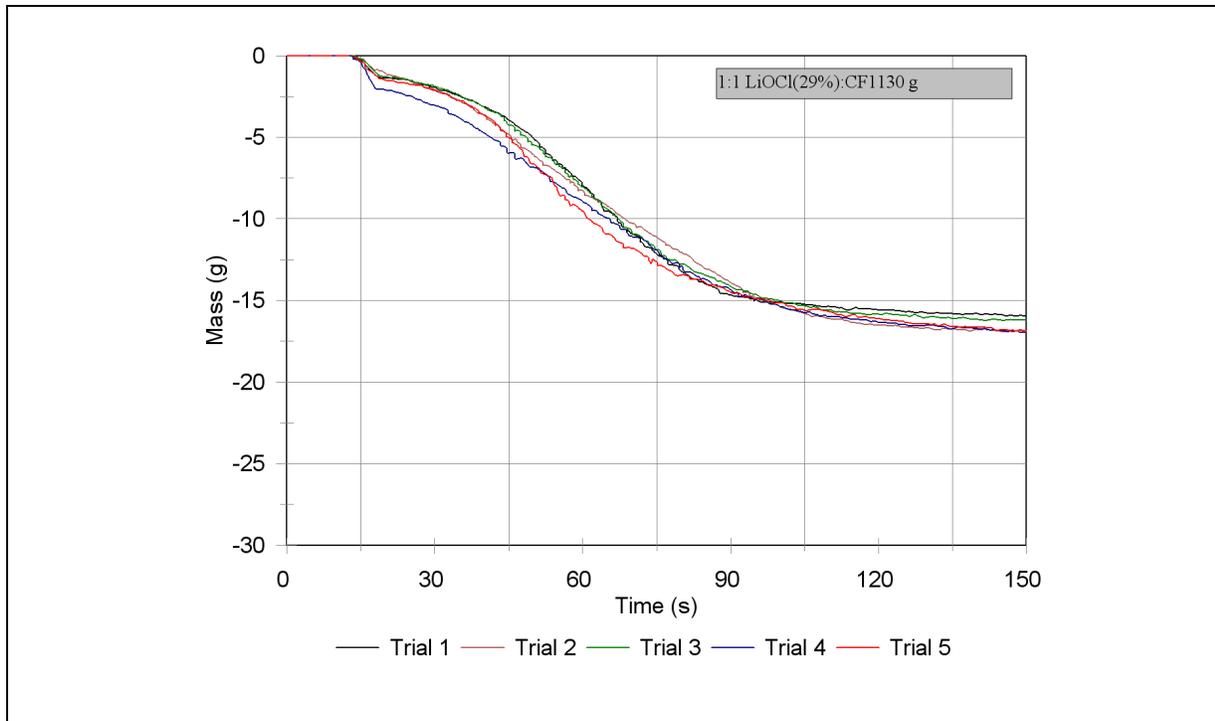


FIGURE 12. 1:1 LiOCl(29%):CF11 mass loss profiles.

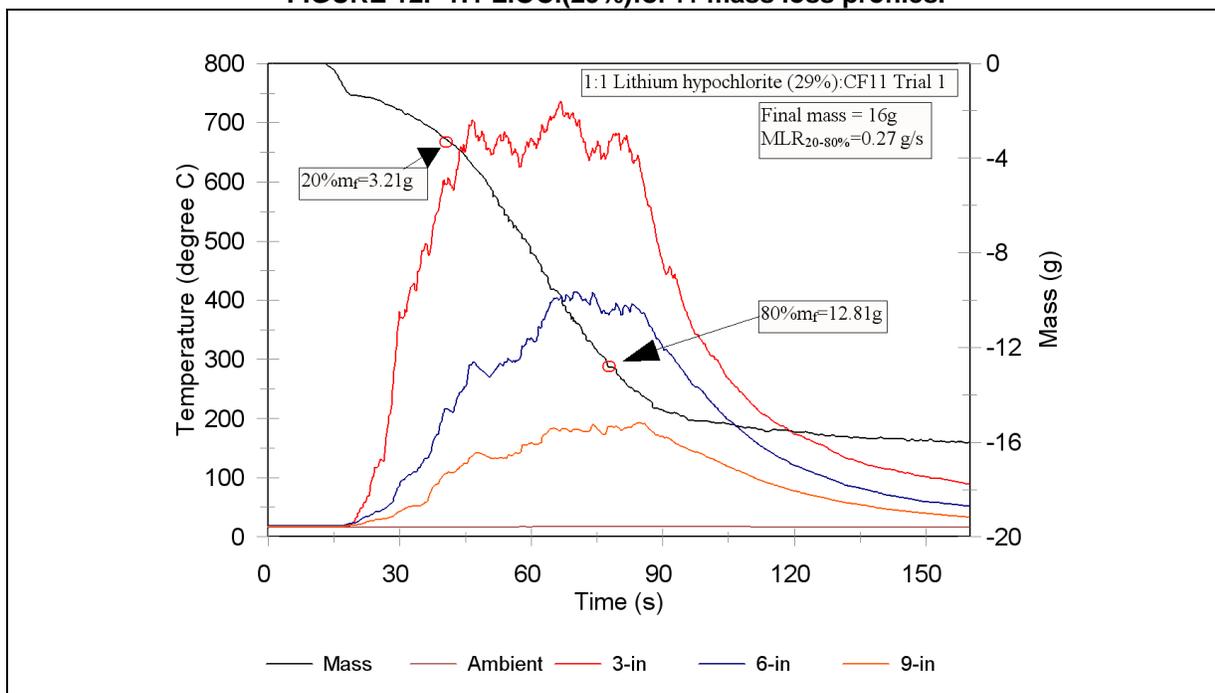


FIGURE 13. Representative mass loss and temperature profile of 1:1 LiOCl(29%):CF11.

TABLE 2. 1:1 LiOCI(29%):CF11 Test Data

Trial		T1	T2	T3	T4	T5	Avg	St Dev
Lab Temp	°C	18	18	18	18	18		
Lab RH	%	50	49	48	48	48		
Mi lithium hypochlorite	g	15.05	15.02	15.05	15.06	15.02		
Mi CF11	g	15	15.01	15.02	15.03	15.01		
Mass of residue	g	13.81	13.63	13.91	13.68	13.5		
Power to wire	Watt	150	150	150	149	149		
Igniter opens	s	106	96	91	91	149		
End of visible flaming combustion	s	116	113	106	116	176	125	29
Calculated Test Data								
Active burning time	s	64.6	74.4	68.2	71.8	68.3	69.5	3.8
MLR20-80%	g/s	0.274	0.2042	0.253	0.204	0.252	0.24	0.03
R ²		0.99	0.99	0.99	0.99	0.98	--	--
Time to 50% consumed	s	47.3	46.7	47	44.7	42	45.5	2.2
Tpeak @ 6 in	°C	415	408	450	361	371	401	36
Mass loss	%	53	56	54	57	56	55	2
<i>Data collection interval was 0.2 s.</i>								
<i>Nr- not recorded. Data shown as average ± sample standard deviation.</i>								

Bench-Scale Test Criteria

The bench scale screening test criteria, shown in Figure 14, are based on the results of intermediate-scale fire exposure tests with oxidizers in combustible test packaging. The maximum mass loss rate of the oxidizer-fuel mixture is determined from the reduced 1:1, 4:1 and 9:1 mixture test data. If the burning behavior depends on the duration of wire continuity, the highest mass loss rate is used instead of the average; or a sufficient number of trials should be conducted to average tests with similar wire continuity durations.

If the maximum mass loss rate is less than or equal to 0.3-g/s and the active burning time is greater than or equal to 30 seconds, the oxidizer is assigned to Class 1. If the maximum mass loss rate is greater than or equal to 3g/s and the active burning time is 6 seconds or less, the oxidizer is assigned to Class 3. If the mass loss rate and active burning time do not meet the criteria for Class 1 or Class 3, the oxidizer is assigned to candidate Class 2. The candidate Class 2 materials should be subjected to intermediate-scale fire exposure testing in order to confirm the Class as Class 1, Class 2 or Class 3. A Class 4 oxidizer generally meets the Class 3 burning rate criteria plus evidence to support explosive reaction due to contamination or exposure the thermal or physical shock. If confirmatory intermediate-scale fire exposure testing is not done, the material is Class 2.

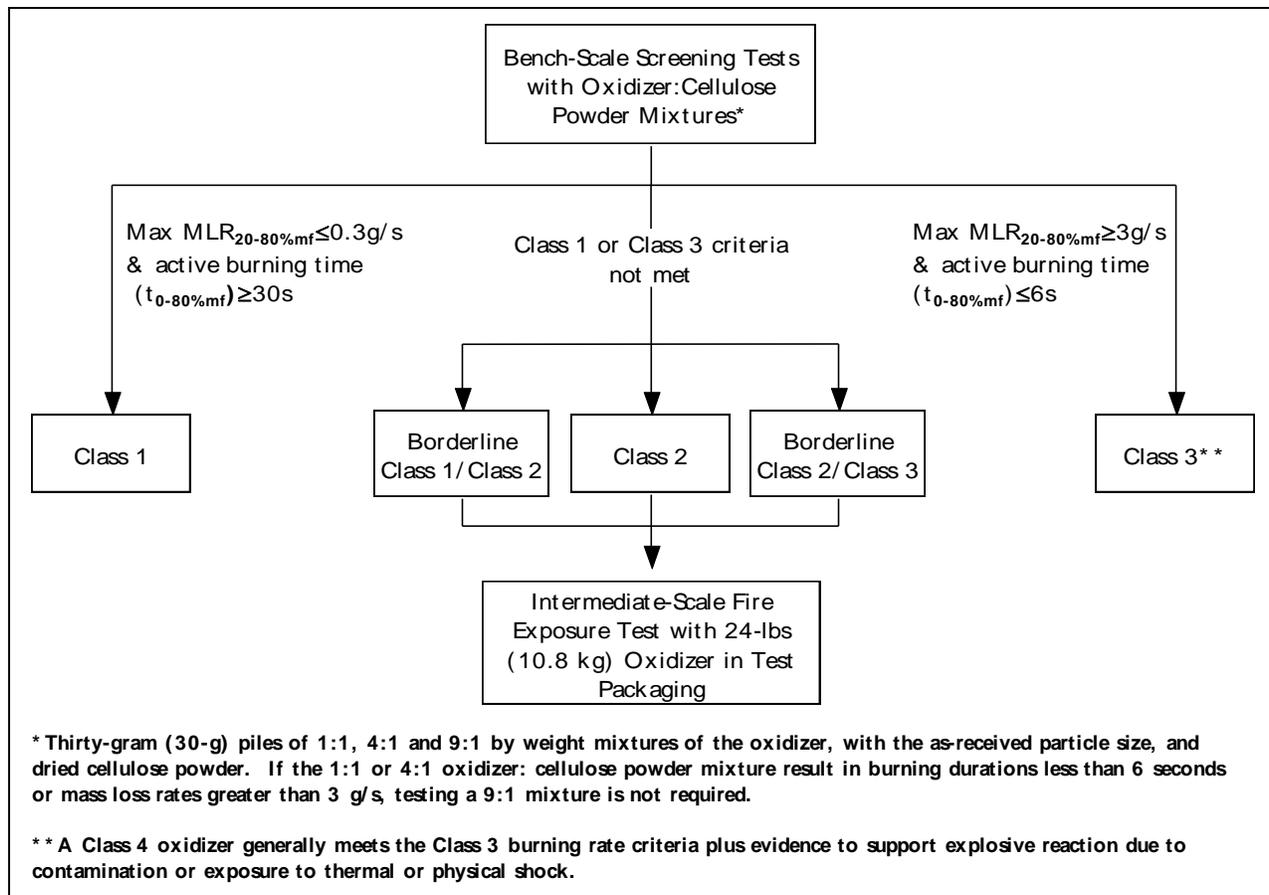


FIGURE 14. Bench-scale screening test criteria for assigning an oxidizer to Class 1, Class 2 or Class 3.