

# APPENDIX 4

## INTERMEDIATE-SCALE FIRE EXPOSURE TESTS

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### Overview

Twenty-four pounds of granular, crystalline, powder or tablet-form oxidizer in combustible test packaging was exposed to a  $38\pm 3$  kW fire under an instrumented 2-MW hood. The external fire or burner was constant. The burner ignited the combustible test packaging and oxidizer and remained on a sufficient time after any enhanced burning. The burning behavior of the packaged test material and the burner baseline were monitored visually and with data profiles. The burning behavior of various oxidizers in the same packaging was characterized by the intensity or peak heat release rate and the duration of enhanced burning. Other data collected from ignition to the end of any enhanced burning involving the oxidizer included dynamic mass loss, product and surface temperatures, radiant heat flux, heat release rate, and smoke production. The burner was subtracted from the heat release rate and radiant heat flux profiles to arrive at the peak heat release rate and peak radiant heat flux from the packaged oxidizer. Each test was documented with photographs, thermal images and high resolution video recordings.

The instrumentation, test methodology and data reduction described below will be the basis of a step-by-step intermediate-scale fire exposure test method for assigning an oxidizer to a NFPA Class.

### Description of Test Set-Up

The ignition and fire exposure testing of a solid oxidizer in combustible packaging under an instrumented exhaust collection hood and duct is called open calorimetry. The set-up required the material to burn freely from ignition to consumption or extinguishment while the gaseous products of combustion and decomposition were captured by the hood. Air flow to and around the test specimen was unobstructed and symmetrical from all sides. Figure 1 is a photograph showing an overview of the test set-up and instrumentation under the 2-MW hood at Southwest Research Institute. A schematic drawing/plan view of the test set-up is shown in Figure 2.

The 2-MW hood, with a variable-speed blower (up to  $4\text{m}^3/\text{s}$ ), was designed and equipped with instrumentation described ASTM E 2067 (2008) *Standard Practice for Full-Scale Oxygen Consumption Calorimetry Fire Tests*. This practice provides details on the general requirements for construction, instrumentation, calibration and use of full-scale calorimeters when used to measure fire-test-response characteristics. Following the practices minimizes discrepancies between laboratories.

Instrumentation at the test material under the hood included a load cell, burn table, burner, two radiant heat flux targets, and a high temperature pyrometer. These were arranged and centered under the exhaust collection duct opening. The 3-ft x 3-ft steel

diamond plate deck, 400 pound capacity electronic load cell<sup>1</sup> was calibrated to 0.05 pound gradation and leveled on the test facility's floor. The load cell had a digital weigh indicator with analog output to a data acquisition. The surface of the load cell was protected with one sheet of ½ inch thick drywall. The welded metal burn table, with a removable tray, was centered and leveled on top of the protected load cell. The bottom of the tray was lined with ¼-inch thick drywall (3-ft x 3-ft). Four short walls were made using strips of drywall and taped to the tray bottom to contain and prevent the flow of material from the tray during a test. The packaged test material (e.g., carton or pail) was centered on the table and inside a U-shaped burner. The burner does not contact the protected load cell, table or tray. The U-shaped burner was constructed from threaded black gas pipe (1 inch NPS) and cast iron threaded fittings. The distance between the two legs of the U-shaped burner was 17¼ inches (43.8 cm); the length of each leg was 20 inches (50.8 cm). Each leg of the burner had 38 openings each with diameter 0.0135 inch (0.34 mm); the pipe joining the two legs had 26 drilled openings. When centered, the distance from the burner to the carton (16 in x 8 in x 8 in) was 4 inches (10.2 cm) on three sides; the third side was open.

The distance from the floor to the tray (on the protected load cell) was 3 feet 8 inches (1.1 meter); the distance from the tray to the opening of the exhaust collection duct was 10 feet 7 inches (3.2 meter).

Two calibrated, water-cooled, Schmidt-Boelter incident radiant heat flux (0-50 kW/m<sup>2</sup>) transducers<sup>2</sup> were affixed to a rigid stand and positioned next to the table with the sensors centered at the carton between the legs of the U-shaped burner. The distance from the floor to the lower target was 4 feet 2 inches (1.2 meters); the distance between the lower and upper target was 18 inches (0.45 meter). The distance from the heat flux sensors to the center of the table where the carton was positioned was 3 feet 2 inches (0.95 meter). With respect to the test packaging, the lower heat flux transducer was located at the height of the top section of the carton (or pail). The second heat flux transducer was located at 16 inches (0.4 meter) above the carton. A high temperature pyrometer<sup>3</sup> was positioned next to the heat flux transducer stand. Its circular target area was focused on the side of the carton centered on the table. The distance from the pyrometer lens to the closest face of the carton was 4 feet 10 inches (1.4 meters). The diameter of the surface temperature target area was 2¾ inches (70 mm). A witness pole with painted 12-inch (0.3 meter) markers was positioned next to the table as a visual aid for estimating flame height from the packaged test material. Figure 3 and Figure 4 are photographs showing the arrangement of the burn table, burner, combustible test packaging, heat flux transducer stand and pyrometer under the hood. The mass, radiant heat flux, temperature and pyrometer data were collected at 2 or 5 second intervals. Duct instrumentation data was collected a 5 second intervals. An FTIR spectrum of the gaseous products in a heated gas cell was collected every 30 seconds.

LP gas was supplied to the burner from a 100 pound (23.6 gallon) cylinder. The flow rate to the burner was adjusted using a regulator at the cylinder outlet. The burner's

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<sup>1</sup> Ricelake Scale (SN 139427) with 4 INCELL weigh cells (Model SBC 1-1K, SN A18667 CC 03-050, Class 111M) and a programmable with analog output IQ Plus 355 Digital Weight Indicator.

<sup>2</sup> Schmidt-Boelter, Model No. 64-5SB-20, S/N 149192 and 149191; Outer dia = 1 inch (2.5cm); sensor surface= 3.9 inches (10 cm), Medtherm Corp., Huntsville, AL.

<sup>3</sup> Infrared Radiation Pyrometer KT19II Serie, SN 2696, Spectral range 8-10um, temperature range 0-1500 degree C, Lens S925AR, 1\*ZR2 spacer, detector type B, 1s response time, Heitronics,

heat release rate and radiant heat flux average and sample standard deviation test data are summarized in Table 1. For a 38 kW fire and using the effective heat of combustion of propane (46.4kJ/g), the mass flow rate of propane gas to the burner was 0.82 g/s.

Two video cameras<sup>4</sup> were used to record each test. One video camera was a close-up view of the carton and burner; the second video camera recorded an overview of the test under the hood. A thermal imaging video camera<sup>5</sup> with a high temperature aperture was positioned at 10 feet (3.0 m) from the table. Still photographs were used to document the test set-up, test materials, and each test.

**TABLE 1. Propane Burner Baseline Heat Release Rate and Radiant Heat Flux**

Burner Baseline	HRR O <sub>2</sub> (kW)	HRR CO <sub>2</sub> (kW)	HRR Conv. (kW)	Lower HFT (kW/m <sup>2</sup> )	Upper HFT (kW/m <sup>2</sup> )
Initial	36±3	34±1	31±1	0.86±0.07	0.64±0.06
Final	39±4	38±3	32±1	0.62±0.06	0.51±0.04

*HFT=heat flux transducer*

*Data shown as average ± sample standard deviation over 3 minutes.*



<sup>4</sup> Sony Handycam w/ Hard Disk Drive, DCR-SR40 and DCR-SR220.

<sup>5</sup> FLIR T400 w/ high temperature option.



FIGURE 1. Photograph showing an overview of 2MW hood and the test set up and instrumentation under the hood.

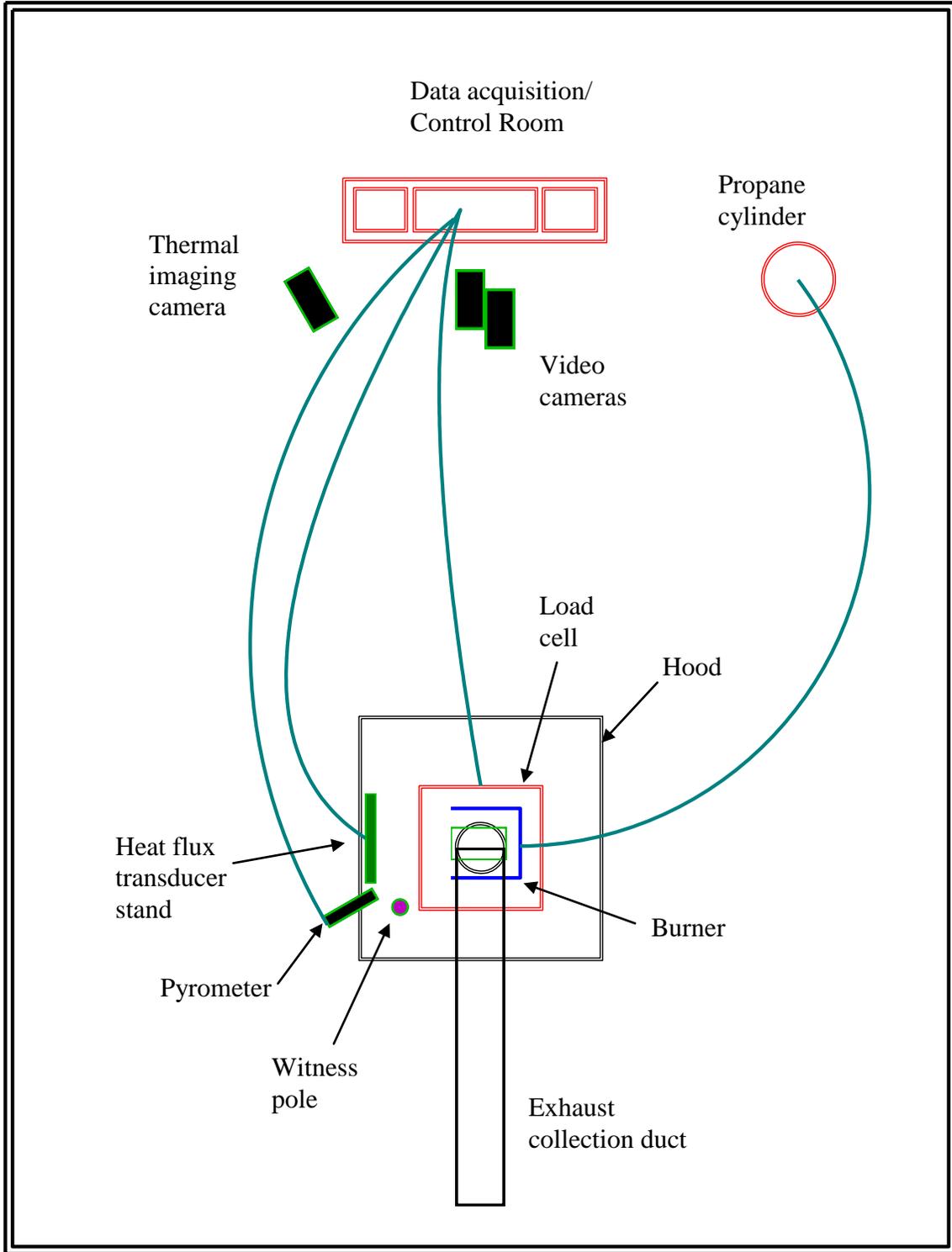


FIGURE 2. Top-down view of test set up and instrumentation.

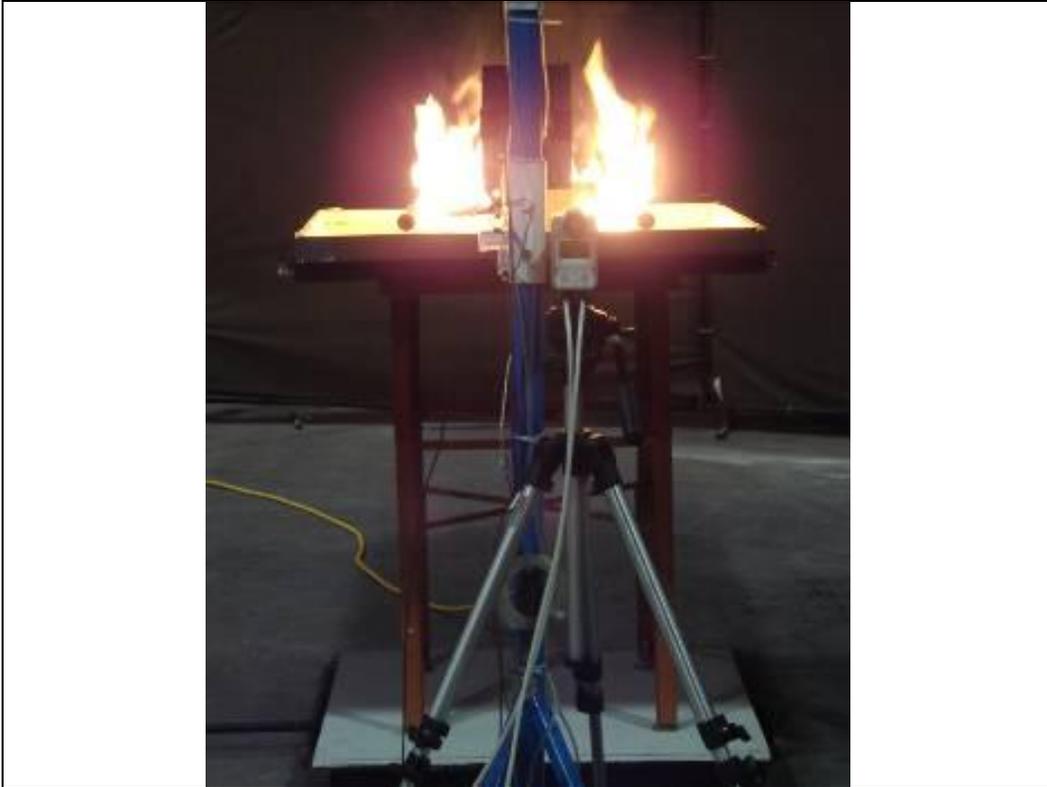


FIGURE 3. Radiant heat flux transducer stand and pyrometer directed at the center of the burner.



FIGURE 4. Profile of the burner and carton with test material centered on the tray and load cell.

## Oxidizer and Test Packaging

The oxidizer's original shipping packaging was documented. Twenty-four pounds of material (i.e., common salt or oxidizer) were transferred inside a laboratory hood into the test packaging. For a typical test, four pounds (1814 grams) of the oxidizer were transferred to each of six HDPE containers utilizing a laboratory balance. During transferring, a sample of test material was collected in labeled, 4-ounce glass jar with a Teflon lined lid. After transferring, the packaged oxidizer's initial mass was measured using a calibrated, 200-lb (90.7 kg) capacity (18 in x 18 in) electronic load cell<sup>6</sup>.

Six, 60 ounce HDPE containers inside a double-walled carton was used in a majority of the tests. Figure 5 shows the test packaging and two other packaging configurations that were evaluated. The other combustible packaging included a DW carton with twenty-four, one pound bags and a 2.5 gallon HDPE pail. The pail could accommodate 25 pounds of dry material.

Four, K-Type fiberglass insulated thermocouples (TC) were attached to each carton or pail. One TC was taped to the exterior bottom center of the carton or pail; a second TC was taped to the exterior top center of the carton or pail. When the packaging was a carton, a hole was punched through one side of the carton through which two TCs were inserted. If the carton contained HDPE containers, a hole was drilled through the lid of one container and a TC was inserted into the material inside. The hole in the lid was sealed with tape. The second TC was 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 was exposed (e.g., not taped). If packaged in 1-lb bags, the orientation of the bags was mixed vertically and horizontally inside the carton. A slit was made in one of the horizontal bags in the center of the carton through which a TC was inserted into the material. The TC was secured and the opening in the bag was sealed with tape. A second TC was taped to the exterior of one bag. If the packaging was a pail, a circular hole was drilled in the top of the pail through which two TCs were inserted. One TC was positioned inside the test material; the second TC was taped to the top inside of the lid. The opening in the lid of the pail was taped after securing the TCs. The individual thermocouples were joined to K-type extension wire to the data acquisition system.

Clear packing tape was used to seal the carton. An identification label with the test material and date were affixed to the exterior of the carton or pail.

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<sup>6</sup> Ricelake Scale (Stainless steel benchmark, SN 151671) calibrated to 0.01 lb gradation, with a programmable with analog output IQ Plus 355 Digital Weight Indicator



FIGURE 5. Combustible test packaging consisting of 6 HDPE containers in a DW carton(top). Other packaging evaluated were twenty four 1-lb bags in a DW carton (middle) and 2.5 gallon HDPE pails (bottom).

## **General Test Procedure**

The hood and instrumentation were calibrated with a propane sand-burner. The intermediate-scale fire exposure series of tests began and ended with a propane burner baseline.

Before each test, the temperature and relative humidity inside the burn facility were recorded. At the start of a test, a two-minute baseline of data was collected. The gas burner was ignited with a plumber's torch at two minutes and a digital clock was activated. The products of combustion and/or decomposition were entrained in the exhaust collection duct. The burner remained 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 inert materials and weaker oxidizers, the burner remained on for up to 20 minutes. For stronger oxidizers, the burner remained on for 10 to 12 minutes. During a test, the establishment of burner baseline values was determined visually and from the data collection profiles.

At the conclusion of a test, the regulator at the propane cylinder supplying fuel to the burner was closed. Video recordings and data acquisition continued for up to five minutes. The test material was observed for residual fire or decomposition. Minor residual fire was extinguished by smothering the flames with the test material and/or common salt. The table tray with the solid remains of the test material was moved to a staging area outside of the burn facility and allowed to cool. The post test residue was 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 was transferred to hazardous waste drums for disposal. Granular and tablet common salt were disposed of as non-hazardous waste.

## **Data Reduction**

The video recordings of each test were reviewed and used to create a timeline of events and observations. Timed events included the ignition and sustained burning of the combustible packaging, the first audible and/or visual involvement of the oxidizer, the peak in visual burning behavior, a clear reduction in flame height and when burner baseline values were reached. Other notable observations included the generation, relative quantity and color of gaseous products, flame color and the presence or absence of residual flaming or decomposition after the burner was extinguished.

The data collected during each test was used with standard equations to calculate the mass loss rate, heat release rates and smoke release rate. The rate at which combustion or decomposition products are generated is based on the change in pressure and temperature in the duct (Equation 1). On the floor, the material's dynamic mass loss rate was simultaneously measured (Equation 2).

$$\dot{m} = C \sqrt{\frac{\Delta p}{T_g}} = 22.1A \sqrt{\frac{\Delta p}{T_g}} \quad \text{Equation 1}$$

where,

$\dot{m}$  = mass flow rate (kg/s)

$p$  = pressuredrop at bidirectional probe (Pa)

$T_g$  = gas temperature (K)

$A$  = cross sectional area of duct (m<sup>2</sup>)

$$\dot{m}_{mat} = \frac{dm}{dt} \quad \text{units (kg/min)} \quad \text{Equation 2}$$

The heat release rate can be estimated from gas temperature (Equation 3) and from the composition of the combustion gas or gaseous species generation (Equation 4). Annexes A1 and A2 in ASTM E 2067 provide the derivation of and equations for the heat release rate based on the composition of the combustion gas and smoke measurement equations. Smoke measurement from light obscuration provides an additional measure of the burning rate and the quantity of gaseous products generated (Equation 5).

$$\dot{q}_{conv} = \dot{m}c_p(T_g - T_a) \quad \text{Equation 3}$$

where,

$\dot{q}_{conv}$  = convective heat release rate (kW)

$\dot{m}$  = 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)

$$\dot{q}_{CO_2} = \dot{m}_{CO_2} \Delta H_{CO_2}^* + \dot{m}_{CO} \Delta H_{CO}^* \quad \text{Equation 4}$$

where,

$\dot{q}_{CO_2}$  = heat release rate (kW)

$\dot{m}_{CO_2}$  = carbon dioxide mass flow in the exhaust duct (kg/s)

$\Delta H_{CO_2}^*$  = net heat of combustion per unit mass of CO<sub>2</sub> generated (MJ/kg)

$\dot{m}_{CO}$  = carbon monoxide mass flow in the exhaust duct (kg/s)

$\Delta H_{CO}^*$  = net heat of combustion per unit mass of CO generated (MJ/kg)

$$RSR = \dot{V}_s k = \left( \frac{\dot{m}_e T_s}{\rho_0 T_0} \right) \left( \frac{1}{D} \ln \frac{I_0}{I} \right) \quad \text{Equation 5}$$

where,

$RSR$  = smoke release rate ( $\text{m}^2/\text{s}$ )

$\dot{m}_e$  = mass flow at the bidirectional probe ( $\text{kg/s}$ )

$T_s$  = gas temperature at the smoke meter (K)

$\rho_0$  = density of air ( $\text{kg/m}^3$ )

$T_0$  = standard temperature of air (K)

$D$  = duct diameter (m)

$I$  = light intensity with combustion products in the duct (cd)

$I_0$  = light intensity in a smoke - free environment (cd)

The heat release rate and radiant heat flux from the propane burner were subtracted from the peak heat release rate and peak radiant heat flux to arrive at the heat release rate from the packaged oxidizer (Equation 6).

$$\dot{q}_{oxidizer} = \dot{q}_{conv} - \dot{q}_{conv, burner} \quad (\text{kW}) \quad \text{Equation 6}$$

The different data profiles generated from each test are illustrated in Figures 6 through 16 below using one oxidizer in the test packaging. The test material's convective heat release rate and chemical heat release rate profiles and total heat released during the fire exposure test are shown in Figure 6 and Figure 7, respectively. The heat release rate profiles are based on the temperature of the gaseous products of combustion or decomposition (Fig. 8) and the composition of specific gaseous products of combustion (Fig. 9). The generation or production rate profiles for carbon monoxide and carbon dioxide are shown in Figure 10 and Figure 11. The volume flow rate in the duct and optical density profiles are shown together in Figure 12. Figure 13 shows the smoke release rate and total smoke released. The radiant heat flux at two targets and mass loss data profiles from the same oxidizer are shown in Figure 14. The width of the curve at one-half the peak radiant heat flux was used to calculate the duration of active burning. Linear regression was used to calculate the mass loss rate over the duration of active burning. The total mass loss was calculated using the initial and final measured mass. The oxidizer and carton temperatures (Figure 15) and the surface temperature (Figure 16) data were reduced to temperature profiles. Additional temperature data reduced included the duration of surface temperature greater than  $700^\circ\text{C}$ .

Table 2 is a summary of data collected and reduced from each intermediate-scale fire exposure test. The peak convective heat release rate of the material in the combustible test packaging and the duration of active burning from the radiant heat flux profiles were determined to be the best fire-test-response characteristics for evaluating the degree to which an oxidizer increases or accelerates the burning rate of a combustible material. The intermediate-scale peak heat release rate and burning duration criteria to assign an oxidizer to Class 1, Class 2, or Class 3, is shown in Figure 17.

**TABLE 2. Summary of Data Recorded for Each Intermediate-Scale Fire Exposure Test.**

Concentration of oxidizer	%
Product form	Crystalline, granular, powder, or tablet
Initial mass of packaged test material	kg (lb)
Burn facility temperature	°C
Burn facility relative humidity	%
Burner heat release rate	kW
Burner radiant heat flux	kW/m <sup>2</sup>
Peak convective heat release rate	kW
Time to the peak convective heat release rate	s
Total convective heat released	MJ
Peak chemical heat release rate	kW
Time to the peak chemical heat release rate	s
Total chemical heat released	MJ
Peak radiant heat flux	kW/m <sup>2</sup>
Duration of active burning from the width of the curve at ½ the peak radiant heat flux	s
Mass loss rate during active burning	kg/min
Total mass loss	kg
Percent material consumed	%
Peak smoke release rate	m <sup>2</sup> /s
Total smoke released	m <sup>2</sup>
Maximum temperature in the oxidizer in the HDPE container	°C
Maximum carton temperature	°C
Peak surface temperature	°C

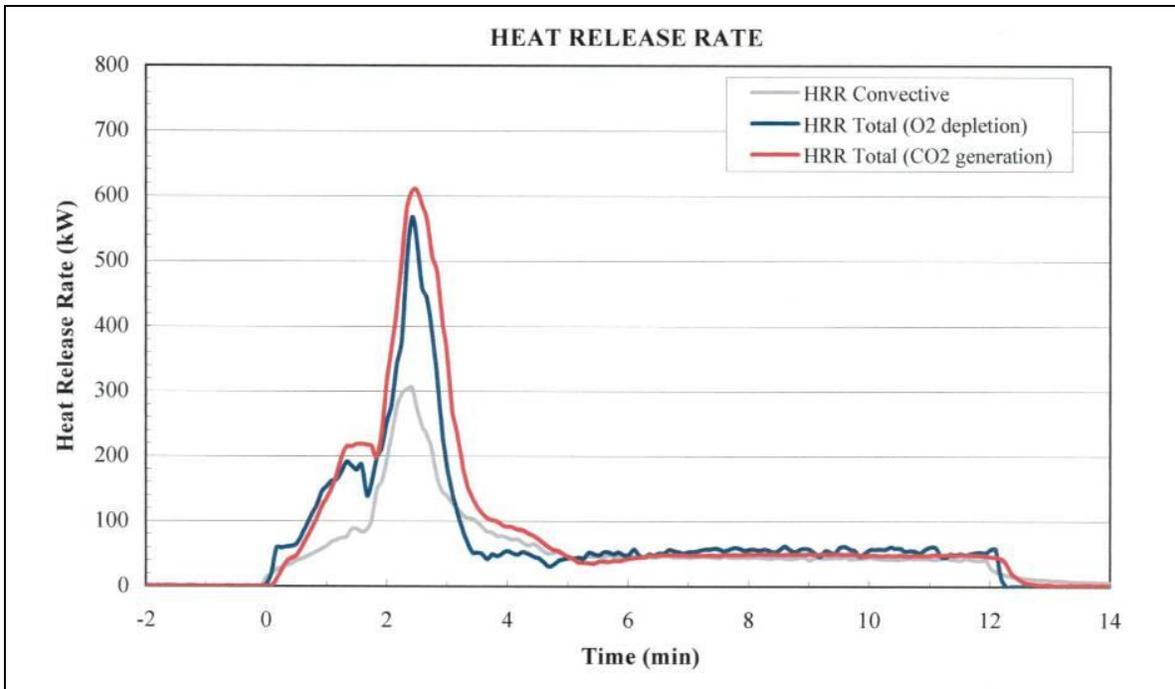


FIGURE 6. Rate of heat release profiles.

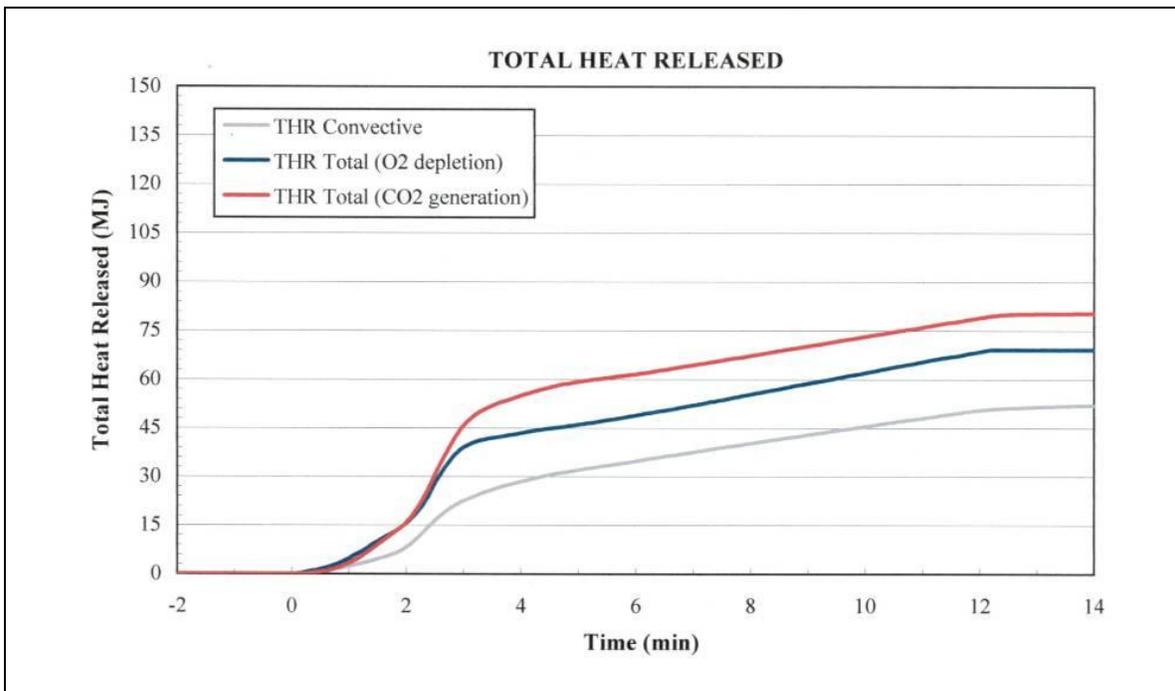


FIGURE 7. Total heat released.

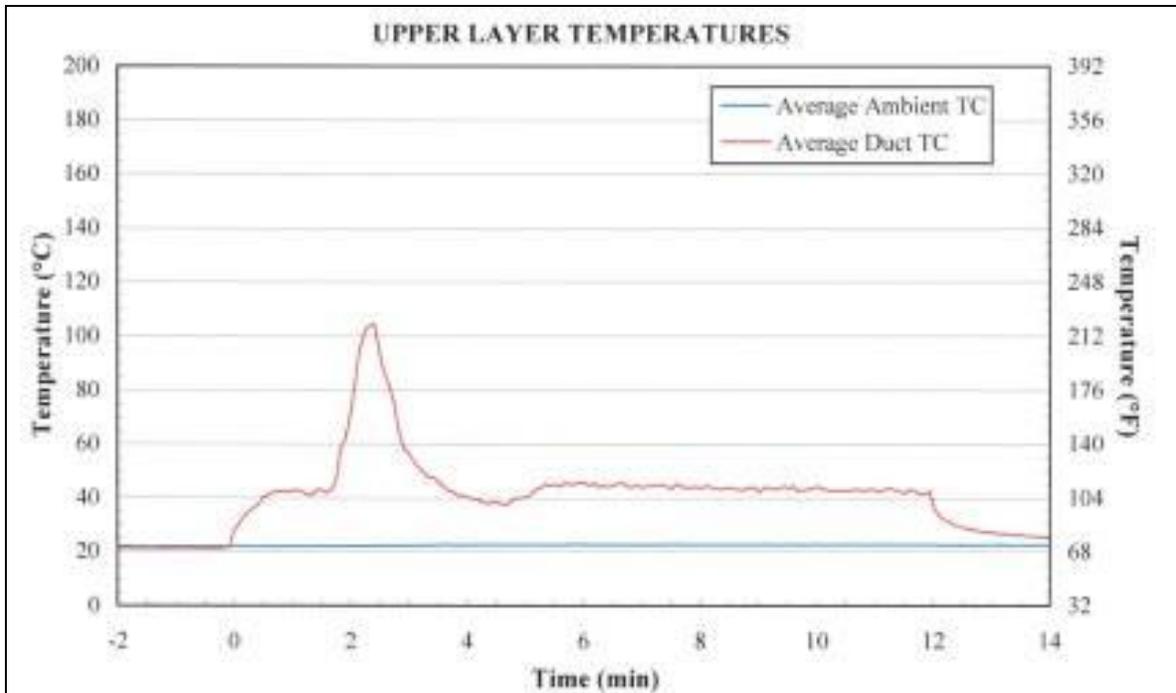


FIGURE 8. Gas temperature profile.

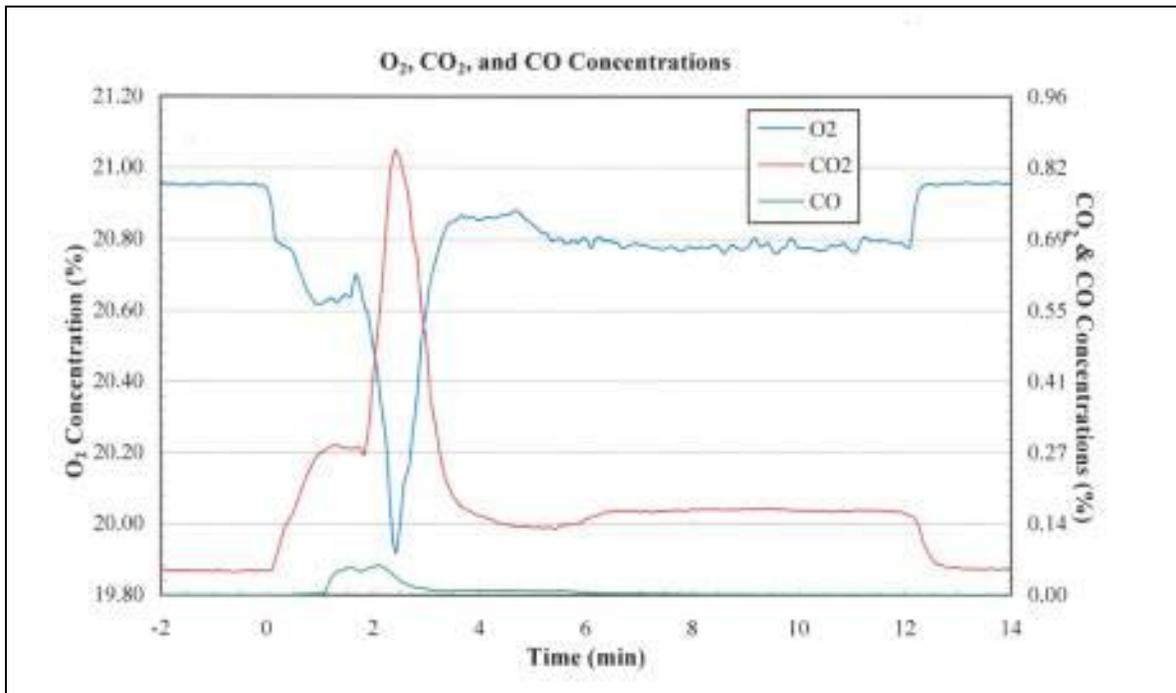


FIGURE 9. Oxygen (O<sub>2</sub>), carbon dioxide (CO<sub>2</sub>) and carbon monoxide (CO) concentration.

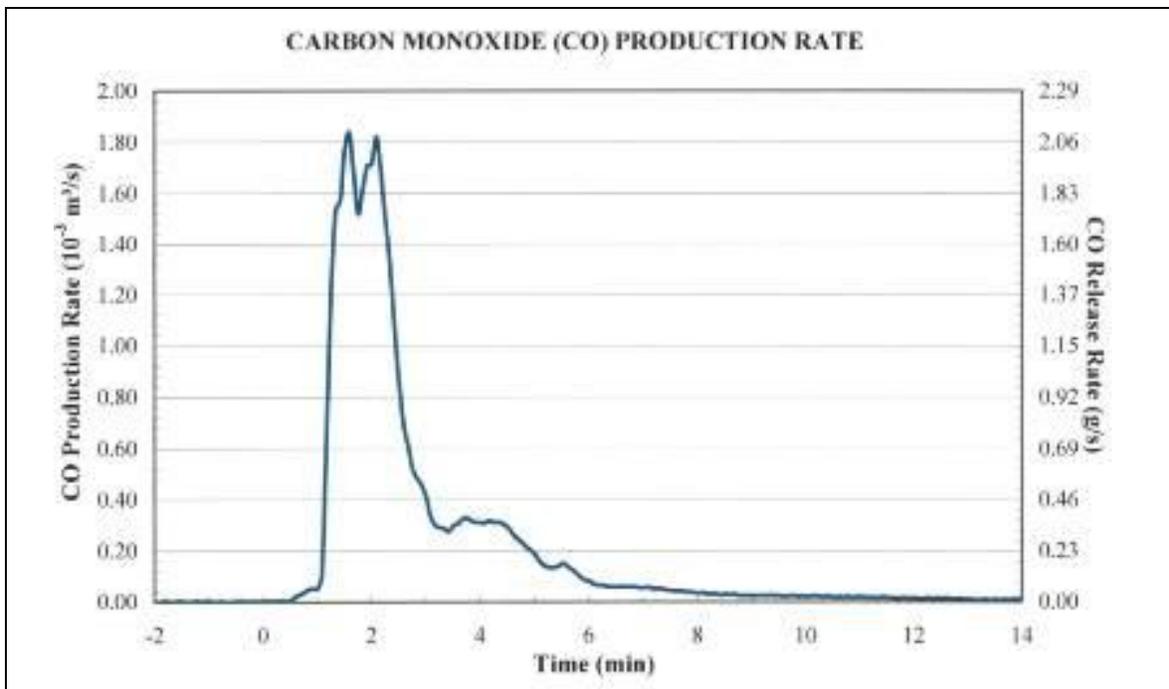


FIGURE 10. Carbon monoxide (CO) production rate.

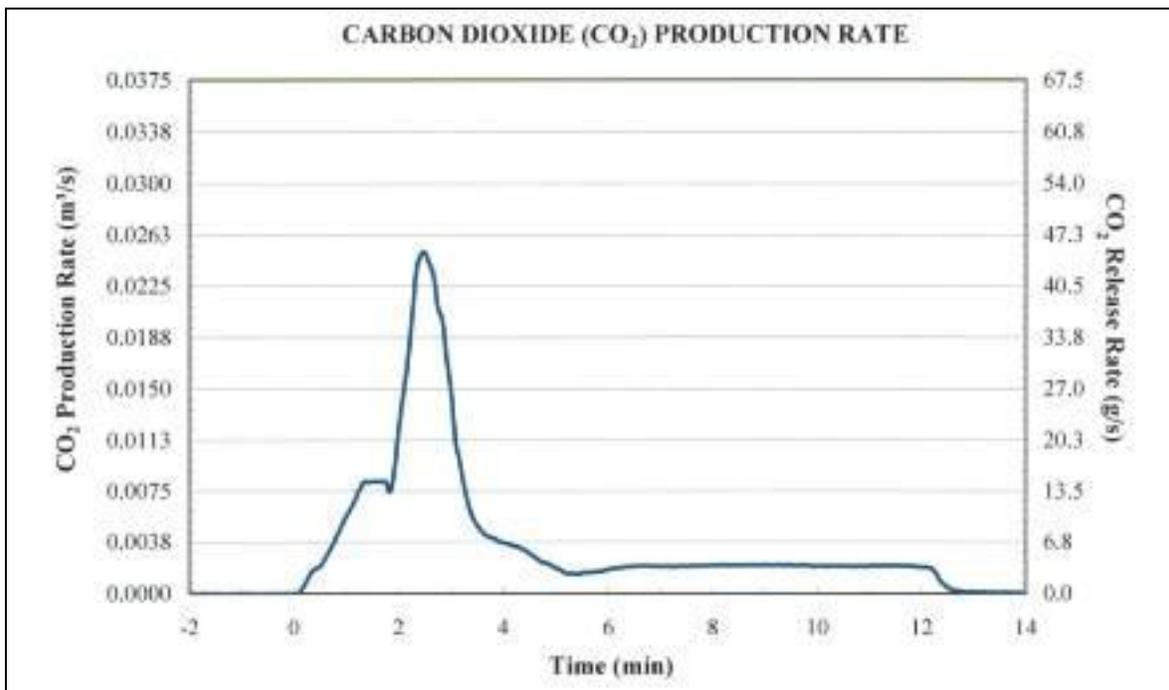


FIGURE 11. Carbon dioxide (CO<sub>2</sub>) production rate.

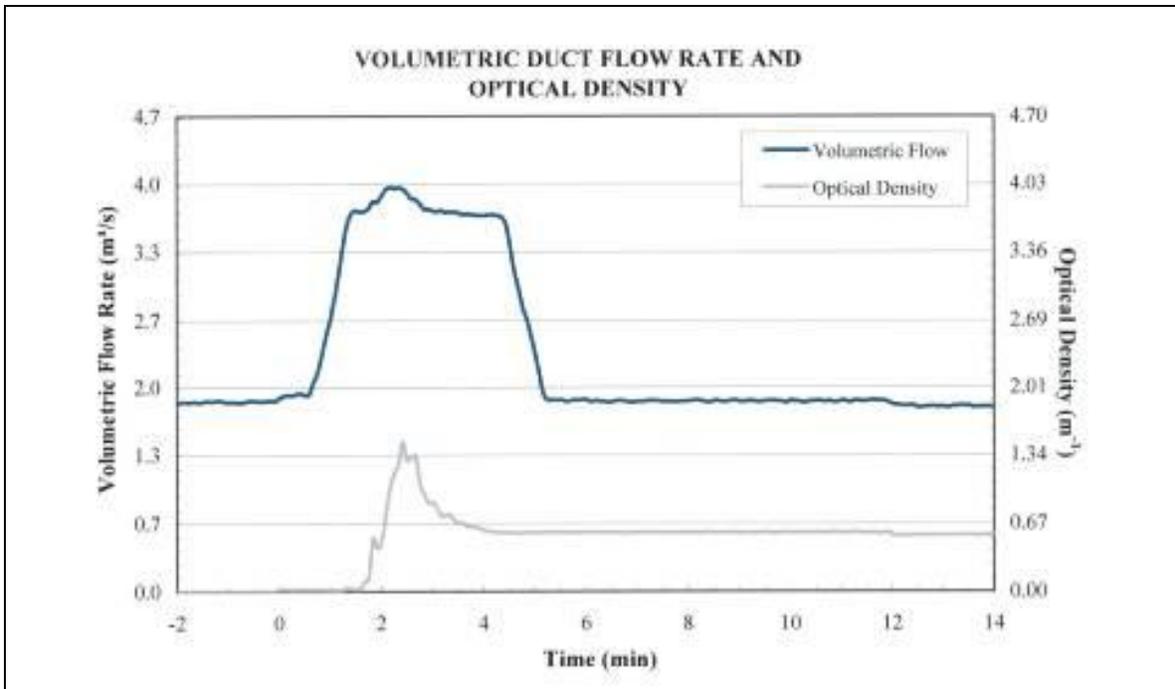


FIGURE 12. Volumetric flow rate and optical density profiles.

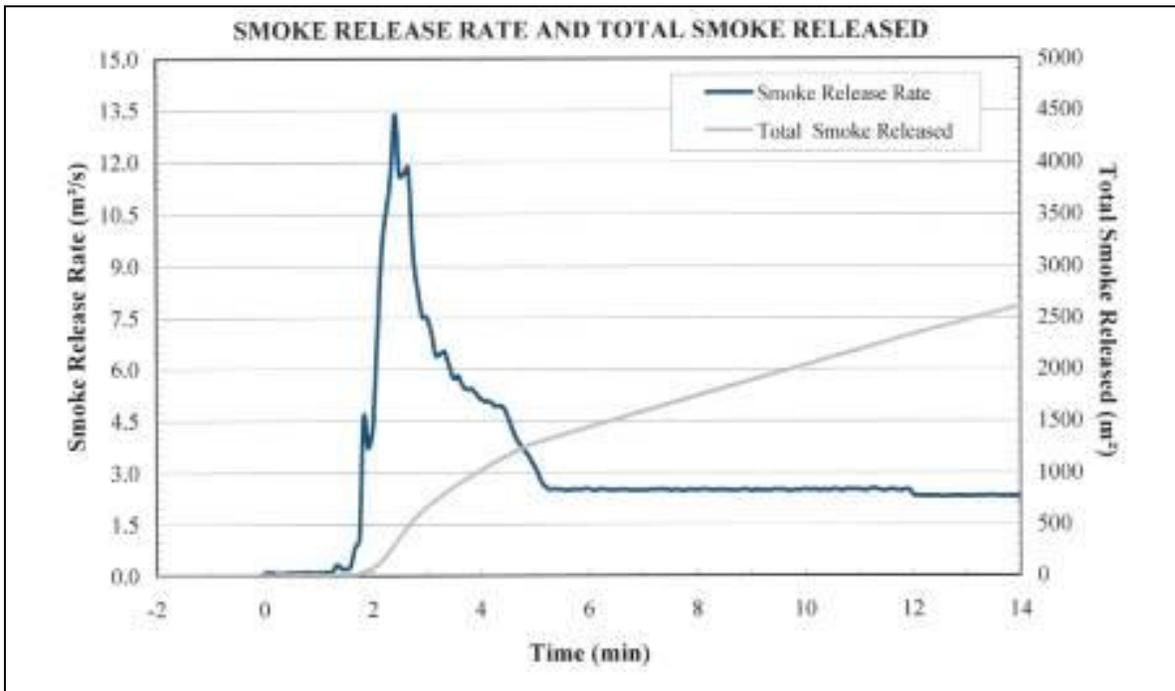


FIGURE 13. Smoke release rate and total smoke released.

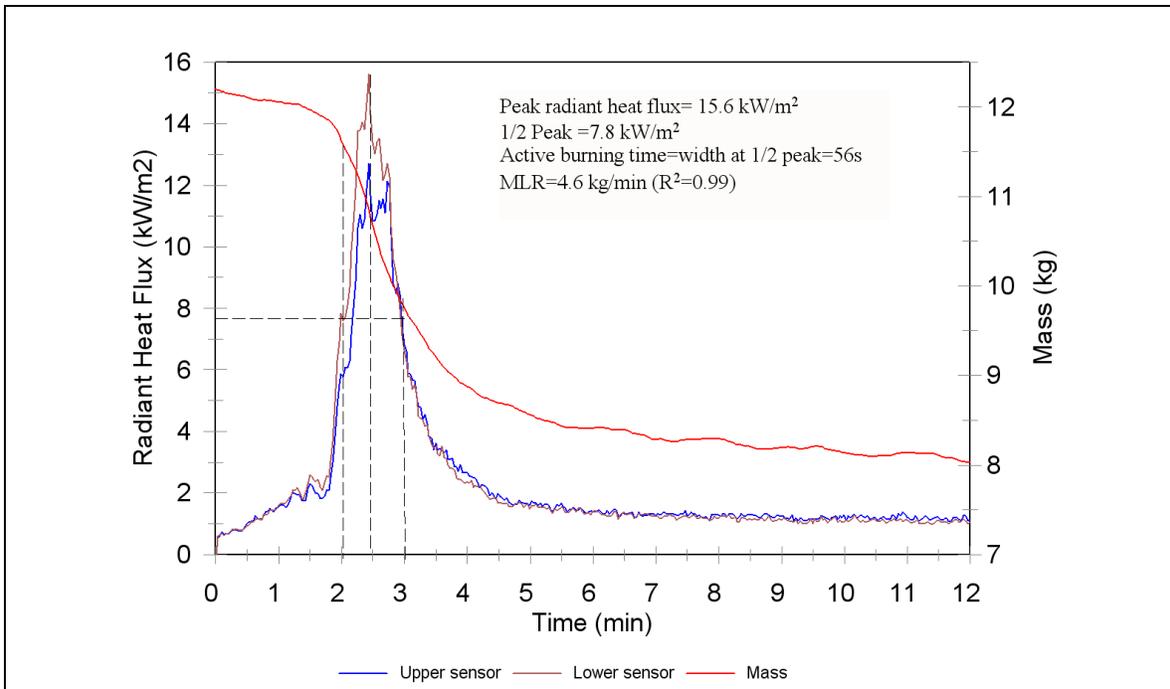


FIGURE 14. Radiant heat flux and mass loss profile with dashed lines showing the duration of active burning.

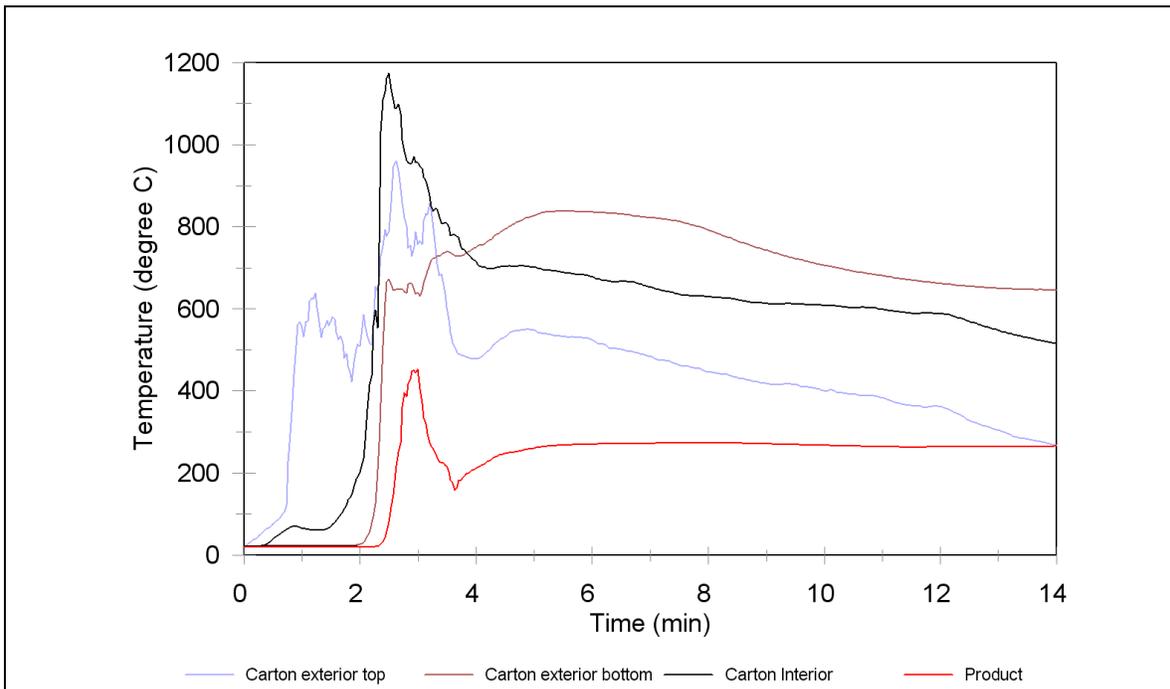


FIGURE 15. Temperature profile from thermocouples inside the oxidizer, inside the packaging, and on the exterior of the carton.

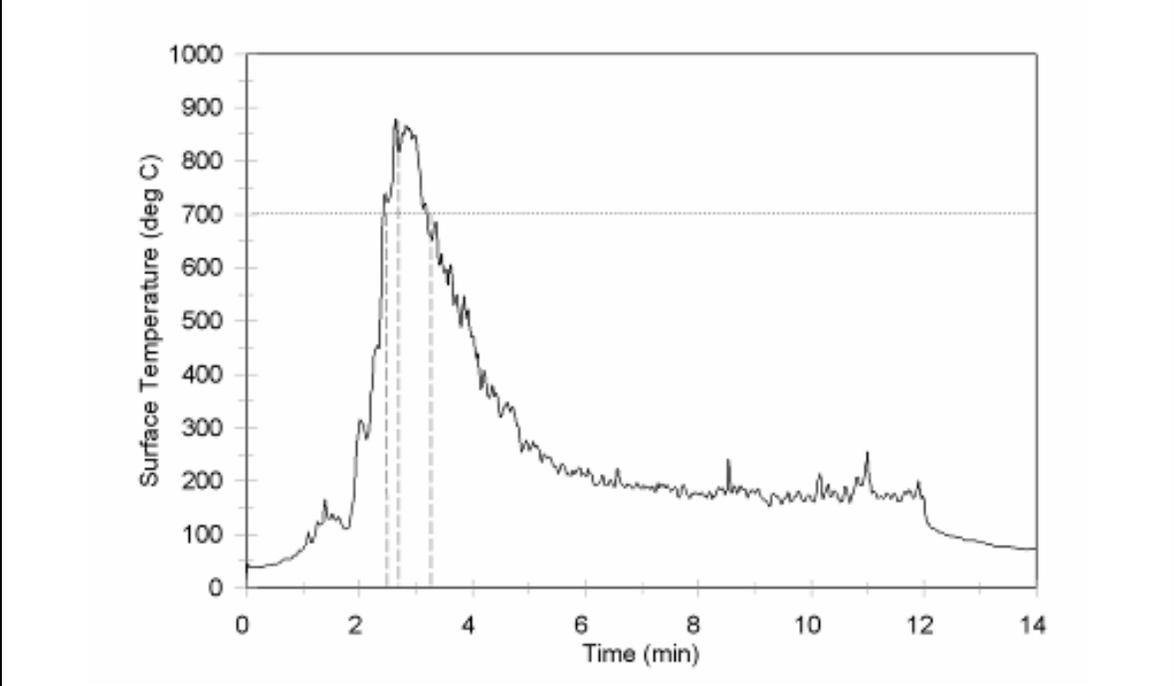


FIGURE 16. Surface temperature profile.

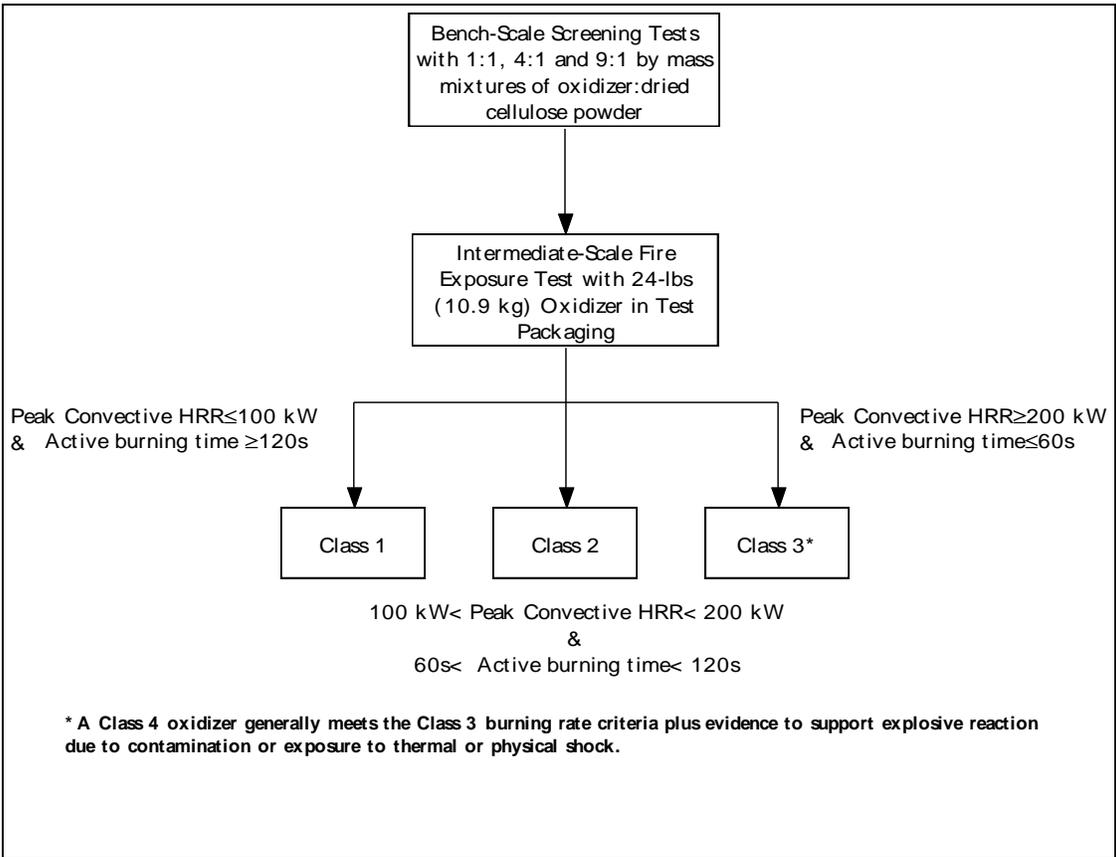


FIGURE 17. Intermediate-scale fire exposure test criteria for assigning an oxidizer to Class 1, Class 2, Class 3 or Class 4.