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Ignitability Measurements with the Cone Calorimeter*

Vytenis Babrauskas and William J. Parker
Center for Fire Research, National Bureau of Standards, Gaithersburg, MD 20899, USA

The Cone Calorimeter is a new-generation instrument developed primarily for making rate of heat release measurements. This instrument, containing a uniform and well-characterized irradiance source, was also seen to be useful for making measurements of radiant ignition on materials. Data have now been collected for a wide range of materials. The effects of various apparatus dependencies are discussed. Also, some comparative data are available illustrating the performance of similar materials in other apparatuses. Finally, for a selected material, Douglas fir particle board, a detailed comparison with an ignition model has been made.

Key words: Cone Calorimeter; ignitability; plastics; radiant ignition; upholstered furniture; wood materials.

INTRODUCTION

Early ignitability testing was generally based on a furnace exposure to a small specimen, which was assumed to be of nearly uniform temperature.[1] The test procedure consisted of determining the furnace temperature at which ignition was first observed. A widely used procedure was the Setchkin furnace, developed in the late 1940s.[2] Such a test could not be used to study composite materials, nor to compare specimens of varying thicknesses.

Later, it was judged to be more useful in assessing the performance of materials and products if a substantially larger specimen were used, and if a relationship of the length of time needed to achieve ignition for a given heating flux was determined. Consequently, ignitability apparatuses have been designed where the heating source was primarily (1) radiant heat, (2) convective heat and (3) heating from direct flame impingement. Heating from direct flame impingement is common in a Bunsen burner-type test, a large number of which have been developed through the years.[3,4] The limitation of using this type of test is that the heat fluxes presented to the specimen are highly non-uniform, and consequently are difficult to analyze mathematically. Convective heating is understood to be a flow of hot gases directed at the specimen, without the nearby presence of flame. While convective heating apparatuses of various sorts have been built, this has not been commonly done for flammability testing purposes, both since convective heating is less important in room fires than radiative heating[5] and also due to practical difficulties. Thus, radiant heating can be considered to be the preferred form for an ignitability test.

The radiant heater

The most important feature of a radiant ignition test is its heater. In general, such a heater should be able to achieve adequately high irradiances, have a relatively small convective heating component, present a highly uniform irradiance over the entire exposed face of the specimen and be designed so as not to change its irradiance when the mains voltage varies, when heater element aging occurs or when the apparatus retains some residual heat from the exposure given to a prior specimen.

A room fire burning near its maximum rate can show gas temperatures over 1000°C, producing corresponding irradiances to walls and contens of 150 $kW\,m^{-2}$. Testing under such extreme conditions may not be required; nonetheless, if ignition in post-flashover fires is to be simulated, irradiances of over 75 $kW\,m^{-2}$ should be available, and preferably closer to 100 $kW\,m^{-2}$. A significant convective component would negate the purpose of having a radiant ignition test. Rather low convective fluxes can be achieved for specimens oriented horizontally, face up, and with the prevailing air flow being upwards. When a vertical specimen orientation is considered, it becomes evident that a boundary layer will normally be expected to develop which will add some convective component. The convective boundary layer component is not uniform over the height of a specimen. Thus it is seen that better uniformity can also be expected under conditions where the convective component is minimized.

During an accidental fire, the actual ignition source that can be expected will, in most cases, be a fire in the vicinity of the target object. Its radiation spectrum will depend on the size of the fire. A very small fire can show a substantial fraction of its radiation at wavelengths characteristic of H_2O, CO_2 and other combustion products.[6] For larger fires—certainly for room fires reaching a hazardous condition—the radiation from the soot tends to dominate: the result is an approximation to a grey body radiation.[7] For such a grey body radiation the temperature will typically be in the vicinity of 1000°C.[8] Experimentally, heater choices for test apparatuses have included gas-fired panels, electric resistance heaters, flames and high-temperature lamps. Electrical heaters tend to have a near-grey body characteristic and, assuming a dull or oxidized surface condition, a high emissivity. Gas-fired panels derive a substantial portion of their radiation from the ceramic face. Thus, while there are discrete molecular wavelength peaks, overall the radiation shows a grey-body continuum, typically in the range of 700–1000°C temperatures.[9] High-temperature lamps, which have been used by several investigators,[6,10] typically have radiating temperatures of 2200–3000°C. The spectral

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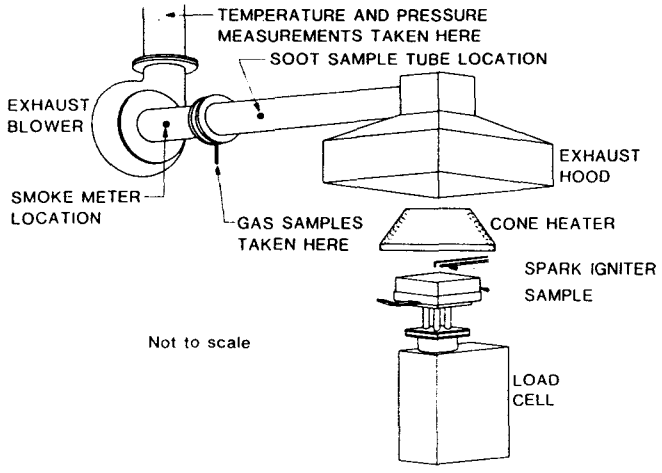

Figure 1. General view of Cone Calorimeter.

specifically designed for testing radiant ignitability. That apparatus, even in its earliest form, contained an especially appropriately designed thermal radiation source in the form of a truncated cone. This design eliminated the difficulty with most conventional designs, namely, that the specimen center is heated more than the edges. For ignitability measurements this was the main advantage for this heater geometry.

A few years later, at NBS, following experiments with a number of heat release rate apparatuses it was desired to develop a new instrument which, while still economically affordable for commercial testing laboratories, would alleviate most of the known difficulties of existing instruments. A significant shortcoming of a number of the existing designs that were capable of operating at high fluxes was that specimen irradiance uniformity was poor. The geometry of the ISO cone in the ignitability apparatus appeared to be ideal for use also within a heat release rate apparatus. The apparatus, as developed at NBS using the conical heater (Figs 1 and 2), became known as the Cone Calorimeter, and has been described in detail.[16,17] This apparatus has been put forth by the American Society for Testing and Materials (ASTM) as a proposed standard.[18]

Used in the Cone Calorimeter, the conical shape of the heater was seen to have additional advantages: it allowed the flame to exit the apparatus directly when used in the horizontal orientation (the Cone Calorimeter, unlike the ISO cone, is designed to also be usable with a vertical specimen and cone orientation), and it provided a geometry which deflected the flame plume in such a manner that a cold air sheath kept the combustion products from directly impinging on the heater elements. In addition to the ability to test in both orientations, the Calorimeter cone was designed with a different heating element and different mechanical layout details, which permit measurements to be made at irradiance of up to 110 kW m^{-2}, instead of being restricted to 50 kW m^{-2}, as is specified for the ISO cone.

A number of other features serve to differentiate the Cone Calorimeter from the ISO ignitability apparatus, in addition to the obvious one of being able to make heat release, mass loss and smoke measurements. The ISO ignitability apparatus uses a specimen 165 mm by 165 mm, which is exposed only at a circular opening of 140 mm diameter in the center. This is not objectionable *per se* for ignitability purposes alone, but for heat release rate testing would not be desirable since the difference between actual specimen surface area and radiatively exposed surface area would be substantial. The specimen holder in the Cone Calorimeter was designed so that there is only a very small, 3 mm, lip around the face of the specimen. Finally, the piloting arrangements are of a very different nature.

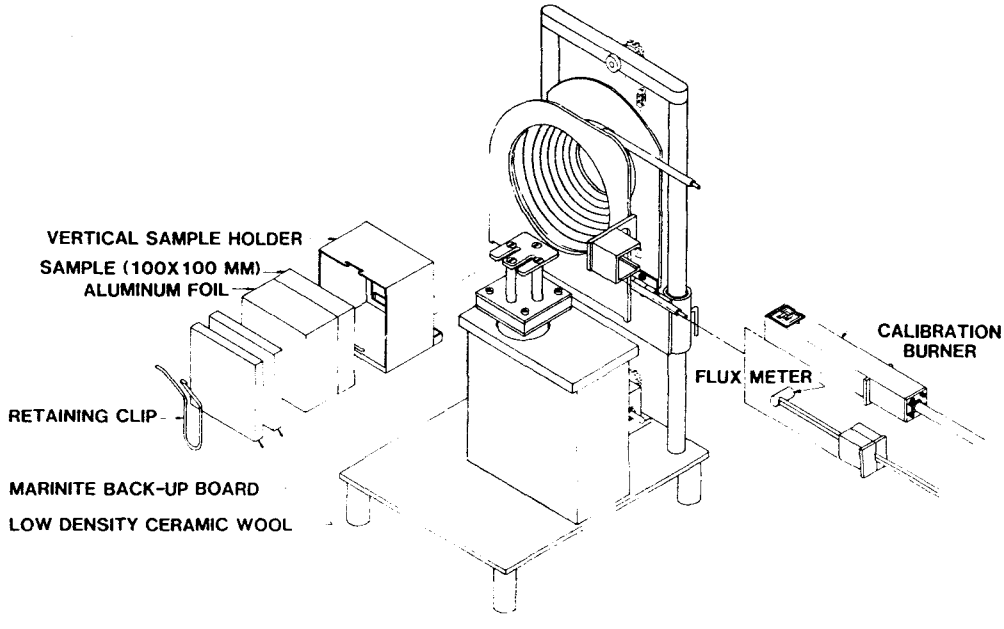

Figure 2. Detailed view of Cone Calorimeter (vertical orientation).

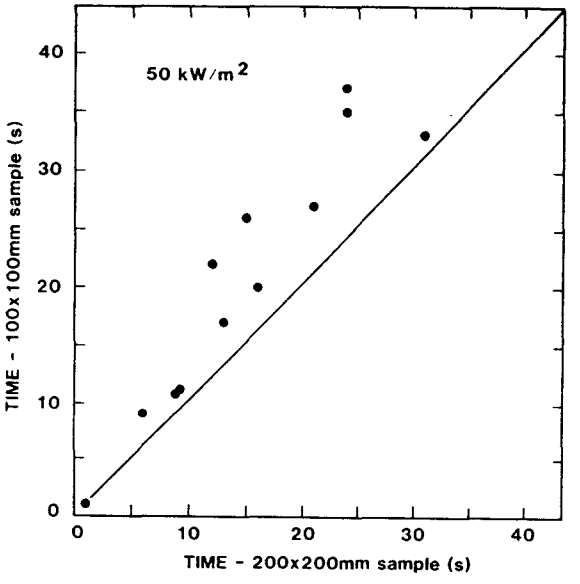

Figure 4. Comparison of ignition times for a variety of materials at 50 kW m^{-2} for two different specimen sizes.[21]

insure that the specimen is thermally thick can be represented by:

$$l = 0.6\rho/\dot{q}'' \quad (1)$$

where l is the thickness(mm), ρ is the density (kg m^{-3}) and $\dot{q}''$ is the heat flux (k W m^{-2}). This is probably a reasonable rule of thumb for other materials as well. The proportionality of the required thickness to $\rho/\dot{q}''$ is derived from classical heat conduction theory by equating the time for the front surface to reach the ignition temperature to the time for the rear surface temperature to begin to rise and assuming that the thermal conductivity is proportional to the density. Numerical calculations were necessary to determine a suitable constant because of the impact of the front surface heat losses.

For materials which are not thermally thick at the time of ignition the nature of the backing material or substrate can influence the measured value of the ignition time. In the Cone Calorimeter the substrate is a blanket of refractory ceramic fiber material, having a nominal density of 65 kg m^{-3}. In use, the material assumes a more compacted density of roughly 100 kg m^{-3}. Whenever possible, materials, whose thicknesses are less than the minimum suggested in the above formula, should be mounted on that substrate material over which they will actually be used. As a practical guide for testing unknown commercial samples it is desirable to specify that any specimens less than 6 mm thick should always be considered as needing to be tested over their in-use substrate.

Fabrics are a special case. Thin fabrics are sometimes used for constructing air-supported structures; these should be tested with an air space in back, simulating the usage conditions. A special holder has been constructed (Fig. 5) which allows the fabrics to be pulled taut and held above a dead air space.

Air flow rates

In the Cone Calorimeter, experiments with varying the exhaust hood air flow rates have been done with two types of materials, black PMMA and redwood. The results are listed in Table 1. The conclusion is that over the complete feasible air flow range, from zero (which, of course, would not be practical for operation), through the normal value[18] of 24 l s^{-1}, up to the apparatus maximum of 41 l s^{-1}, the effects of air flow rates on ignition times in the Cone Calorimeter are minor and do not consistently trend in one direction. Since the thermoplastic PMMA material and the charring redwood represent very different types of degradation behavior it is expected that these conclusions have some generality.

Edge effects

In the vertical specimen orientation, the specimen has to be restrained against falling out: thus, the vertical specimen holder incorporates a small lip covering 3 mm along the edges. In the horizontal orientation, no special

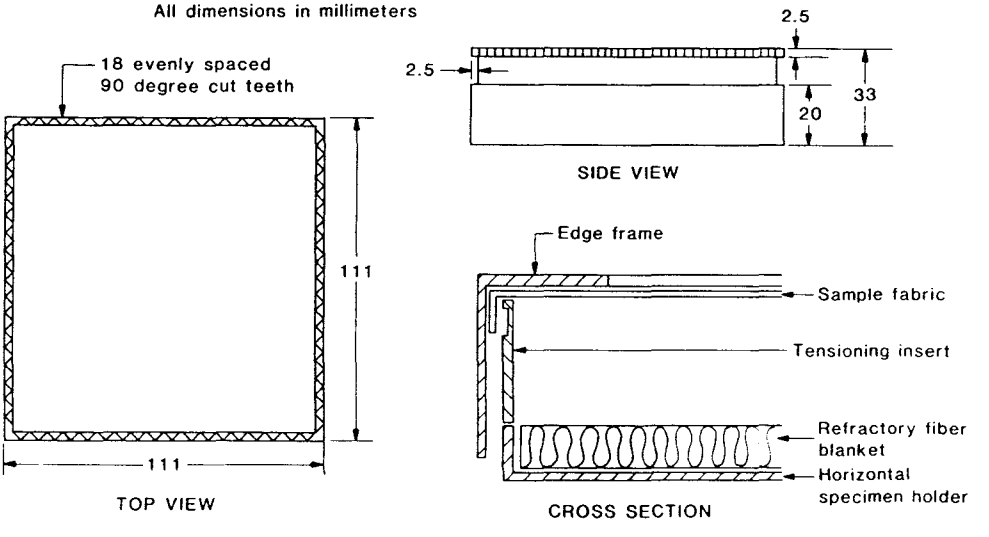

Figure 5. Holder as used for tests of air-supported assemblies.

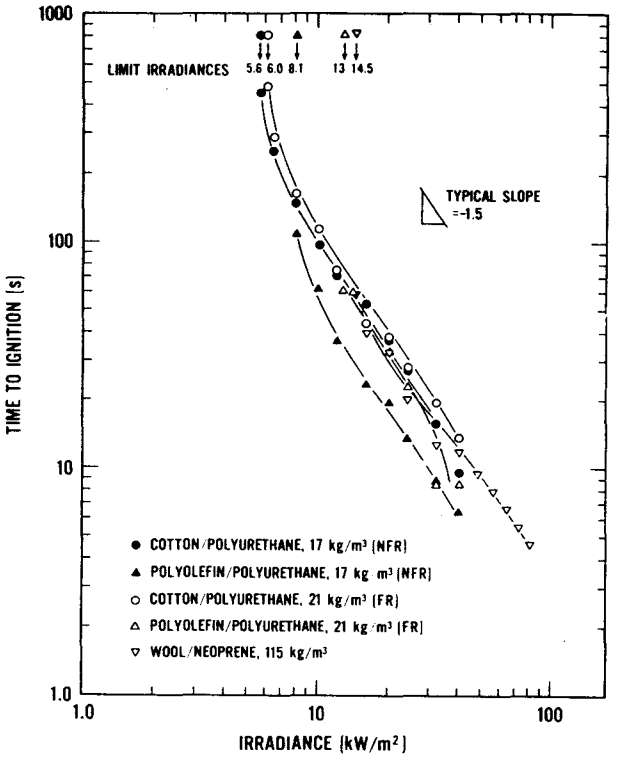

Figure 6. Ignitability curves for various fabric/foam assemblies.

resistant to very small ignition sources, but that the ignition and burning rate behavior is essentially unchanged when larger ignition sources are used.[24] While these conclusions are limited to the furniture foams tested, they are, nonetheless, indicative.

A more extensive investigation was made of foam/fabric composites, representing specimens taken from upholstered furniture. Figure 6 gives these results. The specimens studied encompassed the whole range of expected furniture fire behaviors—from a wool/neoprene specimen, typically showing the least flammable possible behavior of the commercial materials studied, to an unretarded (NFR) polyurethane foam, covered with a polyolefin fabric, showing among the most flammable behaviors.[24] Various fire retarded combinations (FR) are intermediate. A sufficient number of tests was made to characterize the complete ignitability curve, including the determination of the minimum irradiance required to achieve piloted ignition (limit irradiance). It is striking to note that the range of variations in ignitability for all these

Table 6. Comparative ignitability results for red oak and black PMMA, horizontal orientation

Irradiance ($kW m^{-2}$)	Ignition time (s) Red oak (20 mm, with edge frame)	Black PMMA (25 mm)
25	216	161
50	24.3	38
75	11.8	19.5
100	6.5	11.5

materials is only about a factor of 2 to 3. This small range of behavior is most likely due to the fact that the effective density of the test material is a dominant factor in determining its ignitability. For furniture applications, the range of acceptable densities is rather small.

Tests have also been conducted on fabrics intended for air-supported buildings. These fabrics include an impermeable coating, which is typically polytetrafluoroethylene, silicone or PVC. These were tested using the holder illustrated in Fig. 5. Table 5 gives the results for two irradiances—35 $kW m^{-2}$, chosen to represent the typical peak heat fluxes from limited ignition sources,[24] and 75 $kW m^{-2}$, which can represent heat fluxes in a flashed-over room. It can be seen that a satisfactory discrimination can be made between materials likely to ignite from a limited fire, and ones which would get involved only in the event of a fully flashed-over room fire.

Data have also been obtained on red oak, which has often been used as a standard calibration material. Table 6 gives these results over a wide range of irradiances: also given are the comparative results for black PMMA, which is being considered for use as a standard calibration material, representing simply pyrolyzing, non-melting plastics. Figure 7 shows plots of both sets of results on a log–log scale. It can be seen that, for irradiances of 50 $kW m^{-2}$ and greater, a power law can be fit very well to the results for both materials. It shows a dependence of the ignition time on approximately the -1.9 power of the irradiances for red oak and the -1.7 power for PMMA.

COMPARISON WITH EMPIRICAL IGNITABILITY MODELS

PMMA is a convenient combustible to use in ignitability studies and has been examined by other investigators. The

Table 5. Ignitability of air-supported fabrics

Specimen	F/T	F/Sa	F/Sb	F/Sc	F/Sd	PE/PVC
Fabric type	Fiberglass	Fiberglass	Fiberglass	Fiberglass	Fiberglass	Polyethylene
Surface coating	PTFE	Silicone	Silicone	Silicone	Silicone	PVC
Mass ($kg m^{-2}$)	1300	450	1130	1080	1230	950
Time to ignition (s) at irrad.						
$= 35 kW m^{-2}$	N.I.[a]	N.I.	225	N.I.	N.I.	17
$75 kW m^{-2}$	44	12	19	19	28	7

[a]No ignition, for test exposure of 600 s.

Table 7. Comparison of experimental PMMA results to simple predictive rules

Method	Ignition time (s) at irradiance ($kW m^{-2}$) 25	50	75	100
Measured in cone calorimeter	161	38	19.5	11.5
Measured by Hallman	145–58	35–7	15–18	8–10
Simple theory (Eqn. (4))	140	20.8	8.0	4.2
Hallman's correlation (Eqn. (6))	360	90	40	23

conduction equation; unfortunately, the equation then no longer possesses an analytical solution. For estimation, one can set T_s at the fixed value of T_{ig} for all times. The expression for PMMA then becomes

$$t_{ig} = 0.346 \times 10^5 (\dot{q}'' - 9.28)^{-2} \quad \text{(s)} \qquad (4)$$

Hallman considered, instead, a number of somewhat more complex models, but did not use them when he found poor agreement with his data. For practical calculations use he proposed an empirical relationship, based on fitting all of his plastics data, of which PMMA was only a small portion. His recommendation, in the same units as above, is that

$$t_{ig} = 1035 \frac{(T_{ig} - T_o)^{1.04} (k\rho C)^{0.75}}{(\alpha \dot{q}'')^2} \qquad (5)$$

When evaluated for PMMA using the same properties as above, this gives

$$t_{ig} = 2.22 \times 10^5 \, \dot{q}''^{-2} \quad \text{(s)} \qquad (6)$$

Both of the above relationship are evaluated in Table 7. Neither is particularly good at representing the experimental data closely. It is interesting to note that Hallman's expression (Eqn. (6)) does not represent his data any better than does Eqn. (4). This is presumed to be because his correlation was optimized as the best fit for all of the plastics tested, and not just for PMMA. To achieve better predictive results, it is clear that a more advanced model of ignition is needed.

COMPARISON WITH DETAILED NUMERICAL MODELS: DOUGLAS FIR

Ignitability is but one of the responses of a material to a fire exposure. A numerical model for the total fire response of wood was developed by Parker.[27]

This model includes heat release rate, surface temperatures and mass loss rate predictions. The heat release rate is determined by intergrating the product of the instantaneous mass loss rate and the instantaneous heat of combustion at each depth over the thickness of the exposed wood slab. When the calculated heat release rate reaches the minimum required to maintain a flame on the surface, ignition is assumed to occur. Prior to that time the heat release rate is set equal to zero. This minimum flux is taken to be $30 kW m^{-2}$. The model takes into account the degree of char, the char shrinkage and the variation in thermal conductivity, specific heat and density with the temperature. It also accounts for thermal radiation and convective cooling from the front surface. The surface is assumed to be black for both absorption and emission of thermal radiation. The model was initially checked out with Douglas fir particle board. In this case it was assumed that the kinetics were governed by a single first-order reaction. The activation energy, frequency factor, heat of combustion of the volatiles, char contraction factors and the thermal conductivity were all experimentally determined on specimens taken from the same batch of material as those tested in the Cone Calorimeter. The calculated and measured heat rates have been examined in detail earlier.[27]

The ignition times of Douglas fir particle board were measured in the Cone Calorimeter at incident radiant heat fluxes of 25, 50, 75 and $100 kW m^{-2}$ using vertically oriented specimens. The calculated ignition times were taken to be equal to the times of appearance of a calculated heat release rate of $30 kW m^{-2}$. These times are compared in Table 8. Since the convective cooling term in the model is based on steady state laminar free convection, while the ignition process occurs before a steady state has been established, calculations are also included for the case where the convective cooling is assumed to be equal to zero. Thermal radiation from the surface is

Table 8. Measured and computed ignition times for Douglas fir particle board

Irradiance ($kW m^{-2}$)	Ignition time in Cone Calorimeter (s)	Calculated ignition times (s)[a]: Based on $\dot{m}'' = 2.5 g m^{-2}$ w/o conv. cooling	with conv. cooling	Based on $\dot{q}'' = 30 kW m^{-2}$ w/o conv. cooling	with conv. cooling	Based on $T_c = 380°C$ w/o conv. cooling	with conv. cooling
25	100	143 (375°C)	172 (372°C)	168 (388°C)	203 (385°C)	153	189
50	25	30.4 (396°C)	33.0 (395°C)	36.5 (415°C)	39.4 (413°C)	25.7	28.2
75	12	13.6 (409°C)	14.4 (409°C)	16.4 (430°C)	17.4 (429°C)	10.2	10.9
100	8.5	7.7 (418°C)	8.0 (417°C)	9.4 (441°C)	9.8 (440°C)	5.4	5.6

[a]Computed surface temperatures at ignition are shown in parentheses.

Table 10. Ignition of building materials determined by four test methods[34]

Specimen	Thick. (mm)	Mass ($kg\,m^{-3}$)	Mass ($kg\,m^{-3}$)	Time to ignition (s) for $50\,kW\,m^{-2}$ irrad. Cone Calor. Hor.	Cone Calor. Vert.	ISO[c] Hor.	OSU[d] Vert.	STFI[e] Vert.
Insulating fiber board	13	250		1	5	12	5	10
Medium-density fiberboard	12	600		15	20	22	15	25
Particle board	10	750		15	25	30	20	25
Gypsum plaster board	13	700		25	23	34	18	30
PVC wall covering on gypsum board	0.7[a]		240	1	10	9	10	8
Paper wall covering on gypsum board	0.6[a]		200	5	10	14	8	20
Textile wall covering on gypsum board	0.7[a]		370	8	15	22	15	15
Textile wall covering on mineral wool	50	100		10	2	13	5	12
Melamine faced particle board	1.2[a]	810		100	145	26	—[f]	—[f]
Polystyrene foam	50	20		25	83	2	20	105
Rigid polyurethane foam	30	30		1	3	2	8	3
Wood panel, spruce	11	530		10	15	18	10	15
Paper wall covering on particle board	0.6[b]		200	20	24	19	15	25

[a]Over 13 mm substrate.
[b]Over 10.5 mm substrate.
[c]International Organization for Standardization test.[15]
[d]Ohio State University test.[11]
[e]Swedish Forest Products Research Laboratory test.[34]
[f]Poorly defined time to ignition.

laboratory comparison of building materials (discussed below) are also shown in Table 10. In this series, the ignition times for the horizontal orientation, with one exception, are shorter than in the vertical orientation. This may suggest that, in general, it is somewhat easier to achieve the requisite lower flammability limit above a burning pool than it is in the thinner, higher velocity boundary layer which results in the vertical orientation. On the other hand, the convective cooling of the horizontal specimens is less than it is for the vertical specimens, so that the same surface temperature is reached earlier for the horizontal specimens.

Relation to heat release

Not much exploration has been done on the relationship between time to ignition and the rate of heat release. In general, it can be expected that these are not highly correlated properties. In at least one situation, however, a significant correlation can be seen. White pine specimens were tested for ignitability and rate of heat release. The data in Table 11 show that at higher irradiances ignition time data exhibit little scatter, and there is no correlation to heat release rate. At an irradiance of $20\,kW\,m^{-2}$, however, the ignition is very slow and there is a large amount of sample-to-sample variation. Under those circumstances an inverse relationship can be seen between the ignition time and the peak value of the heat release rate. The correlation in Table 11 may possibly be explained on the basis of a delayed ignition allowing a larger accumulation of combustible vapors, which, when ignited, give rise to a larger peak heat release rate (wood materials specimens generally show the peak rate of heat release just shortly after ignition). In a general case, for natural products, ignition and heat release rate variability can be attributed to specimen density variations.[33] In the present series, however, the specimen density variations were less than 3% among the specimens.

Table 11. Effect of random variations of ignition time on peak heat release rate for single specimens

Irradiance = $20\,kW\,m^{-2}$ Ignition time[a] (s)	Peak $\dot{q}''$ ($kW\,m^{-2}$)	Irradiance = $40\,kW\,m^{-2}$ Ignition time[a] (s)	Peak $\dot{q}''$ ($kW\,m^{-2}$)
145	196	13.9	220
266	135	17.4	241
444	96	18.0	223

[a]For 20 mm thick white pine specimens, vertical orientation.

COMPARISON WITH OTHER TEST METHODS

A series of materials was produced by the Swedish National Testing Laboratories (Statens Provningsanstalt) for use in test standardization. Tests were conducted at NBS in the Cone Calorimeter. Östman *et al.*[34] provided data taken in three other ignition apparatuses: the ISO ignitability test, the OSU apparatus and a radiant panel test devised by the STFI (Swedish Forest Products Research Laboratory). Some of the relevant test para-

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