The Correlation of Heat Release Calorimetry Measurements

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16. Abstract

The Ohio State University (OSU) Rate of Heat Release Apparatus specified in FAR Part 25.853(a-1) defines both apparatus test conditions and pass/fail criteria for large surface area aircraft interior materials, such as sidewall panels, bulkheads, and stowage bins. The cone calorimeter (ASTM E-1354 Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen consumption Calorimeter) similarly measures heat release rates from materials subjected to a constant external heat flux. However, heat release from the cone calorimeter is calculated by measuring the decrease in oxygen concentration in a stream of air flowing over the test specimen, rather than the temperature (enthalpy) increase of the air stream leaving the device, as is done with the OSU Rate of Heat Release Apparatus. This report will examine the differences between measurement of convective, radiant, and total heat release rates, and discuss techniques to obtain substantial conformity between the two techniques. Results for both methods are compared for single-ply, bisphenol-A epoxy-impregnated glass cloth.

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INTRODUCTION

The Ohio State University (OSU) Rate of Heat Release Apparatus [1] and the cone calorimeter [2] are test devices that measure the rate of heat release from burning materials. While comparisons between data generated from the two instruments is found in the literature [3-6], no systematic attempt has been made to show conformity, of results between the two test methods.

It was recognized during the Federal Aviation Administration (FAA) adaptation of the OSU as a proposed regulatory requirement and subsequent round-robin testing that oxygen depletion and thermal methods of measuring heat release measurements produced different values, although the two methods are generally proportional [7-8]. Before adoption of the regulation, the FAA and other laboratories involved in the initial round robin examined oxygen depletion as a measurement technique for heat release rate using the OSU. At that time, there were two different sets of pass/fail criteria for each method. For the thermopile, a material must have less than 65 kW/m^2 for a peak heat release rate and less than 65 kW*min/m^2, for the 2-minute total heat release, for the average of triplicate sample measurements. For oxygen depletion, a material must have less than 100 kW/m^2, for the peak heat release rate, and less than 100 kW*min/m^2, for the 2-minute total heat release, for the average of triplicate sample measurements. Oxygen depletion was later abandoned because it was more problematic. In addition, two sets of pass/fail criteria could lead to inconsistency. It was also found that a properly constructed thermopile would generate virtually identical calibration factors year in and year out, whereas an oxygen sensor can degrade and need replacement or recalibration. It must be remembered that the thermopile captures only the convective portion of the heat released, which means that the thermopile always resulted in lower reported values of heat release than oxygen depletion. Thus, the OSU has been criticized for errors introduced by absorption of radiant and convective heat into the apparatus itself. The convective heat absorption errors at steady state are generally accounted for through calibration of the apparatus. As long as airflow rates and leakage rates remained constant, these errors were small (less than 5%) and appeared as the slight nonlinearity of the calibration factor seen with increasing methane flow rates. The radiant heating of the apparatus, however, is instantaneous and can be quite substantial, particularly in instances where materials burn with a flame of high emissivity, i.e., a smoky flame.

In the mid-1990s, the OSU inventor, Dr. Edwin E. Smith of Ohio State University, sought to address thermopile errors by compensating for radiant heat absorption by the walls of the apparatus [9]. Basically, an electrically powered ceramic panel with known voltage and amperage input was inserted into the apparatus to provide a quantitative heat release rate source. The convective heat leaving the apparatus was measured using the thermopile, and the radiant heat absorbed by the apparatus was measured using a thermocouple attached to the apparatus wall. This work examines this technique and compares the results with oxygen depletion measurements for the cone calorimeter and the OSU fire calorimeters.

EXPERIMENTAL

MATERIALS.

Test specimens were prepared using the materials shown in table 1. The DGEBA was warmed to melting, and two parts by weight of EMI-24 per hundred parts resin (phr) was added to the

DGEBA and mixed until homogeneous. The mixture was hand impregnated into E-glass fabric (0.22-mm-thick 6781 S. Glass "Griege" weave 8HS 8.95 oz/sq yd, BGF Industries Inc.) and cured in a Carver press under 10,000 psi and 150ºC for 45 minutes to make a single-ply glass lamina.

TABLE 1. EPOXY DESCRIPTION AND PREPARATION

Component	Trade Name [CAS Registry No.]	Supplier	Equiv. Weight (g/eq)
diglycidylether of bisphenol-A (DGEBA)	DER-322 [001675-54-3]	Dow Chemical	174
2-ethyl-4 methyl-imidazole (2,4-EMI)	Imicure 24 [931-36-2]	Air Products and Chemicals	N/A

METHODS.

WALL TEMPERATURE-CORRECTED THERMOPILE MEASUREMENTS. A 26-gauge Chromel/Alumel thermocouple (Type K) was silver-soldered to the outside wall of the OSU, 150 millimeters below the air manifold and 50 millimeters in front of the plane of the sample face. Early in the experiment, a source of error was discovered in the heat lost to the surroundings term, H_l , of Smith's work. Inserting the panel to the chamber altered the heat leaving the apparatus, as measured on the thermopile. In effect, heat from the Glo-bars (radiant heat source for the OSU) that previously escaped through the radiation doors and into the plenum was absorbed by the panel assembly. Instead of escaping, the heat was absorbed by the air passing through the OSU and then measured by the thermopile in the exhaust stack. This phenomenon caused an upward shift in the equilibrium temperature of the OSU so that the steady-state initial and final temperatures were not experimentally comparable.

The radiant heater used was a 152 by 152 mm (6 in. by 6 in.) Watlow Type 5 ceramic fiber heater (VF506A06S). It was adapted to a standard OSU specimen holder, in much the same way as the Gardon gauge is mounted for radiant heat calibration of the Glo-bars. Two Fluke 77 Series II digital multimeters were used to measure the voltage and amperage supplied to the panel. The power input to the panel was calculated as the product of volts and amperes to determine input watts. The panel was allowed to preheat outside the OSU for 15 minutes. The outer door to the OSU was opened, and the electric panel assembly was clamped into place with sheet metal vise-grip pliers. The electric panel was then inserted into the test chamber after a 1-minute hold period in the plenum. The OSU was then allowed to come to temperature equilibrium with the additional heat input. After attaining equilibrium, the power was turned off and the OSU was allowed to attain equilibrium. Figure 1 shows a typical experiment with the radiant panel. Notice that ΔT, the difference between the initial and final equilibrium temperatures, is approximately 10°C due to the presence of the panel assembly. The increased wall temperature was accompanied with a small increase in thermopile voltage output when comparing thermopile signal at initial equilibrium versus the value of final equilibrium after cool down.
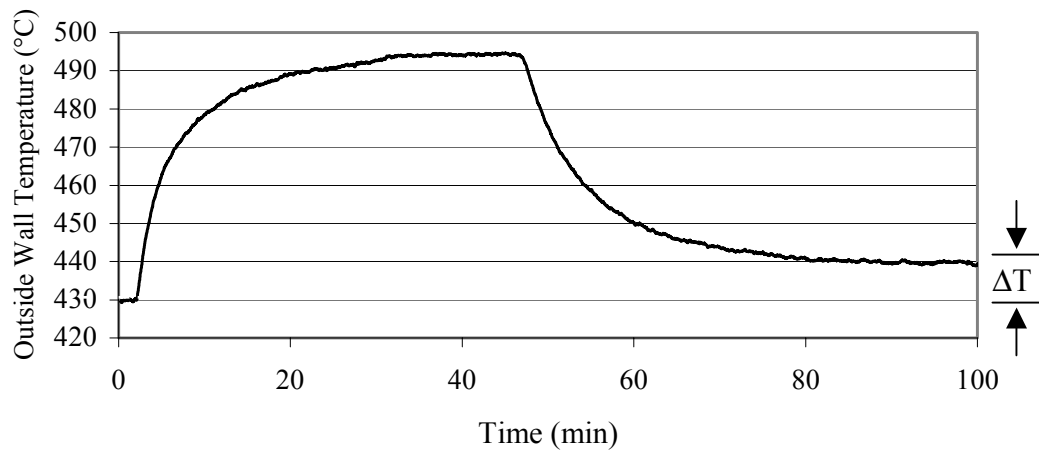
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FIGURE 1. RADIANT PANEL TEST—1031 WATTS

From the moment the panel was injected, the wall temperature immediately climbed, even though the thermopile showed the typical voltage reduction due to the relatively cool air entering the OSU from the plenum during injection. However, at low-power input, the wall temperature of the OSU decreased, as the "cold" sample holder absorbed radiation from the walls and the Glo-bars.

A series of radiant panel tests was conducted to determine the relationship of the rate of wall temperature change as a function of radiant power input. Figure 2 shows a series of wall temperature measurements conducted, as in figure 1, from the time of panel insertion into the environmental chamber until the power is turned off prior to equilibrium.
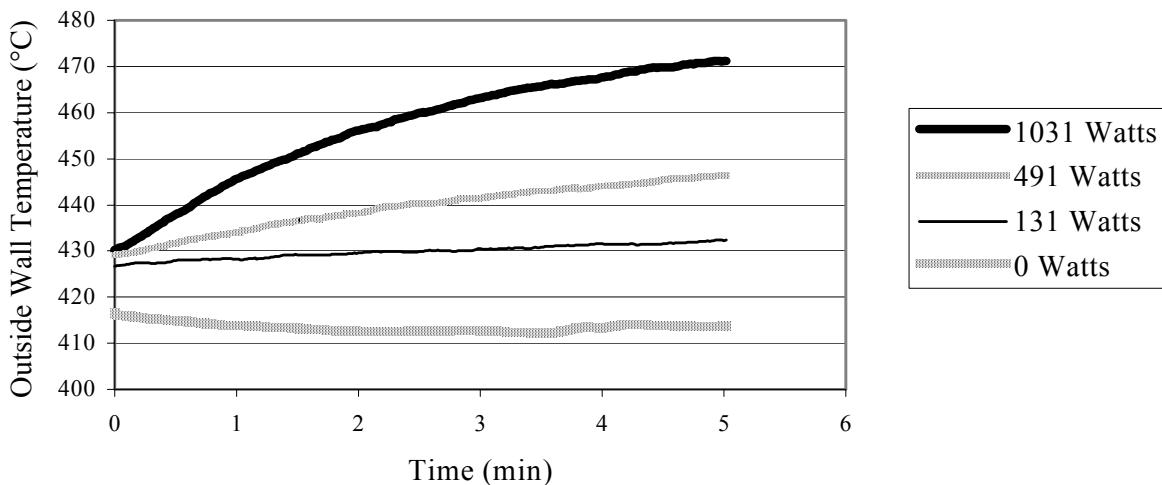

FIGURE 2. RADIANT PANEL TEST SERIES

The four sets of temperature data were each reduced to determine the temperature rate of change by subtracting each data point from the point that preceded it and plotting the resultant time temperature derivative. The data set is clipped from t=0 to t=2 minutes. Using the Excel

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trendline function, the linear best fit (least squares) is generated. The y-intercept (t=0) value is used as the maximum rate of temperature change in degrees C per second, corresponding to the radiant panel input power. An example is shown in figure 3.
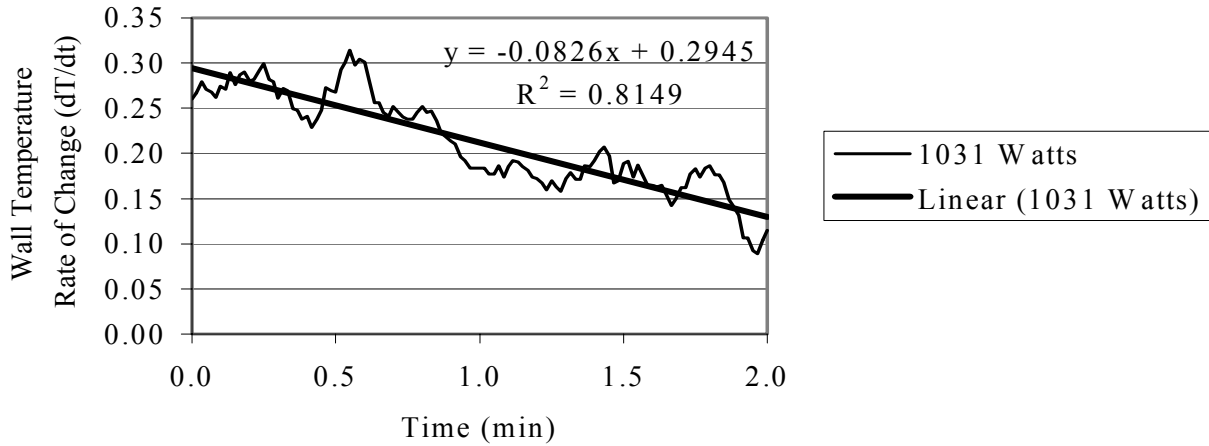

FIGURE 3. RATE OF WALL TEMPERATURE CHANGE

In the same way, the maximum rate of wall temperature change was obtained for the entire series of radiant panel experiments. The test series shown in figure 2 is reduced as shown in figure 3, and the resultant y-axis intercepts are plotted versus radiant panel input wattage in figure 4. The Excel trendline function then performed the least squares function to generate the best-fit linear equation shown in figure 4.
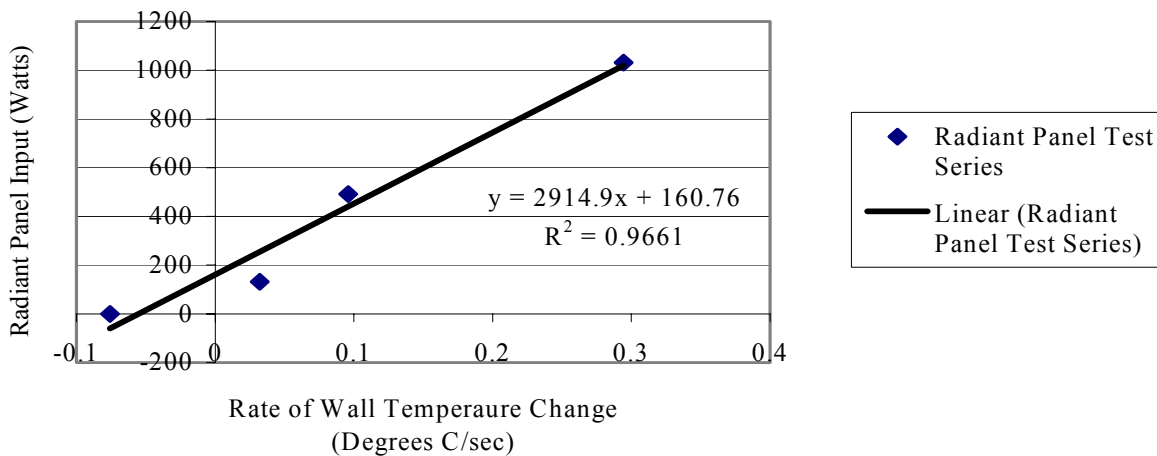

FIGURE 4. WALL TEMPERATURE CHANGE VERSUS RADIANT PANEL INPUT

The standard method of calibrating the OSU for compliance with FAR 25.853 is used to calibrate the OSU. The wall temperature calibrations are a set of experiments performed totally independent from the thermopile calibration. However, the methane flame used for calibrating

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the thermopile has associated with it a radiant component that changes the temperature of the wall. When calibrating the OSU with methane, an additional step must be performed for the wall temperature correction method. In the standard calibration, a methane flow-step change is done from the 1 liter per minute baseline to 4 liters per minute. The thermopile voltage data is taken after 2 minutes, for 10 seconds at a rate of one data point per second, with the 10 data points averaged. The thermopile baseline voltage is then subtracted from the high-flow voltage average. The actual flow rate of methane at baseline, as measured on the wet-test meter, is subtracted from the actual higher flow rate. This flow rate difference can be converted to heat energy based on the heat of combustion of methane and dividing this by the voltage change that yields a calibration factor in units of watts per millivolt. However, the thermopile has not fully reached equilibrium during the 2 minutes. This must be accounted for during the calibration of the wall temperature method. The standard FAA calibration requires doing step changes from 4, to 1, to 6, to 1, to 8, to 1,to 6, to 1, and 4 liters per minute, with the average of these sets of values used as the OSU calibration factor. An additional step change was done at the end, after the OSU equilibrates at 1 liter per minute by changing the flow rate to 6 liters per minute, and allowing the OSU to equilibrate at the higher flow, approximately 15 minutes. This calibration factor was smaller because the millivolt value was higher after equilibration. The standard factor was used for the FAA regulatory requirement, but the smaller factor was used for the wall temperature-corrected method. This was used to calculate only the convective component of heat release and the rate of wall temperature change from the electric panel experiments to calculate the radiant component of heat release.

OXYGEN DEPLETION MEASUREMENTS. The OSU apparatus was equipped with oxygen depletion equipment similar to that originally proposed during the early round-robin testing by the FAA and industry. A drill size "F" 6.42-mm (0.253-in.) hole was made along the front centerline of the pryamidal sections of the OSU at a point 76.2 mm (3 in.) below where air recombines in the OSU just below the exhaust stack. The hole passed through the inner and outer pyramidal sections. The hole in the outer pyramidal section was reamed to accommodate a 6.35-mm (1/4-in.) stainless steel Swagelok® fitting that was drilled out for clearance of the sample probe, and had the male National Pipe Thread (NPT) threads removed on a lathe. The fitting was attached to the outer pyramidal section wall using Saueriesen refractory cement. A sample probe made of 6.35-mm (1/4-in.) stainless steel tubing with a wall thickness of 0.531 mm (0.0209 in.) was fashioned by capping one end with a silver-soldered 6.35-mm (1/4-in.) stainless steel disc. Three no. 20 drill 4.09-mm (0.161-in.) holes were made 12.7 mm (1/2 in.) from the capped end and 19.05 mm (3/4 in.) apart. The probe was held in place using a Vespel® graphite ferrule with the holes oriented upward and pointed away from the direction of the airflow. When inserted, the probe holes were 12.7 mm (1/2 in.) from the inner pyramidal section walls, with the center hole being in the geometric center of the plane, perpendicular to the direction of the airflow.

The probe was connected to the oxygen system with 6.4 mm (1/4 in.) Teflon® tubing. A KNF Laboport Model N86KT all-Teflon® pump with a nominal flow rate of 4 liters per minute drew the gas sample through the probe. After the pump, the flow was split with the majority of air dumped to exhaust through a large orifice needle valve, Whitey Model SS-1RS4. The other leg of the Teflon® tee was adapted to 12.7-mm (1/2-in.) fittings and connected to a 120.7 mm (4 3/4 in.) length of 12.7-mm (1/2-in.) Teflon® tubing. The tube was packed halfway with 20-mesh

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drierite and a small plug of glass wool, with the remaining length filled with 20-mesh Ascarite and oriented so the flow passed first through the drierite, then the Ascarite. This served as the water and CO_2 scrubber. The 12.7-mm (1/2-in.) tubing was then adapted to 3.2-mm (1/8-in.) Teflon® tubing and attached to a Panametric Series 350 Zirconia oxygen analyzer. The outlet of the analyzer was attached to a rotameter. The flow to the analyzer was adjusted by slowly closing the Whitey valve until the rotameter showed a flow of 100 ml/min. A diagram of the oxygen system is shown in figure 5.
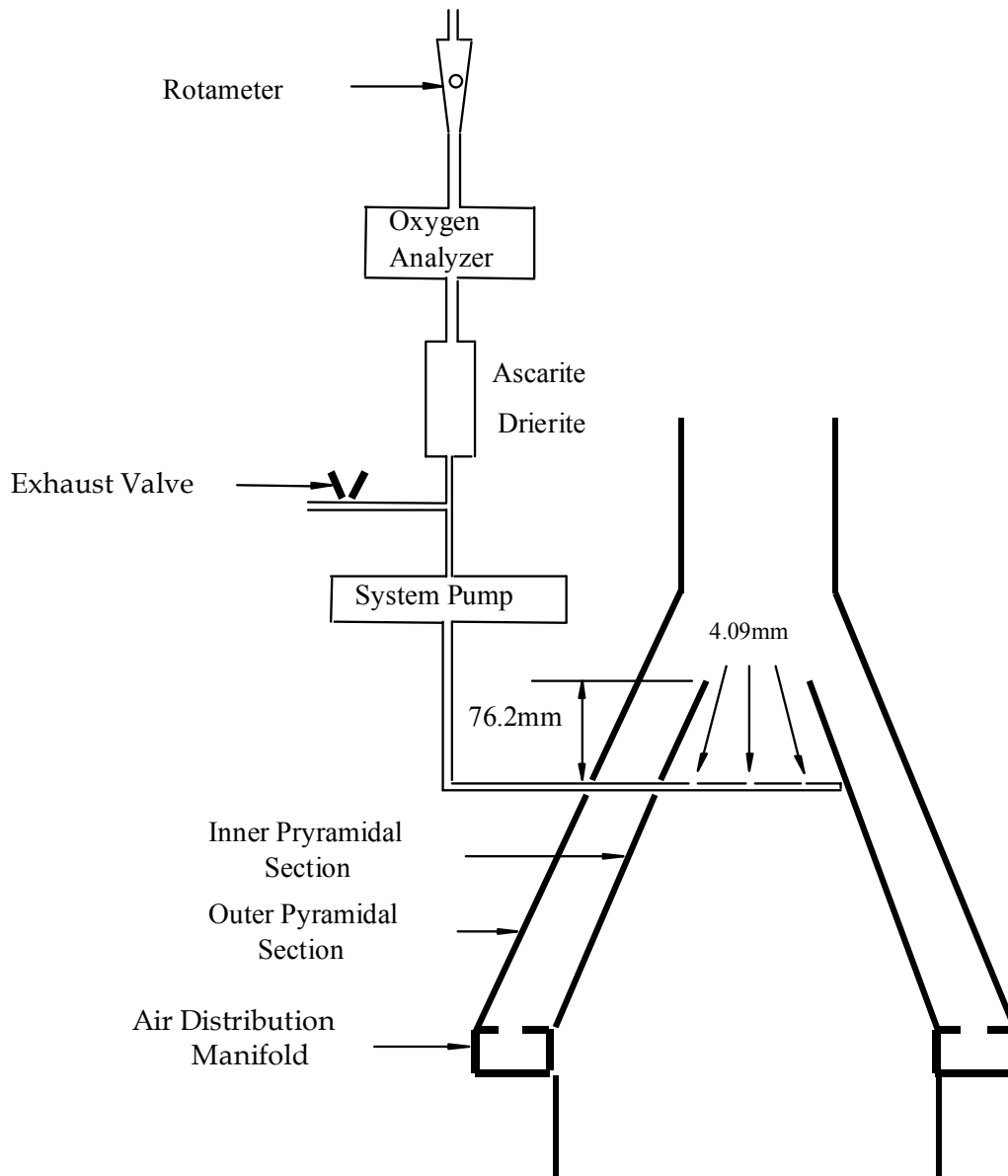

FIGURE 5. OXYGEN DEPLETION SYSTEM

The output of the analyzer is log linear, with respect to oxygen concentration. Therefore, enhanced calibration of the analyzer is necessary. Three analyzed tanks of oxygen in nitrogen were used to determine the analyzer millivolt output corresponding to the tank concentration.

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The data points were entered into an Excel spreadsheet, and the trendline function was used to determine the following calibration curve:

$$y = 19.08e-0.042x$$

where

x = analyzer millivolt output and y = % oxygen

The curve fit had a variance (R^2) of 0.9992.

Calibration of the oxygen system was done simultaneously with the thermopile. A known amount of methane was burned in the usual manner, and the oxygen concentration was measured at the baseline flow and at the three higher flows. The heat release versus the change in oxygen concentration was calculated for the step changes and the values were averaged. The relative standard deviation (RSD) for the three flow changes fell within the 5% criteria specified for the thermopile. Total oxygen system response showed that the analyzer reached 90% of the final equilibrium value within 6 seconds of the step change in methane flow. Turning the oxygen system pump on and off did not effect the thermopile signal. All three methods of measuring and calculating the heat release rate are independent of each other and have no influence on the regulatory requirement of thermopile usage. The FAA pass/fail criteria of 65 kW/m^2, as a peak, and an integrated value of 65 kW-min/m^2, over the first 2 minutes of the test, applies only to FAR Part 25.853(a-i) standard thermopile without a wall temperature correction.

DATA REDUCTION. The wall temperature correction defines separate convective and radiant heat release components.

$$\Delta H_{thermal} = \Delta H_{radiative} + \Delta H_{convective} \tag{1}$$

The thermopile is used to measure the convective portion of heat release. The method of calibrating the thermopile for wall temperature-corrected measurements was similar to the standard FAA method, but the calibration constant was determined after the OSU had equilibrated at the higher flow rather than at the end of a 2-minute interval.

$$\Delta H_{convective} = \frac{(V_m - V_b)}{0.02323m^2} \times K_{h(equilibrated)} \tag{2}$$

where V_m was the measured thermopile voltage during the test and V_b was the thermopile baseline voltage averaged over 20 seconds immediately before test specimen injection. For the FAA OSU, on which these experiments were conducted, the K_h was 0.28258 kW/millivolt and the convective $K_{h(equilibrated)}$ was 0.23401 kW/millivolt, approximately 20% smaller.

The radiant portion of heat release is determined by measuring the wall temperature at 1-second intervals and determining the wall temperature rate of change, dT_{wall}/dt. The rate of wall temperature change, dT_{wall}/dt, is the y-axis intercept generated by the Excel trendline function shown in figure 3 and corresponds to each power input value for the radiant panel. The series of

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panel experiments generated the function shown in figure 4, watts of radiant energy per C°/second of wall temperature change. Equation 3 standardizes the radiant heat release rate to units of kW/m^2 during material burns.

$$\Delta H_{radiative} = \frac{\left(2914.9\,\frac{dT_{wall}}{dt} + 160.76\right)}{1000 \times 0.02323 m^2} \tag{3}$$

It is important to note that the series of radiant panel experiments used to determine equation 3 was specific to the particular OSU. Small changes in location of the thermocouple, or the size of the silver-solder connection, changes the values of the constants in the wall temperature correction equation 3.

Oxygen depletion measurements were also used to calculate $\Delta H_{chemical}$ for comparison with the wall temperature-corrected thermopile method. The oxygen method is calibrated simultaneously with the thermopile. Step changes in methane flow produced a corresponding decrease in oxygen concentration. Values obtained from the five step changes in methane flow are averaged together, as was done with the thermopile, and data reproducibility falls within the 5% RSD specified for the thermopile. The K_{oxygen} for the OSU used in these experiments was determined to be -2.5358 kW/%O$_2$. The heat release rate was calculated using equation 4.

$$\Delta H_{chemical} = \frac{\left(C_m - C_b\right) \times K_{oxygen}}{0.02323\ m^2} \tag{4}$$

where C_m was the measured concentration of oxygen during the test and C_b was the baseline concentration determined by averaging the 20 data points immediately before test specimen injection into the OSU. Units for total, convective, and radiant ΔH are kW/m^2.

RESULTS

OSU RATE OF HEAT RELEASE CALORIMETER.

One way of checking the validity of the above equations was to do an energy balance for the methane calibration procedure. During calibration, the heat release rate from the methane flame was known, calculated from the heat of combustion of methane and the measured flow rate through the wet-test meter. Figure 6 was calculated from the methane flow rate, thermopile signal, and wall temperature rate of change using equations 1, 2, and 3, with the exception that the sample area factor 0.02323 is not included. It was shown that the radiant correction is greatest at the outset of step flow change and becomes smaller as the OSU equilibrates. When the step flow changed from a greater to smaller flow rate, the radiant component was negative. This was indicative of heat flow from the chamber walls into the air flowing through the chamber. The convective portion of heat, sensed by the thermopile, showed the opposite effect. Convective heat measurements lagged behind the actual heat release rate until equilibrium was reached.
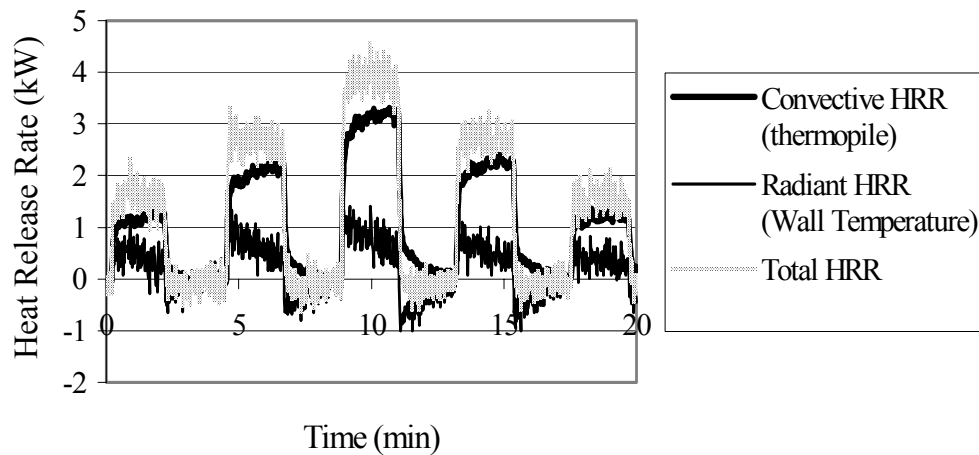
FIGURE 6. METHANE CALIBRATION HEAT RELEASE RATES

The heat release rates, calculated from the flow changes of methane, were 1.54, 2.55, and 3.57 kW.   The system showed much better dynamic response; however, the change of wall temperature increased the signal-to-noise ratio compared to the conventional thermopile technique. With the methane calibration flame, about 80% of the heat released was convective and about 20% was radiant. Figure 7 shows the wall temperature-corrected thermal calibration method compared to the oxygen depletion chemical calibration method. Within experimental error, $\Delta H_{thermal}=\Delta H_{chemical}$.
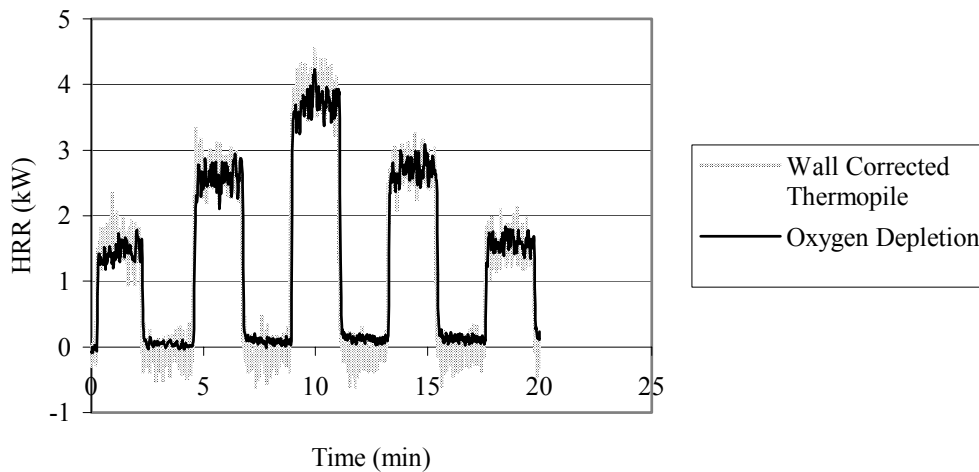

FIGURE 7. CHEMICAL VERSUS WALL-CORRECTED THERMAL CALIBRATION

An example of the independent data reduction methods is shown in figures 8 and 9. Figure 8 shows the results for a 1.52-mm (0.060-in.) -thick sample of transparent, bisphenol-A (BPA) polycarbonate. This particular specimen is not required by regulation to meet the pass/fail criteria of FAR 25.853, but illustrated the improvement thermal measurement methods can achieve relative to oxygen depletion. Figure 9 shows the results for a 1.52-mm (0.060-in.) -thick

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sample of bisphenol-C polycarbonate. It is perhaps the only structural polymer transparency that meets FAR 25.853 criteria.
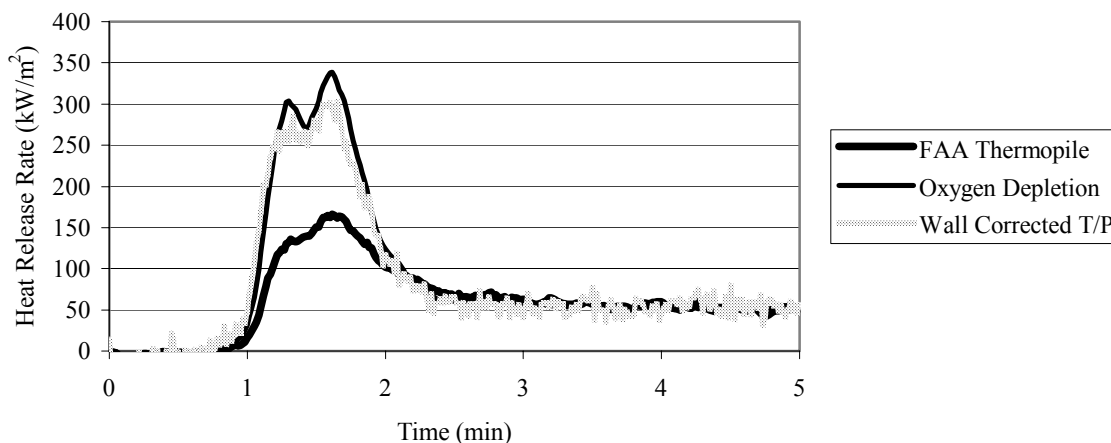

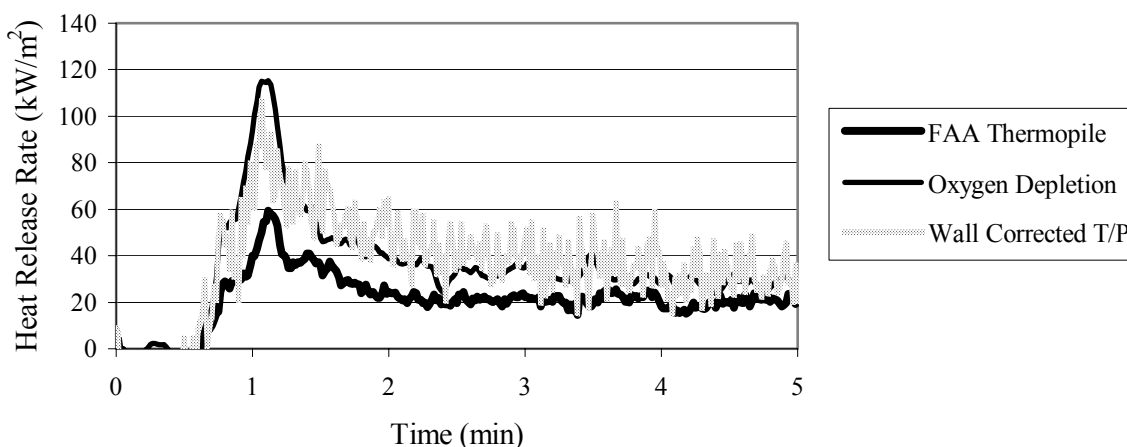FIGURE 8. HEAT RELEASE RATE COMPARISON—BPA POLYCARBONATE

FIGURE 9. HEAT RELEASE RATE COMPARISON—BPC POLYCARBONATE

While agreement between the peak heat release rate was quite good, the increased noise of the signal was also apparent. The polycarbonates burned with a smoky flame, i.e., one with high emissivity, and demonstrated that a much larger portion of total heat release, approximately 50%, is given off as radiant energy, compared to the methane calibration flame where approximately 25% is radiant energy.

ASTM E 1354—THE CONE CALORIMETER.

An Atlas Fire Science Products CONE2 Combustion Analysis System was operated in accordance with ASTM E 1354 and the manufacturer's instructions. A 29-mm orifice plate was used, and the measured system flow rate was 0.006 m^3/sec. The software was such that all calculations are performed internally with system prompts, assuring that the operator must perform each step successfully in order for the device to be operated.

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Additionally, an Iotech Personal Daq/55 data acquisition system was piggybacked onto each combustion gas analyzer to allow independent retrieval and subsequent analysis of analog data. Since the FAA is interested in the measurement of the fire performance of thin, light-weight, low-heat release materials, some initial instrument characterization experiments were run. One of the first experiments conducted was to examine the square-wave response of the oxygen analyzer to step changes in methane flow. As mentioned above, the OSU oxygen depletion system achieved 90% of the equilibrium final concentration to a step change of methane flow in 6 seconds. For the OSU, this was a combination of both sample transport and oxygen analyzer response parameters. In the cone experiment, the calibration burner was removed from the cone calibration system and attached to a methane tank with a rotameter and high-accuracy needle valve. Methane flow rate was set approximating the calibration flow. The data acquisition system was started and the burner was lit. After a 1-minute baseline measurement, the burner was inserted into the cone system and held in place for 15 seconds and then removed. One minute later, the burner was again inserted into the cone and held for 30 seconds then removed. One minute later, the burner was inserted into the cone and held for 1 minute then removed.

Figure 10 shows that the analyzer response lagged the test event significantly. Although the oxygen analyzer showed that the insertion and removal events had the proper widths of 15, 30, and 60 seconds, the lag time until analyzer response was 48 seconds. The response of the analyzer itself was such that 68% of the equilibrium value is achieved after 9 seconds. Since the fire-resistant materials of interest to the FAA have peak heat release rate events of approximately 15 seconds, the standard cone software generating the reduced data was unsuitable for use.
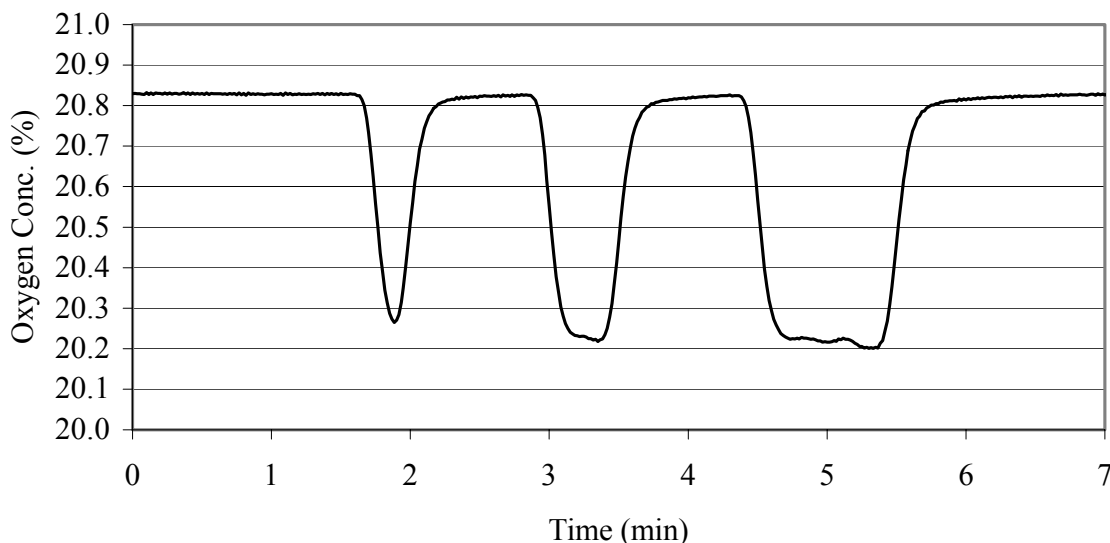

FIGURE 10. CONE OXYGEN ANALYZER RESPONSE TO STEP CHANGES IN METHANE FLOW

This problem was rather easily overcome, however, by having the cone raw data file transferred into an Excel spreadsheet. Although a time shift calculation was done by the cone software, an additional 19 seconds had to be added to match actual analyzer lag time. The heat release data

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generated by the cone was then time deconvoluted [10] for analyzer response using the formula $HRR_{deconvoluted} = HRR + 9*dHRR/dt$, with $dHRR/dt$ calculated using a smoothing formula [11]:

$$dHRR/dt_{(t)} = (HRR_{(t+2)} + 8*HRR_{(t+1)} + 8*HRR_{(t-1)} + HRR_{(t-2)})/12$$

During the round-robin testing of the OSU, for regulatory purposes, it was suspected that calibration of the incident heat flux was the largest contributor to the initial lack of data reproducibility between laboratories and was likely to be a problem here as well. Unfortunately, the physical dimensions of the Gardon gauge used to calibrate the cone were such that the transfer standard (i.e., direct simultaneous comparison with a black-body calibrated Gardon gauge) used for calibration of OSU Gardon gauges could not be used. Instead, a slug calorimeter [12] was constructed similar to one used for verification of OSU heat flux, with the exception that the slug was sized to fit the smaller cone specimen holder. The Gardon gauge supplied with the cone was used to set the heat flux at 25, 35, 50, and 75 W/m^2, with the Gardon gauge millivolt output recorded. After stabilization at the heat flux, the slug was inserted as if it were a test specimen. The data acquisition system was used to measure the slug temperature at a sample rate of 1.0 Hz. The rate of temperature change was used to calculate the actual heat flux, and the new calibration constant determined and entered into the cone software. The previous Gardon gauge calibration constant was approximately 8% low, i.e., 32.2 kW/m^2, instead of the desired 35 kW/m^2.

Two variations of the cone test were conducted for comparison purposes with the OSU. First, the cone was run in normal horizontal mode with the spark igniter. Data was collected in the normal manner; however, the heat release rate curve was deconvoluted to correct for the slow oxygen analyzer response. Second, to simulate the OSU test conditions, the cone was run in vertical mode with a methane pilot for surface ignition. A section of 3.4-mm (1/8-in.) stainless steel tubing was bent so that the tubing passed through the hole in the cone heater and the pilot flame impinged the test specimen 3 millimeters above the center bottom of the specimen holder. Air and methane flow rates were adjusted to be comparable to the OSU pilot flame. The volumetric flow was scaled to the pilot tube orifice size so that the linear velocity of the gases passing through the tube and the resultant flame length were equivalent. Since a baseline measurement was made pretest and subtracted from test concentrations, the small change in oxygen concentration due to the pilot was corrected for by the cone software.

A test series of single-ply fiberglass cloth impregnated with BPA epoxy was run on the OSU and both cone calorimeter configurations. A complete description of the test panel materials is shown in table 1. Because of the hand layup characteristics of the test specimens, the weight pickup varied substantially. One of the objectives of the testing was to see the relationship between peak heat release rate and specimen weight loss. Since the burn event is basically concluded in 2 minutes, it was expected that the total heat release would be proportional to weight loss.

Although the cone calorimeter measured specimen weight for the duration of the test with the mass loss transducer, specimens for both the cone and OSU were weighed manually. This consisted of recording the weight of the test specimen, wrapping the specimen in aluminum foil, recording the weight, and then inserting the specimen and the foil into the holder and recording

the total weight of the assembly. The specimen holder with residue was again weighed posttest. The residue was then removed. The char was oxidized off the glass cloth in a muffle furnace at 600ºC and the char yield determined. During the course of the experiments, it was discovered that cone specimens lost more weight during testing than was possible, based on the initial and final manual weight measurements. This was attributed to small amounts of water absorbed on the rock wool backing material. The discrepancy between the weight loss reported by the cone mass loss transducer versus manual weighing averaged 0.48 grams. More weight loss was consistently registered on the mass loss transducer. When multiple runs for a given day were examined, the first run always had the largest difference, as much as 0.77 grams, which decreased later in the day to as low as 0.11 grams. Until manual weight data was used, little correlation was apparent. While the test procedure calls for environmental conditioning of the test specimen, no requirement is called out for conditioning or storage of the mill board and rock wool backing material. The standard procedure was modified so that the insulation backing material was stored in a dessicator after testing was conducted.

The peak heat release per gram of weight loss and the 2-minute heat release per gram of weight loss for the OSU and cone calorimeters are shown in figures 11 and 12. Weight loss data for the cone was scaled to the OSU specimen size, 0.01 m^2 versus 0.02323 m^2.
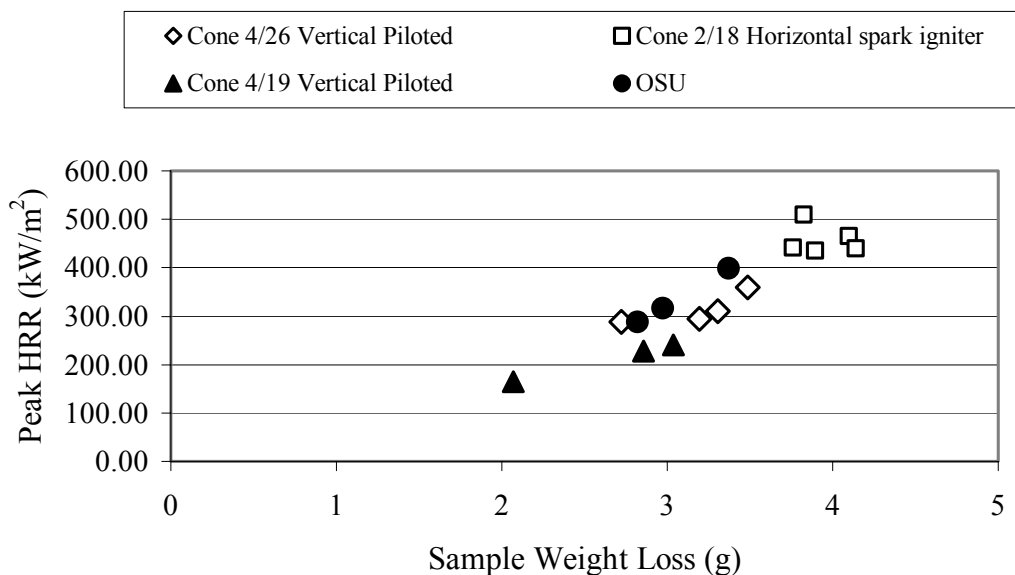

FIGURE 11. OSU AND DECONVOLUTED CONE DATA FOR PEAK HEAT RELEASE RATE OF BPA EPOXY/FIBERGLASS SPECIMENS

As can be seen in figures 11 and 12, there was virtually no difference in the peak and total heat release per gram of sample between the OSU and cone calorimeter data for the BPA epoxy. Sample orientation also had virtually no effect on results when a pilot flame was used in vertical orientation to insure consistent specimen ignition. The 2-minute heat release showed excellent correlation between the OSU and cone calorimeter for the single-ply BPA fiberglass laminate, well within the variability expected of any fire test. (This is especially true where hand layup techniques introduced specimen-to-specimen variations in resin thickness across the specimen face.) Peak heat release rate also correlated very well.
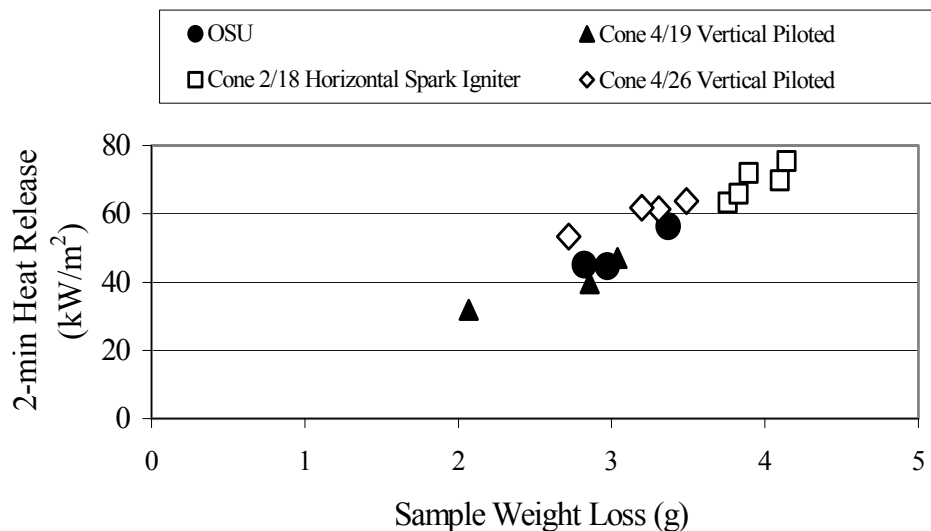
13

FIGURE 12. OSU AND DECONVOLUTED CONE DATA 2-MINUTE TOTAL HEAT
RELEASE OF BPA EPOXY/FIBERGLASS SPECIMENS

CONCLUSIONS

The Ohio State University (OSU) Rate of Heat Release Apparatus and cone calorimeters have shown reproducible results when using the oxygen depletion technique for moderately flammable materials. When both instruments are properly calibrated and the cone calorimeter is adjusted or time deconvoluted for the response time delay of the paramagnetic oxygen analyzer, the techniques generated comparable data within the expected accuracy of fire test measurements (±15%).

The thermal and chemical heat release rates are in good agreement for the OSU when correction of the thermopile signal is accomplished for radiant heat release absorbed into the body of the apparatus. Wall temperature-corrected thermopile data had considerably more signal noise and variability than either the oxygen depletion technique examined or the standard thermopile measurement technique used for regulatory purposes.

REFERENCES

1.   "Heat Release Rate Test for Cabin Materials," Aircraft Material Fire Test Handbook, Section 5, DOT/FAA/CT-89/15, September 1990, pp. 37-66.

2.   "Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter," ASTM E 1354-90, ASTM Fire Standards, 3rd ed., Philadelphia, PA; American Society for Testing of Materials, 1990, pp. 803-817.

3.   Ostman, A. L. and Svensson, I.G., "Comparison of Three Test Methods for Measuring Rate of Heat Release," *Fire and Materials,* Vol. 9 No. 4, 1985, pp. 176-184.

4. Babrauskas, V., "Comparative Rates of Heat Release From Five Different Types of Test Apparatuses," *Journal of Fire Sciences,* Vol. 4, No. 2, 1986, pp. 148-159.

5. Tsuchiya, Y. and Mathieu, J. F., "Measuring Degrees of Combustibility Using an OSU Apparatus and Oxygen-Depletion Principle," *Fire Safety Journal*, 17, 1991, pp. 291-299.

6. Babrauskas, V., "Comparative Heat Release Rates for Aircraft Materials Measured in Different Apparatuses," Babrasukas, V. and Grayson, S.J. eds., *Heat Release in Fires*, Elsevier Applied Science, New York, NY, 1992, Chapter 17(c), pp. 583-590.

7. Hill, R.G. and Fitzgerald, L. T., "Interlaboratory Comparison of Heat Release Data From Aircraft Panels," DOT/FAA/CT-TN86/3, March 1986.

8. Sarkos, C. P., et al., "Implementation of Heat Release Measurements as a Regulatory Requirement for Commercial Aircraft Materials," *50th Calorimetry Conference*, National Institute of Standards and Technology, Gaithersburg, MD, July 23-28, 1995.

9. Smith, E. E., "Heat Release Rate Calorimetry," *Fire Technology*, Vol. 32, No. 4, 1996, pp. 333-347.

10. Lyon, R.E. and Abramowitz, A., "Effect of Instrument Response Time on Heat Release Measurements," *Fire and Materials*, Vol.19, 1995, pp. 11-17.

11. Salvadori, M.G., *Numerical Methods in Engineering*, Prentice-Hall, Englewood Cliffs, NJ, 1961, Chapter 2, pp. 85-87.

12. Filipczak, R.A. and Lyon, R. E., "Heat Flux Measurements in the Ohio State University Rate of Heat Release Apparatus," DOT/FAA/AR-TN00/38, August 2000.