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Heat Release and Mass Loss Rate Measurements for Selected Materials

William D. Walton William H. Twilley

U.S. DEPARTMENT OF COMMERCE National Bureau of Standards National Engineering Laboratory Center for Fire Research Gaithersburg, MD 20899

December 1984

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, Secretary NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director



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NOMENCLATURE

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AH effective he	eat of combustion
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- L heat of gasification
- m mass
- Am change in mass
- q heat transfer
- Q energy release
- r stoichiometric oxygen/fuel ratio

Subscripts

b burning

Superscripts

- Agent

- ()" per unit area
- (') per unit time

HEAT RELEASE AND MASS LOSS RATE MEASUREMENTS FOR SELECTED MATERIALS

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Abstract

The purpose of this study was to measure fire parameters for a selected group of materials. These parameters are to be used in a continuing study of flame spread. The parameters measured are rate of heat release, rate of mass loss, heat of gasification, effective heat of combustion, stoichiometric ratio and time to ignite. Heat release rates and mass loss rates are given as a function of time for several external heat flux levels. The rate of heat release is also given as a function of the total heat released. The experimental results and analysis are shown for six diverse materials representative of aircraft (interior panels, carpeting, and seat cushions) and buildings (particle board, polymethyl methacrylate (PMMA) and rigid foam).

Key words: rate of heat release, ignition, fire tests, fire safety, aircraft interior materials.

1. INTRODUCTION

A variety of fire parameters have been considered important in predicting the fire performance of materials in full scale fires. Quintiere [1] has assessed the importance of a number of these parameters and developed relationships for use in predicting flame spread. In addition, flame spread measurements have been conducted for a selected group of materials [2]. This paper reports measurements of fire parameters conducted on the same group of

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materials, as part of a continuing effort to predict full-scale fire performance from laboratory measurements. The six materials tested are particle board, polymethyl methacrylate (PMMA), rigid foam, flexible foam, carpet and aircraft panel. A description of the materials can be found in table 1 [2].

A series of non-flaming and flaming experiments were conducted in a heat release rate calorimeter to determine the fire parameters. The fire parameters that were measured are rate of heat release (\dot{Q}) , rate of mass loss (\dot{m}) , heat of gasification (L), effective heat of combustion (Δ H), and stoichiometric ratio (r) and time to ignite. Terminology relating to fire is not always standardized and several of the parameters mentioned above may be described by different names. Rate of heat release may also be called rate of energy release. The effective heat of combustion may be referred to as heat of combustion or heat of reaction. The term effective is used to signify the heat of combustion during actual fire conditions as opposed to the maximum theoretical value.

2. DESCRIPTION OF EXPERIMENTAL APPARATUS

A detailed description of the experimental apparatus, known as the NBS II calorimeter, has previously been reported [3]. In brief, the apparatus consists of a steel box with dimensions of approximately one meter on each side. The top of the box opens to a 0.46 m diameter exhaust stack which vents to the outside of the test building. The bottom of the box has an opening to permit the insertion of a calibration burner or a sample holder. When either is in place the box is fully enclosed. Air is introduced into the apparatus through a controlled, fan powered air dilution line and distributed through

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-2-
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air cooling towers. Energy is supplied to the test specimen through premixed air - natural gas radiant panels located around the specimen on the four sides of the box. These are capable of providing irradiance levels from approximately 25 to 80 kW/m². Lower irradiances may be obtained through the use of shades. One half of the radiant panels can be operated to irradiate a single side of a vertical specimen or all panels can be operated to irradiate both sides of a vertical sample or the top face of a horizontal specimen. Specimens in either the vertical or horizontal position can be weighed continuously with an electronic weigh cell. Diagrams of the apparatus are shown in figures 1-2.

Several modifications were made to the apparatus and associated instrumentation for this experimental series. The calorimeter was originally designed to measure rate of heat release using a substitution burner. For this method a gas fired substitution burner is connected via a feedback control system to a grid of thermocouples in the exhaust stack. As the specimen releases heat the gas flow to the substitution is reduced such that the temperature as measured by the thermocouple grid remains constant. The reduction in gas flow is directly related to the rate of heat release. For this test series it was decided to use the oxygen consumption technique to determine the rate of heat release. This technique has been successfully used in a number of applications [4,5], and overcomes some of the problems of the substitution burner method such as thermal transients when the sample is inserted into the test chamber.

A detailed explanation of the oxygen consumption technique can be found in ref. [6]. It is based on the concept that, to a reasonable engineering

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approximation, the heat produced per unit mass of oxygen consumed is nearly constant for materials commonly used in construction [7]. Reference [6] describes a number of possible measurement options which can be used to implement the oxygen consumption technique. For the NBS II calorimeter total air flow into the system was measured. The concentrations of O_2 , CO_2 and CO in the stack were measured with water vapor trapped out. The gas concentrations as well as all other data were collected on a digital data acquisition system at a 5 second interval and processed with a computer data reduction program.

The normal vertical specimen holder used in the apparatus was reconstructed to accommodate back-to-back vertical specimens with a total exposed area of 0.1515 m². Two total heat flux gauges were installed in the plane of the specimen adjacent to the specimen holder. One of these gauges faced in the direction of each of the sample faces. The gauges were calibrated with a gauge mounted in the face of a blank specimen. This allowed a measurement of the incident heat flux to the specimen to be made prior to each experiment. A similar arrangement with a single heat flux gauge was used for the horizontal specimens. The exposed area of the horizontal specimen was 0.0465 m^2 .

An air-acetylene pilot was used to ignite the specimens in the flaming experiments. For vertical specimens the pilot was in the plane of the specimen approximately one centimeter above the specimen holder. In this position the pilot would ignite the combustible gas from the specimen but would not impose a heat flux on it. If back-to-back specimens were used a pilot was placed over each of the specimens. For horizontal specimens a single pilot was placed perpendicular to each of the specimens and approximately two centimeters above the specimen surface.

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The purpose of the non-flaming experiments was to determine the external heat flux required to generate a unit mass of vapors. This can be defined as a heat of gasification (L). Mass loss data for determining heat of gasification should preferably be taken with the surface bathed in an inert atmosphere such as nitrogen. This prevents oxidation and thus heat release at the material surface during the experiment. The use of an inert atmosphere was not practical in the experimental apparatus used. Care was taken however, to attempt to exclude data in the analysis during the time at which a specimen was glowing or flaming. Figures 3-7 are the characteristic mass loss rate data for the materials. To simplify presentation, data for experiments with approximately the same irradiance have been shown as a single curve. An error bar has been used to indicate the average range of the data contributing to a single curve. Data was collected until the specimen fell from the holder. Data for the flexible foam could not be taken in this apparatus because of excessive melting and dripping.

All experiments, except the aircraft panel, were conducted on back-toback vertical specimens irradiated from both sides. The aircraft panel was mounted vertically and exposed on a single side. The carpet specimens were held in place with 19 gauge (1.04 mm), $1/2 \times 1/2$ inch (13 x 13 mm) mesh galvanized hardware cloth placed over the specimen face. A 1/2 inch (13 mm) thick calcium silicate board was placed between the specimens to maintain rigidity. All specimens were conditioned at 55% relative humidity and a temperature of 22° C.

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The heat of gasification has been used previously as both a time dependent and average parameter. For the data collected here it was decided to use an average value. Table 2 shows the values of external heat flux or irradiance and the average mass loss rate per unit area. In addition, the percent of the peak mass loss rate over which the average mass loss rates were computed is shown. The percentages were chosen to describe an average peak mass loss rate in a systematic manner. Figures 8-12 are the data in table 2, plotted with a least squares linear fit. The slope of the line has been defined as the heat of gasification. These values along with the intercept are shown in table 3. It should be noted that insufficient data were available for the PMMA to have confidence in the curve fit.

4. FLAMING EXPERIMENTS

This group of experiments was designed to measure the time to ignition, the rate of heat release and mass loss, the effective heat of combustion, and the stoichiometric ratio. The time to ignition was measured in the same experiments as the other fire parameters. The ignition data were taken to compare with ignition times measured previously in another apparatus on the same materials [2]. The ignition time was measured by visual observation and defined as the time at which flames were sustained over the surface. The observed ignition times at various external heat flux levels are listed in table 4. The data have also been plotted along with the data previously taken in figures 13-18. The number of experiments with ignition times reported is larger than the number of experiments described in other parts of this section. This is a result of instrument failure which affected the calculations of the other fire parameters but did not interfere with the observation of ignition time.

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The rate of heat release measurements were made using the oxygen consumption technique which was described earlier. Since the gas fired radiant panels used to irradiate the specimen release heat, a correction was required. Prior to each experiment a baseline heat release rate was established for the radiant panels and subtracted from the subsequent measurements for the specimen. This procedure was handled automatically through the computer data reduction program. The magnitude of the heat release from the panels was significant and in many cases greater than the heat released by the specimen. In the apparatus, the heat release from the radiant panels at the 25, 50 and 75 kW/m² incident flux levels to the specimen was approximately 280, 450 and 600 kW/m², respectively. These baseline levels must be considered when analyzing the results of the experiments.

In order to provide the highest level of confidence practical in the experimental results a number of calibration procedures were used. Prior to conducting the experiments, the individual instruments were calibrated and checked over a period of time for drift. A number of experiments were conducted in which the heat release of a metered quantity of technical grade methane was measured. After calibration, the measured heat release corresponded to the value calculated by the oxygen consumption technique to within ± 5%. Each day prior to conducting heat release measurements, a methane calibration test was conducted as a check of instrumentation. For each experiment in which the heat release was measured the mass of the sample was also measured. Since the density of materials varied widely, the electronic weigh cell was adjusted and calibrated each time a different series of specimen materials were tested.

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Figures 19-36 are the characteristic curves of the heat release and mass loss rate measurements grouped by material. In these curves, rates for experiments with approximately the same irradiance have been shown as a single curve with an error bar to indicate the average variation. The heat release data have been shown both as a function of time and of total heat released. The latter is one method of describing release rates at different irradiance levels without the effects of ignition delay times. The data shown in all curves is over the time in which the specimen remained intact and in the sample holder. All experiments, except the flexible foam, were conducted on back-to-back vertical specimens irradiated from both sides. The flexible foam specimens were mounted horizontally and irradiated from the top only. The carpet specimens were held in place with 19 gauge (1.04 mm), $1/2 \times 1/2$ inch $(13 \times 13 \text{ mm})$ mesh, galvanized hardware cloth placed over the specimen face. A 1/2 inch (13 mm) thick calcium silicate board was placed between the carpet specimens to maintain rigidity.

The rate of heat release measurements were used to calculate the effective heat of combustion and the stoichiometric ratio. The effective heat of combustion, which is sometimes referred to as the heat of reaction, can be defined as either a time dependent or average parameter. As a time dependent parameter

$$\Delta H = \frac{Q''}{m_{h}''}$$

 \dot{Q} is the rate of heat release and \dot{m}_{b} is the rate of mass loss for the burning material. Both of these values are time dependent and thus the effective heat of combustion may vary with time. For most applications an average effective heat of combustion is desired and can be defined as

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$$\Delta H = \frac{Q''}{\Delta m''_{b}}$$

where Q is the heat released and Δm_b is the change in mass during the time over which that heat was released. From the effective heat of combustion the stoichiometric oxygen/fuel mass ratio r can be computed as

$$r = \frac{\Delta H}{13.1} (MJ/kg) (MJ/kg)$$

where 13.1 (MJ/kg) is the heat produced per unit mass of oxygen consumed, a constant for all materials [7]. Table 5 shows the effective heat of combustion and stoichiometric ratio for the materials at various irradiance levels. Also given are the average values of these quantities for each material.

5. ANALYSES OF TEST RESULTS

Very little data, of the type measured, is available for the materials tested with the exception of the PMMA and the rigid foam. In fact several of the materials are unique and no data for these are known to exist. The curves for the non-flaming experiments show that the materials demonstrate a number of different mass loss profiles. The particle board shows a single rise and a gradual decline. The rigid foam carpet and aircraft panel show two peaks indicating that lighter fractions of the material may be released quickly. The PMMA demonstrates a nearly constant rise in rate and at the higher irradiance levels appeared to boil on the surface.

The heat of gasification measurements for particle board, PMMA and rigid foam can be compared to the data of Tewarson [8]. He has measured fire para-

meters for oak, PMMA and rigid foam. Data for the rigid foam can also be found in [7] much of which was measured by Tewarson. The rigid foam tested by Tewarson is reported to be the same as that tested in this study. The heat of gasification values from Tewarson were determined from measurements at a single irradiance. Tewarson's measurements of heat of gasification for oak is 1.7 MJ/kg for an irradiance less than or equal to 46 kW/m² and 5.5 MJ/kg for an irradiance above 46 kW/m². Although oak is quite a different material from particle board, these values span the average value of 4.52 MJ/kg determined for particle board. Tewarson reports a value of 1.63 MJ/kg for PMMA as compared to the 1.81 MJ/kg measured here. These are within the expected agreement but the values for the rigid foam are not. Tewarson reports 3.11 MJ/kg and this study 7.77 MJ/kg. Since the results for the rigid foam are the most consistent, they indicate that the rigid foam used in this study may not be the same as that tested by Tewarson.

The ignition data in figures 13-18 shows good agreement between the time to ignite measured in the NBS II calorimeter and flame spread apparatus. There seems to be a trend for NBS II times to be slightly shorter. This may be a result of the different airflow characteristics in the two pieces of apparatus.

The heat release and mass loss curves reveal several attributes of the materials and test apparatus. Those heat release curves which do not go to approximately zero, such as the particle board and PMMA, indicate the experiments were terminated prior to complete consumption of the material. Those materials fell from the sample holder and the test was terminated at that point. The total heat released versus heat release rate curves for rigid

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foam, flexible foam and carpet indicate that at the higher irradiances the total heat released is almost independent of irradiance. It can also be observed that the mass loss curves correspond well with the heat release curves. The exception to this is for PMMA. The mass loss curves show an intermediate peak at all irradiance levels. This peak appears to correspond to the delamination of the two sheets of PMMA that were glued together to form the specimen. This peak is only barely visible in the heat release curves. It can also be seen for the PMMA that the mass loss and heat release rates were continuing to rise after the test was terminated. Sample delamination resulted in the specimen falling from the holder before a substantial portion of the sample had been consumed. As a result, neither a peak nor a steadystate value could be obtained.

As with the heat of gasification there is only limited data for effective heat of combustion and stoichiometric ratio with which to compare. Since the stoichiometric ratio was derived from the effective heat of combustion the comparison need only be made with one of these parameters. The effective heat of combustion data from Tewarson for oak ranges from 11.2 - 12.6 MJ/kg. This compares with an average 11.6 MJ/kg measured for particle board (table 5). Tewarson's values for PMMA range from 23.9-24.9 MJ/kg with a maximum value measured in the oxygen bomb calorimeter of 25.2 MJ/kg. These compare well with the values at an irradiance level of approximately 50 kW/m² but the values at other levels exceed the maximum theoretical value. This may be due in part to the relatively short time of the PMMA experiments and the delamination of the samples. The average effective heat of combustion measured for the rigid foam was 19.0 MJ/kg. The oxygen bomb value as reported by Tewarson was 25.0 MJ/kg and he measured effective heats of combustion from

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10.8-15.0 MJ/kg. As with the heat of gasification there is considerable difference between the results of this study and those from Tewarson.

It is generally assumed that the effective heat of combustion is basically independent of irradiance level. This was found to be true for all materials except the aircraft panel. It showed a strong dependence of irradiance level on effective heat of combustion. This may be due to the fact the material is a composite and does not burn well at lower irradiance levels. As a result the heat of combustion measurement may show the effect of different materials burning at different irradiance levels.

6. ACKNOWLEDGMENTS

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Table 1. Description of materials

Material	Description	Thickness (cm)
Particle board	Douglas Fir wood with particle size range of 0.97-2.6 mm and ureaformaldehyde binder	1.28
Poly (methyl methacrylate) [PMMA]	Rohm & Haas* - Type G, two 0.635 cm thick samples bonded with methyl ethyl ketone	1.28
Rigid foam	Polyurethane low density rigid foam - GM 31 ⁺	2.54
Flexible foam	Polyurethane low density flexible foam - Custom Products, Inc.* HD54CA low density	2.54
Carpet	Wool-nylon looped fibers with a rubberized backing	0.634
Aircraft panel	Aircraft interior lining (#2), a composite material consisting of a phenolic-polyamide honeycomb core with apoxy fiberite face sheets and a tedlar coating on the exposed face	2.54

* Use of trade names implies no endorsement by the National Bureau of Standards.

⁺ GM 31 is a material from the Material Bank of the Product Research Committee currently maintained at the National Bureau of Standards ("Materials Bank Compendium of Fire Property Data", Product Research Committee, February 1980).

	Irradiance kW/m ²	Average Mass Loss Rate g/s-m ²	Percent of Peak Mass Loss Rate Over Which Average Mass Loss Rate Was Computed
Particle Board	14.7	6.6	90
	30.0	9.9	
	30.4	10.2	
	31.0	10.1	
	35.6	11.3	
PMMA	10.5	1.3	90
	24.8	10.1	
	28.5	10.6	
Rigid Foam	10.0	0.8	70
·	25.4	2.9	
	25.7	3.0	
	30.3	3.5	
	36.8	4.3	
Carpet	13.9	1.2	70
	25.3	3.1	
	25.9	3.4	
	34.4	6.1	
	35.0	7.9	
	58.5	16.9	
	60.5	18.5	
Aircraft Panel	11.8	1.3	50
	36.3	5.9	
	37.5	7.6	
	42.3	9.5	
	42.7	9.9	
	47.4	10.6	
	53.4	11.2	
	64.3	15.0	

Table 2. Average mass loss rate for non-flaming experiments

Table 3. Heat of gasification

Material	Heat of Gasification MJ/kg	Intercept kW/m ²
Particle Board	4.52	-15.0
PMMA	1.81	8.1
Rigid Foam	7.77	3.1
Carpet	2.50	15.8
Aircraft Panel	3.72	9.1

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	Irradiance kW/m ²	Time to Ignition S		Irradiance kW/m ²	Time to Ignition s
Particle Board	11.6	611	Flexible Foam	24.8	12
	13.0	440,475,510		25.1	17
	19.9	180		25.2	12
	20.9	165		49.8	2
	24.7	118		49.8	2
	24.9	120		50.2	3
	25.2	115		74.8	1
	25.4	105		75.0	1
	49.7	23		75.2	1
	50.0	31			
	50.1	25	Carpet	20.4	225
	50.3	28		25.0	215
	74.7	10		25.1	206
	74.8	10		25.4	221
				49.9	27
PMMA	20.0	194		50.2	27
	24.7	173		74.6	13
	24.8	113		74.8	13
	25.1	113			
	25.3	111	Aircraft Panel	25.2	64
	49.7	28		25.4	73
	50.1	26		50.2	10
	50.1	31		50.4	12
	50.4	30		74.6	3
	74.9	14		74.9	7
	75.2	13			
Rigid Foam	13.4	105			
	14.0	100			
	15.5	78			
	20.2	55			
	20.6	51			
	25.2	3			
	25.4	4			
	49.6	1			
	50.2	1			
	74.8	1			
	75.7	1			

	Irradiance kW/m ²	Effective Heat of Combustion MJ/kg	Stoichiometric Ratio r	Time for Average s
Particle Board	24.9 25.4 49.7 50.1	11.2 11.1 11.4 12.7	0.86 0.85 0.87 0.97	100-400 100-400 10-250 10-250
	74.7 74.8	11.8 <u>11.4</u> 11.6	0.90 <u>0.87</u> <u>0.89</u>	5-250 5-250
РММА	25.1 25.3 50.1 50.4 74.9 75.2	26.9 27.2 24.2 24.5 25.8 <u>25.1</u> 25.6	2.05 2.08 1.85 1.87 1.97 <u>1.92</u> 1.95	150-390 145-420 90-225 85-245 55-150 55-185
Rigid Foam	13.4 25.2 25.4 49.6 50.2 74.8 75.7	22.4 18.7 20.0 18.1 18.3 17.5 <u>18.0</u> 19.0	1.71 1.43 1.53 1.38 1.40 1.33 <u>1.37</u> 1.45	115-200 5-105 5-105 5- 75 0- 75 0- 65 10- 50
Flexible Foam	24.8 25.1 25.2 49.8 49.8 50.2 74.8 75.0 75.2	21.0 20.1 19.2 15.8 16.6 21.6 20.8 17.6 <u>16.9</u> 18.8	1.60 1.54 1.47 1.20 1.26 1.65 1.59 1.35 <u>1.29</u> 1.44	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$
Carpet	25.0 25.4 49.9 50.2 74.6 74.8	15.8 16.8 17.0 17.1 17.7 <u>16.2</u> 16.8	1.20 1.28 1.30 1.31 1.35 <u>1.24</u> 1.28	180-515 185-505 5-160 5-160 5-155 5-155
Aircraft Panel	25.2 25.4 50.2 50.4 74.6 74.9	11.5 12.0 11.3 14.9 17.2 <u>16.0</u> 13.8	$ \begin{array}{r} 0.88\\ 0.91\\ 0.86\\ 1.14\\ 1.31\\ \underline{1.22}\\ 1.05\\ \end{array} $	60-330 65-330 5-200 5-200 5-150 5-150

Table 5. Effective heat of combustion and stoichiometric ratio







Figure 2. Sectional view A-A of calorimeter



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800 700 10.5 kW/m² 600 24.8 kW/m² 500 TIME (s) 400 28.5 kW/m² 300 34.8 kW/m² 200 100 10 2 12 9 S 3 0 ດ ω 4 ~



Figure 4. Mass loss rate for PMMA, non-flaming



MASS LOSS RATE (g/s/m²)

Figure 5. Mass loss rate for rigid foam, non-flaming



Figure 6. Mass loss rate for carpet, non-flaming

(²m/s/g) **JTAR 2201 S2AM**

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Figure 7. Mass loss rate for aircraft panel, non-flaming







Figure 10. Average mass loss rate for rigid foam, non-flaming

















Figure 16. Time to ignite for flexible foam



Figure 17. Time to ignite for carpet

Figure 18. Time to ignite for aircraft panel





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Figure 19. Heat release rate for particle board



(^{Sm/s/g}) **JTAR SSOJ SSAM**



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Figure 21. Total heat released for particle board







(Sm/s/g) JTAR 2201 SSAM









(^{Sm\s\g}) STAR SSOJ SSAM

Figure 27. Total heat released for rigid foam







Figure 29. Mass loss rate for flexible foam



Figure 30. Total heat released for flexible foam



Figure 31. Heat release rate for carpet





Figure 32. Mass loss rate for carpet



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Figure 34. Heat release rate for aircraft panel





Figure 35. Mass loss rate for aircraft panel



HEAT RELEASE RATE (KW/m²)

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