Assessing the List of Typical Oxidizers

Final Report

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FOREWORD

The purpose of this research project, was to subject solid oxidizers from NFPA 400 Hazardous Materials, Annex G.3 Typical Oxidizers to the bench-scale solid oxidizer test method recently added to the Annex of NFPA 400 to determine if the listed Class assignment is consistent with the established burning rate criteria.

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The content, opinions and conclusions contained in this report are solely those of the authors.

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Assessing the List of Typical Oxidizers

FPRF Code Fund Project

Report Date: December 30, 2013

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Executive Summary

The purpose of this Code Fund project was to subject solid oxidizers from NFPA 400 (2013) Hazardous Materials Code Annex G.3 Typical Oxidizers to the bench-scale solid oxidizer test method described in Annex G.1.4.3 to determine if the listed Class assignment is consistent with the established burning rate criteria in Annex Table G.1.2.

Seven solid oxidizers were selected from Annex G.3 Typical Oxidizers: one (1) Class 1, five (5) Class 2 and one (1) Class 3. Each oxidizer was mixed, in at least two concentrations, with dry cellulose powder and ignited with a glowing wire. The burning duration and mass loss rate data collected indicated that the classification of one (1) oxidizer remained unchanged, four (4) may be assigned to a higher Class and one (1) may be assigned a lower Class. Ammonium persulfate principally decomposed with minor flaming combustion; the resulting maximum mass loss rate resulted in a higher Class assignment based on test data. However, because the test data show that ammonium persulfate principally decomposed, it is recommended that the oxidizer remain Class 1. The listed Class 3 anhydrous sodium dichlor also principally decomposed; its maximum mass loss rate captured both decomposition and combustion and still resulted in a lower assignment to Class 2. Stopwatch burning time and temperature profiles were used to establish mass loss from burning and from decomposition. Four oxidizers exhibited a severe increase in burning rate with mass loss rates exceeding 15 grams per second—three of them are currently listed as Class 2 in NFPA 400. The results of the research project establish additional inconsistencies in NFPA 400 Annex G.3 Typical Oxidizers.

The bench scale test is useful to identify and distinguish between strong oxidizers and oxidizers that principally decompose. While one test and criteria are better than no data; one test and criteria are not meant to prevent, reduce or replace comprehensive hazard characterization, fire and thermal decomposition testing of oxidizers and formulated products containing oxidizers. It is recommended the intermediate scale burn test be performed when there are borderline Class results or questions regarding combustion and decomposition that may not be captured at the bench scale.

1. Background

Before the development of tests and criteria for assigning solid oxidizers to an NFPA Class was established and published in NFPA 400 (2013) Annex G, oxidizers were classified based on the subjective NFPA definitions and in some cases the list of typical oxidizers in the Annex of the Code. According to Annex G.3.1, the list of oxidizers in G.3.2 through G.3.5 are “provided to clarify how the committee has classified typical oxidizers”. Over various editions of NFPA 430 Code for the Storage of Solid and Liquid Oxidizers, the subjective definitions of Class 1, Class 2, Class 3 and Class 4 oxidizers changed while the list of typical oxidizers in the Annex remained the same. Test data was required to change the Class of an oxidizer or formulated oxidizer in the Annex.
The industry-supported, Fire Protection Research Foundation (FPRF) oxidizer research project, published in 2009, developed objective tests and criteria to assign solid oxidizers to Class 1, Class 2, or Class 3 based on burning rate defined by mass loss rate and active burning time of ignited mixtures. The project resulted in a bench-scale test where 30 gram mixtures of the oxidizer and dry cellulose powder are exposed to a glowing wire ignition source and, an intermediate-scale test with 10.9 kg (24-lbs) of solid oxidizer in combustible test packaging exposed to a propane fuel burner under a calibrated hood equipped with instrumentation to measure heat release rate based on gas temperature rise data. The tests and criteria are summarized in NFPA 400 Annex Table G.1.2, reproduced in Table 1 below, with a detailed description of the test methodologies in NFPA 400 Annex G.1.2 though G.1.5.6 (see Appendix 1).

At the bench scale, burning rate is measured as mass loss and as active burning time calculated from the time stamped mass loss data. This method of collecting mass data, or gravimetry, during combustion involving oxidizers has been subject to Round Robin evaluation and is the basis of a new UN Test O.3 Gravimetric test for oxidizing solids.1,2 From the Round Robin evaluation, statistical analysis of the test data concluded that "mass loss based parameters yield improved results regarding the discriminatory power and the probabilities of wrong classification compared to time-based (only) parameters....and is highly recommended”. At the intermediate-scale, the Class criteria are based on the peak convective heat release rate, based on gas temperature rise data, and active burning time from radiant heat flux transducer data. The heat release rate is one of the most important parameters in evaluating the potential fire hazard of a material because its maximum value is the numerical representation of the peak intensity of a fire.

The oxidizer project resulted in tests to assign oxidizers with a range in burning behavior to a Class for storage where before, there were ad hoc test data to no test data. One conclusion of the FPRF oxidizer project was that there were several inconsistencies between various oxidizers’ listed Class and the Class assigned after testing.

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1 BAM 2011 Final Report Evaluation of the 3rd Round Robin on Solid Oxidizer Test (UN O.1) with Calcium Peroxide, Sodium Nitrate, Sodium Perborate Monohydrate
Table 1. NFPA Oxidizer Class Tests and Criteria (Metric Units)

<table>
<thead>
<tr>
<th>Class</th>
<th>Maximum Mass Loss Rate * (g/s)</th>
<th>Active Burning Time ** (s)</th>
<th>Peak Convective Heat Release Rate (kW)</th>
<th>Active Burning Time Based on Radiant Heat Flux Profile*** (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class 1</td>
<td>≤0.3</td>
<td>≥30</td>
<td>≤100</td>
<td>≥120</td>
</tr>
<tr>
<td>Class 2</td>
<td>0.3&lt;max MLR&lt;3</td>
<td>6&lt; t&lt;sub&gt;active&lt;/sub&gt; &lt;30</td>
<td>100–200</td>
<td>60&lt; t&lt;sub&gt;active&lt;/sub&gt; &lt;120</td>
</tr>
<tr>
<td>Class 3</td>
<td>≥3</td>
<td>≤6</td>
<td>≥200</td>
<td>≤60</td>
</tr>
<tr>
<td>Class 4</td>
<td>A Class 4 oxidizer meets the Class 3 burning rate criteria plus has evidence to support explosive reaction due to contamination or exposure to thermal or physical shock (as determined from Tests 1(b), 3(a) or 3(b) in the United Nations ST/SG/AC.10/11/Rev.5, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, fifth revised edition, 2009).</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

MLR: Mass loss rate
*Max MLR: the maximum mass loss rate of a test mixture from 20% to 80% the final measured mass
**t<sub>active</sub>: active burning time calculated from first measurable mass loss to time of 80% final measured mass
***Calculated from the mass loss profiles.
Calculated from the width of the curve at ½ the peak radiant heat flux.

2. Oxidizers
Seven solid oxidizers were selected for testing for this study. The oxidizers with their CAS number, source, lot or batch number and the current listed Class in NFPA 400, are listed in Table 2.

Table 2. Oxidizers Selected for Evaluation.

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>CAS No.</th>
<th>Source</th>
<th>Batch/Lot No.</th>
<th>NFPA Class</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium persulfate</td>
<td>7727-54-0</td>
<td>Manufacturer</td>
<td>70101</td>
<td>1</td>
</tr>
<tr>
<td>Sodium dichloro-s-triazinetrione (96-98%)</td>
<td>2893-78-9</td>
<td>Oxy Chem</td>
<td>454C096417</td>
<td>3</td>
</tr>
<tr>
<td>Sodium permanganate monohydrate (&gt;97%)</td>
<td>79048-36-5</td>
<td>Sigma Aldrich</td>
<td>MKBK1584V</td>
<td>2</td>
</tr>
<tr>
<td>Sodium peroxide (97%)</td>
<td>1313-60-6</td>
<td>Sigma Aldrich</td>
<td>MKBJ2816V</td>
<td>2</td>
</tr>
<tr>
<td>Potassium dioxide</td>
<td>12030-88-5</td>
<td>Sigma Aldrich</td>
<td>11119DCV</td>
<td>2</td>
</tr>
<tr>
<td>Sodium chlorite (40%)</td>
<td>7758-19-2</td>
<td>Sigma Aldrich</td>
<td>BCBF36304</td>
<td>2</td>
</tr>
<tr>
<td>Sodium chlorite (80%)</td>
<td>7758-19-2</td>
<td>Sigma Aldrich</td>
<td>BCBF36304</td>
<td>3</td>
</tr>
</tbody>
</table>
Anhydrous sodium dichloro-s-triazinetrione (96-98%) (sodium dichlor) is an algaecide, microbiocide/microbiostat, disinfectant, sanitizer, bactericide, fungicide and bleaching agent.³ Anhydrous sodium dichlor is listed as a Class 3 oxidizer in Annex G 3.4; sodium dichlor dihydrate is listed in G 3.2 and established by testing to be a Class 1 oxidizer.

Ammonium persulfate is used in various products and chemical processes. Examples include use as a radical polymerization initiator in polymer chemistry, as an etchant and cleaner of printed circuit boards, and as a booster in hair bleaching formulations.⁴ Before potassium bromate and calcium peroxide, ammonium persulfate was an UN Test O.1 packing group (PG) III reference oxidizer. The burning behavior of ammonium persulfate was studied extensively by Hasegawa.⁵

Sodium permanganate is used in aqueous solutions as etchants in printed circuitry and is gaining popularity in water treatment.

Sodium peroxide was used to bleach wood pulp for the production of paper and textiles. Presently it is mainly used for specialized laboratory operations, e.g. the extraction of minerals from various ores.⁶

Potassium dioxide is used as an oxidizing agent in industrial chemistry, as a carbon dioxide scrubber, water dehumidifier and oxygen generator in rebreathers, spacecraft, submarines and spacesuit life support systems.

Sodium chlorite is used in the manufacture of paper and as a precursor for generation of chlorine dioxide gas.

3. Methodology
The bench-scale solid oxidizer test method was used to evaluate the burning behavior of the oxidizers listed in Table 2. An overview photograph of the test set-up in a laboratory hood is shown in Figure 1. Additional photographs of the bench scale test equipment and set up are contained in Appendix 2. Time stamped mass data was collected at 0.2 second intervals; temperature data at each thermocouple was collected a 1 second intervals. The tests were documented with photographs and video recordings. The cellulose powder moisture content after drying in an oven at 108°C for minimum four hours was less than 0.5 weight percent. Notes and observations recorded during each trial included the current and voltage

⁶ Wikipedia
of the wire; a description of the burning behavior such as ease of ignition, swelling and discoloration of the pile, approximate flame height, color of gaseous products evolved; the stopwatch burning time from energizing the wire to the end of visible flaming; and the time when the wire opened, if at all. The laboratory temperature and relative humidity during testing were 18°C to 20°C and 51 percent to 65 percent, respectively.

The combustion of 30 gram mixtures of 1:1 and 4:1 potassium dioxide with cellulose powder and the 1:1 and 4:1 40% and 80% sodium chlorite mixtures with cellulose powder overwhelmed the hood operating on the high blower setting and/or was too energetic. Energetic mixtures were ignited outdoors in a three-sided, drywall lined enclosure. The tests were documented with video recordings. Mass data was not recorded when using the drywall lined enclosure.

Figure 1. Bench-scale test set-up in laboratory hood.
4. Summary of Results

The results of the bench scale test with oxidizer cellulose powder mixtures are summarized in Table 3.

<table>
<thead>
<tr>
<th></th>
<th>1:1</th>
<th>4:1</th>
<th>9:1</th>
<th>Listed Class</th>
<th>Class after testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium persulfate</td>
<td>0.3±0.02</td>
<td>29.9±1.8</td>
<td>1.8±0.5</td>
<td>8.8±2.5</td>
<td>1.2±0.3</td>
</tr>
<tr>
<td>Sodium dichloro-s-triazinetrione</td>
<td>0.5±0.1</td>
<td>43.7±4.7</td>
<td>1.0±0.1</td>
<td>23.2±0.9</td>
<td>0.8±0.1</td>
</tr>
<tr>
<td>Sodium permanganate monohydrate (&gt;97%)</td>
<td>2.1±0.2</td>
<td>10.7±1.3</td>
<td>11.3±1.3</td>
<td>2.0±0.4</td>
<td>-</td>
</tr>
<tr>
<td>Sodium peroxide (97%)[note 1]</td>
<td>2.9</td>
<td>6.2</td>
<td>5.2±1.0</td>
<td>3.0±1.8</td>
<td>-</td>
</tr>
<tr>
<td>Potassium dichlorate[note 2]</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>-</td>
</tr>
<tr>
<td>Sodium chloride (40%)[notes 3,4]</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>-</td>
</tr>
<tr>
<td>Sodium chlorite (80%)[note 4]</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>&gt;15</td>
<td>&lt;1</td>
<td>-</td>
</tr>
</tbody>
</table>

*Data shown as average ± standard deviation calculated over five trials unless noted otherwise.*

[Note 1] 1:1 data from two trials; 4:1 data averaged over four trials
[Note 2] The data for potassium dichlorate is based on visual assessment and burning time not mass loss data.
[Note 3] The 40% NaClO3 mixture was made by diluting the 80% NaClO2 using (NaCl) salt.
[Note 4] The data for the sodium chlorite mixtures are based on visual assessment and burning time and not mass loss data.

5. Detailed Description of Oxidizers

A detailed description of each oxidizer’s bench-scale test, burning behavior and resulting Class assignment is provided below. Each oxidizer trials test data, mass loss and temperature profiles, and photographs are provided in the appendices. The data collected and/or calculated from each trial for each mixture with dried cellulose performed in the hood in the referenced appendices include the time the wire opened in seconds, the final measured mass from the mass loss profiles, the calculated mass at 20% and 80% the final measured mass, the mass loss rate from data points closest to the calculated 20% to 80% the final measured mass, the mass loss rate linear regression r-squared value, the active burning time in seconds and the maximum temperature above the pile.

**Ammonium persulfate.** Ammonium persulfate (NH₄)₂S₂O₈, FMC Lot No. 70101) is a clear, yellow crystalline solid. The 1:1 mixture swelled, spread black/brown discoloration and low flaming over the pile; the flames extinguished and the pile continued to decompose without flaming combustion. The stopwatch burning times of the 1:1 mixtures ranged from 33 to 37 seconds. The wire opened in four of the five trials. The time at which the wire opened ranged from 2 minutes 16 seconds to 4 minutes 11 seconds—well after flaming at the pile ceased. The rate of gas evolution during decomposition visibly decreased when the wire opened. The peak flame height from the 1:1 mixtures was approximately 6 inches above the test platform. The average peak temperature above the pile was 472±84°C. The mass loss rate, calculated from the mass at 40 seconds after the first measured mass loss and during which
flaming combustion was observed, was 0.31±0.02g/s; the active burning time was 29.9±1.8s. When the residue after testing was measured and compared with the initial 30-gram mixture, the average total mass loss was 51wt%.

The 4:1 mixtures spread black discoloration and low flaming while swelling until the flames went out; the stopwatch burning times ranged from 15 to 22 seconds. The pile continued to evolve gaseous products until the wire opened, which occurred in four of the five trials. The shortest time at which the wire opened was 2 minutes 3 seconds well after flaming combustion had ceased. Peak flame heights were less than 6-inches above the pile. The average maximum temperature above the pile was 316±80°C. In four of five trials, swelling of the pile involved the wire coinciding with and causing an inflection in the mass data. The final measured mass was taken as the mass at the point of inflection. The average mass loss rate was 1.8±0.5 g/s; the average active burning time was 8.8±2.5s.

The 9:1 mixtures swelled black while burning with low flames never reaching three inches above the pile. The persulfate-rich mixture continued to evolve gaseous products while the wire was energized. The peak temperature at the thermocouples above the pile ranged from 62°C to 140°C. In four of five trials, swelling of the pile involved the wire coinciding with and causing an inflection in the mass data. The final measured mass was taken as the mass at the point of inflection. The average mass loss rate was 1.2±0.3 g/s; the average active burning time was 9.4±1.1s.

Representative photographs during peak burning of the 1:1, 4:1 and 9:1 mixtures are shown in Figure 2. Data tables, corresponding mass and temperature profiles, and additional photographs taken during the bench scale tests with ammonium persulfate are contained in Appendix 3.

Based on the 4:1 mixture maximum mass loss rate and active burning time, ammonium persulfate is assigned to Class 2. However, ammonium persulfate exhibited Class 1 burning behavior e.g., it did not cause a moderate increase in burning rate. Visual observations, low peak temperatures, off gassing during and after flaming combustion from the ammonium persulfate rich mixtures indicate that the mass loss was due primarily to decomposition rather than combustion; therefore, it is recommended that the oxidizer remain Class 1.
Sodium dichlor. Sodium dichloro-s-triazinetrione (NaC₃N₃O₃Cl₂, Occidental Chemical Corporation OxyChem ACL® 60 Chlorinating Composition, 96 to 98% sodium dichlor, 0.5 to 3% water, 0.1 to 1% sodium chloride) is a white granular solid with a strong chlorine odor. Mixtures with cellulose were prepared with the material as-received. As this material is somewhat hygroscopic, it was stored in a desiccator until use.

The 1:1 sodium dichlor-cellulose powder mixtures ignited slowly and often at the rear of the pile where the glowing wire was exposed. Both low flames and decomposition evolving gaseous products spread over the pile. Flame height increased and the rate of gas evolution decreased with increasing time. However, flame spread was slow. Peak flame height of the 1:1 mixtures was greater than 9 inches from the test platform. The peak temperature from the temperature profiles ranged from 785°C to 1,171°C. Flaming combustion ceased at 82 seconds to 112 seconds after energizing the wire followed by decomposition. The average active burning time from mass loss data was 43.7±4.7 seconds. After burning and decomposing, the average final mass remaining from the initial 30-grams was 5.3 grams of low density black residue. The average mass loss rate was 0.49±0.06 with all mass loss profiles from 20 to 80% the final measured mass having r-squared values 0.97 and greater.
Upon energizing the wire, the 4:1 mixtures evolved copious yellow-white gaseous products with minor to no visible flame. In two of the five trials, there were either intermittent or no flames observed. The wire used for the 4:1 mixtures typically opened in less than 36 seconds. For the mixtures that did ignite, measuring the stopwatch burning time was difficult since the flames were intermittent. The maximum temperature above the pile during the trials ranged from 84°C to 167°C. The 4:1 mixture exhibited an average mass loss rate double (1.04±0.09 g/s) the 1:1 mixture and half the active burning time (23±0.9 seconds) compared to the 1:1 mixtures. Visual observations, low peak temperatures, copious off gassing and intermittent flames from the 4:1 mixture indicate that the mass loss was due primarily to decomposition rather than combustion.

Upon energizing the wire, the 9:1 mixtures evolved copious white gaseous products with minor to no visible flame. The wire was continuous in two of five trials; the wire opened in less than one minute, after 20 seconds and after 35 seconds during the remaining three trials. The maximum temperature above the pile during the trials ranged from 115°C to 135°C. The average mass loss rate was intermediate at 0.78±0.6g/s between the 1:1 and 4:1 mixtures. The active burning time from mass data was also intermediate at 27.8±2.2 seconds between the 1:1 and 4:1 mixtures.

The mass loss of sodium dichlor mixtures were both the result of decomposition and flaming combustion. There clearly was more decomposition than combustion with increasing concentration of sodium dichlor in the mixtures. The mass loss profiles exhibited exponential type decay over time. While sodium dichlor anhydrous is listed in Annex G.3 as a Class 3 oxidizer, the maximum mass loss rate and active burning time of the 4:1 mixture met the Class 2 test criteria.

Representative photographs are shown in Figure 3. Data tables, corresponding mass and temperature profiles, and additional photographs taken during the bench scale tests with anhydrous sodium dichlor are contained in Appendix 4.
**Sodium permanganate.** Sodium permanganate monohydrate (NaMnO₄·H₂O, Sigma Aldrich, Lot No. MKBK1584V) is a dark, crystalline solid. The 1:1 mixtures ignited readily, first momentarily releasing gaseous products then burning with flames and minor smoke. The peak flame height exceeded 9 inches above the test platform. The stopwatch burning times averaged 35 seconds from energizing the wire to the end of flaming combustion; the active burning time from the first measurable mass loss to 80% final measured mass was 10.7±1.3 seconds. Peak temperatures above the pile ranged from 1050°C to 1,270°C. The mass loss rate was 2.09±0.23 g/s; each profile had corresponding linear regression r-squared value greater than 0.95. After one of the trials, the mass remaining on the test platform after cooling was measured. The final mass was 11.57g compared with the initial 30-g, or 62% consumed.
The 4:1 mixtures ignited readily and burned rapidly. The mass loss rate of the 4:1 mixtures (11.32±1.31 g/s) was greater than the 1:1 mixtures (2.09±0.23 g/s). The stopwatch time of flaming combustion was less than 4 seconds; the average active burning time was 2.0±0.4 seconds. The peak temperatures above the pile ranged from 816°C to 967°C. Representative photographs are shown in Figure 4. Data tables, corresponding mass and temperature profiles, and additional photographs taken during the bench scale tests with sodium permanganate are contained in Appendix 5.

Sodium permanganate, listed as Class 2, exhibited Class 3 burning behavior. Combustion was more vigorous than potassium permanganate (1.6 g/s and 3.5 g/s for 1:1 and 4:1 potassium permanganate mixtures, respectively). Both the maximum mass loss rate and active burning time of sodium permanganate readily met the Class 3 criteria.

Sodium peroxide. Sodium peroxide (Na₂O₂, Sigma Aldrich, Lot MKBJ2816V, 97% granular +140 mesh), a yellow particulate solid, mixed with dry cellulose powder had delayed ignition from energizing the wire to

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7 Potassium permanganate was assigned to Class 3 based on the intermediate scale burn test data.
first visible flame on the order of 3 seconds. Once ignited, the mixture burned rapidly with peak flame heights exceeding 24 inches and copious generation of gaseous products. Two trials of the 1:1 mixture were burned in the drywall lined enclosure; two trials were performed in the hood with mass loss data. The 1:1 mixtures burned intensely at the test platform with orange flames and white gaseous products. The stopwatch burning time of the 1:1 mixtures ranged from 49 to 109 seconds. The active burning time from mass loss data for two trials were 5.8 and 6.6 seconds. The stopwatch burning times were long due to sustained low flames after peak burning.

One 4:1 mixture had a 2 to 3 second delayed ignition yet once ignited burned rapidly with bright burning at the test platform and peak flame heights well over 24 inches from the test platform. During the other four trials, the sodium peroxide rich mixtures ignited readily. The 4:1 mixture had significantly shorter burning durations than the 1:1 mixture. The stopwatch burning time of the 4:1 mixture ranged from 13.6 to 26.8 seconds. The average active burning time based on mass loss data was 3.0±1.8s; the corresponding mass loss rate was 5.2±1.0g/s. Peak temperatures for both the 1:1 and 4:1 mixtures were greater than 500°C. The sodium peroxide mixtures were observed to liquefy and flow as a result of combustion; lingering low flames were concentrated on the perimeter of the pool. Representative photographs of sodium peroxide are shown in Figure 5. Appendix 6 contains mass loss and temperature profiles and additional photographs taken during tests with sodium peroxide.

Sodium peroxide exhibited Class 3 burning behavior. Both the maximum mass loss rate and active burning time data met the Class 3 criteria.
Potassium dioxide. Potassium dioxide (KO₂, Sigma Aldrich, Lot 11119DV) is a fine, yellow powder. The first trial with a 30-gram mixture of 1:1 potassium dioxide:cellulose overwhelmed the hood with gaseous products of combustion (Fig. 6). A bright, pink-colored ball of flame was observed. Additional 30-gram 1:1 and 4:1 mixtures were ignited in the drywall test enclosure and video recorded. The burning behavior was the same as observed in the hood. The stopwatch burning time of the 1:1 and 4:1 mixtures were both less than 1 second. The 1:1 mixture combustion was a bright flash fire followed by a gaseous cloud of combustion products; the remains were a black dispersed residue. The 4:1 mixture combustion was a flash accompanied by an audible ‘boom’ followed by a gaseous plume. There were minor remains after combustion (e.g., near 100% consumed). The 1:1 mixture mass loss and temperature loss profiles are contained in Appendix 7. The 4:1 mixtures were documented with video recordings; mass loss and temperature data were not collected.

Potassium dioxide, listed as typical Class 2, clearly exhibited Class 3 burning behavior. Both the maximum mass loss rate and active burning time of the oxidizer met the Class 3 criteria.

Sodium chlorite. The oxidizer sodium chlorite (NaClO₂, 80%, Sigma Aldrich Lot BCBF36304) is a fine white powder. A 40% concentration was prepared by diluting 80% sodium chlorite with ground salt (NaCl) before mixing with dried cellulose powder. The 1:1 mixtures were tested in the laboratory hood with mass loss and temperature data; the 4:1 mixtures were tested in the drywall test enclosure and videorecorded. The 1:1 sodium chlorite (40%) mixtures ignited readily and burned rapidly. Bright, orange flames reached heights well in excess of 24 inches from the test platform; the reaction left a low density
black residue dispersed in the hood. Combustion was accompanied by copious amounts of gaseous products. Representative photographs of ignition and near peak burning of a 1:1 and 4:1 sodium chlorite 40% mixture are shown in Figures 7 and 8, respectively. The mass loss profile from 4 trials with 1:1 mixtures is shown in Figure 9. The data and profiles show that 23 to 27 grams of the material were lost during combustion in 0.4 to 0.6 seconds.

The 4:1 mixture ignited readily producing a ball of flame followed by a cloud of gaseous products (Figure 8). The duration of combustion was less than 1 second. The 40% sodium chlorite mixtures dispersed flaming particles from the test platform. Additional photographs, mass loss and temperature profiles of the 1:1 40% sodium chlorite:cellulose powder mixture are contained in Appendix 8. The combustion reaction was so fast that elevated temperatures were not recorded by the thermocouples.

Sodium chlorite (40%) is listed as a Class 2 oxidizer. Both the maximum mass loss rate and active burning time of the 40% sodium chlorite oxidizer met the Class 3 criteria.
Figure 9. Mass loss profiles from 1:1 sodium chlorite (40%): cellulose mixtures.

The sodium chlorite (80%): cellulose mixtures were tested in the drywall lined test enclosure. Video recordings were made during the tests. Appendix 9 contains photographs during the tests. Both mixtures ignited readily and burned rapidly. The 4:1 mixtures were accompanied by an audible boom. The 80% sodium chlorite is listed as a Class 3 oxidizer consistent with its severe increase in burning behavior.

6. Discussion
The maximum allowable quantities (MAQ) for oxidizers per control area in NFPA 400 Hazardous Materials Code Chapter 5 are based on the oxidizer Class. The determination of Class is based on information available to the technical committee in the form of a list of typical oxidizers or, more recently, on test data. The list of typical oxidizers, while provided for informational purposes only, is widely used and reproduced.
NFPA 400 now includes tests and criteria for users to assign solid oxidizers and products containing oxidizers to Class 1, Class 2 or Class 3. The purpose of this Code Fund project was to evaluate select solid oxidizers from the list of typical Class 1, Class 2, and Class 3 oxidizers to determine if their current Class is consistent or conflicts with the Class assigned based on test data. From the current project evaluating seven solid oxidizers, the data indicate that one (1) was consistent with its assigned Class (Class 3 sodium chlorite (80%)); four (4) resulted in higher Class/higher hazard assignment (ammonium persulfate, sodium permanganate, sodium peroxide and 40% sodium chlorite), and one (1) oxidizer resulted in a lower Class assignment (anhydrous sodium dichlor-s-triazinetrione). While test data indicated assignment to Class 2, ammonium persulfate exhibited significant decomposition after the main combustion period.

The results of the current project are consistent with the results of previous testing in that a number of oxidizers exhibited characteristics from a Class different than assigned under Annex G.3 typical oxidizers. The results of previous testing showed five (5) oxidizers had Class assignment after testing higher than the assigned Class prior to testing. Class 1 oxidizers calcium peroxide (75%) and sodium persulfate (99%) met the Class 2 criteria; Class 2 potassium perchlorate (99%), calcium hypochlorite (48% available chlorine) and potassium permanganate (>97.5%) met Class 3 criteria.  

Users of NFPA 400 now have the option of testing or utilizing the list of typical oxidizers published in the Annex G.3. The results of previous and current testing have shown a number of inconsistent oxidizer Class assignments between the listed Class and actual burning behavior. In a majority of the oxidizers tested, the oxidizer was assigned to a higher hazard Class. The maximum allowable quantities per control area between Class 1, Class 2 and Class 3 oxidizers can be orders of magnitude different. If an oxidizer is not properly classified, the protection criteria used to establish the control area may not be adequate. In contrast, if an oxidizer by testing is found to be a lower Class assignment than the listed Class then the protection criteria per control area should be more than adequate.

Oxidizers that principally decompose can present challenges when determining Class. The maximum mass loss rate may reflect decomposition as opposed to combustion whereas the current Class criteria are based on burning rate. Burning rate and decomposition are identified and recognized as separate oxidizer hazards. From NFPA 400 (2013 Edition), an oxidizer is defined as any solid or liquid material that readily yields oxygen or other oxidizing gas or that readily reacts to promote or initiate combustion of combustible materials and that can, under some circumstances, undergo a vigorous self-sustained decomposition due to contamination or heat exposure. A number of oxidizers decompose when exposed to heat or flame. The specific hazards of decomposing oxidizers include toxic, corrosive and/or explosive gaseous products and potential to over-pressurize a container or building. The hazards associated with decomposition of

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specific oxidizers are often contained in safety data sheets. For example, the safety data sheet for anhydrous sodium dichlor states: if heated by outside source to temperatures above 240°C, this product will undergo self-sustaining decomposition with the evolution of heat and dense noxious gases but no visible flame. Ammonium persulfate’s safety data sheets similarly states: decomposes in storage under conditions of moisture and/or excessive heat causing release of oxides of sulfur, nitrogen and oxygen that supports combustion.

The bench- and intermediate- scale tests are based on burning rate and the data clearly distinguishes between slow burning, low mass loss rate Class 1 oxidizers and vigorous burning, higher mass loss rate Class 3 oxidizers. Oxidizers that decompose have corresponding mass loss and the mass loss from combustion and decomposition are distinguishable when mass loss data is supplemented with temperature profiles and video recordings. The test report for bench scale testing of oxidizers should include a description of burning behavior including decomposition. Oxidizers that principally decompose during the bench scale test may be subject to additional testing at the intermediate scale or listed in a separate Annex of both.

7. Conclusion
The solid oxidizer test protocol is referenced in the NFPA 400 (2013) Hazardous Materials Code definitions of Class 1, Class 2 and Class 3 oxidizers and is detailed in Annex G. Oxidizer Class is used to determine the MAQ per control area based on use and occupancy. From the current project’s test results of seven solid oxidizers, one (1) was consistent with its assigned Class (Class 3 sodium chlorite (80%); four (4) resulted in higher Class/higher hazard assignment (ammonium persulfate, sodium permanganate, sodium peroxide and 40% sodium chlorite), and one (1) oxidizer resulted in a lower Class assignment (anhydrous sodium dichlor-s-triazinetrione). While assigned to Class 2 based on test data, ammonium persulfate exhibited Class 1 burning behavior and it is recommended ammonium persulfate remain listed as Class 1. A number of oxidizers tested that are currently listed as Class 2 exhibited Class 3 burning behavior.

Currently, NFPA 400 Annex G contains conflicting data: Annex G.2 lists oxidizers based on test data while Annex G.3 is the historical list of oxidizers typical of Class 1, Class 2, Class 3 and Class 4. The G.3 list is flawed in part because the definition of Class 1, Class 2 and Class 3 oxidizer have changed over time without concurrent evaluation of those oxidizers listed as typical. The conflicting data leads to orders of magnitude difference in MAQs for the oxidizer based on Class. The data is provided for the technical committee’s use for listing or classifying typical oxidizers in Annex G.3.

One test (e.g., the bench scale test) and criteria are not meant to limit, prevent, reduce or replace a complete hazard characterization of oxidizers and formulated products to include enhanced burning rates and self-accelerating decomposition. It is recommended the intermediate scale burn test be performed.
when there are borderline or questions regarding combustion and decomposition that may not be captured at the bench scale.

8. Appendices
Appendix 1
NFPA 400 Annex G.1  General.

G.1.1 Solid Oxidizers. Oxidizers can have both physical and health hazards. The tests and criteria described in sections G.1.2 - G.1.5 are based on burning rate only and do not address other physical hazards of oxidizers, such as thermal instability and chemical reactivity, or health hazards of gaseous products generated during combustion or decomposition. If confined, gaseous products generated from oxidizer decomposition can result in overpressure events and explosions. Cumulative research test data indicate the burning rate of solid oxidizers is principally a function of the chemical composition of the oxidizer and its concentration, and to a lesser degree, its physical form (e.g., powder, granular or tablets). Inclusive in the influence of the oxidizer composition are the amount and type of additives (if any) as well as the moisture content of the oxidizer or hydrated salt additives. The tests and criteria apply to oxidizers or formulations containing oxidizers that are found in commerce, use, and storage and do not apply to the evaluation of ammonium nitrate, oxidizer-containing explosive mixtures, and pyrotechnic mixtures. The tests and criteria assign an oxidizer to an NFPA Class for storage and are not applicable to and should not be confused with Department of Transportation (DOT) and United Nations (UN) Packing Group (III, II, I) assignments and vice versa. The UN and DOT Division 5.1 solid oxidizer packing groups are different from, and not synonymous with, the NFPA Class (1, 2, 3, 4) assignments for storage, handling and use. The NFPA Classes for solid oxidizers are also different from the hazard categories (Categories 3, 2, 1) for oxidizing solids defined by the Global Harmonization System (GHS) adopted by the revised OSHA Hazard Communication Standard in March, 2012.

The oxidizer classification method consists of two reaction-to-fire tests: (1) a bench-scale test with 30 g mixtures of oxidizer and dried cellulose exposed to a glowing wire and (2) an intermediate-scale test with 10.9 kg of oxidizer in combustible test packaging exposed to a constant external fire source. The key physical indicators of burning rate measured in the intermediate-scale test are peak convective heat release rate and the burning time calculated from the radiant heat flux profile. The key physical indicators of burning rate measured in the bench-scale test are mass loss rate and burning time calculated from the mass data. Either of these tests may be used to classify a solid oxidizer. If both of these tests are conducted and the results differ, then the results from the intermediate scale fire exposure test data should be used as the basis for the classification.

Referenced Documents:


ASTM E 2067 Standard Practice for Full-Scale Oxygen Consumption Calorimetry Fire Tests


Medium-Scale Burn Testing: Determination of Total (Oxygen Consumption and Carbon Dioxide Generation) and Convective (Temperature Rise) Heat Release Rates of Various Chemical Oxidizers, Final Report, Jason P. Huczek, Southwest Research Institute, San Antonio TX, January 7, 2009, SwRI Project No. 01.13538.01.303.


G.1.2 Tests and Criteria. Oxidizers and formulated products containing an oxidizer shall be assigned to Oxidizer Class 1, Class 2, Class 3, or Class 4 according to the tests and criteria specified in Table G.1.2(a) and Table G.1.2(b).

<table>
<thead>
<tr>
<th>Class</th>
<th>Bench-Scale Test [30 g mixtures with cellulose powder]</th>
<th>Intermediate-Scale Fire Exposure Test [10.9 kg in test packaging]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Maximum Mass Loss Rate * (g/s)</td>
<td>Peak Convective Heat Release Rate (kW)</td>
</tr>
<tr>
<td></td>
<td>Active Burning Time ** (s)</td>
<td>Active Burning Time Based on Radiant Heat Flux Profile *** (s)</td>
</tr>
<tr>
<td>Class 1</td>
<td>( \leq 0.3 )</td>
<td>( \leq 100 )</td>
</tr>
<tr>
<td>Class 2</td>
<td>( 0.3 &lt; \text{maxMLR} \leq 3 )</td>
<td>( 100 - 200 )</td>
</tr>
<tr>
<td>Class 3</td>
<td>( \geq 3 )</td>
<td>( 60 &lt; t_{\text{active}} &lt; 120 )</td>
</tr>
<tr>
<td>Class 4</td>
<td>A Class 4 oxidizer meets the Class 3 burning rate criteria plus has evidence to support explosive reaction due to contamination or exposure to thermal or physical shock (as determined from Tests 1(b), 3(a) or 3(b) in the United Nations ST/SG/AC.10/11/Rev.5, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, fifth revised edition, 2009).</td>
<td>( \geq 200 )</td>
</tr>
</tbody>
</table>

MLR: Mass loss rate

*Max MLR: the maximum mass loss rate of a test mixture from 20% to 80% the final measured mass

\( t_{\text{active}} \): active burning time calculated from first measurable mass loss to time of 80% final mass loss

** Calculated from the mass loss profiles.

***Calculated from the width of the curve at ½ the peak radiant heat flux.
Table G.1.2(b) NFPA Oxidizer Class Tests and Criteria (U.S. Units)

<table>
<thead>
<tr>
<th>Class</th>
<th>Bench-Scale Screening Test [0.066 lb mixtures with cellulose powder]</th>
<th>Intermediate-Scale Fire Exposure Test [24 lb in test packaging]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Maximum Mass Loss Rate *(lb/min)</td>
<td>Active Burning Time <strong>(min)</strong></td>
</tr>
<tr>
<td>Class 1</td>
<td>≤0.04</td>
<td>≥0.5</td>
</tr>
<tr>
<td>Class 2</td>
<td>0.04&lt;MLR &lt;0.4</td>
<td>0.1 &lt; t&lt;sub&gt;active&lt;/sub&gt; &lt;0.5</td>
</tr>
<tr>
<td>Class 3</td>
<td>≥0.4</td>
<td>≤0.1</td>
</tr>
<tr>
<td>Class 4</td>
<td>A Class 4 oxidizer meets the Class 3 burning rate criteria plus has evidence to support explosive reaction due to contamination or exposure to thermal or physical shock (as determined from Tests 1(b), 3(a) or 3(b) in the United Nations ST/SG/AC.10/11/Rev.5, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, fifth revised edition, 2009).</td>
<td></td>
</tr>
</tbody>
</table>

MLR: Mass loss rate
*Max MLR: the maximum mass loss rate of a test mixture from 20 to 80% the final measured mass
** Active: active burning time calculated from first measurable mass loss to time of 80% final mass loss
***Calculated from the mass loss profiles.
****Calculated from the width of the curve at ½ the peak radiant heat flux.

G.1.3 Definitions.

G.1.3.1 Active Burning Time. Duration of burning with flaming combustion, or decomposition with minor flaming combustion, calculated from test data. From the bench-scale test data, the active burning time is calculated as the time from first measurable mass loss to the time at which 80 percent of the total mass loss has occurred. From the intermediate-scale radiant heat flux test data, the active burning time is the width of the curve at one-half the peak radiant heat flux.

G.1.3.2 Burning Behavior. The complete characterization of a material or mixture’s reaction to fire including both visual descriptors and test data.

G.1.3.3 Burning Rate. The principal physical hazard of oxidizer fires described by the intensity or quantity of energy release, in the form of heat and light or accelerated fire, and time duration over which the energy is released.

G.1.3.4 Fire-test-response Characteristic. A response characteristic of a material, product, or assembly to a prescribed source of heat or flame, under controlled fire conditions; such response characteristics may include, but are not limited to, ease of ignition, flame spread, heat release, mass loss, smoke generation, fire resistance, and toxic potency of smoke [ASTM E 176-13, Standard Terminology of Fire Standards].

G.1.3.5 Formulated Product. A product that contains a solid or liquid oxidizer as one of its constituents, including admixtures. Some oxidizers are not sold in pure form (e.g., lithium
hypochochlorite, calcium hypochochlorite). Formulations include chemical by-products and unreacted raw materials inevitably present in the product and intentionally added diluents and exothermic control agents, such as hydrated salt crystals coarsely blended with the manufactured oxidizer.

G.1.3.6 Heat Release Rate (HRR). The thermal energy released per unit time by an item during combustion under specified conditions. [ASTM E 176-13, Standard Terminology of Fire Standards].

G.1.3.7 Mass Loss. The change in mass of a solid material to gaseous products during combustion or decomposition expressed per unit quantity of material (g/g, %).

G.1.3.8 Mass Burning Rate. Mass loss per unit time by materials burning under specified conditions. [ASTM E 176-13, Standard Terminology of Fire Standards].

G.1.3.10 Test Packaging. Packaging for solid (granular, tablet, or powder) oxidizers during the intermediate-scale fire exposure test consisting of six, high-density polyethylene (HDPE) containers in a double-wall corrugated board carton.

G.1.3.11 Visual Burning Duration. Duration (s, min) of visible flaming combustion, as measured with a stopwatch.

G.1.4 Bench-Scale Test. The solid oxidizer (granular, powder, crystalline) is evaluated in the particle size and distribution intended for storage, handling, and use. For tablets, the pre-tablet or raw material is tested. Thirty gram (30 g) mixtures (1:1, 4:1, and 9:1 by mass) of the oxidizer or formulated product and dry cellulose powder are exposed to a glowing wire energized to 150 Watt on a datalogging laboratory balance in a laboratory hood. The mass loss rate of each mixture is calculated from test data. The mixture with the highest or maximum mass loss rate is compared to the mass loss rate and burning rate duration criteria in Table G.1.2. The bench-scale test is capable of distinguishing weak oxidizers (Class 1), strong oxidizers (Class 3), and oxidizers that principally decompose.

G.1.4.1 Test Materials. Cellulose Powder. Dried fibrous cellulose with a fiber length between 50 microns and 250 microns and a mean diameter of 25 microns is used as the combustible material. The cellulose powder is spread in a pan so the depth of powder is less than or equal to 0.25 in. and dried in a laboratory oven at 221°F (105°C) for 4 hours. After drying, the powder is transferred to a desiccator cabinet with desiccant. When cool, the moisture content is measured using an analytical moisture analyzer or by gravimetric analysis. The moisture content must be below 0.5 percent w/w when measured at 105 °F. Suitable cellulose is currently available as Technocel 75 from CFF GmbH & Co. KG, Arnstädter Strasse 2, 98708 Gehren, Germany, www.dff.de, and was previously available as CF-11 from Whatman.

Oxidizer. A solid (granular, crystalline, powder, prill or tablet) containing an oxidizer functional group requiring evaluation for assignment to Class 1, Class 2, Class 3 or Class 4. The oxidizer should be documented as-received to include: CAS number of the oxidizer(s), chemical composition if a mixture or formulation, form, and, if granular, the particle size and distribution measured with certified sieves and recorded as mass percent of each particle size fraction.

G.1.4.2 Instrumentation and Test Set-Up.
**Laboratory Balance.** A laboratory balance is positioned and leveled inside a clean laboratory hood. The hood air stream velocity should be 0.5 m/s (1.6 ft/s) or less measured at the face of the hood and capable of capturing all gaseous products generated. An apparatus such as a metal frame, constructed of welded steel and having two pins at the diagonal of the weighing surface, is used to center scale protection materials. The pins do not contact the laboratory balance but function to center protection materials. The laboratory balance programmable response-time settings are optimized to establish the data output interval. Data collection at 0.2, 0.5 or 1 second intervals are typical. Data-logging parameters are similarly set at the computer interface and software to collect and record time-stamped mass data. Auto-calibration functions of the balance, if any, are disabled during tests.

**Protection Materials.** The laboratory balance weighing pan is protected with three or more layers of high-temperature ceramic fiberboard. For example, a balance weighing surface with dimensions 0.15 m x 0.15 m (6 in. x 6 in.) would be protected with a bottom or base layer of Kaowool HT board (0.30 m x 0.30 m x 0.013 m; 12 in. x 12 in. x \(\frac{1}{8}\) in.). The base layer extends beyond the balance to capture material ejected from the test platform. Two openings in the base layer correspond to the location of the centering pins of the apparatus. The middle layer is Kaowool HT board (0.178 m x 0.178 m x 0.013 m; 7 in. x 7 in. x \(\frac{1}{8}\) in.), and the top layer, or test platform, is Kaowool PM board (0.114 m x 0.114 m x 0.006 m; 4 \(\frac{1}{2}\) in. x 4 \(\frac{1}{2}\) in. x \(\frac{1}{4}\) in.). The base layer of protection remains on the balance after each trial.

Due to heat retained from the glowing wire and/or test mixtures, the two top layers of ceramic board on the base layer are removed after each test. The middle and top layers of HT board can be reused after cleaning and cooling to room temperature.

A suitable barrier is constructed around the laboratory balance and protection materials to prevent air currents from the operating hood from influencing balance stability. For example, a barrier may consist of up to three 0.0127 m (\(\frac{1}{2}\)-in) thick fiber boards — one on each side of the balance extending from the back of the hood to the front of the hood and one in the back and clear plastic in the front secured to the sides with tape. The hood air velocity should not interfere with balance stability.

**Thermocouples (Optional).** Three, K-type air thermocouples (TC) are secured to a ring stand to one side of the protected lab balance. The tips of the thermocouples are centered above the test platform. The distance from the surface of the test platform recommended to capture the range in plume temperatures are 7.6 cm (3 in.), 15.2 cm (6 in.), and 22.8 cm (9 in.). A fourth K-type thermocouple monitors the ambient temperature. The TCs are attached to the data acquisition system programmed to record the plume temperatures over the same time interval as the lab balance (e.g., 0.2, 0.5 and 1 second intervals). The fixed-height thermocouples are also used to estimate peak flame height during a test.

**Ignition source.** A wire (for instance Nichrome) is formed as specified in Figure G 1.4.2. The length of the wire is 30±1 cm (11.8 in.); the wire diameter is <1 mm (<0.039 in.). A variable laboratory power supply is used to regulate the voltage and current to the wire to achieve 150±7 Watts when energized. It is very important that the power connection to the heating wire is flexible enough to avoid any influence on the proper operation of the balance. For example, conductors from the power supply output may be secured to one or more ceramic terminal blocks. The first terminal block, secured with a clamp at the rear of the lab balance, has
conductors to a second ceramic terminal block near the test platform, and the looped wire is inserted in the second terminal block to complete the circuit. The looped portion of the wire is centered over the test platform.

**G.1.4.3 Test Methodology.**

*Test Mixtures.* Three concentrations of oxidizer-cellulose powder mixtures evaluated are (1) 1:1 by mass mixtures containing 15 g oxidizer and 15 g dried cellulose powder; (2) 4:1 by mass mixtures containing 24 g oxidizer and 6 g dried cellulose powder; and (3) 9:1 by mass mixtures containing 27 g oxidizer and 3 g dried cellulose powder. Five trials of each concentration are tested. Each mixture should be prepared individually and not taken from a pre-mixed batch.

*Single Test Procedure.* Each mixture of oxidizer and dried cellulose powder is prepared immediately prior to each trial in a test series. A second laboratory balance is used to weigh the required amounts of cellulose powder and oxidizer to the nearest 0.1 gram. The materials are transferred to and combined in a clean, labeled container (500 mL) with a screw top lid. Mixing of the contents is done by inverting (not shaking) the container no less than 25 times. After mixing, the contents of the container are transferred into a funnel (70 mm diameter, 102 mL volume, 60 degree angle) with a sealed stem. The funnel with the mixture is tamped lightly. The funnel with material is inverted so that the conical pile of material is centered over the looped ignition wire. This may be done by putting the wire and test platform over the funnel and then inverting the entire assemblage. Alternatively, using cardstock paper, the funnel with the mixture is inverted over the looped wire ignition source on the test platform. When the cardstock is removed, gently twisting the funnel over the wire allows for uniform settling of the mixture around the ignition wire on the test platform. The funnel is removed, leaving the conical pile of material centered on the wire.

The balance is tared to zero. The sash of the hood is lowered and the hood blower is energized. The laboratory temperature and relative humidity are recorded. If necessary, the balance is re-tared to zero. Once balance stability is confirmed, data acquisition is initiated and baseline data is collected for at least 10 seconds before the wire is energized at the power supply. A stopwatch is activated when the wire is energized. Mass loss and the temperature data are collected until spreading discoloration and/or visible flaming combustion cease. In the event of little or no flaming combustion, mass and temperature data are collected for 5 minutes.

After each trial, whether the wire is open or continuous, the electrical power to the wire is shut off, the wire is disconnected, and the top two layers of balance protection materials with the test residue are removed.

A new wire shall be used for each test.

A video-recorded demonstration of the single test procedure is available at www.nfpa.org/foundation.

**G.1.4.4 Test Sequence.** The oxidizer:cellulose powder mixtures are tested in the following sequence: 1:1 (5 trials), 4:1(5 trials), then 9:1(5 trials). If either the 1:1 or 4:1 mixture results in vigorous burning with stopwatch burning durations less than 6 seconds and/or the calculated peak mass loss rate is greater than 3 g/s, the 9:1 mixture does not need to be tested.
G.1.4.5 Test Data. **Visual.** Photographs and/or video recordings are used to document, at a minimum, one representative trial in a test series. Visual observations should be recorded and may include: the peak flame height above the test platform; 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.

**Calculated.** The profile of each burning test is examined by plotting the mass loss data as a function of time. The percent material consumed is calculated from the final measured mass loss (m_f) over the initial mass (m_i or 30.0 g) of the mixture times 100%. The mass loss rate is then calculated from the time at 20% and 80% the final measured mass loss. For example, the balance with a 30.0 g mixture is tared to and starts with a reading of 0.0 g and, at the completion of combustion, ends with a reading of -20.0 g. The percent material consumed is 20.0/30.0g x 100 or 67%. The mass loss rate is calculated from the time at 0.20 x 20.0 g or 4.0 g to the time at 0.80 x 20.0 g or 16.0 g. The mass loss rate is preferably calculated by performing a least squares linear fit of the mass loss data between 0.20 m_f and 0.80 m_f. The coefficient of correlation (R^2) of the mass curve from each trial should be at least 0.90 between 20% to 80% mass loss. The active burning time is the time from first measurable mass loss to the time at 0.80 m_f. First measurable mass loss is defined as, upon energizing the wire, the first time-stamped mass data that leads a continuous series of decreasing mass at the pile as ignition, combustion, or decomposition occurs.

Mass loss rate and active burning time data are calculated for each trial within a series and are then used to calculate an average and sample standard deviation for the series of trials (e.g., 5 trials for 1:1 mixture). If the burning behavior is found to depend on the duration of wire continuity, the highest mass loss rate is used instead of the average; or a sufficient number of trials are conducted to obtain trials where the wire break occurs after the main reaction time.

G.1.4.6 Test Criteria. The bench-scale test criteria, based on the maximum mass loss rate from the averages) and the active burning time, are shown in Tables G.1.2(a) and G.1.2(b). The average active burning time and mass loss rate data for an oxidizer in 1:1, 4:1 and, if necessary, 9:1 oxidizer:cellulose powder mixtures are compared after completing the series of trials. The mixtures with the greater mass loss rate is identified; the maximum mass loss rate within that mixture is compared with the test criteria. For example, a 1:1 oxidizer:cellulose powder mixture have average mass loss rate and active burning time 2.1 +/- 0.2 g/s and 10.7 +/- 1.3 s, respectively. The 4:1 mixture of the same oxidizer with cellulose powder result in average mass loss rate and active burning time 11.3 +/-. 1.2 g/s and 2.0 +/- .4 s, respectively. The 4:1 mixture has the greater mass loss rate with a maximum mass loss rate 11.3 g/s + 1.2 g/s or 12.5 g/s. Using the maximum mass loss rate, active burning time and criteria in Table G.1.2(a), the oxidizer is assigned to Class 3.

G.1.5 Intermediate-Scale Fire Exposure Test. If the bench scale test results in borderline Class assignment (e.g., borderline Class 1-Class 2 or borderline Class 2-Class 3), the intermediate scale test should be performed. The intermediate scale test can be performed instead of the bench scale test. Otherwise, the intermediate scale test is optional. Twenty-four pounds (10.9 kg) of the solid oxidizer in the form and concentration intended for storage,
handling, and use are transferred to the combustible test packaging, is centered on a drywall-lined table and inside the opening of a U-shaped propane-fueled burner and ignited under a calibrated 2 MW hood instrumented in accordance with ASTM E 2067. The peak convective heat release rate, less the burner contribution, is calculated from gas temperature rise calorimetry equations. The duration of active burning is calculated from radiant heat flux transducers, less the burner contribution, at a fixed distance from the packaged test material. The peak convective heat release rate of the oxidizer in test packaging is compared to the peak convective heat release rate and active burning time criteria in Table G.1.2(a) or Table G.1.2(b).

G.1.5.1 Test Materials.

*Oxidizer.* The chemical composition and physical properties of the oxidizer or formulated product containing an oxidizer being evaluated are recorded. The oxidizer or formulated physical form is characterized as granular, crystalline, powder, prill, or tablet. The test material is not ground or subject to any mechanical attrition or sieving to reduce the particle size. The original packaging of the oxidizer is recorded.

*Combustible Test Packaging.* High-density polyethylene (HDPE) wide mouth containers with screw top lids in corrugated board cartons are used as combustible test packaging. The HDPE containers should not contain additives that would reduce the burn rate. The HDPE container specifications are summarized in Table G.1.5.1 (a). Suitable containers are currently supplied by Consolidated Plastics Inc. (Twinsburg, OH).

<table>
<thead>
<tr>
<th>Table G.1.5.1 (a). Description of HDPE Containers with Lids</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Volume</strong></td>
</tr>
<tr>
<td>Ounce (cm³)</td>
</tr>
<tr>
<td>60 +/- 0.5 (1.774)</td>
</tr>
</tbody>
</table>

The corrugated board carton should be double wall construction and not contain additives that would reduce the burn rate. The carton specifications are summarized in Table G.1.5.1 (b). Suitable cartons are made by JM Packaging-Detroit Tape and Label (Warren, MI).

<table>
<thead>
<tr>
<th>Table G.1.5.1 (b). Description of Cartons</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Height</strong></td>
</tr>
<tr>
<td>Inches (cm)</td>
</tr>
<tr>
<td>8 +/- 0.1 (20.3)</td>
</tr>
</tbody>
</table>

Although oxidizers and formulations containing oxidizers may be sold in other packaging, the standard test method utilizes combustible packaging consisting of six, HDPE containers (60 oz or 85 oz) with lids in a corrugated board carton. The 1,774 cm³ (60 oz) HDPE containers accommodate most oxidizer solids based on density; the 2,514 cm³ (85 oz) containers can be used for low density (i.e., powder form) oxidizers. Four pounds (1.8 kg) of oxidizer are transferred to each HDPE container. The HDPE containers are then arranged in the corrugated board carton with doubled-wall construction. The dimensions of the carton that enclose six,
1,774 cm$^3$ (60 oz) HDPE containers are 40.6 cm \times 25.4 \text{ cm} \times 20.3 \text{ cm} (16 \text{ in.} \times 10 \text{ in.} \times 8 \text{ in.}); the dimensions of the carton to enclose six 2,514 cm$^3$ (85 oz) HDPE containers are 40.6 cm \times 20.3 \text{ cm} \times 20.3 \text{ cm} (16 \text{ in.} \times 10 \text{ in.} \times 10 \text{ in})

G.1.5.2 Instrumentation and Test Set-Up.

Laboratory Balance. A laboratory balance is used to measure the oxidizer from the as-received packaging for transfer into the test packaging. The mass of oxidizer in each container and/or bag is measured and recorded.

Hood. A minimum 2 MW capacity hood designed and equipped with instrumentation described in ASTM E 2067, Standard Practice for Full-Scale Oxygen Consumption Calorimetry Fire Tests, is required to capture and measure the temperature of the gaseous products of combustion or decomposition. The skirt of the hood should be no more than 6 ft from the floor. For consistency between burns, the hood flow rate should be the same for the calorimetric calibration, the baseline burner and the test materials. Because oxidizers may evolve oxygen during combustion, gas temperature rise and not oxygen consumption calorimetry is used to calculate the heat release rate.

Burn Table. A non-combustible table, with a removable top and tray, is centered and leveled under the hood. The bottom of the tray is lined with $\geq 6.35 \text{ mm} (0.25 \text{ in.})$ thick drywall ($0.91 \text{ m} \times 0.91 \text{ m}; 3 \text{ ft} \times 3 \text{ ft}$). Four short (7.6 cm to 10.1 cm; 3 in. to 4 in.) walls made of non-combustible material are attached to the edges of the tray to contain and prevent the flow of material from the tray during a test. The table height is such that the bottom of the test package is 1.1 m (3ft 8 in.) from the floor.

Ignition Source. A U-shaped burner is constructed from threaded black gas pipe (1 in. NPS) and cast iron threaded fittings. The distance between the two legs of the U-shaped burner is 43.8 cm (17 ¼ in.); the length of each leg is 50.8 cm (20 ¼ in.). Each leg of the burner has 38 uniformly spaced openings oriented towards the interior each with a diameter 3.4 mm (0.13 in.); the pipe joining the two legs has 26 drilled openings. The carton is centered between the parallel burner legs and 4-inches from joining section opposite the open side of the U. The burner does not contact the protected load cell, table, or tray. Propane gas is supplied to the burner from a cylinder. The flow rate to the burner is adjusted using a regulator at the cylinder outlet. The flow rate to the burner is adjusted using the regulator so that the output of the burner is 38 kW (convective heat release rate). Note: Using the effective heat of combustion of propane (46.4 kJ/g), for a 38 kW fire, the mass flow rate of propane gas to the burner will probably be approximately 0.8 g/s.

Radiant Heat Flux Transducers. Two calibrated, water-cooled, Schmidt-Boelter or Gardon Gage incident radiant heat flux (0-50 kW/m$^2$) transducers are affixed to a rigid stand and positioned next to the table with the sensors center with the carton between the legs of the U-shaped burner. The distance from the floor to the lower target is 1.2 m (4 ft 2 in.); the distance between the lower and upper target is 0.45 m (18 in.). The distance from the heat flux sensors to the center of the table where the carton is positioned is 0.95 m (3 ft 2 in.). With respect to the test packaging, the lower heat flux transducer is located at the height of the top section of the carton (or pail). The second heat flux transducer is located at 0.4 m (16 in.) above the top of the carton.
Digital clock. A digital clock that will be visible and readable in the video recording of the test can be used.

Ambient Air Thermocouple (Required). The ambient air temperature is measured continuously during the test at the same data acquisition rate as the exhaust gas temperature. The ambient air thermocouple should be placed outside of the hood, far enough away to prevent interference from the burn test, but close enough to represent the ambient air in the room. The thermocouple should be protected from radiant heat from the burn test.

Thermocouples (Optional). Four, K-type fiberglass insulated thermocouples (TC) are attached to the carton. One TC is taped to the exterior bottom center of the carton; a second TC is taped to the exterior top center of the carton. A hole is punched through one side of the carton, through which two TCs are inserted. A hole is drilled through the lid of one HDPE container and a TC is inserted into the material inside. The hole in the lid is sealed with tape. The second TC is secured to the side exterior center of one HDPE container with the bead positioned between the container and the carton wall. The bead of the TC is exposed (e.g., not taped). Temperature data is recorded using a data acquisition system.

Data shall be collected at least every second for the calibration, baseline and test burns.

G.1.5.3 Test Methodology.

Before each test, the temperature and relative humidity inside the burn facility are recorded. At the start of a test, the hood is turned on and a 2-minute baseline of data is collected. The data should be collected at a frequency rate of at least 1 second; 5 second intervals is typical. The gas burner is ignited at 2 minutes and a timer is activated. The gaseous products of combustion and/or decomposition from the packaged oxidizer are entrained in the hood and exhaust collection duct. The burner remains on for the duration of a test and for sufficient time after enhanced burning of the packaged test material to return to burner baseline values. For example, for inert materials and weaker oxidizers, the burner remains on for up to 20 minutes. For stronger oxidizers, the burner remains on for 10 minutes. At the conclusion of a test, the regulator at the propane cylinder supplying fuel to the burner is closed. The test material is observed for residual fire or decomposition. The post-test residue is photographed and examined — specifically noting evidence of a phase change (i.e., molten) and the presence of unburned combustible packaging material(s). After cooling, the post-test residue is prepared for disposal.

A new test tray shall be used for each test.

G.1.5.4 Test Sequence. It is recommended that bench scale tests are performed prior to intermediate-scale testing to understand the potential burning behavior. A typical test series includes hood calibration, empty test packaging, then the oxidizer or formulation containing an oxidizer in the combustible test packaging. The following is test sequence is recommended each day of testing:

<table>
<thead>
<tr>
<th>Sequence</th>
<th>Burner Output, kW</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hood calibration per ASTM E2067</td>
<td>0, 40, 160, 0</td>
</tr>
<tr>
<td>Burner baseline with U-shaped burner only</td>
<td>38</td>
</tr>
<tr>
<td>Sample runs (1 or more) with U-shaped burner and packaged oxidizer</td>
<td>38</td>
</tr>
<tr>
<td>Burner baseline with U-shaped burner only</td>
<td>38</td>
</tr>
</tbody>
</table>
The calibration runs are used to calculate the C factor (see calculations). The baseline should be obtained at the same hood and gas flow rates as the test. The baseline runs should be conducted so that at least 5 minutes of stable data is obtained.

**G.1.5.5 Test Data.** *Visual.* Photographs and video recordings document the intermediate-scale fire exposure test of the oxidizer in test packaging, specifically the visual increase in burning behavior when the test packaging is compromised and the oxidizer is involved. Other notable observations include the generation, relative quantity, and color of gaseous products, flame color, and the presence or absence of residual flaming or decomposition after the burner is extinguished. Time resolved combustion gas analysis by Fourier Transform Infrared spectroscopy is optional.

*Calculated.* The convective heat release rate (Equation 1) is calculated from the mass flow rate (Equation 2) and temperature of combustion gases in the exhaust duct. The width of the curve at one-half the peak radiant heat flux is used to calculate the duration of active burning. The propane burner convective heat release rate is subtracted from the peak convective heat release rate to arrive at the heat release rate from the packaged oxidizer (Equation 3).

\[
q_{\text{conv}} = m c_p (T_g - T_a) \quad \text{Equation 1}
\]

where:
- \(q_{\text{conv}}\) = convective heat release rate (kW)
- \(m\) = mass flow in exhaust duct (kg/s)
- \(c_p\) = specific heat of combustion gas (kJ/kg-K)
- \(T_g\) = gas temperature (K)
- \(T_a\) = ambient temperature (K)

\[
m = \frac{C \sqrt{\Delta p}}{T_g} \quad \text{Equation 2}
\]

where \(C\) is the calibration constant measured for the calorimeter
\(\Delta p\) is the pressure drop at the bidirectional probe (Pa)

\[
q_{\text{oxidizer}} = q_{\text{conv}} - q_{\text{conv,burner}} \quad \text{kW} \quad \text{Equation 3}
\]

ASTM E 2067 Standard Practice for Full-Scale Oxygen Consumption Calorimetry Fire Tests provides details regarding hood instrumentation and calibration. Since combustion products consist primarily of excess air and nitrogen, it is reasonable to approximate the specific heat of the gas mixture in the exhaust duct, \(c_p\), by that of air. The specific heat of air is nearly independent of temperature and on the order of \(c_p,\text{air} = 1.01\) kJ/kg K.

The calibration constant is calculated using the following procedure from Trevino *et al*:

1. An “initial” calibration constant is calculated using the following equation:
   \[C = 22.1 \times A,\] where \(A\) is the area of the duct (in m²).
2. Propane or methane fuel is burned at a constant rate for a specific time interval (e.g. 10 min). The heat release rate is measured using oxygen consumption calorimetry and the “initial” value for C. The mass of fuel consumed is measured directly or from a calibrated mass flowmeter.
3) The total heat release is calculated from the mass of fuel consumed and net heat of combustion. Multiply this value by 0.98 to take into account efficiency of combustion.
4) Adjust the calibration constant, \( C \), so that the total heat release from the system calculations agrees with that calculated from the mass of fuel consumed.
5) Use this new constant for subsequent tests for calculations of HRR and volumetric flow rate.

Typically, a spreadsheet with equations are used to generate calibration and test material heat release rate profiles. The equations needed to calculate the total heat released for the calibration only may be found in ASTM E 2067.

\[
q_{T,O2} = [E \phi - (E - E_{CO}) \frac{1-\phi}{2} \frac{X_{O2}^A}{X_{O2}^o} \frac{M_{O2}}{M_a} \frac{m_e}{1+\phi(a-1)}] X_{O2}^o
\]

where

\[
\phi = \frac{X_{O2}^A (1 - X_{CO2}^A - X_{CO}^A) - X_{O2}^A (1 - X_{CO2}^A - X_{CO}^A)}{X_{O2}^A (1 - X_{O2}^A - X_{CO2}^A - X_{CO}^A)}
\]

The following values are measured:
- \( X_{O2}^o \) = measured mole fraction of O\(_2\) in incoming air
- \( X_{CO2}^o \) = measured mole fraction of CO\(_2\) in incoming air
- \( X_{CO}^o \) = measured mole fraction of CO in incoming air
- \( X_{O2}^A \) = measured mole fraction of O\(_2\) in exhaust flow
- \( X_{CO2}^A \) = measured mole fraction of CO\(_2\) in exhaust flow
- \( X_{CO}^A \) = measured mole fraction of CO in exhaust flow

The following constants are used in the calculations:
- \( E \) = net heat released per unit mass of O\(_2\) consumed, generic (13.1 MJ/kg)- this value should be used for burning of samples other than oxidizers
- \( E \) = net heat released per unit mass of O\(_2\) consumed, propane (12.8 MJ/kg)- this value should be used for burning of propane
- \( E_{CO} \) = net heat release per unit mass of O\(_2\) consumed, for CO (17.6 MJ/kg)
- \( M_a \) = molecular weight of incoming air (29 kg/kmol)
- \( M_{CO} \) = molecular weight of carbon monoxide (28 kg/kmol)
- \( M_{CO2} \) = molecular weight of carbon dioxide (44 kg/kmol)
- \( M_{O2} \) = molecular weight of oxygen (32 kg/kmol)
- \( a \) = combustion expansion factor (nondimensional) (1.084)
- \( \Delta H^\circ_{CO2} \) = net heat of complete combustion per unit mass of carbon dioxide generated (13.3 MJ/kg)
- \( \Delta H^\circ_{CO} \) = net heat of complete combustion per unit mass of carbon monoxide generated (11.1 MJ/kg)
- \( \rho_o \) = density of air at 273.15 K (1.293 kg/m\(^3\))
The baseline burner convective heat release rate ($q_{\text{conv}}$) is calculated using the Equation from ASTM 2067 then averaging over at least a 5 minute steady-state period. The two baseline averages (start and end) should then be compared and the average used for the baseline value used in the calculation of the oxidizer convective heat release rate (Equation 3). The active burning time is calculated from the width of the radiant heat flux-time profile at one-half the peak radiant heat flux.

**Test Report.** The following information and data to report should include:
- Any chemical analysis information available on the test sample
- SDS of test sample
- Weight of each individual HDPE container, empty and filled with sample.
- Total weight of sample, HDPE containers and carton.

For the calibration burns:
- Heat release rate versus time profile
- All measurements, calculations and constants used to calculate the $C$ factor.

For the baseline and test burns:
- Ambient and exhaust gas temperature versus time profiles
- Radiant heat flux versus time profile
- Convective heat release versus time profile with peak convective heat release indicated
- Burn times calculated from each heat flux transducer versus time profile
- All measurements, calculations and constants used to calculate the convective heat release
- Unusual burning behavior

**G.1.5.6 Test Criteria.** The intermediate-scale fire exposure test criteria, based on the peak convective heat release rate, are summarized in Table G.1.2(a) and Table G.1.2(b). If the peak convective heat release rate and active burning times result in assignment to different Classes, the heat release rate should be used to assign the oxidizer to a Class. A Class 4 oxidizer meets the Class 3 burning rate criteria plus there is evidence (such as results from in Tests 1(b), 3(a) or 3(b) in the United Nations ST/SG/AC.10/11/Rev.5, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, fifth revised edition, 2009) to support explosive reaction due to contamination or exposure to thermal or physical shock.
Appendix 2
Test Set Up
Test Set Up

- 6 -
Appendix 3
Ammonium Persulfate

1:1 Ammonium persulfate:CF11

Mass loss (g) vs. Time (s)

1:1 Ammonium Persulfate:CF11

Mass loss (g) vs. Time (s)
## 1:1 Ammonium Persulfate:Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened</th>
<th>Mf at 40s (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R²</th>
<th>Active burning time (s)</th>
<th>Max temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>251</td>
<td>10.5</td>
<td>2.1</td>
<td>8.4</td>
<td>0.29</td>
<td>0.98</td>
<td>27.2</td>
<td>553</td>
</tr>
<tr>
<td>2</td>
<td>136</td>
<td>9.42</td>
<td>1.84</td>
<td>7.53</td>
<td>0.28</td>
<td>0.99</td>
<td>29.4</td>
<td>451</td>
</tr>
<tr>
<td>3</td>
<td>140</td>
<td>8.05</td>
<td>1.61</td>
<td>6.44</td>
<td>0.31</td>
<td>0.98</td>
<td>30.2</td>
<td>460</td>
</tr>
<tr>
<td>4</td>
<td>230</td>
<td>9.71</td>
<td>1.94</td>
<td>7.76</td>
<td>0.33</td>
<td>0.99</td>
<td>32.2</td>
<td>348</td>
</tr>
<tr>
<td>5</td>
<td>199</td>
<td>8.7</td>
<td>1.74</td>
<td>6.96</td>
<td>0.31</td>
<td>0.99</td>
<td>30.6</td>
<td>551</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.31±0.02</td>
<td>-</td>
<td>-</td>
<td>29.9±1.8</td>
<td>472±84</td>
</tr>
</tbody>
</table>

## 4:1 Ammonium persulfate:CF11

![Graph showing mass loss over time for different series of experiments.](image)
### 4:1 Ammonium Persulfate: Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R²</th>
<th>Active burning time (s)</th>
<th>Max temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>9.35</td>
<td>1.87</td>
<td>7.48</td>
<td>0.82</td>
<td>0.97</td>
<td>13.2</td>
<td>312</td>
</tr>
<tr>
<td>2</td>
<td>159</td>
<td>15.85</td>
<td>3.17</td>
<td>12.68</td>
<td>2.18</td>
<td>0.99</td>
<td>7.2</td>
<td>470</td>
</tr>
<tr>
<td>3</td>
<td>Continuous</td>
<td>13.18</td>
<td>2.63</td>
<td>10.54</td>
<td>2.04</td>
<td>0.99</td>
<td>6.2</td>
<td>250</td>
</tr>
<tr>
<td>4</td>
<td>Continuous</td>
<td>13.09</td>
<td>2.61</td>
<td>10.47</td>
<td>1.87</td>
<td>0.99</td>
<td>7.4</td>
<td>300</td>
</tr>
<tr>
<td>5</td>
<td>123</td>
<td>15.19</td>
<td>3.04</td>
<td>12.15</td>
<td>2.20</td>
<td>0.98</td>
<td>10.2</td>
<td>252</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.8±0.5</td>
<td>-</td>
<td>8.8±2.5</td>
<td>316±80</td>
</tr>
<tr>
<td>Trial</td>
<td>Time wire opened (s)</td>
<td>Mf (g)</td>
<td>20%Mf (g)</td>
<td>80%Mf (g)</td>
<td>20-80% MLR (g/s)</td>
<td>20-80% MLR $R^2$</td>
<td>Active burning time (s)</td>
<td>Max temp above pile (°C)</td>
</tr>
<tr>
<td>-------</td>
<td>---------------------</td>
<td>--------</td>
<td>-----------</td>
<td>-----------</td>
<td>-----------------</td>
<td>-----------------</td>
<td>------------------------</td>
<td>-------------------------</td>
</tr>
<tr>
<td>1</td>
<td>Continuous</td>
<td>12.8</td>
<td>2.56</td>
<td>10.24</td>
<td>1.14</td>
<td>0.99</td>
<td>10.4</td>
<td>111</td>
</tr>
<tr>
<td>2</td>
<td>Continuous</td>
<td>8.85</td>
<td>1.77</td>
<td>7.08</td>
<td>1.24</td>
<td>0.98</td>
<td>7.8</td>
<td>68</td>
</tr>
<tr>
<td>3</td>
<td>214</td>
<td>6.98</td>
<td>1.39</td>
<td>5.58</td>
<td>0.57</td>
<td>0.97</td>
<td>10.4</td>
<td>140</td>
</tr>
<tr>
<td>4</td>
<td>Continuous</td>
<td>9.16</td>
<td>1.83</td>
<td>7.32</td>
<td>1.47</td>
<td>0.96</td>
<td>8.4</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>continuous</td>
<td>11.31</td>
<td>2.26</td>
<td>9.04</td>
<td>1.67</td>
<td>0.99</td>
<td>9.8</td>
<td>130</td>
</tr>
<tr>
<td>Avg±std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.21±0.37</td>
<td>-</td>
<td>9.4±1.1</td>
<td>105±30</td>
<td>-</td>
</tr>
</tbody>
</table>
1:1 Ammonium Persulfate:CF11

1:1 Ammonium Persulfate:CF11 T3

1:1 Ammonium Persulfate:CF11 T4
9:1 Ammonium Persulfate:CF11

9:1 Ammonium Persulfate:CF11 T3

9:1 Ammonium Persulfate:CF11 T4
9:1 Ammonium Persulfate: CF11 T5

![Graph showing data for 9:1 Ammonium Persulfate: CF11 T5]
Ammonium Persulfate
Ammonium Persulfate
Ammonium Persulfate
Ammonium Persulfate
Appendix 4
Sodium dichloro-s-triazinetrione, anhydrous

1:1 Sodium Dichlor: Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened (s)</th>
<th>Mf (g)</th>
<th>20% (g)</th>
<th>80% (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R^2</th>
<th>Active burning time (s)</th>
<th>Max temp. above pile (C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Continuous</td>
<td>23.76</td>
<td>4.75</td>
<td>19.00</td>
<td>0.49</td>
<td>0.99</td>
<td>45.4</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>66</td>
<td>23.20</td>
<td>4.64</td>
<td>18.56</td>
<td>0.59</td>
<td>0.99</td>
<td>35.8</td>
<td>874</td>
</tr>
<tr>
<td>3</td>
<td>105</td>
<td>27.88</td>
<td>5.57</td>
<td>22.30</td>
<td>0.48</td>
<td>0.99</td>
<td>44.0</td>
<td>815</td>
</tr>
<tr>
<td>4</td>
<td>94</td>
<td>25.00</td>
<td>5.00</td>
<td>20.00</td>
<td>0.47</td>
<td>0.97</td>
<td>43.2</td>
<td>785</td>
</tr>
<tr>
<td>5</td>
<td>60</td>
<td>22.80</td>
<td>4.56</td>
<td>18.24</td>
<td>0.39</td>
<td>0.97</td>
<td>50.4</td>
<td>1,171</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.49±0.06</td>
<td>-</td>
<td>-</td>
<td>43.7±4.7</td>
<td>911±177</td>
</tr>
</tbody>
</table>
### 4:1 Sodium Dichlor: Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened (s)</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80%MLR $R^2$</th>
<th>Active burning time (s)</th>
<th>Max temp. above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>38</td>
<td>20.9</td>
<td>4.18</td>
<td>16.72</td>
<td>1.17</td>
<td>0.99</td>
<td>22.6</td>
<td>84</td>
</tr>
<tr>
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<td>25</td>
<td>22.2</td>
<td>4.44</td>
<td>17.76</td>
<td>1.05</td>
<td>0.99</td>
<td>22.4</td>
<td>113</td>
</tr>
<tr>
<td>3</td>
<td>25</td>
<td>23.5</td>
<td>17.4</td>
<td>18.80</td>
<td>1.09</td>
<td>0.99</td>
<td>22.4</td>
<td>167</td>
</tr>
<tr>
<td>4</td>
<td>36</td>
<td>21.0</td>
<td>4.20</td>
<td>16.83</td>
<td>0.98</td>
<td>0.99</td>
<td>24.0</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>23</td>
<td>24.1</td>
<td>4.82</td>
<td>19.28</td>
<td>0.91</td>
<td>0.99</td>
<td>24.4</td>
<td>-</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.04±0.09</td>
<td>-</td>
<td>23±0.9</td>
<td>121±42</td>
<td></td>
</tr>
</tbody>
</table>
9:1 Sodium Dichlor Anhydrous:CF11

![Graph showing mass loss over time for different trials.]

### 9:1 Sodium Dichlor: Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R²</th>
<th>Active burning time (s)</th>
<th>Max. temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Continuous</td>
<td>22.1</td>
<td>4.42</td>
<td>17.68</td>
<td>0.75</td>
<td>0.99</td>
<td>30.8</td>
<td>135</td>
</tr>
<tr>
<td>2</td>
<td>58</td>
<td>22.1</td>
<td>4.42</td>
<td>17.68</td>
<td>0.88</td>
<td>0.99</td>
<td>26.8</td>
<td>125</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>18.2</td>
<td>3.64</td>
<td>14.56</td>
<td>0.76</td>
<td>0.98</td>
<td>25</td>
<td>129</td>
</tr>
<tr>
<td>4</td>
<td>35</td>
<td>20.0</td>
<td>4.00</td>
<td>16.00</td>
<td>0.81</td>
<td>0.99</td>
<td>27.2</td>
<td>117</td>
</tr>
<tr>
<td>5</td>
<td>Continuous</td>
<td>21.5</td>
<td>4.3</td>
<td>17.20</td>
<td>0.73</td>
<td>--</td>
<td>29.4</td>
<td>115</td>
</tr>
<tr>
<td>Avg±std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.78±0.06</td>
<td>27.8±2.27</td>
<td>124±8</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Sodium Permanganate

1:1 Sodium Permanganate: CF11

![Graph showing mass loss over time for 1:1 Sodium Permanganate: CF11]

1:1 Sodium Permanganate: Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened (s)</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R²</th>
<th>Active burning time (s)</th>
<th>Max temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No</td>
<td>18.31</td>
<td>3.66</td>
<td>14.64</td>
<td>2.20</td>
<td>0.97</td>
<td>9.0</td>
<td>1,270</td>
</tr>
<tr>
<td>2</td>
<td>No</td>
<td>21.29</td>
<td>4.25</td>
<td>17.03</td>
<td>2.11</td>
<td>0.98</td>
<td>12.0</td>
<td>1,109</td>
</tr>
<tr>
<td>3</td>
<td>No</td>
<td>20.56</td>
<td>4.11</td>
<td>16.44</td>
<td>1.82</td>
<td>0.97</td>
<td>12.0</td>
<td>1,210</td>
</tr>
<tr>
<td>4</td>
<td>No</td>
<td>20.82</td>
<td>4.16</td>
<td>16.65</td>
<td>1.93</td>
<td>0.98</td>
<td>10.6</td>
<td>1,050</td>
</tr>
<tr>
<td>5</td>
<td>No</td>
<td>19.80</td>
<td>3.96</td>
<td>15.84</td>
<td>2.43</td>
<td>0.97</td>
<td>10.0</td>
<td>1,050</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2.09±0.23</td>
<td>-</td>
<td>10.7±1.3</td>
<td>1,137±99</td>
<td></td>
</tr>
</tbody>
</table>

Note: The graph and table data are used in the context of the text. The graph shows the mass loss over time for sodium permanganate, while the table provides specific test data for each trial.
**4:1 Sodium Permanganate: Cellulose Powder Test Data**

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR $R^2$</th>
<th>Active burning time (s)</th>
<th>Max temp. above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>continuous</td>
<td>18.8</td>
<td>3.76</td>
<td>15.04</td>
<td>10.39</td>
<td>0.98</td>
<td>2.0</td>
<td>917</td>
</tr>
<tr>
<td>2</td>
<td>continuous</td>
<td>18.7</td>
<td>3.74</td>
<td>14.96</td>
<td>11.84</td>
<td>0.99</td>
<td>2.2</td>
<td>967</td>
</tr>
<tr>
<td>3</td>
<td>continuous</td>
<td>17.0</td>
<td>3.40</td>
<td>13.60</td>
<td>13.33</td>
<td>0.99</td>
<td>1.6</td>
<td>816</td>
</tr>
<tr>
<td>4</td>
<td>continuous</td>
<td>17.3</td>
<td>3.46</td>
<td>13.84</td>
<td>10.07</td>
<td>0.99</td>
<td>2.6</td>
<td>927</td>
</tr>
<tr>
<td>5</td>
<td>continuous</td>
<td>16.5</td>
<td>3.30</td>
<td>13.2</td>
<td>10.97</td>
<td>0.99</td>
<td>1.6</td>
<td>893</td>
</tr>
<tr>
<td>Avg±Std</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>11.32±1.31</td>
<td>-</td>
<td>2.0±0.42</td>
<td>904±56</td>
</tr>
</tbody>
</table>

**4:1 Sodium Permanganate:CF11**

![Graph showing mass loss over time for 4:1 Sodium Permanganate: Cellulose Powder Test Data]
1:1 Sodium Permanganate: CF11

1 to 1 NaMnO4:CF11 T5

Graph showing the concentration over time for 1:1 NaMnO4:CF11 T5.
Sodium Permanganate
Sodium Permanganate
Sodium Permanganate
Appendix 6
1:1 Sodium Peroxide: CF11

<table>
<thead>
<tr>
<th>Trial</th>
<th>Time wire opened</th>
<th>Mf (g)</th>
<th>20%Mf (g)</th>
<th>80%Mf (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR $R^2$</th>
<th>Active burning time (s)</th>
<th>Max temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No</td>
<td>19.2</td>
<td>3.84</td>
<td>15.36</td>
<td>1.33</td>
<td>0.95</td>
<td>5.8</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>No</td>
<td>12.5</td>
<td>2.5</td>
<td>10.0</td>
<td>4.47</td>
<td>0.99</td>
<td>6.6</td>
<td>-</td>
</tr>
</tbody>
</table>
## 4:1 Sodium Peroxide:Cellulose Powder Test Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Wire opened</th>
<th>MF (g)</th>
<th>20%MF (g)</th>
<th>80%MF (g)</th>
<th>20-80% MLR (g/s)</th>
<th>20-80% MLR R²</th>
<th>Active burning time (s)</th>
<th>Max temp above pile (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>no</td>
<td>13.5</td>
<td>2.7</td>
<td>10.8</td>
<td>3.5</td>
<td>0.98</td>
<td>5.8</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>yes</td>
<td>10.4</td>
<td>2.08</td>
<td>8.32</td>
<td>6.2</td>
<td>0.98</td>
<td>5.8</td>
<td>&gt;1200</td>
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<tr>
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<td>yes</td>
<td>nr</td>
<td>-</td>
<td>-</td>
<td>nr</td>
<td>-</td>
<td>1.083</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>yes</td>
<td>13.5</td>
<td>2.7</td>
<td>10.8</td>
<td>5.5</td>
<td>0.97</td>
<td>2.4</td>
<td>930</td>
</tr>
<tr>
<td>5</td>
<td>yes</td>
<td>12.6</td>
<td>2.52</td>
<td>10.08</td>
<td>5.9</td>
<td>0.99</td>
<td>2.4</td>
<td>1,323</td>
</tr>
<tr>
<td>Avg±Std</td>
<td>-</td>
<td>-</td>
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<td>5.2±1.0</td>
<td>-</td>
<td>3.0±1.8</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
1:1 Sodium Peroxide: CF11

1 to 1 40Na2O2:CF11 T1

1 to 1 40Na2O2:CF11 T2
4:1 Sodium Peroxide: CF11
4:1 Sodium Peroxide: CF11
Sodium Peroxide
Sodium Peroxide
Sodium Peroxide
Appendix 7
1:1 Potassium dioxide: CF11
Potassium Dioxide
Appendix 8
1:1 Sodium chlorite (40%): CF11

Mass loss (g) vs. time (s) for Series 1, 2, 3, and 4.
1:1 Sodium chlorite (40%):CF11
40% Sodium Chlorite
40% Sodium Chlorite
40% Sodium Chlorite
40% Sodium Chlorite
Appendix 9
80% Sodium Chlorite
80% Sodium Chlorite