



Standard Test Method for Measuring Smoke Toxicity for Use in Fire Hazard Analysis¹

This standard is issued under the fixed designation E 1678; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

INTRODUCTION

The pyrolysis or combustion of every combustible material or product produces smoke that is toxic. It is, therefore, desirable to establish a test method for the development of data characterizing smoke toxicity as an element of fire hazard analyses for both pre-flashover and post-flashover fires. The test method includes quantification of the toxicity of the smoke and ascertain whether the observed toxicity can be attributed to the major common toxicants.

1. Scope

1.1 This fire-test-response standard covers a means for determining the lethal toxic potency of smoke produced from a material or product ignited while exposed to a radiant heat flux of 50 kW/m² for 15 min.

1.2 This test method is limited to test specimens no larger than 76 by 127 mm (3 by 5 in.), with a thickness no greater than 51 mm (2 in.). Specimens are intended to be representative of finished materials or products, including composite and combination systems. This test method is not applicable to end-use materials or products that do not have planar, or nearly planar, external surfaces.

1.3 Lethal toxic potency values associated with 30-min exposures are predicted using calculations that use combustion atmosphere analytical data for carbon monoxide, carbon dioxide, oxygen (vitiation) and, if present, hydrogen cyanide, hydrogen chloride, and hydrogen bromide. The calculation method is therefore limited to those materials and products whose smoke toxicity can be attributed to these toxicants.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 This standard ~~should be used to measure~~ measures and describes the ~~properties~~ response of materials, products, or assemblies in response to heat and flame under controlled conditions and ~~should~~ conditions, but does not be used to describe or appraise the ~~by itself~~ incorporate all factors required for fire hazard ~~or~~ of fire risk assessment of the materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire-hazard assessment or a fire-risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard or fire risk of a particular end-use. conditions.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations (particularly with regard to the care and use of experimental animals) prior to use.* For specific hazards statements, see Section 7 and Note X1.1.

2. Referenced Documents

2.1 *ASTM Standards:*

¹ This test method is under the jurisdiction of ASTM Committee E-5 E05 on Fire Standards and is the direct responsibility of Subcommittee E05.21 on Smoke and Combustion Products.

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E 176 Terminology—Relating to of Fire Standards²

E 800 Guide for Measurement of Gases Present or Generated During Fires²

2.2 ISO Document:

TR 9122 (Parts 1–5) Toxicity Testing of Fire Effluents³

3. Terminology

3.1 *Definitions*—For definitions of general terms used in this test method, refer to Terminology E 176.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *carboxyhemoglobin saturation, n*—the percent of blood hemoglobin converted to carboxyhemoglobin from reaction with inhaled carbon monoxide.

3.2.2 *concentration-time curve, n*—a plot of the concentration of a gaseous toxicant as a function of time.

3.2.3 *Ct product, n*—the concentration-time product in ppm-min obtained by integration of the area under a concentration-time curve.

3.2.4 *fractional exposure dose (FED), n*— the ratio of the Ct product for a gaseous toxicant produced in a given test to that product of the toxicant which has been determined statistically from independent experimental data to produce lethality in 50 % of test animals within a specified exposure and postexposure time. Since the time values in this ratio numerically cancel, the FED is also simply the ratio of the average concentration of a gaseous toxicant to its LC₅₀ value for the same exposure time. When not used with reference to a specific toxicant, the term FED represents the summation of FEDs for individual toxicants in a combustion atmosphere.

3.2.5 *LC₅₀, n*—a measure of lethal toxic potency; the concentration of gas or smoke calculated statistically from concentration-response data to produce lethality in 50 % of test animals within a specified exposure and postexposure time.

3.2.6 *mass loss concentration, n*—the mass loss of a test specimen per unit exposure chamber volume in g-m⁻³.

3.2.7 *post-flashover, adj*—the stage of a fire at which the average air temperature in the upper half of the room exceeds 600°C.

4. Summary of Test Method

4.1 This test method subjects a test specimen to ignition while it is exposed for 15 min to a radiant heat flux of 50 kW/m². (See X1.2.2.) The smoke produced is collected for 30 min within a 200-L chamber communicating with the combustion assembly through a connecting chimney. Concentrations of the major gaseous toxicants are monitored over the 30-min period, with Ct products for each being determined from integration of the areas under the respective concentration-time plots. The Ct product data, along with the mass loss of the test specimen during the test, are then used in calculations to predict the 30-min LC₅₀ of the test specimen. The predicted LC₅₀ is then confirmed in comparable tests by exposing six rats, restrained for head-only exposure, for 30 min to the smoke produced from that mass of the test specimen whose mass loss concentration during the 30-min period is approximately (±10 %) equivalent to 70 % and to 130 % of its estimated LC₅₀. If no more than one rat dies during the 30-min exposure, or within 14-days postexposure to the mass loss concentration corresponding to 70 % of the LC₅₀, and at least five rats die during the 30-min exposure, or within 14-days postexposure to the mass loss concentration corresponding to 130 % of the LC₅₀, the predicted LC₅₀ is considered to be confirmed. Confirmation ensures that the monitored toxicants account for the observed toxic effects.

4.2 For calculation of hazard from pre-flashover, flaming fires, the toxicant gas yields and LC₅₀ values are intended to be used as experimentally determined. For calculation of hazard from post-flashover fires, the yields of carbon monoxide are augmented to reflect the higher yields produced in such fires. The experimental LC₅₀ values are then adjusted using a specified calculation to produce LC₅₀ (post-flashover) values.

5. Significance and Use

5.1 This test method has been designed to provide data for the mathematical modeling of fire hazard as a means for the evaluation of materials and products and to assist in their research and development.

5.2 This test method is used to predict, and subsequently confirm, the lethal toxic potency of smoke produced upon the exposure of a material or product to specific fire test conditions. Confirmation determines whether certain major gaseous toxicants account for the observed toxic effects and lethal toxic potency. If a predicted lethal toxic potency value is not confirmed adequately, indicating a potential for unusual or unexplained toxicity, the lethal toxic potency will need to be investigated using other methodology, such as conducting an experimental determination of the LC₅₀ using the apparatus described. (See X1.3.1 and X1.3.2.)

5.3 This test method produces lethal toxic potency values that are appropriate for use in the modeling of both pre-flashover and post-flashover fires. Most fire deaths due to smoke inhalation in the U.S. occur in areas other than the room of fire origin and are caused by fires that have proceeded beyond the room of fire origin. It is assumed that these are flashover fires. Therefore, the principal emphasis is placed on evaluating toxic hazard under these conditions. In post-flashover fires, large concentrations of

² Annual Book of ASTM Standards, Vol 04.07.

³ Available from American National Standards Institute, 11 W. 42nd St., 43rd Floor, New York, NY 10036.

carbon monoxide results from reduced air supply to the fire plume and other room-scale factors. Bench-scale tests do not have the capacity to simulate these phenomena. The lethal toxic potency values determined in this test method are obtained from fuel/air ratios more representative of pre-flashover, rather than post-flashover conditions. In cases where a pre-flashover fire representation is desired in fire hazard modeling, these LC₅₀ values are appropriate. Lethal toxic potency and carbon monoxide yield values determined in this test method require adjustment for use in modeling of the hazard from post-flashover conditions. (See X1.4.1.)

5.4 The lethal toxic potency values determined in this test method have a level of uncertainty in their accuracy when used to predict real-scale toxic potencies. (See X1.4.2.)

5.4.1 The accuracy of the bench-scale data for pre-flashover fires has not been established experimentally. The combustion conditions in the apparatus are quite similar to real pre-flashover fires, although the mass burning rate may be higher at the 50 kW/m² irradiance of the test method.

5.4.2 Comparison of the toxicant yields and LC₅₀ (post-flashover) values obtained using this method have been shown in limited tests (22) to reproduce the LC₅₀ values from real-scale, post-flashover fires to within an accuracy of approximately a factor of three. Therefore, LC₅₀ (post-flashover) values differing by less than a factor of three are indistinguishable from each other. (See X1.4.2.)

5.5 This test method does not attempt to address the toxicological significance of changes in particulate and aerosol size, smoke transport, distribution, or deposition or changes in the concentration of any smoke constituent as a function of time as may occur in a real fire.

5.6 The propensity for smoke from any material to have the same effects on humans in fire situations can be inferred only to the extent that the rat is correlated with humans as a biological system. (See X1.2.5.)

5.7 This test method does not assess incapacitation. Incapacitation must be inferred from lethal toxic potency values.

5.8 The effects of sensory irritation are not addressed by this test method.

6. Apparatus

6.1 *Animal Exposure Chamber*—Shown in Figs. 1 and 2, a transparent polycarbonate or polymethylmethacrylate chamber with a nominal volume of 0.2 m³ (200 L). (See X1.2.6.) Its inside dimensions are 1220 by 370 by 450 mm (48 by 14½ by 17¾ in.). The six animal ports, intended for head-only exposure, are located in a horizontal row, approximately half way from the bottom to the top of the chamber, in the front wall. A plastic bag with an approximate volume of 0.05 m³ (50 L or approximately 13 gal) is attached to the port at the end of the chamber during a test to provide for gas expansion. The exposure box is equipped with a gas sampling port at the animal nose level in the geometric center of the exposure chamber and with a port for returning gases in the end wall closest to the gas analyzers. A thermocouple shall be provided to monitor the temperature at the level of the animal ports. There are two doors in the exposure chamber, in the front wall near the connection to the combustion cell and in the end wall nearest the animal ports. The purpose of the doors is to allow for cleaning and maintenance of the chamber, chimney, and smoke shutter and to provide fresh air during calibration of the heat lamps and immediately prior to testing.

6.2 *Smoke Shutter*, made of stainless steel plate and situated inside the animal exposure chamber. It is positioned so that it will close over the chimney opening. It is hinged and provided with a positive locking mechanism. The purpose of the shutter is to seal the combustion chamber and chimney from the exposure chamber at the end of irradiation. A wire attached to the shutter and a simple push rod are provided for gentle closing of the shutter. A wire attached to a clamp locks the shutter in place. To produce a gas-tight seal, the underside of the shutter is covered with a 12-mm (0.5-in.) thick blanket of low-density ceramic fiber insulation (approximately 65 kg/m³), which is further covered with stainless steel foil.

6.3 *Chimney* (Fig. 3)—A stainless steel assembly approximately 30 by 300 mm (1¼ by 11¾ in.), inside dimensions, and 300 mm (11¾ in.) wide. It connects the combustion cell to the animal exposure chamber. The chimney is divided into three channels by stainless steel dividers. The center channel is approximately 150 mm (6 in.) wide. The purpose of the dividers is to induce smoke

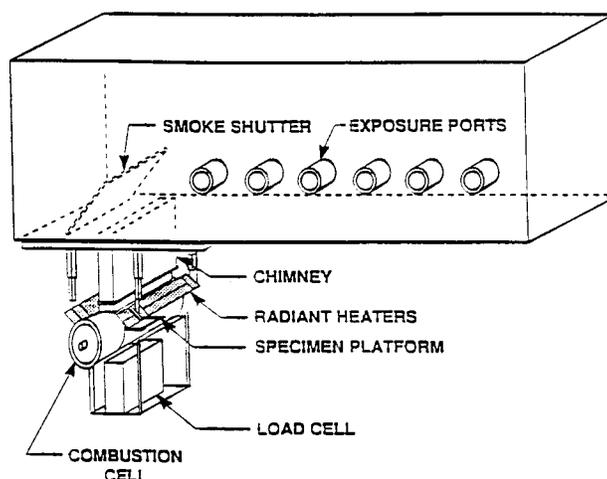
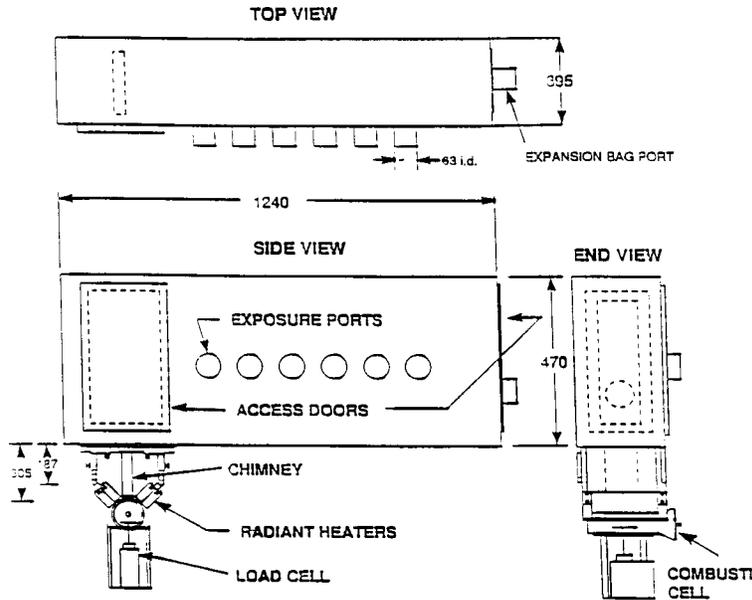


FIG. 1 Overall View of the Apparatus



DIMENSIONS SHOWN ARE IN MILLIMETERS
 FIG. 2 Schematic Drawing of the Apparatus

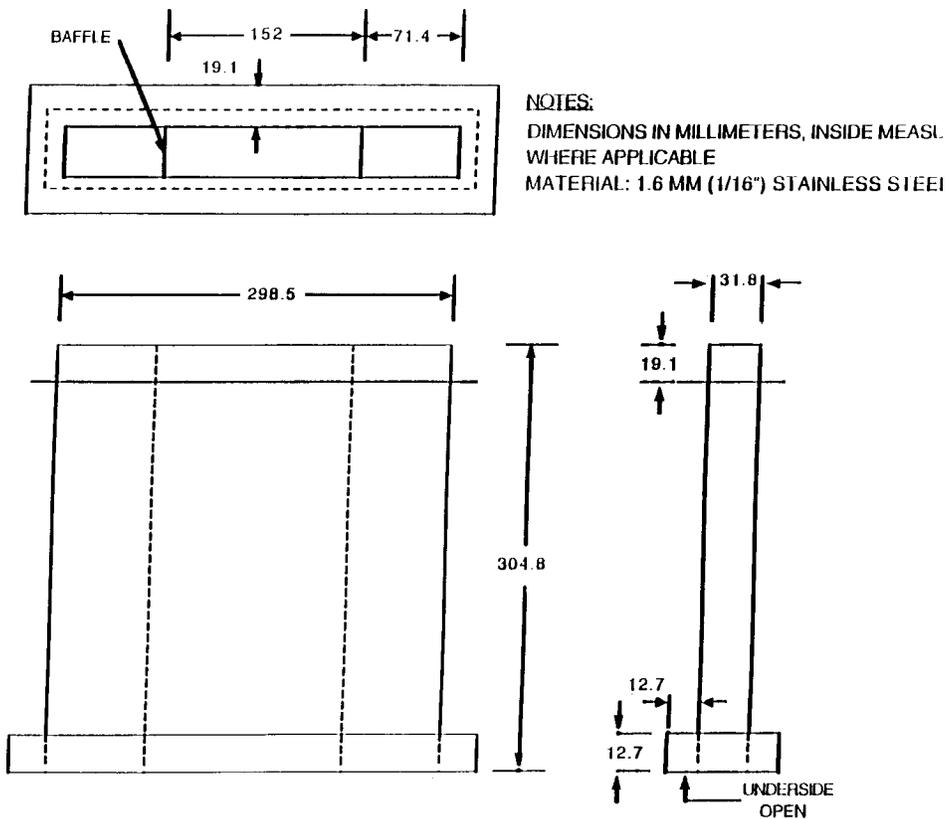


FIG. 3 Stainless Steel Chimney

to travel up through the center portion of the chimney, while air from the animal exposure chamber is drawn down through the outside channels to provide air to the combustion cell. The chimney is connected to the underside of the animal exposure chamber by clamps, permitting its removal for cleaning. It is sealed to the animal chamber by low-density ceramic fiber insulation (approximately 65 kg/m^3). The other end of the chimney is sealed to the combustion cell by an H-shaped trough with a small quantity of the same fiber insulation in the trough.

6.4 *Combustion Cell*— Shown in Figs. 4-6, a horizontal quartz tube with a 127-mm (5-in.) inside diameter and approximately 320 mm (12½ in.) long. It is sealed at one end and has a large standard taper outer joint at the other end. A sealed inner joint serves

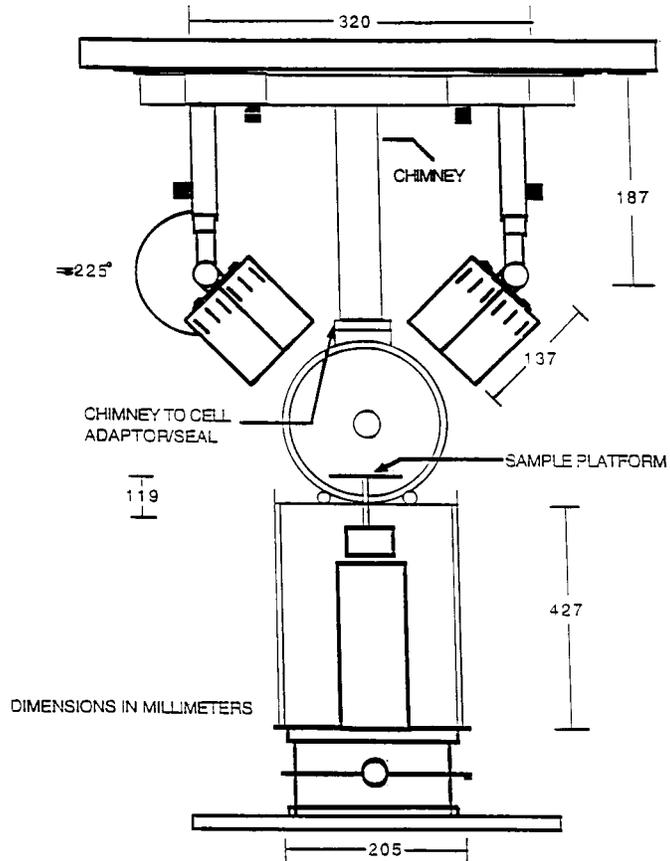


FIG. 4 Front View of the Combustion Zone

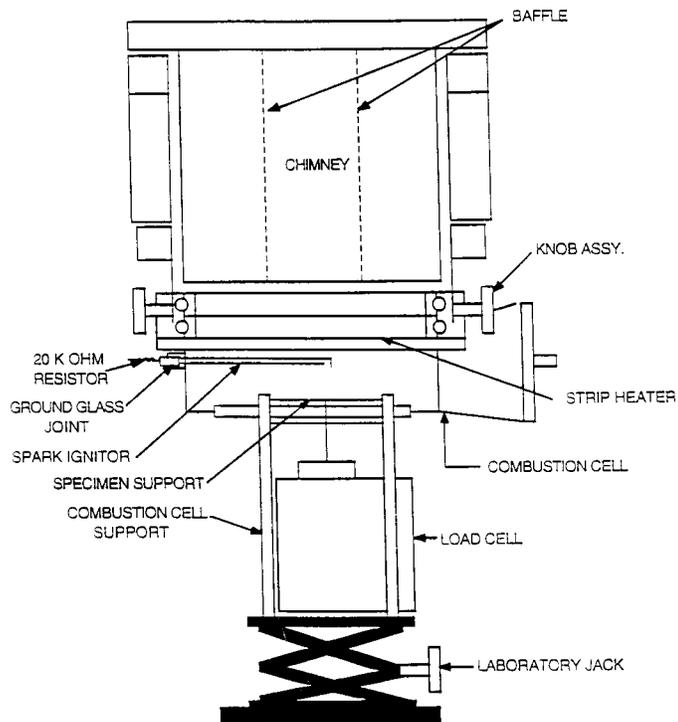


FIG. 5 Side View of the Combustion Zone

as a removable plug for the open end (Fig. 6). The combustion cell has a rectangular opening on the top parallel to the axis of the cylinder with a "collar" that allows it to fit securely into the chimney. The bottom of the cell has a hole for the rod connecting the

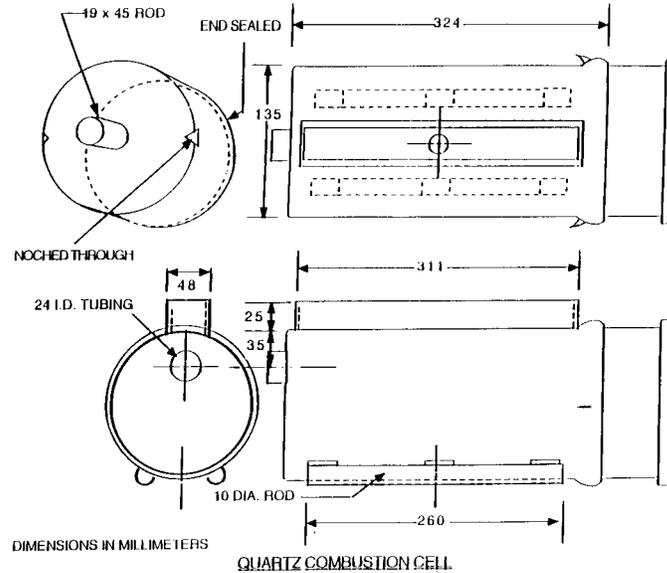


FIG. 6 Combustion Cell

specimen support platform and load cell. The sealed end of the combustion cell is fitted with a glass collar to accommodate the electric sparker.

6.4.1 The combustion cell is supported by a metal frame that also holds the load cell (Figs. 4 and 5). This entire frame is supported by a laboratory jack that holds the combustion cell tightly to the chimney during experimentation and allows the cell to be lowered for removal and cleaning. The load cell is always at a fixed distance from the combustion cell.

6.5 Radiant Heaters:

6.5.1 The active element of the heater consists of four quartz infrared lamps (with tungsten filaments), rated at 2000 W at 240 V. The lamps (two on each side) are encased in water-cooled holders with parabolic reflectors. These holders (Fig. 4) are attached to adjustable metal frames, which allow the lamps to be moved vertically and laterally and rotated in such a way as to provide a uniform flux field across the sample surface. Cooling water must be circulated through the lamps' respective holders to keep them from overheating. The lamps must not be operated without the cooling water.

6.5.2 The irradiance of the lamps must be held at a preset level. One method entails a temperature controller and two thermocouples (Type K) that are placed between the lamps and the combustion cell and wired in parallel.

6.5.3 The irradiance from the lamps shall be uniform within the central area of the specimen holder to within $\pm 10\%$. Fig. 7 shows the calibration holder to be used when determining the uniformity of the radiant field from the lamps. The lampholders must be repositioned, as necessary, if the field is found not to be adequately uniform.

6.6 Temperature Controller (Optional)—When a temperature controller is used for maintaining the required radiant flux, the quartz lamp output is controlled by a thermocouple signal to the temperature controller. The outputs from the two Type K

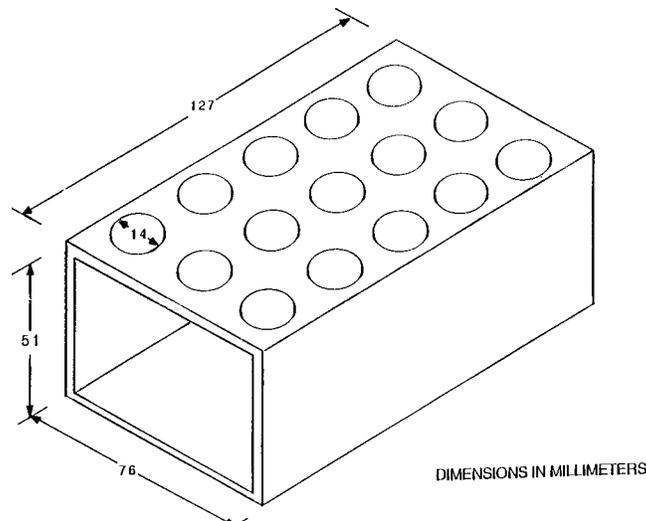


FIG. 7 Calibration Jig

thermocouples are averaged by means of a parallel-wired connection, and this averaged value is used as the input to the controller. The temperature controller must be a three-term type and must provide an output signal suitable for driving the power controller. The temperature controller must also incorporate a means for setting the maximum output to prevent the power controller from being driven wide-open, if needed. The power controller is selected to be compatible with the radiant heat lamps used.

6.7 Heat Flux Meter:

6.7.1 The total heat flux meter shall be of the Schmidt-Boelter (thermopile) type or equivalent, with a design range of at least 75 kW/m². The target receiving radiation shall be flat, circular, approximately 12.5 mm in diameter, and coated with durable matt-black finish. The target shall be water cooled. The flux meter shall have an accuracy of within ± 3 % and a repeatability within 0.5 %.

6.7.2 The calibration of the heat flux meter must be checked periodically. This is accomplished most readily by having two flux meters, one used for routine testing and another used only for calibration purposes.

6.7.3 The flux meter shall be used to calibrate the radiant heater temperature controller. It shall be positioned in a rigid support device to ensure repeatable readings. The surface of the heat flux meter must be located at a position equivalent to the center of the specimen face. Fig. 7 indicates a calibration bracket suitable for this purpose.

6.8 Ignitor—A spark ignitor is constructed of two 3.2-mm (0.125-in.) stainless steel rods. One of these two rods is bent at 90°, flattened on the end, and positioned to have the appearance of the tip of an automotive spark plug. The gap between the two rods shall be 2 ± 0.5 mm. The two rods are connected to the high-voltage spark system, which uses a 10-kV transformer (Fig. 8). A 20 000-Ω, 5-W resistor is connected in series with one of the electrodes to reduce the propagation of radio frequency interference into the instrumentation. The spark gap is positioned approximately 25 mm (1 in.) above the center of the top surface of the specimen, inside the combustion cell. In one operation method, the rods comprising the spark ignitor pass through a 29/42 male ground glass stopper, forming a gas-tight seal with a mating joint in place of the collar on the combustion cell (Fig. 6). Otherwise, the electrical leads shall be sealed in the glass collar in a gas-tight manner.

6.9 Specimen Holder—A stainless steel assembly approximately 76 by 127 mm (3 by 5 in.), inside dimensions, and 50 mm (2 in.) deep (Fig. 9). The specimen is backed by a layer of ceramic fiber blanket of nominal 65-kg-m⁻³ density. The specimen holder is positioned for testing on the specimen platform, inside the combustion cell.

6.10 Load Cell—The general arrangement of the load cell and specimen holder is illustrated in Fig. 5. The load cell is installed under the combustion cell and is insulated against heating from the lamps. The specimen and holder are located on a support plate and a rigid rod. The load cell shall have an accuracy of 0.01 g, and it shall have a measuring range of at least 100 g.

6.11 Gas Sampling:

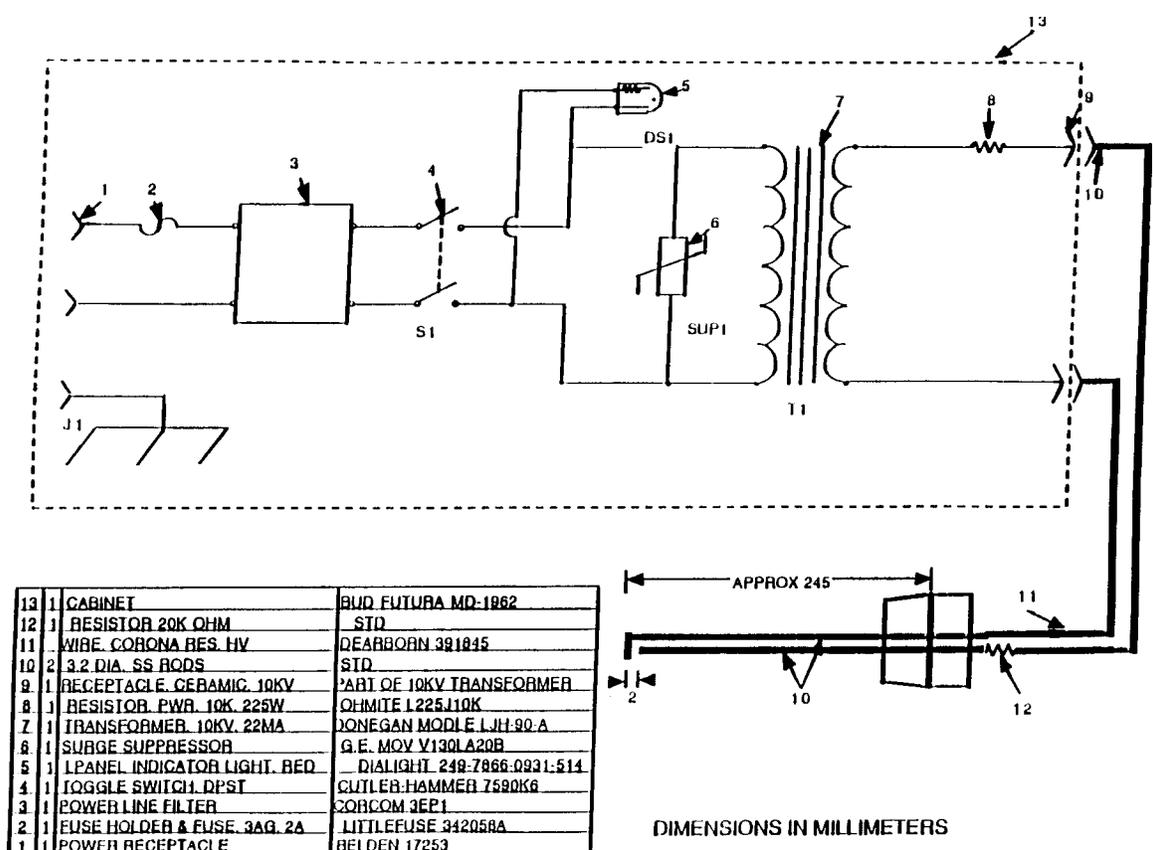
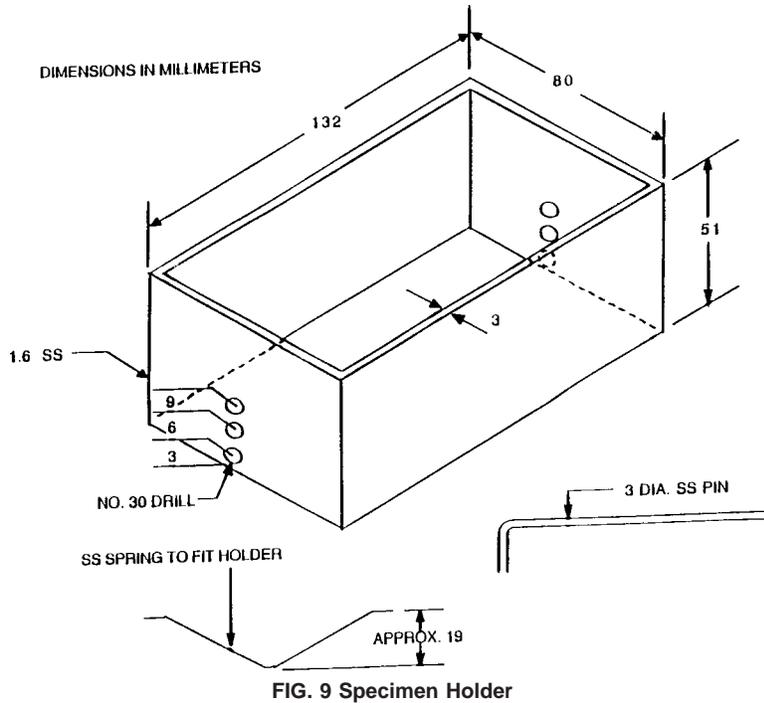


FIG. 8 Spark Igniter System



6.11.1 The gas sampling system shall be designed in accordance with the requirements specified in Guide E 800. Gases that are removed for chemical analysis and that can be recirculated to the animal exposure chamber are returned since this is a closed system. A suitable gas sampling arrangement includes a pump, glass wool filter at the sampling port, cold trap to remove soot and moisture, and pressure relief valve that returns all flow not required by the CO, CO₂, and O₂ gas analyzers. The flow to these analyzers is also returned to the animal exposure chamber through separate return lines. The return lines shall be closed during calibration of the instruments to prevent the accumulation of calibration gases in the animal exposure chamber.

6.11.2 Gas Analyzers:

- 6.11.2.1 The oxygen analyzer shall have a range from 0 to 21 %.
- 6.11.2.2 The carbon dioxide analyzer shall have a range from at least 0 to 10 %.
- 6.11.2.3 The carbon monoxide analyzer shall have a range from at least 0 to 10 000 ppm.

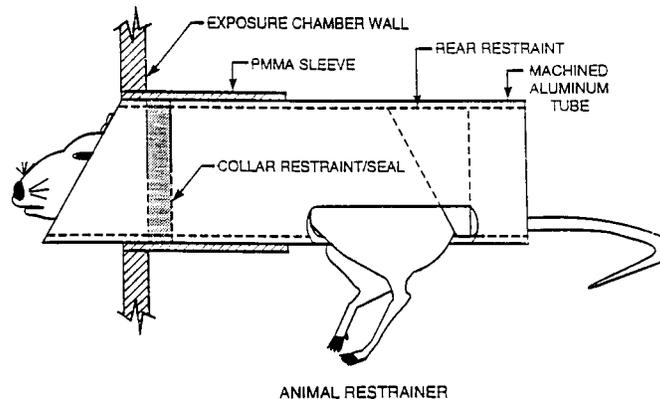
6.11.2.4 Additional gas analysis for HCN, HCl, or HBr shall be performed when the nature of the test specimen indicates the possibility of these gases being present in the combustion products. Analysis for these gases shall follow the instructions given in Guide E 800. For any gases with which analysis methods are used that involve chemical reaction, such products are not returned to the animal exposure chamber but, rather, disposed of in an environmentally correct manner.

6.12 Data Collection— The data collection system must have the capability of recording the output from the gas analyzers, thermocouple(s) in the chamber, and load cell and shall have an accuracy corresponding to 0.01 % of full-scale instrument output.

6.13 Animal Restraint—Animal restrainers made of aluminum and designed to permit head-only exposures shall be used. A detailed illustration of an animal restrainer meeting this requirement is shown in Fig. 10.

7. Hazards

7.1 This test method involves bright lights, high temperatures, and combustion processes. Hazards may, therefore, exist for eye



ANIMAL RESTRAINER
FIG. 10 Animal Restraint

injuries, burns, ignition of extraneous objects, and inhalation of combustion products. To prevent the accidental leakage of toxic combustion products into the surrounding atmosphere, the entire exposure system should be placed into a chemical hood or under a canopy hood. If under a canopy hood, an accessory exhaust trunk for any combination gases escaping through the load cell hole on the bottom of the combustion cell is required. An exhaust line to evacuate the exposure box at the end of a test is recommended. The operator must use safety tongs for removal of the specimen holder. While hot, the combustion cell must be touched only with protective gloves. Due to the intense light from the infrared lamps used, dark safety glasses must be worn by the operator, or a darkened polymethylmethacrylate or polycarbonate shield must be placed in front of the combustion cell.

7.2 The venting system for the exposure chamber must be checked for proper operation before testing and must discharge into an exhaust system with adequate capacity.

8. Test Specimens

8.1 Test specimens shall be cut to an appropriate area (see Section 12), no larger than 76 by 127 mm (3 by 5 in.) and no thicker than 50 mm (2 in.) (see X1.2.3), representing the end-use product. Raw materials (for example, paints, adhesives, wall coverings, etc.) shall be tested on the substrate to which they are normally applied. Wrap the specimens for testing on all sides except for the top face with either aluminum or stainless steel foil.

8.2 The test specimens shall be conditioned at an ambient temperature of $23 \pm 3^{\circ}\text{C}$ ($73 \pm 5^{\circ}\text{F}$) and relative humidity of $50 \pm 10\%$ for at least 24 h prior to testing.

9. Animals

9.1 The test animals shall be inbred 3 to 4 month old male rats obtained from a reputable supplier that certifies its animals to be free of major respiratory pathogens. Appropriate weight ranges at the time of use are 225 to 325 g for Fischer 344 rats and 300 to 375 g for Sprague-Dawley rats.

9.2 The maintenance and care of animals shall be performed by qualified personnel in accordance with guidelines of the National Institutes of Health Guide for the Care and Use of Laboratory Animals (1).⁴ The animal housing facilities shall be suitable for studies of this type.

9.3 The animals shall be identified, weighed, and housed upon receipt in a separate quarantine area for a minimum of seven days prior to testing. The animals shall be weighed and observed daily during the quarantine period. Animals that are unsuitable by reason of size, health, or other criteria are not to be used. Cage assignments shall be made according to a randomization routine.

9.4 The animals shall be housed one to a cage. The environment shall have proper ventilation and be controlled to a temperature of $23 \pm 3^{\circ}\text{C}$ ($73 \pm 5^{\circ}\text{F}$) and have a relative humidity of $50 \pm 15\%$. The animal room shall have a 12-h light/dark cycle.

9.5 Animals are to be weighed daily from the day of arrival to the end of the 14-day postexposure observation period. Normally, one rat in five are to be used as controls.

9.6 The animals shall be weighed prior to exposure and be secured in individual restrainers for placement in the animal exposure chamber.

9.7 After testing, surviving animals shall be housed in an animal room separate from the pretest animal room for the postexposure observation period.

10. Calibration of Apparatus

10.1 The following parts of the test apparatus require calibration: radiant heaters, gas analyzers, load cell, and temperature controller (if used).

10.2 Heat Flux Calibration:

10.2.1 For heat flux calibration, secure the heat flux meter into the proper position. The target surface of the flux meter must be centered at the location equivalent, both horizontally and vertically, to that of the top of the specimen when the specimen holder is in place on the platform. (The ignitor shall be removed from its position during this procedure.) If used, set the temperature controller to the desired flux temperature and turn on the radiant heat lamps, adjusting the temperature controller until the desired irradiance (that is, $50 \text{ kW/m}^2 \pm 10\%$) is achieved. If one is using manual control of the heat lamps, develop a calibration curve of heater controller setting as a function of time required to maintain the desired flux level.

10.2.2 Check the orientation of the radiant heat lamps whenever the heaters have been moved or a lamp replaced, using the following procedure. Install the heat flux calibration jig shown in Fig. 7. The top face of the calibration jig should be at the same height where the top of a test specimen is placed. Estimate a power setting for the lamps that will produce the desired level (for example, 50 kW/m^2) at the center hole. Adjust the power, and allow at least 5 min for equilibration. Adjust the orientation of the radiant heat lamps so that no measurement at seven locations across the face of the specimen deviates by more than 10% from the average.

10.3 Gas Analyzer Calibration:

10.3.1 At the beginning of each series of tests, the O_2 , CO_2 , and CO analyzers shall be calibrated by using nitrogen gas for “zeroing” and an appropriate gas mixture near to, but less than, the analyzer full-scale reading. The gas shall be set to flow at the

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

same rate and pressure as the sample gas for all calibrations. Ambient air (20.9 % O₂) shall be used for calibration of the O₂ analyzer, while bottled gases containing CO₂ and CO at known concentration are required for the CO₂ and CO analyzers. A single mixture containing both CO and CO₂ may be used. The gas return lines must be diverted during the calibration procedure into an exhaust duct in order to prevent inadvertent accumulation of CO and CO₂ in the exposure chamber.

10.3.2 The calibration of devices used for analysis of other gases (for example, HCN, HCl, and HBr) shall be performed in accordance with Guide E 800.

10.4 *Load Cell Calibration:*

10.4.1 The load cell shall be calibrated with standard weights in the range of the test specimens periodically and when first setting up the apparatus or after making adjustments for sensitivity and range.

10.4.2 The load cell is checked routinely before each test with at least two analytical quality weights over the effective range of measurement. Any deviation of the load cell output, as compared to these weights, shall be recorded, and appropriate compensation shall be made for the specimen mass loss readings.

10.5 *Calibration of Optional Temperature Controller*—To set up the controller, first install the flux meter so that the sensing surface is at the exact center of where the top of the specimen is placed in normal testing. Lamp adjustment to obtain a uniform flux field over the sample shall have been completed previously. Next, connect the output of the heat flux meter to a strip chart recorder running at a trace speed sufficiently fast to detect any changes in the flux. Using the output from the heat flux meter, follow the instructions of the controller manufacturer for adjusting the controller in order to obtain, as closely as possible, a square wave output from the heat flux meter when the lamps are turned on and then turned off. Because the lamps respond quickly, while the temperature at the thermocouples rises more slowly, it is important to avoid a significant overshoot, which can occur if the controller is not tuned optimally. It is also important to avoid using settings that result in an unstable, oscillating output. If such a problem is noted, the solution is to limit the maximum output from the slave controller. This can be accomplished by either using the “load line out” function of the temperature controller, if so equipped, or by installing a voltage divider at the output of the temperature controller. When setting up the controller with this function, it is always necessary to have the thermocouples reading room air temperature and not some elevated temperature. By correct adjustment of the temperature controller, 90 % of the desired flux can be reached within 2 s, with 100 % being reached within 20 s and a deviation of within ± 5 % for the rest of the test.

11. Procedure

11.1 *General*—Test procedures for smoke toxicity data are initially to be followed without the exposure of test animals in order to produce analytical data for CO, O₂, CO₂ and, if present, HCN, HCl, and HBr. The choice of specimen size for the initial tests is made with consideration of anticipated toxicant yields such that FEDs from 0.5 to 1.5 are obtained (see Section 13). In the absence of appropriate information for such choices, an area equal to one fourth of the maximum area of 96.5 cm² is selected initially. Analytical data from at least two initial tests are used for the prediction of an average LC₅₀ for the test specimen (see Section 12).

11.1.1 Comparable tests are then conducted, but with the exposure of six rats to the smoke produced from that amount of the test material whose mass loss concentration during the 30-min period is approximately (± 10 %) equivalent to 70 and 130 % of its average predicted LC₅₀. The predicted LC₅₀ is considered to be confirmed if no more than one rat dies during the 30-min exposure, or within 14 days postexposure, to the mass loss concentration corresponding to 70 % of the LC₅₀ and at least five rats die during the 30-min exposure, or within 14-days postexposure to the mass loss concentration corresponding to 130 % of the LC₅₀. If the confirmation is not successful, or if unexplained or unusual toxicity is suspected, other test methodology must be used to investigate the lethal toxic potency of the test material. (See X1.3.1 and X1.3.2.)

11.2 *Preparation for Tests:*

11.2.1 Turn on the coolant water for the heat flux meter (at least 750 mL/min) and for the tungsten lamps (at least 600 mL/min).

11.2.2 Verify that all lines, filters, and traps for the gas analyzers have been serviced and that the flow rates are satisfactory.

11.2.2.1 Check the moisture trap in the gas analyzer stream. Dry the trap and replace the glass wool. The normal operating temperature of the moisture trap is 0°C.

11.2.2.2 Place a glass-wool filter before the gas sampling port.

11.2.3 Verify that the spark ignition circuit is operational.

11.2.4 Perform the required calibration procedures specified in Section 8.

11.2.5 Weigh the specimen on a laboratory balance capable of ± 0.01 g. Wrap the specimen in either aluminum or stainless steel foil, leaving the top surface exposed, and determine the combined weight of the specimen with the foil and after mounting in the specimen holder. Verify that the load cell readout corresponds to the appropriate weight of the specimen plus holder.

11.3 *Test Procedure:*

11.3.1 If the animals are to be exposed, they shall be weighed and placed in their restrainers.

11.3.2 Insert the specimen, mounted in the specimen holder, into the combustion cell, and replace the standard taper plug (use no grease or sealant on the ground glass). Secure the plug with wire or springs. Place the animals into the ports in the exposure chamber immediately prior to the beginning of an animal exposure test. Close all exposure chamber doors and ports, if not used for animals. Ascertain that the smoke shutter is open.

11.3.3 Turn on the sparker. Activate the power to the radiant heat lamps simultaneous with the start of data collection.

11.3.4 Record the time at which ignition of the specimen occurs and turn off the sparker. Record the time of flameout. For

specimens that have a tendency to self-extinguish soon after ignition, the sparker shall be left on until flaming ceases.

11.3.5 Switch off the power to the radiant heat lamps and close the smoke shutter at the end of 15 min.

11.3.6 Collect data for a total of 30 min from initiation of the test.

11.3.7 Cease collecting data at the end of 30 min. If animals were exposed, they are to be removed from the exposure chamber. Vent the exposure chamber with a high capacity exhaust system.

11.3.8 Blood samples should be taken from any dead animals and analyzed for carboxyhemoglobin saturation in tests using the exposure of animals. Blood sampling and analyses are to be conducted in accordance with generally accepted methodologies.

11.3.9 In tests using the exposure of animals, those surviving shall be checked daily for any signs of toxic effects (for example, difficulty in breathing and convulsions), exploratory behavior, and eye and righting reflexes. The status and weights (at 7 and 14 days) of the animals shall be followed for a 14-day postexposure period. Any deaths during this time period shall be recorded.

11.3.10 Remove the sample holder from the combustion chamber, and cool it to ambient temperature in an exhaust hood. Disassemble the specimen holder and determine the weight of the stainless steel foil and residue after the specimen has cooled.

11.3.11 Remove and clean the combustion chamber and chimney after each test. Clean the exposure chamber after each test. Ethyl alcohol is a satisfactory solvent. There must be no residue on the inside of any of the pieces of the apparatus.

12. Calculation

12.1 *General*—The lethal toxic potency (LC_{50}) of the test specimen is predicted from the combustion atmosphere analytical data for CO, CO₂, O₂, and, if present, HCN, HCl, and HBr. (See X1.2.7.) This is accomplished for a given specimen mass loss by first calculating the FED for the test. The LC_{50} is then calculated as that specimen mass loss which would yield a FED = 1 within a chamber volume of 1 m³.

NOTE 1—Although the theoretical value of the FED associated with 50 % lethality is 1.0, a median value of 1.1 has been found experimentally (2).

12.2 The 30-min FED for a given specimen mass loss is calculated from Eq 1:

$$\begin{aligned}
 FED &= \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{21 - LC_{50}O_2} + \frac{[HCN]}{LC_{50}HCN} \\
 &+ \frac{[HCl]}{LC_{50}HCl} + \frac{[HBr]}{LC_{50}HBr} \\
 &= \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{(21 - 5.4)\%} \\
 &+ \frac{[HCN]}{150 \text{ ppm}} + \frac{[HCl]}{3700 \text{ ppm}} + \frac{[HBr]}{3000 \text{ ppm}} \quad (1)
 \end{aligned}$$

$$\begin{aligned}
 FED &= \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{21 - LC_{50}O_2} + \frac{[HCN]}{LC_{50}HCN} \\
 &+ \frac{[HCl]}{LC_{50}HCl} + \frac{[HBr]}{LC_{50}HBr} \\
 &= \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{(21 - 5.4)\%} \\
 &+ \frac{[HCN]}{150 \text{ ppm}} + \frac{[HCl]}{3700 \text{ ppm}} + \frac{[HBr]}{3000 \text{ ppm}} \quad (1)
 \end{aligned}$$

where the values of all gas concentrations are the integrated Ct product values under their respective concentration-time curves taken over the 30-min test period divided by 30. All of the values are in parts per million except O₂, which is in %. The values of *m* and *b* depend on the concentration of CO₂. If [CO₂] ≤ 5 %, *m* = -18 and *b* = 122 000. If [CO₂] > 5 %, *m* = 23 and *b* = -38 600. For each individual toxicant, the LC_{50} values shown were determined statistically from independent experimental data to produce lethality in 50 % of the test animals (rats) within a 30-min exposure plus 14-days postexposure.

12.3 The 30-min LC_{50} for a test specimen is calculated from Eq 2:

$$LC_{50} = \frac{\text{specimen mass loss}}{FED \times \text{chamber volume}} \quad (2)$$

where the specimen mass loss is in g and the chamber volume is 0.2 m³. The resulting LC_{50} has the units of g·m⁻³.

12.4 The yields of gases produced (mass of gas per mass of fuel consumed) at 25°C are calculated from Eq 3:

$$Y_x = \frac{[X] \cdot 8.2 \cdot MW_x}{10^6 [m_{100}]} \quad (3)$$

$$Y_x = \frac{[X] \cdot 8.2 \cdot MW_x}{10^6 [m_{100}]} \quad (3)$$

where [X] is the concentration of gas X in ppm, MW_x is its gram molecular weight, and m₁₀₀ is the mass of sample consumed, also in grams.

12.5 The lethal toxic potency value for hazard analysis of post-flashover fires is calculated from Eq 4:

$$LC_{50}(\text{post-flashover}) = \frac{1}{\frac{1}{LC_{50}} + 44 \times 10^{-3} - 5.0 \times 10^{-5} \frac{[CO]}{m_{100}}} \quad (4)$$

where: $LC_{50}(\text{g}\cdot\text{m}^{-3})$ is the value determined from Eq 2, m_{100} is the mass (g) of specimen lost during the test at the FED=1 condition, and [CO] (ppm) is the concentration of CO at the FED-1 condition. This equation is based on a post-flashover CO yield of 0.2 g/g of fuel burned. (See Appendix X1.4.1.)

12.6 The post-flashover CO yield for the specific product under evaluation may also be obtained from an appropriate full-scale test. A value of LC_{50} (post-flashover) is then determined by substituting $22 \times 10^{-2} Y_{CO}$ for 44×10^{-3} in Eq 4, resulting in Eq 5:

$$LC_{50}(\text{post-flashover}) = \frac{1}{\frac{1}{LC_{50}} + 0.22Y_{CO} - 5.0 \times 10^{-5} \frac{[CO]}{m_{100}}} \quad (5)$$

$$LC_{50}(\text{post-flashover}) = \frac{1}{\frac{1}{LC_{50}} + 0.22Y_{CO} - 5.0 \times 10^{-5} \frac{[CO]}{m_{100}}} \quad (5)$$

13. Report

13.1 Report the following information:

13.1.1 Laboratory.

13.1.2 Test identification and date.

13.1.3 Laboratory ambient conditions (temperature and humidity).

13.1.4 Description of specimen.

13.1.5 Specimen dimensions.

13.1.6 Irradiation time and heat flux conditions.

13.1.7 Maximum exposure chamber temperature and time when attained (see X1.2.6).

13.1.8 Initial specimen mass and mass loss during the test in $\text{g}\cdot\text{m}^{-3}$ of chamber volume (see X1.3.3).

13.1.9 Time to ignition and flame out.

13.1.10 *Observations of Specimen*—Required observations are times to smoke evolution, ignition, and flame out. Other observations would include melting, char formation, spalling, unusually vigorous burning, and re-ignition.

13.1.11 *Gas Analysis Data*—Required exposure chamber data include integrated Ct product values over the 30-min test for CO, O₂, HCN, HCl, and HBr; minimum O₂ concentration and maximum CO₂ concentration; and times to reach minimum O₂ and maximum CO₂. The methods used for analyses are to be identified.

13.1.12 *Calculation*:

13.1.12.1 Ct product for each analyzed toxicant in each test,

13.1.12.2 Yield of each analyzed toxicant in each test.

13.1.12.3 FED value for each test,

13.1.12.4 Predicted LC_{50} value for pre-flashover use, for each test, and

13.1.12.5 *Best Overall Predicted LC_{50} Value* to one significant figure—A least squares regression analysis of FED versus mass loss values for all tests is used to determine the best overall predicted LC_{50} value.

13.1.12.6 Calculated value of LC_{50} (post-flashover) to one significant figure.

13.1.13 Optional plots are those of individual toxicant concentrations, specimen mass loss, and temperature as functions of time.

13.2 Include the following information in the report for each test using the exposure of animals:

13.2.1 Strain of rat and identity of supplier.

13.2.2 Weight of each animal when received, prior to test, and at 7 and 14-days postexposure for surviving animals.

13.2.3 Number of animals dying during the test (including up to 10-min posttest) and number of animals that die up to 14 days posttest.

13.2.4 Blood carboxyhemoglobin saturation values for animals dying during the test.

13.2.5 Animal observations, for example, unusual behavior during the test; immediate posttest observations of live animals such as tremors, convulsions, difficulty in breathing, severe eye irritation, etc.

13.3 State in the report whether the animal tests did or did not confirm the value of the LC_{50} obtained from Eq 2.

14. Precision and Bias

14.1 *Precision*—The precision of this test method has not yet been established. A precision statement will be prepared and included in the test method after the completion of an interlaboratory test series.

14.2 *Bias*—The bias of this test method has not been measured since there is no accepted reference material for use in making such measurements.

15. Keywords

15.1 combustion toxicity; fire-hazard analysis; fire tests; fire toxicity

APPENDIX

(Nonmandatory Information)

X1. COMMENTARY

X1.1 *Introduction*—This commentary is provided to give insight into the development of this test method, to describe the rationale for the unique features of this test method, and to describe the proper use of the data provided. NIST Special Publication 827 is recommended (2) for a more comprehensive treatment, along with the presentation of data and results obtained on typical materials.

X1.2 *Development of the Test Method*—A test method for assessing the acute inhalation toxicity of combustion products has three basic components: a combustion system, a chemical analysis system, and an animal exposure system (3). Additionally, there must be a rational and accepted strategy for the incorporation of raw experimental data into a quantified expression for toxic potency.

X1.2.1 This test method uses as the combustion system that developed at Southwest Research Institute (SwRI) for the National Institute of Building Sciences (NIBS) (4). Representing a significant improvement over an earlier radiant heat device first used at the Weyerhaeuser Company (3), the combustion system was adopted jointly by the National Institute of Standards and Technology (NIST) and SwRI for the development of this test method (5). Its main features is that of providing for combustion of a test specimen under the realistic conditions of radiant heat within an apparatus designed especially for ruggedness and ease of operation.

X1.2.2 For a small, developing fire, the bench scale specimen in the radiant apparatus is a reasonable representation of the full-scale fire. The thermal boundary conditions are appropriate, being radiative and from one face only. A small fire will impose approximately 35 kW/m² on an adjacent unburned surface (6,7), although values around 48 kW/m² are common and values over 100 kW/m² can be measured. Thus, while an irradiance of 50 kW/m² for a pre-flashover test may be somewhat high, it is by no means out of line. If a specific scenario involves a heating flux other than 50 kW/m², it can be accommodated readily in this test method.

X1.2.3 In the real scale fire, the combustion products will generally contain contributions from portions of the burning product that are burning near the front surface, those that are burned partway through, and those coming from nearly burned out portions. Thus, a bench scale test should represent this mixture of gases as closely as possible. The physical constraints of the test method are a maximum specimen thickness of 51 mm and a radiant heating time of 15 min. The actual thickness of non-layered products should be selected so that thermal decomposition is complete when 15 min has elapsed and the shutter is closed. This can be determined by examination of the generation of CO, which should have ceased before the shutter is closed. A preliminary trial run where neither the animals nor the gas analyzers need to be used should suffice. Homogeneous products can then be prepared for actual testing by cutting to the appropriate thickness dimension. Layered composites may also generally be reduced in thickness, as required. However, separate tests are indicated, with each face being exposed in separate testing, if the two face materials are not identical.

X1.2.4 The chemical analysis system used in the test method employs methodology commonly accepted by those skilled in such procedures and as presented in Guide E 800 and in ISO/TR 9122, Part 3.

X1.2.5 Within the scope and significance of this test method, the use of rats as an acceptable model for human exposure has been well documented in ISO/TR 9122, Part 5.

X1.2.6 The animal exposure system is that used in the NBS Cup Furnace Method (8). It has been used widely in a number of laboratories and found to be highly satisfactory. An important consideration when conducting animal exposures is that the biological effects on the animals' condition during a test should be affected adversely as little as possible by causes other than specimen toxicity. This includes the following: providing a sufficient size of animal exposure chamber so that the animals' exhaled CO₂ does not affect them adversely; making sure that heating conditions from specimen heaters do not create an excessive heat burden to the animals; and providing a restraint system that does not cause undue physical stress. One should still exercise judgment regarding exposure chamber temperature excursions above 40°C. Such excursions have not been regarded as detrimental to the resulting test data for the most part. However, there could be exceptions for extreme cases.

X1.2.7 The strategy used in this test method for the quantification of smoke toxic potency represents use of the latest in state-of-the-art understanding of the prediction of the toxic effects of fire effluents as reported in ISO/TR 9122, Part 5. It uses methodology for the calculation of toxic potencies from combustion product analytical data without the exposure of experimental animals. The basis for such methodology comes from extensive experimentation using the exposure of rats to the common fire gases, both singly and in combinations, which showed the additivity of FEDs of the individual toxicants (9-19). Expressed mathematically, the principle is shown in Eq X1.1:

$$FED = \sum_{i=1}^n \int_0^t \frac{C_i}{(Ct)_i} dt \tag{X1.1}$$

where:

C_i = concentration of the toxic component i, and

$(Ct)_i$ = specific exposure dose required to produce the toxicological effect (20) .

When the FED = 1, it is expected that the mixture of gaseous toxicants would be lethal to 50 % of exposed animals. Use of the principle in the form given in this test method has been termed the “N-Gas Model” by NIST. The N-Gas Model also takes into account the effect of CO₂ on the toxicity of CO, as expressed empirically from studies conducted at NIST (9). Examination of a series of pure gaseous toxicant experiments in which various percentages of animals died indicated that the mean FED value using the “N-Gas” calculation was 1.07, with 95 % confidence limits of ±0.20 (12).

X1.3 Limitations on Materials and Products:

X1.3.1 All products can be tested using the calculation method described. However, there will be products whose smoke lethal toxic potencies cannot be ascribed to the common toxic gases analyzed. These products may need to be tested according to other, more conventional, animal exposure methodology or, preferably, be subject to further research in order to explain why the lethal toxic potency cannot be estimated from the concentrations of the common toxic gases. Simply requiring the experimental determination of an LC₅₀ value could circumvent the more important aspect of being able to explain the cause of the animal deaths.

X1.3.2 This test method may not be appropriate for testing materials or products containing highly fluorinated compounds and has not been validated for such. Hydrogen fluoride (HF) or other fluorinated compounds are not included in the N-Gas Model at the present time. The predictivity of the model would therefore fail to account for the toxicity of the fluoride content of the material or product. In addition, the thermal decomposition products from such fluorinated materials are well known for their reaction with the silica-based components (for example, glassware) of analytical instruments. Thus, the smoke can potentially damage the analytical instrumentation. There are indications that a number of highly specialized combustion and toxicological phenomena can arise for certain highly fluorinated polymers that make difficult the creation of proper bench scale conditions to represent real scale toxicity (21).

X1.3.3 Certain ash-producing materials (for example, silicones) require the use of appropriate procedures for measuring the quantity of sample burned since the load cell readings are compromised by the deposition of ash resulting from burning of the material.

X1.4 *Test Method Data*—This test method has been designed to provide data for the mathematical modeling of fire hazard as a means for the evaluation of materials and products and to assist in their research and development.

X1.4.1 Studies at NIST (5) have demonstrated that a bench scale toxic potency test can represent many aspects of a post-flashover fire adequately. One exception is the generation of carbon monoxide, which may be governed more by the available air supply in the actual full-scale fire in some cases than by the nature of the material burned. This cannot be simulated in a practical bench scale test method. A search of the published literature has shown that a range of real-scale fire tests, involving a diversity of fuels burning both as walls and free-standing combustibles, produce CO yields with relatively little dependence on the type of fuel(s) burned, as shown in Table X1.1. These CO yield data are well-represented as 0.24 ± 0.09 g CO/g fuel consumed in that 6 of the 8 values fall within ± one standard deviation of the mean. [A summary that also includes a number of dissimilar tests obtains the same mean value. (27)] Due to the limited reproducibility of real-scale fire tests in general and the scatter in the data from each of the cited tests, this mean yield is best used to 1 significant figure, i.e., for example, 0.2 g CO/g fuel consumed. While this is the best representation given current knowledge, research to improve this knowledge is in progress.

X1.4.2 Data from this method have been compared directly to real-scale, post-flashover fires of the same materials: Douglas fir, a formulation of rigid polyurethane foam, and a formulation of PVC (22). These materials challenge the method with a diverse

TABLE X1.1 Yields of Carbon Monoxide from Real-Scale Fire Tests

Fuel	CO Yield	Reference
Plywood walls	0.10 ^A	(23)
Upholstered chair, bed, FR plywood walls	0.36 ^A	(24)
Plywood walls, bed	0.3 ^A	(24)
Chair, TV and business machine cabinets, circuit boards, cables (Test N1)	0.22	(25)
Wood cribs	0.15 ^B	(26)
Flexible PU foam	0.25 ^B	(26)
PMMA	0.3 ^B	(26)
Hexane	0.23 ^B	(26)

^ANIST calculation from CO/CO₂ ratios and estimated burning rates in references 23 and 24.

^BNIST extraction of average yield data in reference 26.

set of test cases: a natural cellulosic and man-made plastics, solid and foam plastics, a material where CO (enhanced by CO₂ and low O₂) is the only toxicant and materials that produce significant amounts of HCl or HCN. The results showed agreement of the post-flashover LC₅₀ data and the bench-scale data to within a factor of 3.

X1.4.3 It is possible to demonstrate the regime of LC₅₀ (post-flashover) values where performance differentiation is not appropriate. The LC₅₀ of CO₂-potentiated CO is about 5 g·m⁻³, and the yield of CO is approximately 0.2 g/g of fuel burned (X1.4.1). Therefore, the LC₅₀ of post-flashover smoke is approximately 25 g·m⁻³. The accuracy of this method for LC₅₀ (post-flashover) values is about a factor of 3 (22). Thus, it is indicated that LC₅₀ (post-flashover) values between 8 g·m⁻³ (i.e., ⁻³ (for example, 25/3) and 25 g·m⁻³ are indistinguishable from each other using this test method and the current state of its accuracy. LC₅₀ values above approximately 25 g·m⁻³ are not possible for post-flashover smoke, since the high CO yield is characteristic of these fires. Thus, it is indicative of the state of the art that LC₅₀ (post-flashover) values greater than 8 g·m⁻³ are indistinguishable from each other using this test method. Most common building and furnishing materials have LC₅₀ values substantially higher than 8 g·m⁻³. Thus, the lethal toxic potency of the smoke will most often be determined by the fire ventilation. For post-flashover fires, this method will identify products that generate smoke of extreme toxic potency. Because of the accuracy limits of the method, such products may be viewed within LC₅₀ (post-flashover) ranges of a factor of 3, e.g., for example, between 8 g·m⁻³ and 3 g·m⁻³, between 3 g·m⁻³ and 1 g·m⁻³, etc. and so forth.

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