

## FLAMMABILITY TESTING STATE-OF-THE-ART

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### ABSTRACT

Traditional material flammability tests are discussed in terms of their empirical foundation and oversimplified interpretation of fire phenomena. More recent rate-of-heat-release tests overcome some of these problems by measuring a material's response to different levels of fire exposure. However, no existing small-scale tests are sensitive to the radiant emission from the material's own flames. This radiant emission controls large-scale fire hazards. As a result, existing flammability tests cannot be expected to adequately characterize large-scale hazards. Some new approaches to this problem are discussed and a specific bench-scale test method is suggested which may overcome the identified problems of existing test methods.

### BACKGROUND

Traditionally, the flammability of a building material has been evaluated by measuring its: 1) ease of piloted ignition; 2) ability to propagate a small creeping flame in the presence of an external radiant source; and/or 3) ability to propagate a larger under-ceiling fire as measured by the ASTM-E84 "tunnel test" which exposes a 25 ft (7.62 m) long sample to a sizeable propane ignition source. This latter test is legally recognized by most building codes. Since the piloted ignition and creeping flame spread phenomena are closely related and depend on similar material properties they are often jointly evaluated by the ASTM-E162 test apparatus which measures the creeping spread rate and extent of maximum flame travel under conditions of a spatially decreasing external radiant flux.

These tests were developed about thirty years ago at a time when building materials were based primarily on cellulose which has a limited range of flame properties. Also, at that time, lacking a basic understanding of fire behavior, it was implicitly assumed that all materials could be ranked on a single flammability scale based on some standard test which subjects a material to a single representative fire environment. In view of the need for some flammability assessment procedure and the absence of obviously contradictory full-scale (or loss) data this oversimplified approach appeared justified at its time. This traditional philosophy has now outlived its usefulness.

### FULL-SCALE TESTING

Around 1970, after experiencing unexpectedly severe losses involving newly introduced fire retarded plastics, various full-scale corner tests were run to check their flammability rankings suggested by the ASTM-E84 test (Castino, 1975). A lack of correlation was observed which was particularly troublesome for those fire-resistant insulation materials having a flame spread rating

less than 25. The ASTM-E84 ranking is based primarily on the extent of flame travel normalized so that red-oak has a rating of 100 and cement-board a rating of zero. Apparently modern polymeric materials and especially fire-resistant foam insulations do not properly fit on this ranking scale.

This lack of correlation has led to a wide-spread mistrust of current standard flammability tests and the reluctant suggestion that one can only rely on full-scale tests for flammability assessment. Consistent with this full-scale test philosophy the ISO (International Standards Organization) and ASTM are developing a "Standard Method for Testing Wall and Ceiling Materials and Assemblies" (ASTM, 1980) which exposes a material to a large 176 kW propane burner flame placed in a lower corner of an 8 ft x 12 ft x 8 ft high (2.4 x 3.6 x 2.4 m) room whose wall and ceilings are lined with the material. The outcome of these corner/room tests is strongly dependent on the rather arbitrarily chosen heat release rate of the ignition source. For exposure heating rates above some (material dependent) critical value the fire will undergo a dramatic transition to flashover when the heat release rate from the burning wall material becomes comparable to the exposure fire heat release rate. Exposure fires smaller than this critical value are insufficient to initiate flashover and usually cause only local damage. Test engineers welcome such clear-cut go/no go tests because they have an indisputable outcome. However, a result from a single test run with a given exposure is relatively uninformative to a potential user interested in the outcome involving other levels of exposure. A potential user probably wishes to rank materials according to their exposure which will just cause run-away ignition (e.g. flashover) of the material. Unfortunately, at present, it is not possible to determine this critical exposure for a given material from a single full-scale test.

Full-scale tests are also very expensive, difficult to reproduce, and require such large quantities of sample materials that they cannot be considered for screening new materials under development. Finally full-scale tests, being empirical, give little guidance for assessing hazards in related situations. Often small changes in geometric details have a profound effect on the outcome of a fire. In conclusion, full-scale tests are generally regarded as essential for corroborating the general claims of standard flammability test methods, but cannot serve as a substitute because of their complexity, cost and large material requirements.

#### FIRE PHENOMENA

It is now generally recognized that various materials can have markedly different flammability rankings in different situations depending on such factors as: 1) fire scale; 2) imposed heat flux levels; 3) geometric arrangement; 4) the presence of other nearby materials, and 5) the temperature, pressure and degree of vitiation of the surrounding atmosphere. Fires generally involve synergistic couplings between a material and its environment. Also, different fire scenarios are often governed by qualitatively different burning mechanisms which in turn are controlled by different combinations of material properties. It is important to understand these differences in burning mechanisms when interpreting flammability test results. In particular, it is important to appreciate the effects of fire-scale, if one wishes to infer full-scale fire behavior from small standard flammability tests.

## SMALL-SCALE

The steady (constant area) burning rate of a small-scale fire is controlled by the convective heat transfer from the flames. Small-scale flames are not thick enough to emit significant radiation. As a result their mass transfer rates are primarily controlled by the heat required to vaporize unit mass of fuel. The overall heat release per unit area is given by the product of the mass transfer rate and the heat of combustion of the fuel volatiles. Other factors controlling small-scale burning rates depend only on geometry for typical organic fuels burning by natural convection in air at atmospheric pressure. The important fuel property - namely the heat required to vaporize unit mass of fuel - can be directly measured by Tewarson's (Tewarson, 1980) well known "FM Flammability Apparatus" which measures the fuel-mass-loss-rate and heat-release-rate under different applied radiant exposures.

Flame-retardants acting by inhibiting gas-phase reactions can significantly reduce, or even prevent, burning at small-scale. The effectiveness of such retardants has often been inferred from the LOI (Limiting Oxygen Index) test which measures the critical ambient oxygen concentration that is just sufficient to permit downward creeping flame-spread on a small sample. Because this test is convenient and requires only a very small test sample, it is widely used in the chemical industry during material development. Unfortunately, the test results can be very misleading because large-hazardous-scale-fires are not significantly influenced by such gas-phase flame retardants (because large-scale flow times are so much longer than reaction times). Innumerable disappointments have occurred in recent years when supposedly non-flammable fire-retardant polymers burned vigorously in large-scale tests. For example, PVC plastics which usually have an excellent LOI rating burn more rapidly at large-scales than acrylics which generally have a poor LOI rating. Also, the flame-retardants encouraged by this test tend to significantly increase the smoke output and toxicity of a fire.

Fire-retardants which act by encouraging char-formation in the solid-phase can be very effective at all fire scales. By preventing transfer of carbon to the gas-phase they are triply effective by: 1) providing a thermally insulating char layer; 2) reducing the gas-phase heat-release-rate and resulting flame heights; and 3) reducing the flame luminosity and consequent radiant heat transfer which is of dominant importance at large-scales. It is speculated that some of these retardants act by encouraging the polymerization of the fuel vapors as they flow through the chemically active char layer (Parker, 1982). The effectiveness of these char-enhancing retardants can be evaluated by a rate-of-heat-release (RHR) apparatus which measures the transient combustion heat release per unit area of a material subjected to a controlled radiant flux. Tewarson's "FM Flammability Apparatus" and Smith's "Ohio State Apparatus" are well known examples of such RHR tests. Tewarson uses a 10 cm diameter sample and Smith uses a 25 x 25 cm square sample. In both cases the material requirements are small enough to permit testing at a variety of imposed flux levels. However, neither test explicitly measures the flame luminosity or radiated fraction of heat release. As a result, one should not directly extrapolate the test results to large-scales where radiation from the flames is a controlling factor.

Many modern polymeric materials are retarded by the simple addition of inert fillers which increase the heat required for fuel gasification and often leave a porous char-like insulating residue. These effects can be measured by the above mentioned RHR tests. In addition, some fillers incorporate a significant amount of water of hydration, which upon vaporization may possibly reduce soot formation and flame radiation. Unfortunately, the current lack of a flame radiation test has prevented measurement of this latter effect.

The rate-of-heat-release test is particularly useful for examining charring flame-retarded materials such as polyurethane or PVC foams. Such materials can have a distinctly non-linear response to an imposed heat flux. Figure 1 shows the peak response of various polyurethane foams (NFPA Handbook, 15th Ed., pg. 4-7). Notice the changes in rankings for various imposed heat fluxes. At very low flux levels the material surface temperature does not increase sufficiently for significant gasification. Above some critical flux level gasification occurs at a rate sufficient to support piloted ignition. Once ignition occurs the sample receives heat both from the external radiant source and the flames themselves. The added heat transfer from the flames often decreases with increasing rates of gasification leading to a less than linear increase of heat release rate with increasing imposed flux.

A rate-of-heat-release (RHR) test has the advantage of providing several important flammability parameters from a single test run versus time. Figures 2a and 2b show a typical RHR test arrangement and results (Ostman, 1982). The sample receives a uniform radiant heat flux. Measurement of oxygen depletion in the exhaust is now typically used to infer the rate-of-heat-release (Huggett, 1980). The initial time delay prior to gasification provides a measure of the ease of ignition. The rapid increase to the peak heat-release-rate is controlled by the material's heat of gasification. The subsequent decrease in heat-release-rate is due to increasing char insulation; while the final secondary peak results from acceleration of the pyrolysis wave as it approaches the thermally insulated back-surface of the sample. Figure 2b shows curves for several externally imposed fluxes. It simulates the effects of flame radiation in much larger fires. The heat flux actually received by the solid is augmented by the heat transfer from the flames produced by the sample itself. All of the above transient phenomena are being actively studied by various fire research groups (Delichatsios and de Ris, 1983). A possible criticism of most current rate-of-heat-release tests is their external radiant heat source. Gas panel radiant heat sources provide heat over a typical infrared wavelength range but their flux levels are too low for realistic view factors; whereas quartz heaters provide plenty of heat but at unrealistically short wavelengths. Solid fuel response times are known to be quite sensitive to the imposed wavelength (Welker, 1969). Improved infrared gas fired radiant heaters using newly available high temperature ceramics may resolve this problem.

Except for the characterization of flame radiation, it is now generally believed that the rate-of-heat-release measurement provides the most meaningful characterization of large-scale flammability.

Before closing this discussion of small-scale fire phenomena, one should mention the wide body of research on the creeping flame spread associated with downward and horizontally propagating fires. This phenomenon is reasonably

well understood for both flame-retarded and non-retarded materials having a smooth surface. It is addressed in part by the LOI test. Also Quintiere, in a series of studies, has shown that the ASTM-E162 flammability apparatus can be used to evaluate downward creeping flame spread rates under the influence of external radiation (Quintiere, et al, 1982). In particular, one can measure the minimum external flux required to sustain propagation. A similar apparatus and technique is now widely used for evaluating carpet flammability. While these advances are significant for the general flammability problem, the creeping fire spread phenomenon is not of central importance to most large-scale fire hazards. The marginal creeping flame-spread is governed by local chemical kinetics, gas phase diffusion and solid conduction, whereas the critical condition for large-scale upward fire spread is governed by solid ignition, the duration and intensity of rate-of-heat-release and the flame radiative heat feed-back. The associated phenomena are quite different and should not be expected to correlate.

#### LARGE-SCALE

As the scale of a fire increases, the flames become thicker and have more material which can radiate. In general, the radiative heat transfer from flames to adjacent surfaces exceeds convective heat transfer for flame heights exceeding 30 centimeters (Orloff, de Ris, Markstein, 1975). For organic fuels this radiation comes primarily from soot in the flames which makes them appear brightly luminous. Generally, the pyrolysis vapors from man-made polymeric materials are high in carbon content and produce more soot than cellulosic fuels whose pyrolysis vapors have a significant amount of oxygen already bound to the carbon atoms. Fuels which generate copious amounts of smoke tend to have highly radiative flames and have higher large-scale burning rates. The black smoke is thought to arise from the flames losing so much heat by radiation that they are extinguished locally by this radiant loss.

All present day small-scale flammability tests attempt to simulate large-scale fire environments by imposing an independently controlled external radiative flux onto the fuel sample. This external flux generally dominates the radiation from the sample's own flames; so that the measured results are insensitive to the sample's own flame radiation and cannot be expected to provide a complete evaluation of the material flammability at large-scales. This insensitivity is advantageous insofar as it can yield a clear picture of the solid response to a controlled external environment. But it leaves out the essential ingredient - namely the flame radiation which typically represents 80% of the heat feedback at large-scales (Orloff, Modak, Alpert, 1977).

How should we cope with these problems? Clearly we cannot do away with standard flammability tests. If possible, we should have tests which require relatively small samples - say 30 cm square or even less - to encourage testing by industry involved in developing new materials. Of course results from such tests must be corroborated at full-scale for a selection of representative fuels. These problems appear surmountable as will be described below.

Rate-of-heat-release tests are clearly essential and several such tests are under development at various fire research institutions. The test measures

the rate of combustion energy released per unit sample area versus time when subjected to various levels of externally supplied radiation. It is essential to evaluate material at various levels of irradiance because many materials have a strong non-linear response. Also, because charring materials typically have a strongly decreasing transient heat release subsequent to ignition, one should evaluate both the peak rate of heat release, maximum average rate-of-heat-release over selected time intervals (say 1, 2, 3 and 5 minutes) as well as the cumulative heat release. Results from these rate-of-heat-release tests can be directly used for estimating the evolved transient heat release rate and corresponding flame heights for the material when subjected to a known source fire in different practical situations of interest.

Knowledge of the rate-of-heat-release leads directly to estimates of flame heights. In general, both laminar and turbulent flame heights are controlled only by the fire geometry and the actual heat release rates and not by other fuel properties such as its stoichiometric requirements (Masliyah and Steward, 1970; Schug, Manheimer-Timnat, Yaccarino and Glassman, 1981).

To evaluate whether the evolved flames are powerful enough to significantly add to the exposure heat flux, and thereby induce a self-propagating wall or corner fire, one must evaluate the radiative properties of the flames. These properties are the effective flame radiation temperature  $T_f$  and the absorption-emission coefficient  $k_f$  which is essentially proportional to the amount of soot per unit volume. The radiation emitted per unit volume is equal to  $4 \sigma k_f T_f^4$  where  $\sigma$  is the classical Stefan-Boltzmann constant.

The accompanying Figure 3 shows a scientific flammability apparatus being constructed at FMRC to evaluate these flame radiative properties for fire-resistive charring wall materials. The charring material on the left is subjected to an externally controlled radiant flux. The transient rate of heat release is measured by chemically sampling the gases leaving the top of the enclosure. A water-cooled heat transfer plate measures the total (radiative plus convective) heat feedback from the flames. It is shielded from the radiant heat source by a series of radiation baffles, so that it measures only the heat flux from the flames. In addition, we have built a dual radiometer which looks through the flames from the side in order to simultaneously measure both the effective flame radiation temperature  $T_f$  and absorption-emission coefficient  $k_f$ .

This apparatus is not intended as a standard flammability test. It is clearly too sophisticated for widespread use. It is a scientific apparatus intended to provide an in-depth analysis of the radiative properties of a few selected fire-resistive fuels; so that we can provide a rigorous scientific foundation for a subsequent simplified standard material flammability measuring apparatus. It also is intended to provide the basic flame property data needed for the development of mathematical models predicting corner and room flashover. In addition, provision has been made for providing vitiated air to the enclosure for studying the effects of oxygen depletion on flame radiation. This apparatus is the outcome of a long-range research program aimed at providing a basic scientific understanding of flame radiation in fires.

NBS is currently developing a similar but simpler test apparatus which measures the total radiative-convective heat feedback flux from the upper flames. While it is not placed within an enclosure and consequently is not suitable for evaluating the effects of vitiation, it may eventually lead to a standard test method.

#### A SUGGESTED BENCH-SCALE FLAMMABILITY TEST

As discussed above, flammability (or fire hazard) of a material at large-scale is governed by three principle factors: 1) its piloted ignition time in response to an imposed heat flux; 2) the subsequent rate-of-heat-release of its pyrolysis vapors in response to the imposed heat flux; and 3) the radiant emission from the flames resulting from the burning of these pyrolysis vapors.

We have already discussed several test devices which can evaluate the rate-of-heat-release and ignitability of a material. Here we discuss a proposed test concept which in addition may evaluate the radiant emission. The suggested apparatus is also sufficiently compact to be placed on a laboratory bench.

As shown in Figure 4, the test examines a buoyant laminar (candle-like) diffusion flame produced by the pyrolysis vapors emerging from the heated test sample. As explained later, the ignition and rate-of-heat-release measurements are directly inferred from the resultant flame height and should produce results similar to existing test methods with the advantage of decoupling the flame heat-feedback from the pyrolysis process.

Of greater significance the test concept allows one to infer the expected radiant emission from material flames at large-scale. It does this by measuring the fuel's so-called "smoke-point". Recent research at FMRC shows there is a close correlation between large-scale flame radiation and the smoke-point for various hydrocarbon fuels\*. The smoke-point is conventionally defined as the maximum height a buoyant laminar flame can attain without releasing soot (i.e. smoke). The aircraft industry has traditionally used the smoke-point of commercial fuels as a measure of their relative smokiness and as well as their radiant output. Standard test methods exist for evaluating the smoke-point of liquid and gaseous fuels. The present concept extends these methods to solid fuels.

It is well-known that the radiation from both large- and small-scale diffusion flames comes principally from their luminous soot. This soot is both formed and oxidized within the flames. Fuels which produce more soot radiate more intensely. The radiative heat loss cools the flames and, if given enough time, can induce local radiative extinguishment accompanied by release of cold soot in the form of visible smoke. By increasing the fuel supply to a small candle-like flame, one increases its flame height and residence time,

\*Specifically the peak soot absorption coefficient in a 50 kW pool fire and the radiative fraction from a buoyant turbulent fuel jet ranging over 10-50 kW are both tightly correlated with the fuel smoke-point (Markstein, 1983).

resulting in an increased fractional radiative heat loss. A sooty fuel such as propylene can maintain only a relatively short buoyant flame (2.9 cm high) without release of visible smoke; where as a less sooty fuel like propane can support a much taller (16.2 cm) diffusion flame without smoke emission (Shug, et al, 1981). These candle-like flames at their smoke-points release approximately one fifth of their chemical energy in the form of radiation. In the case of hydrocarbon fuels, this heat loss reduces the flame tip temperature to about 1550°K at which temperature soot oxidation rates are significantly reduced (Markstein, 1983). Smoke-point heights are easily measured because the flame undergoes a sudden transition to sooting and release of smoke. Measured smoke-points are independent of apparatus details provided the fuel is supplied at a given temperature and provided the buoyant flame is: well ventilated, shielded from stray laboratory air currents by a chimney, and not subjected to excessive induced forced ventilation (Schalla and Hibbard, 1957).

The accompanying figure shows the suggested measuring apparatus for solid fuels. A patent disclosure has been submitted. It is intended to simultaneously measure both the transient heat-release-rate and sootiness of the pyrolysis vapors emerging from a test sample (say 4-6 cm in diameter) placed in an oven at the start of a test run. Auxilliary supplies of fuel and inert gases are added to the pyrolysis vapors under feedback control to maintain a constant overall heat-release-rate and degree of flame sootiness. In general, for organic fuels, the heat-release-rate of a laminar buoyant diffusion flame is directly proportional to its height, regardless of the fuel chemical composition or presence of added inert gas (Shug, 1981). Consequently, as the rate-of-heat-release from the pyrolysis vapors increases, the excess fuel controller will reduce the excess fuel supply while maintaining a constant flame height as seen by the radiometer. This reduction in excess fuel supply provides a direct measurement of the sample's instantaneous heat-release-rate. The substitution technique should be both rapid and precise.

Similarly, the flame can be maintained in its marginal smoke-point state by a smoke detector which increases the supply of inert gas (say N<sub>2</sub>) as the pyrolysis vapors increase in sootiness. An increase in inert gas flow suppresses soot formation without influencing the flame height (Shug, 1981). The added inert flow provides an instantaneous measure of pyrolysis vapor sootiness. The respective heat-release-rate and sootiness measurements are presumably independent of one another and can be performed simultaneously throughout the test run. Certainly the heat release measurement should be independent of the simultaneous soot-point measurements. Recently Calcote and Manos (1983) showed that the relative ranking of hydrocarbon fuels in terms of their sootiness in diffusion flames is not particularly sensitive to the measurement technique. This suggests that the relative sootiness of fuels will not depend importantly on the sample size, or the base point supply rates of excess fuel and nitrogen.

At present, the suggested test concept is in its early stages of development. Further data is needed for relating large-scale radiant fluxes in various fire situations in terms of measured fuel smoke-points. So far we have only used hydrocarbon fuels for evaluating the test concept. We do not know whether the principles can be extended to fuels having gas-phase chemical retardants. Also considerable effort will be required to develop a standard test method.



In spite of these caveats, one has little choice but to further investigate this suggested flammability test; because there are no other suggested alternative tests designed to assess flame radiative properties. Its bench-scale size and minimal material requirements should make it very attractive to the chemical industry; thereby eliminating the principal impediment to the development of truly fire-resistive materials.

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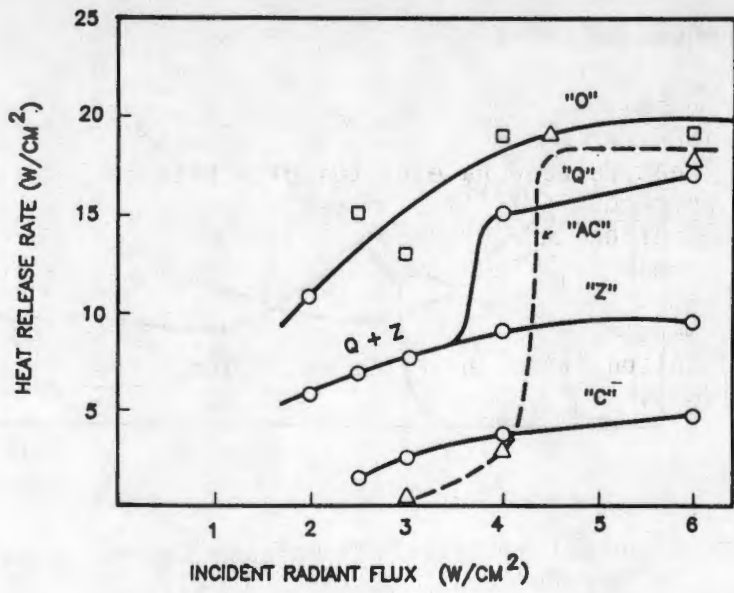


Figure 1: Heat Release Rate of Some Fire-Retarded Polyurethane (Coded According to Castino et al, 1975).

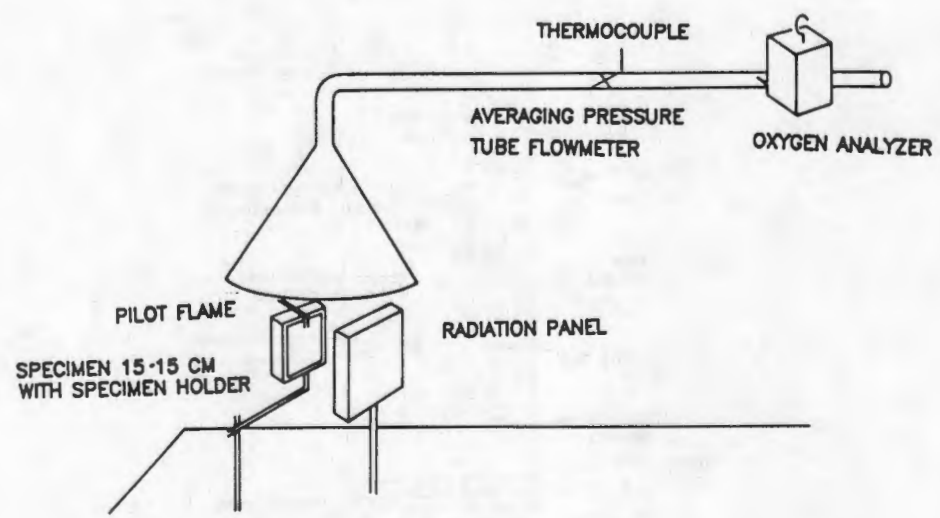


Figure 2A: Typical Rate-of-Heat-Release Apparatus (Ostman, 1982).

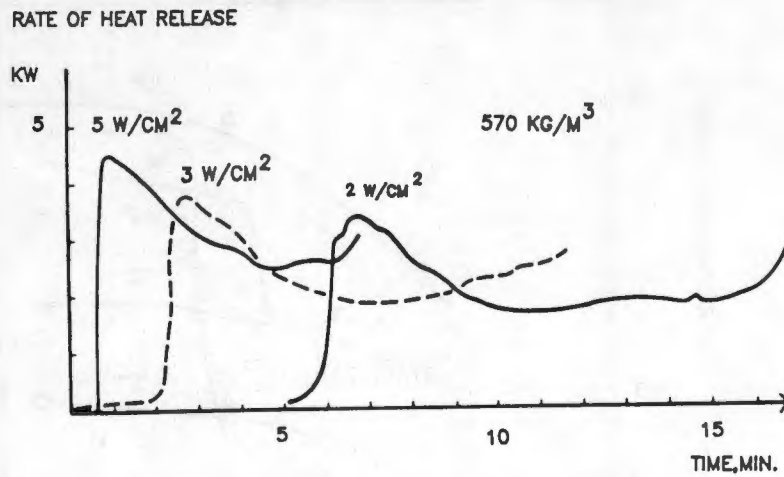


Figure 2B: Typical Rate-of-Heat-Release Curves versus Time for Charring Fuels (Ostman, 1982).

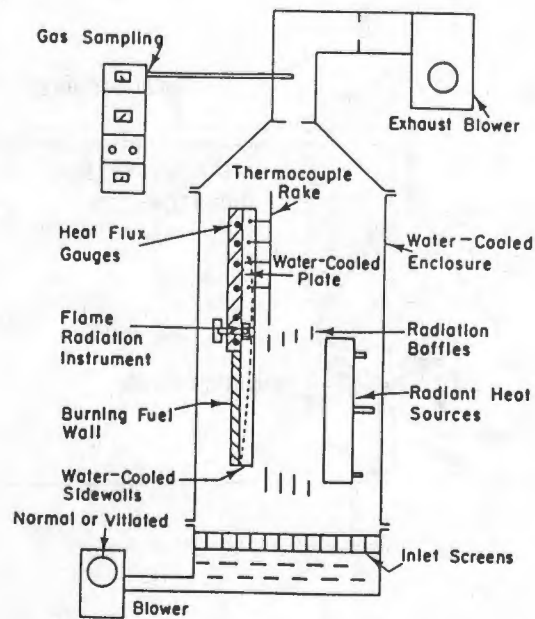
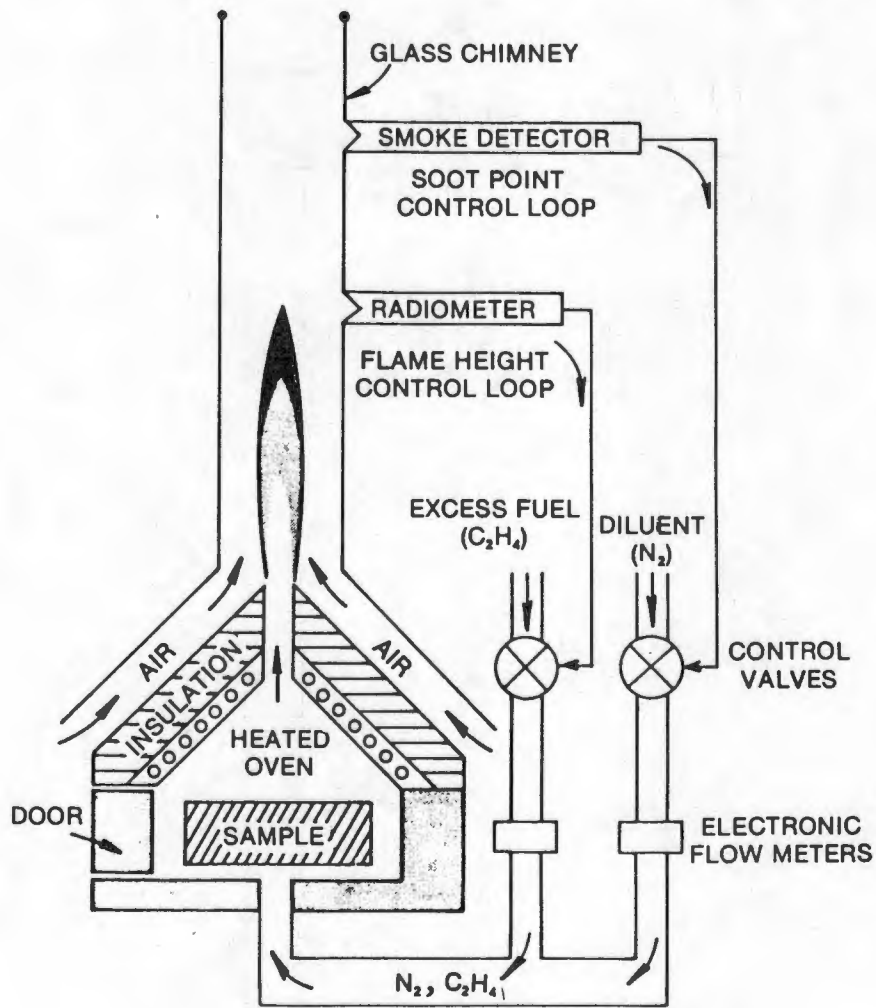


Figure 3: Material Flammability Apparatus of Measuring Flame Radiation in a Vitiated Atmosphere.



MATERIAL FLAMMABILITY TEST

Figure 4: Suggested Bench-Scale Material Flammability Apparatus for Measuring Both Rate-of-Heat Release and Flame Radiation.