NFPA 269

Standard Test Method for Developing Toxic Potency Data for Use in Fire Hazard Modeling

1996 Edition

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NFPA 269

Standard Test Method for

Developing Toxic Potency Data for Use in
Fire Hazard Modeling

1996 Edition

This edition of NFPA 269, *Standard Test Method for Developing Toxic Potency Data for Use in Fire Hazard Modeling*, was prepared by the Technical Committee on Fire Tests and acted on by the National Fire Protection Association, Inc., at its Fall Meeting held November 13-15, 1995, in Chicago, IL. It was issued by the Standards Council on January 12, 1996, with an effective date of February 2, 1996, and supersedes all previous editions.

This edition of NFPA 269 was approved as an American National Standard on February 2, 1996.

Origin and Development of NFPA 269

This is the first edition of NFPA 269. It was desirable to establish a standard test method for the development of data for use in toxic hazard modeling. The basis of this standard is derived from much work completed by the National Institute of Standards and Technology.

Technical Committee on Fire Tests

Jesse J. Beitel, *Chair*
Hughes Assoc. Inc., MD

This list represents the membership at the time the Committee was balloted on the text of this edition. Since that time, changes in membership may have occurred. A key to classifications is found at the back of this document.

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Committee Scope: This Committee shall have primary responsibility for documents on fire testing procedures when such standards are not available; for reviewing existing fire test standards and recommending appropriate action to NFPA; for recommending the application of and advising on the interpretation of acceptable test standards for fire problems of concern to NFPA technical committees and members; and for acting in a liaison capacity between NFPA and the committees of other organizations writing fire test standards. This committee does not cover fire tests that are used to evaluate extinguishing agents, devices, or systems.

Contents

NFPA 269

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NOTICE: An asterisk (*) following the number or letter designating a paragraph indicates that explanatory material on the paragraph can be found in Appendix A.

Information on referenced publications can be found in Footnotes to Appendix B and Appendix C.

Chapter 1 Introduction

1-1 General. The pyrolysis or combustion of every combustible material or product produces smoke that is toxic. A standard test method for the development of data for use in toxic hazard modeling is valuable. Such data include quantification of the toxicity of the smoke. It also is desirable to ascertain whether or not the observed toxicity can be attributed to the major common toxicants.

Chapter 2 General

2-1 Scope.

2-1.1 This method is intended to provide a means for assessing the lethal toxic potency of combustion products produced from a material or product ignited when exposed to a radiant flux.

2-1.2 This method has been designed to generate toxic potency data on materials and products (including composites) for use in fire hazard analysis. It also can be used to assist in the research and development of materials and products.

2-1.3 Lethal toxic potency values associated with 30-minute exposures are predicted using calculations that employ 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.

2-1.4 Specimens shall be exposed to a radiant heating flux with an electric spark ignition.

2-1.5 Specimens tested shall be representative of finished products, including composite and combination systems.

2-1.6 This standard is not intended to address all safety issues associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations (especially with regard to the institutional care and use of experimental test animals) prior to use. For specific hazard requirements, see Section 7-1.

Chapter 3 Purpose

3-1 Significance and Use.

3-1.1 This test method is intended to provide data for the mathematical modeling of fire hazard as a means for the evaluation of materials and products.

3-1.2 This test method shall be used to predict, *and subsequently confirm,* the lethal toxic potency of smoke produced upon exposure of a material or product to specific fire test conditions. Confirmation determines whether certain major gaseous toxicants account for the observed toxic effects as well as the lethal toxic potency. If a predicted lethal toxic potency value is not adequately confirmed, indicating the potential for unusual or unexplained toxicity due to other components, the lethal toxic potency shall be investigated using other methodology, such as conducting an experimental determination of the LC_{50} using the apparatus described. (*See B-3.1 and B-3.2.*)

3-1.3 This test method produces lethal toxic potency data that shall be considered appropriate for use in modeling the hazard 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 fires typically are flashover fires. Therefore, the principal emphasis is placed on evaluating toxic hazard under these conditions. In post-flashover fires, large concentrations of carbon monoxide result from a reduced air supply to the fire plume. Bench-scale tests do not have the capability to simulate this phenomenon. Therefore, the lethal toxic potencies determined in this test method shall require adjustment for use in modeling the hazard from post-flashover conditions (*see Section 12-2.3*). For pre-flashover conditions, the LC_{50} values derived from this method shall be permitted to be used as toxic potency data without adjustment.

3-1.4 Lethal toxic potency values determined in this test method exhibit a degree of uncertainty where used to predict real-scale toxic potencies. (*See B-4.2.*)

3-1.5 Tests shall be conducted on small-size specimens that are representative of the materials, products, or composites in their intended end-use.

3-1.6 This test method does not attempt to address the toxicological significance of changes in particulate/aerosol size, smoke transport, distribution, or deposition or changes in the concentration of any smoke constituent as a function of time that occurs in an actual fire.

3-1.7 The propensity for smoke from any material tested for its effects on a rat to have the same effects on a human in fire situations shall be inferred only to the extent that a rat can be correlated with a human biologically. (*See B-2.5.*)

3-1.8 This test method shall not be used to assess incapacitation. Incapacitation shall be inferred from lethal toxic potency values.

3-1.9 The effects of sensory irritation are not addressed by this test method.

Chapter 4 Definitions

4-1 Definitions of Terms Specific to this Measurement Method.

Carboxyhemoglobin Saturation. The percent of blood hemoglobin converted to carboxyhemoglobin due to reaction with inhaled carbon monoxide.

Concentration–Time Curve. A plot of the concentration of a gaseous toxicant (ppm) as a function of time.

Ct Product. The concentration-time product (in ppm•min) obtained by the integration of the area under a concentration-time curve.

Fractional Effective Dose (FED). The ratio of the Ct product for a gaseous toxicant produced in a given test to that Ct product of the toxicant that has been statistically determined from independent experimental data to produce lethality in 50 percent of test animals within a specified exposure and post-exposure time. Since time values in this ratio numerically cancel, the FED is also the ratio of the average concentration of a gaseous toxicant to its LC_{50} value for the same exposure time. Where not used with reference to a specific toxicant, FED represents the sum of FEDs for individual toxicants in a combustion atmosphere.

LC_{50}. A measure of lethal toxic potency; the concentration of gas or smoke statistically calculated from concentration=response data to produce lethality in 50 percent of test animals within a specified exposure and post-exposure time. LC_{50} is measured in units of $g•m^{-3}$.

Mass Loss Concentration. The mass loss of a test specimen per unit exposure chamber volume ($g•m^{-3}$).

Post-flashover. The stage of a room fire during which average air temperature in the upper half of the room exceeds 600°C.

Shall. Indicates a mandatory requirement.

Should. Indicates a recommendation or that which is advised but not required.

Chapter 5 Test Method

5-1 Summary of Test Method. This method shall subject a 76-mm × 127-mm or smaller test specimen to ignition while exposed to 50 kW/m^2 of radiant heat for 15 minutes. The smoke produced shall be collected for 30 minutes within a 200-L chamber to which the combustion assembly is joined through a connecting chimney. Concentrations of the major gaseous toxicants shall be monitored over the 30-minute period, with Ct products for each determined from the 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, then shall be used in calculations to predict the 30-minute LC_{50} of the test specimen. The predicted LC_{50} then shall be confirmed in comparable tests by exposing six rats, restrained for head-only exposure, for 30 minutes to the smoke produced from that mass of the test specimen whose mass loss concentration during the 30-minute period is approximately equivalent to 70 percent ± 10 percent and equivalent to 130 percent of its estimated LC_{50}. If no more than one rat dies during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to 70 percent of the LC_{50}, and at least five rats

die during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to 130 percent of the LC_{50}, the predicted LC_{50} shall be considered to be validated. This confirmation ensures that the monitored toxicants account for the observed toxic effects. For data representing post-flashover fires, a correction shall be made for the excess CO generated by ventilation-limited fires.

The normal radiant heat flux is 50 kW/m^2. However, other levels of flux shall be permitted to be used for specific evaluations of other fire scenarios, provided the variation is properly recorded and reported.

The test method also allows for measurements of the time to ignition and the rate of mass loss of the test specimens.

Chapter 6 Test Apparatus

6-1 Animal Exposure Chamber. The animal exposure chamber, shown in Figures 6-1(a) and (b), shall be of a transparent polycarbonate or polymethylmethacrylate with a nominal volume of 0.2 m^3. Its inside dimensions shall be 1220 mm × 370 mm × 450 mm. The six animal ports, intended for head-only exposure, shall be located in a horizontal row, approximately halfway from the bottom to the top of the chamber, in the front wall. A plastic bag with a volume of 0.05 m^3 shall be attached to a port at the end of the chamber to provide for gas expansion. The exposure box shall be 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 shall be two doors in the exposure chamber, one in the front wall near the connection to the combustion cell and one in the end wall nearest the animal ports. The purpose of the doors is to allow for cleaning and maintenance of the chamber, the chimney, and the smoke shutter and to provide fresh air during the calibration of the heat lamps and immediately prior to testing.
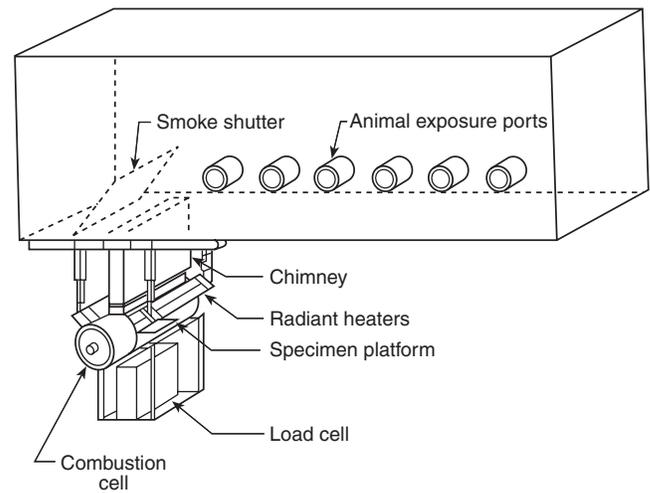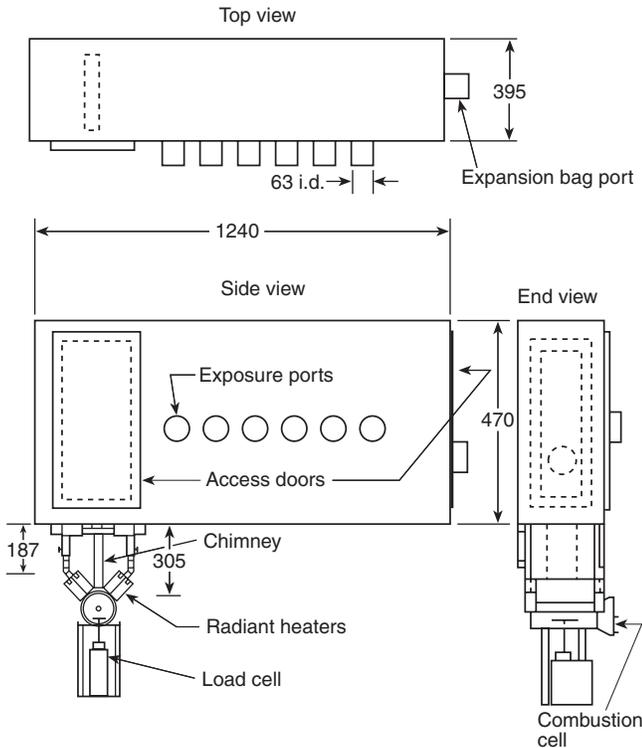

**Figure 6-1a General view of radiant heat apparatus.**

NOTE: All dimensions in mm.

Figure 6-1(b) Main dimensions of the apparatus.

6-2 Chimney. The chimney (*see Figure 6-2*) shall be a stainless steel assembly approximately 30 mm × 300 mm, inside dimensions, and approximately 300 mm wide. It shall connect the combustion cell to the animal exposure chamber. The chimney shall be divided into three channels by stainless steel dividers. The center channel shall be approximately 150 mm wide. The purpose of the dividers shall be to induce smoke 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 shall be connected to the underside of the animal exposure chamber by clamps, allowing its removal for cleaning. It shall be sealed to the animal chamber by low-density ceramic fiber insulation of approximately 65 kg/m^3. The other end of the chimney shall be sealed to the combustion cell by an H-shaped trough with a small quantity of the same fiber insulation in the trough.

6-3 Smoke Shutter. The smoke shutter shall be made of stainless steel plate and shall be situated inside the animal exposure chamber. It shall be positioned so that it closes over the chimney opening. It shall be hinged and provided with a positive locking mechanism. The purpose of the shutter shall be 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 shall be provided to allow the shutter to be closed gently. A wire attached to a clamp shall lock the shutter in place. To produce a gastight seal, the underside of the shutter shall be covered with a 12-mm thick blanket of low-density ceramic fiber insulation of approximately 65 kg/m^3, which shall be covered further with stainless steel foil.
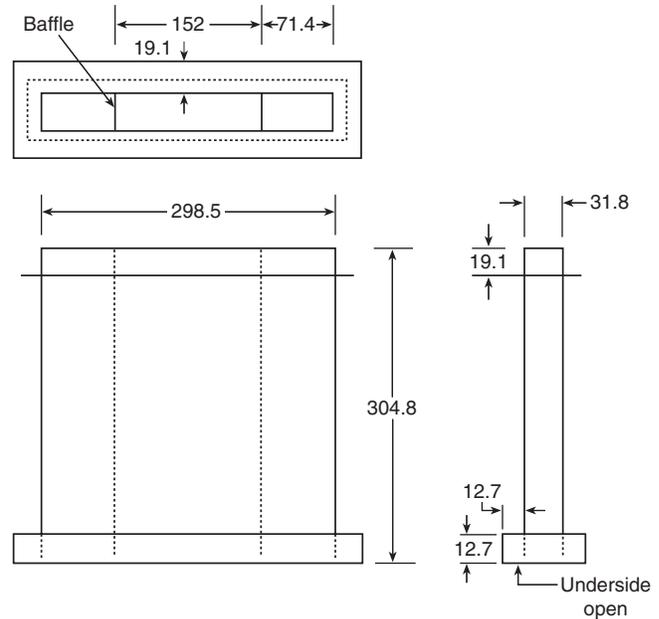
NOTE 1: All dimensions in mm, inside measure where applicable.
NOTE 2: Material is 1.6 mm stainless steel.

Figure 6-2 Chimney.

6-4 Combustion Cell.

6-4.1 The combustion cell [*see Figures 6-4.1(a), (b), and (c)*] shall be a horizontal quartz tube with a 127-mm inside diameter and a length of approximately 320 mm. It shall be sealed at one end and shall have a large standard taper outer joint located at the other end. A sealed inner joint shall serve as a removable plug for the open end [*see Figure 6-4.1(c)*]. The combustion cell shall have a rectangular opening on the top that is parallel to the axis of the cylinder with a "collar" that allows it to fit securely into the chimney. The bottom of the cell shall have a hole for the rod connecting the specimen support platform and the load cell. The sealed end of the combustion cell shall be fitted with a standard tapered glass joint for the electric sparker.

6-4.2 The combustion cell shall be supported by a metal frame that also holds the load cell [*see Figures 6-4.1(a) and (b)*] that monitors mass loss rate. The entire frame shall be 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 shall be located at a fixed distance from the combustion cell.

6-5 Radiant Heaters.

6-5.1 The active element of the heater shall consist of four quartz infrared lamps (with tungsten filaments) rated at 2000 W at 240 V. The lamps (two on each side) shall be encased in water-cooled holders with parabolic reflectors. These holders [*see Figure 6-4.1(a)*] shall be attached to adjustable metal frames that allow the lamps to be moved vertically and laterally and to be rotated in such a way as to give a uniform flux field across the sample surface. To keep the lamps from overheating, cooling water shall be circulated through their respective holders.
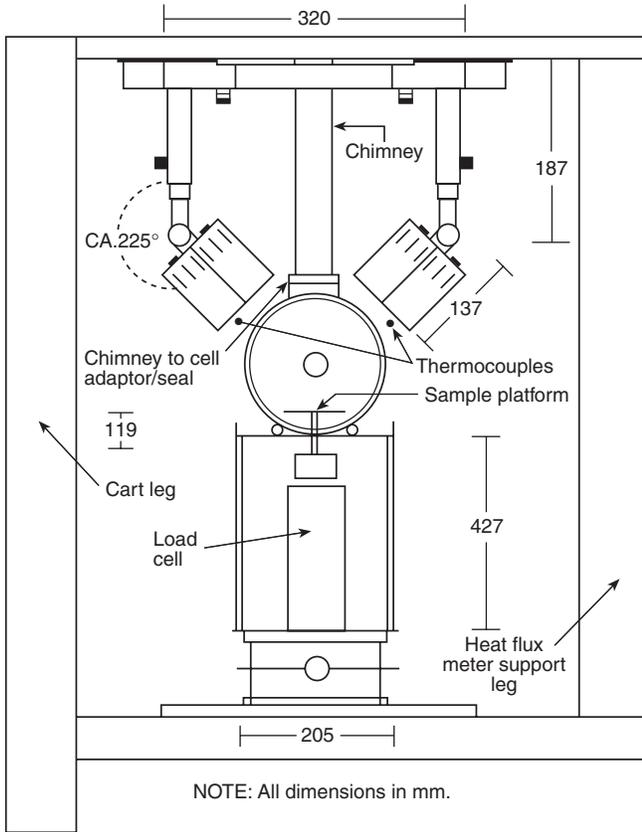
Figure 6-4.1(a) End view showing dimensions pertinent to combustion cell and heaters.
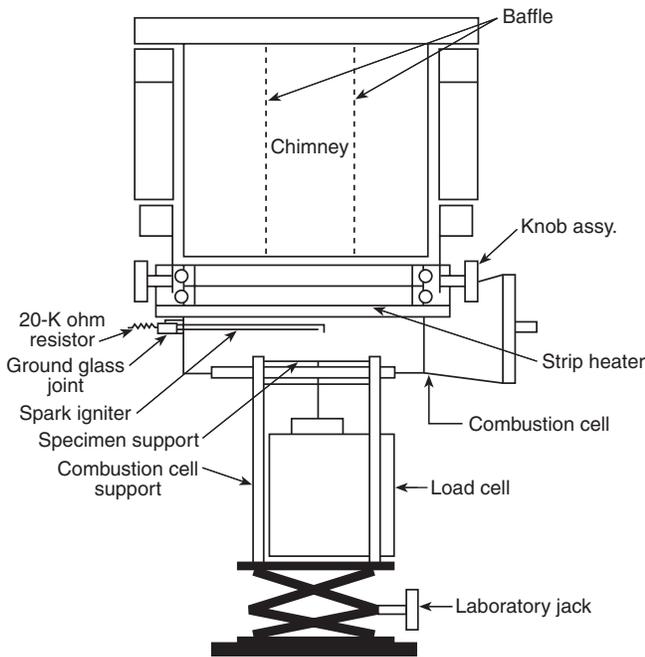
Figure 6-4.1(b) Side view of combustion cell.
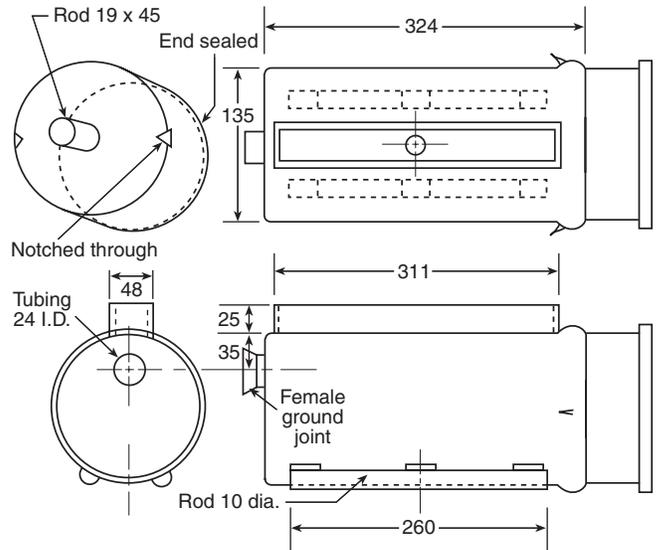
NOTE: All dimensions in mm.

Figure 6-4.1(c) Construction details for the combustion cell.
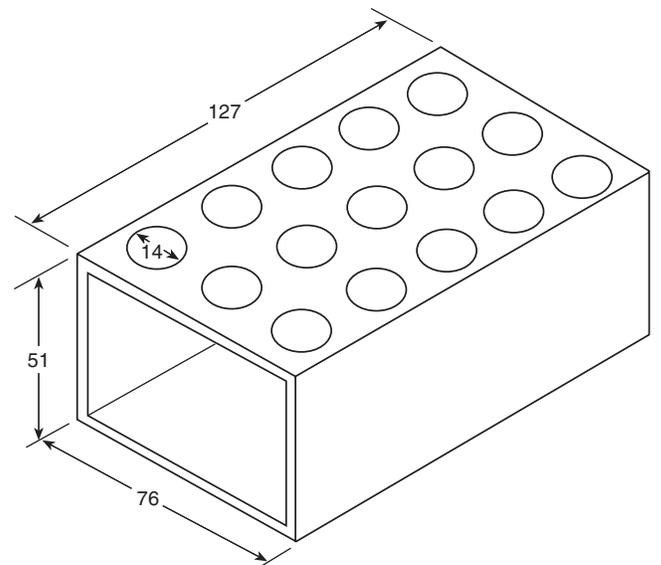
NOTE: All dimensions in mm.

Figure 6-5.3 Calibration jig used for checking the uniformity of irradiance.

6-5.2 The irradiance of the lamps shall be held at a preset level. This shall be accomplished by using a temperature controller and two type K thermocouples 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 sample holder to within ±10 percent. Figure 6-5.3 shows the calibration holder used in determining the uniformity of the radiant field from the lamps. If the field is found to be inadequately uniform, the lamp holders shall be repositioned, as necessary.

6-6 Temperature Controller. Where a temperature controller is used for maintaining the required radiant flux, the out-

put of the quartz lamps shall be controlled by a thermocouple signal to the temperature controller. The outputs from the two type K thermocouples shall be averaged by means of a parallel-wired connection, and this average value shall be used as the input to the controller. The temperature controller shall be a three-term type and shall provide an output signal suitable for driving the power controller. If necessary, the temperature controller also shall incorporate a means for setting the maximum output to prevent the power controller from being driven wide open. The power controller shall be 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/m^2. The target receiving radiation shall be flat, circular, approximately 12.5 mm in diameter, and coated with durable matte-black finish. The target shall be water-cooled. The flux meter shall have an accuracy of within ±3 percent and a repeatability within 0.5 percent.

6-7.2 The calibration of the heat flux meter shall be checked periodically. This shall be accomplished most easily 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 heaters. It shall be positioned firmly in a rigid support device to ensure repeatable readings. The surface of the heat flux meter shall be located in a position corresponding to the center of the specimen face. Figure 6-5.3 shows a calibration bracket suitable for this purpose.

6-8 Igniter. A spark igniter shall be constructed of two 3.2-mm stainless steel rods. One of these two rods shall be bent at 90 degrees, flattened on the end and positioned to have the appearance of the tip of an automotive-type spark plug. The gap between the two rods shall be about 2 mm ± 0.5 mm. These two rods shall be connected to a high voltage spark system that uses a 10-kV transformer (*see Figure 6-8*). To reduce the propagation of radio frequency that could interfere with the instrumentation, a 20,000 $^1/_2$, 5-W resistor shall be connected in series with one of the electrodes. The spark gap shall be positioned approximately 25 mm above the center of the top surface of the specimen, inside the combustion cell. In one method of operation, the rods comprising the spark igniter shall pass through a 29/42 male ground glass stopper, forming a gastight seal with a mating joint in place of the collar on the combustion cell [*see Figure 6-4.1(c)*]. Otherwise, the electrical leads shall be sealed in the glass collar in a gastight manner.

6-9 Specimen Holder. The specimen holder shall be a stainless steel assembly approximately 76 mm × 127 mm, inside dimensions, and 50 mm deep (*see Figure 6-9*). The specimen shall be backed by a layer of ceramic fiber blanket of nominal 65 kg•m^3 density. The specimen holder shall be 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 Figure 6-4.1(b). The load cell shall be installed under the combustion cell and shall be insulated against the heating effects of the radiant heaters. The specimen and holder shall be 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.

6-11.1 A suitable gas sampling arrangement shall include a pump, a glass wool filter at the sampling port, a cold trap to remove soot and moisture, and a pressure relief valve that shall return all flow required by the CO, CO$_2$, and O$_2$ gas analyzers. The flow to these analyzers also shall be returned to the animal exposure chamber through separate return lines (*see Figure 6-11.1*). The return lines shall be closed during the calibration of the instruments to prevent the accumulation of calibration gases in the animal exposure chamber. Since this is a closed system, gases that are removed for chemical analysis and that can be recirculated to the animal exposure chamber are allowed to be returned.

6-11.2 The gas analyzers shall have the following ranges and resolutions:

   (a) Carbon monoxide — 0 to ≥10,000 ppm; 10 ppm;

   (b) Carbon dioxide — 0 to ≥10 percent; 0.1 percent (vol./vol. percent);

   (c) Oxygen — 0 to 21 percent; 0.01 percent (vol./vol. percent).

6-11.3 Gas analysis for HCN, HCL, and HBr shall be performed as required by Section 12-1.

6-12 Data Collection. The data collection system shall be capable of recording the output from the gases monitored by the gas analyzer, the thermocouple(s) in the chamber, and the load cell and shall have an accuracy corresponding to 0.01 percent of full-scale instrument output.

6-13 Animal Restrainers. Animal restrainers that are made of aluminum and designed to allow exposure of only the heads of the animals shall be used. A detailed illustration of one animal restrainer meeting this requirement is shown in Figure 6-13.
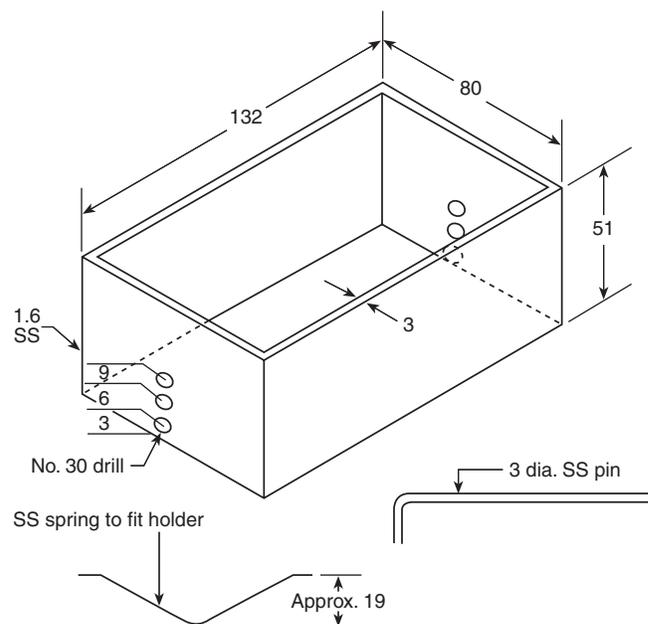
NOTE: All dimensions in mm.

Figure 6-9 Specimen holder.
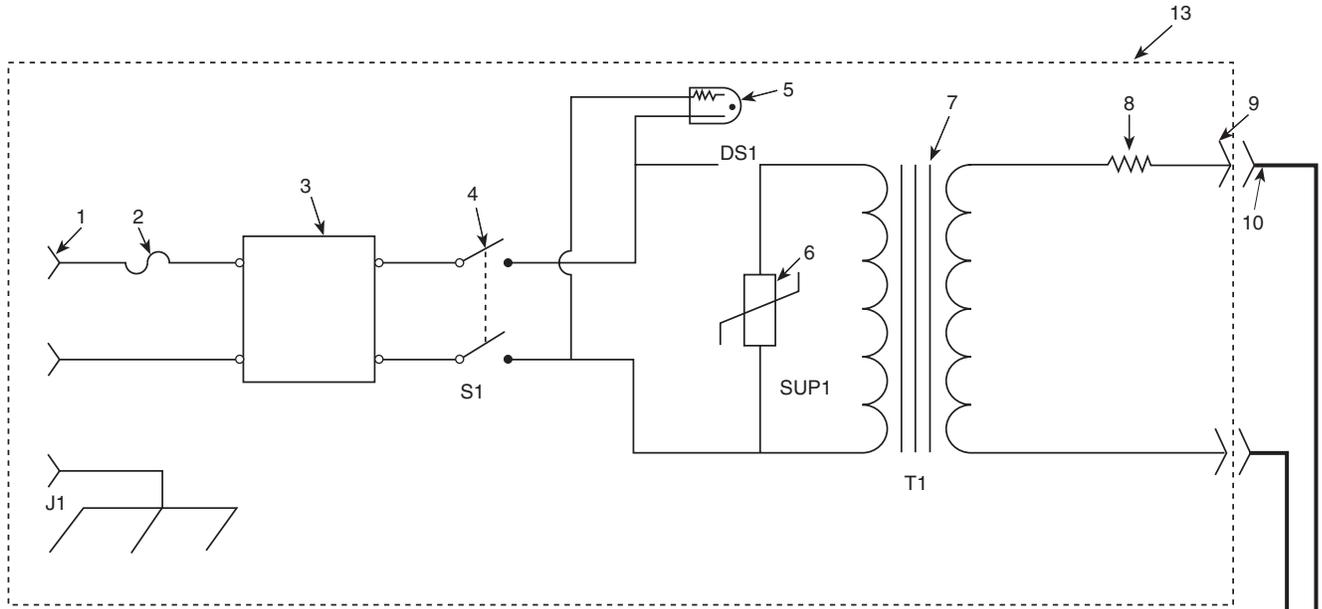
13	1	Cabinet	Bud futura md-1962
12	1	Resistor 20k ohm	STD
11		Wire corona res hv	Dearborn 391845
10	2	3.2 dia. SS rods	STD
9	1	Receptacle ceramic 10kv	Part of 10kV transformer
8	1	Resistor pwr 10k 225w	Ohmite L225J10K
7	1	Transformer 10kV 22ma	Donegan modle LJH-90-A
6	1	Surge suppressor	G.E. mov V130LA20B
5	1	Panel indicator light red	Dialight 249-7866-0931-514
4	1	Toggle switch dpst	Cutler-hammer 7590K6
3	1	Power line filter	Corcom 3EP1
2	1	Fuse holder & fuse 3AG 2A	Littlefuse 342058A
1	1	Power receptacle	Belden 17253

NOTE: All dimensions in mm.

Figure 6-8 Power supply for spark ignition.
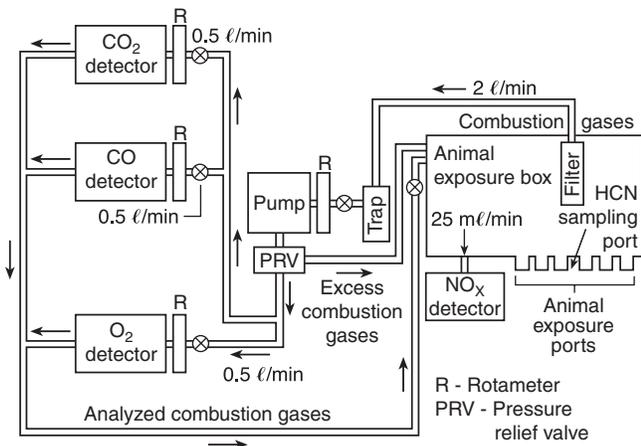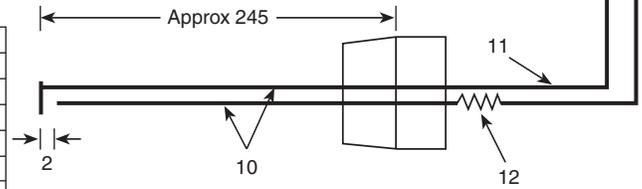
Figure 6-11.1 Gas sampling system.
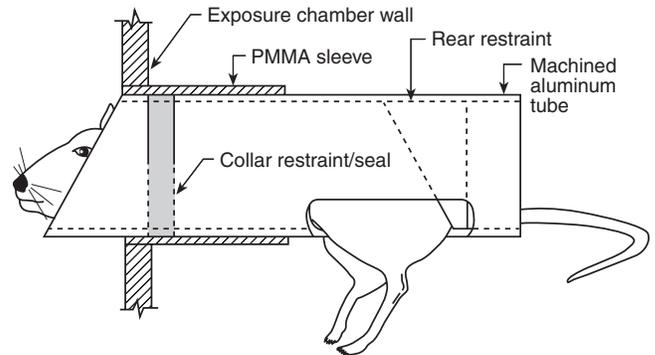
Figure 6-13 Animal restrainer.

Chapter 7 Hazards

7-1 Safety Precautions.

7-1.1 The test procedure involves high temperature and combustion processes. Therefore, precautions shall be exercised against hazards from burns, eye injuries, ignition of extraneous objects, and inhalation of combustion products. To avoid accidental leakage of toxic combustion products into the surrounding atmosphere, the entire exposure system shall be placed into a chemical hood or under a canopy hood. If under a canopy hood, an accessory exhaust trunk for any combustion gases escaping through the load cell hole on the bottom of the combustion cell shall be required. An exhaust line to evacuate the exposure box at the end of a test shall be recommended. The operator shall use safety tongs for the removal of the specimen holder. The combustion cell, while hot, shall be handled only with protective gloves. Due to the intense light from the infrared lamps used, dark safety glasses shall be worn by the operator, or a darkened polymethylmethacrylate or polycarbonate shield shall be placed in front of the combustion cell.

7-1.2 The venting system for the exposure chamber shall be checked for proper operation before testing and shall discharge into an exhaust system with adequate capacity.

Chapter 8 Material Test Specimens

8-1 Test Specimens.

8-1.1 Test specimens shall be cut to an appropriate area and thickness (to be determined by the procedure described in Section 11-1), no larger than 76 mm × 127 mm and no more than 50 mm thick, representing the end-use product. Raw materials (e.g., paints, adhesives, and wall coverings) shall be tested on the substrate to which they are normally applied. For testing, the specimens shall be wrapped on all sides with either aluminum or stainless steel foil.

Exception: The top faces of the specimens shall not be required to be wrapped.

8-1.2 Test specimens shall be conditioned to moisture equilibrium (constant weight) at an ambient temperature of 23°C ± 3°C and a relative humidity of 50 percent ± 10 percent.

Chapter 9 Test Animals

9-1 Test Animal Specifications. The test animals shall be inbred 3-month to 4-month-old male rats, weighing 225 g to 350g. The rats shall be obtained from a reputable supplier that certifies that the animals are free of major respiratory pathogens.

9-1.1 The maintenance and care of the animals shall be performed by qualified trained personnel in accordance with the guidelines of the National Institute of Health Guide for the Care and Use of Laboratory Animals. The animal housing facilities shall be suitable for studies of this type.

9-1.2 Upon receipt, the animals shall be identified, weighed, and housed in a separate quarantine area for a minimum of 7 days prior to testing. During the quarantine period, animals shall be observed and weighed daily. Animals that are unsuitable by reason of health or other criteria shall not be used. Cage assignments shall be made according to a randomization routine. Health criteria for eliminating animals from a study shall include any animal weighing outside ±20 percent of the body weight specified in Section 9-1.

9-1.3 The animals shall be housed one animal to a cage. The environment shall have proper ventilation and shall be controlled at a temperature of 23°C ± 3°C and a relative humidity of 50 percent ± 15 percent. The animal room shall have a 12-hour light/dark cycle.

9-1.4 Each animal shall be weighed when received, prior to test, and surviving animals shall be weighed at 7 and 14 days post-exposure.

9-1.5 Prior to exposure, the animals shall be weighed and secured in individual restrainers for placement in the animal exposure chamber.

9-1.6 After testing, surviving animals shall be housed in an animal room separate from the pre-test animal room for the post-exposure observation period.

Chapter 10 Equipment Calibration

10-1 Calibration of the Apparatus. The following parts of the test apparatus shall require calibration:

    (a) The radiant heaters;

    (b) The gas analyzers;

    (c) The load cell;

    (d) The temperature controller (if used).

10-2 Heat Flux Calibration.

10-2.1 For heat flux calibration, the heat flux meter shall be secured in the proper position. The target surface of the flux meter shall be centered at the location corresponding, both horizontally and vertically, to that of the top of the specimen when the specimen holder is in place on the platform. (The igniter shall be removed from its position during this procedure.) The temperature controller, if used, shall be set to the desired flux temperature and the radiant heat lamps shall be turned on, adjusting the temperature controller until the desired irradiance (i.e., 50 kW/m² ± 10 percent) is achieved. If manual control of the heat lamps is used, the calibration curve of the heater controller setting as a function of the time needed to maintain the desired flux level shall be developed.

10-2.2 The orientation of the radiant heat lamps shall be checked whenever the heaters have been moved or a lamp has been replaced, using the following procedure:

    (a) The heat flux calibration jig shown in Figure 6-5.3 shall be installed.

    (b) The top face of the calibration jig shall be positioned at the same height at which the top of a test specimen is placed.

(c) A power setting for the lamps shall be estimated that produces the desired level (i.e., 50 kW/m^2) at the center hole.

(d) The power shall be adjusted and at least 5 minutes shall be allowed for equilibration.

(e) The orientation of the radiant heat lamps shall be adjusted so that no measurement at seven locations across the face of the specimen (*see Figure 6-5.3*) deviates by more than 10 percent 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 with a full-scale reading that is near to, but less than, that of the analyzer. For all calibrations, the gas shall be set to flow at the same rate and pressure as the sample gas. For calibrating the O_2 analyzer, ambient air (20.9 percent O_2) shall be used, while, for the CO_2 and CO analyzers, bottled gases containing CO_2 and CO at known concentrations shall be required to be used. A single mixture containing both CO and CO_2 shall be permitted to be used. *During the calibration procedure, the gas return lines shall be diverted into the exhaust and not into the exposure chamber to prevent the inadvertent accumulation of CO and CO_2.*

10-3.2 The calibration of apparatus for analysis of optional gases (e.g., HCN, HCl, HBr, and NO_2) shall be performed.

10-4 Load Cell Calibration.

10-4.1 The load cell shall be calibrated with standard weights corresponding to the range of test specimens on a regular basis, and when first setting up the apparatus or after making adjustments for sensitivity and range.

10-4.2 Before each test, the load cell shall be checked routinely using at least two analytical quality weights for the full 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 the Temperature Controller. To set up the controller, the flux meter first shall be inserted into its holder so that the sensing surface is at the exact center of where the top of the specimen is placed during normal testing. The lamp shall have been adjusted to obtain a uniform flux field over the sample. The output of the heat flux meter then shall be connected to a strip chart recorder running at a trace speed that is still fast enough to detect any changes in the flux. Using the output from the heat flux meter, the instructions of the controller manufacturer for adjusting the controller shall be followed 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 shall be to limit the maximum output from the slave controller. This can be done either by 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 for this function, the thermocouples always shall read room air temperature only. Using the correct adjustment of the temperature controller, 90 percent of the desired flux can be reached within 2 seconds, with 100 percent flux reached within 20 seconds, and a deviation of within ±5 percent reached for the remainder of the test duration.

Chapter 11 Procedures

11-1 General. Test procedures for smoke toxicity data initially shall be followed *without* the exposure of test animals in order to produce analytical data for CO, O_2, CO_2, and, if present, HCN, HCl, and HBr. The choice of specimen size for the initial tests shall be made with consideration of anticipated toxicant yields so that total FEDs of 0.5 to 1.5 shall be obtained (*see Section 12-1*). In the absence of appropriate information for such choices, an area equal to $1/4$ of a maximum area of 96.5 cm^2 shall be selected initially. Analytical data from at least two initial tests shall be used for estimation of an average LC_{50} for the test specimen. (*See Section 12-1.*)

Comparable tests then shall be conducted, but *with* the exposure of six rats to the smoke produced from that quantity of the test material whose mass loss concentration during the 30-minute exposure is approximately equivalent to 70 percent (±10 percent) and to 130 percent of its average estimated LC_{50}. If no more than one rat dies during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to 70 percent of the LC_{50} and at least five rats die during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to 130 percent of the LC_{50}, the estimated LC_{50} shall be considered to be validated. If the validation is not successful, or if unexplained or unusual toxicity is suspected, other test methods shall be employed, or further research will be needed to determine experimentally the lethal toxic potency of the test material. (*See B-3.1 and B-3.2.*)

11-2 Preparation for Tests.

11-2.1 Coolant water for the heat flux meter (at least 750 ml/min) and for the tungsten lamps (at least 600 ml/min) shall be turned on.

11-2.2 Verification that all lines, filters, and traps for the gas analyzers have been serviced and that flow rates are satisfactory shall be made.

11-2.2.1 The moisture trap in the gas analyzer stream shall be checked. The trap shall be dried, and the glass wool shall be replaced. The normal operating temperature of the moisture trap is 0°C.

11-2.2.2 A wool filter shall be placed before the gas sampling port.

11-2.3 Operation of the spark ignition circuit shall be verified.

11-2.4 The required calibration procedures specified in Chapter 10 shall be performed. The specimen shall be weighed on a laboratory balance with an accuracy of ±0.01 g.

The specimen shall be wrapped in either aluminum or stainless steel foil, leaving the top surface exposed, and the combined weight of the specimen with the foil and after mounting in the specimen holder shall be determined. The correspondence of the load cell readout to the appropriate weight of the specimen plus the holder shall be verified.

11-3 Test Procedure.

11-3.1 If animals are to be exposed, they shall be weighed and placed in their restrainers.

11-3.2 The specimen, mounted in the specimen holder, shall be inserted into the combustion cell, and the standard taper plug shall be replaced (no grease or sealant shall be used on the ground glass). The plug shall be secured with wire or springs. Immediately prior to beginning the test, the animals shall be placed into the ports in the exposure chamber. All exposure chamber doors and ports shall be closed if not used for animals. It shall be ascertained that the smoke shutter is open.

11-3.3 The spark igniter shall be turned on. The power to the radiant heat lamps shall be activated simultaneously with the start of the data collection.

11-3.4 The time at which ignition of the specimen occurs shall be recorded and the spark igniter shall be turned off. The time of flameout shall be recorded. For specimens that have the tendency to self-extinguish soon after ignition, the sparker igniter shall be left on until flaming ceases.

11-3.5 At the end of 15 minutes, the power to the radiant heat lamps shall be switched off, and the smoke shutter shall be closed.

11-3.6 Data shall be collected for a total of 30 minutes from the initiation of the test.

11-3.7 At the end of 30 minutes, data collection shall cease. If animals were exposed, they shall be removed from the exposure chamber. The exposure chamber shall be vented with a high capacity exhaust system.

11-3.8 In tests where animals are exposed, blood samples shall be taken from any dead animals and analyzed for carboxyhemoglobin saturation. Blood sampling and analyses shall be conducted in accordance with generally accepted methodologies.

11-3.9 In tests where animals are exposed, those surviving shall be checked daily for any signs of toxic effects (e.g., difficulty in breathing, 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 post-exposure period. Any deaths during this period shall be recorded.

11-3.10 The sample holder shall be removed from the combustion chamber and cooled to an ambient temperature in an exhaust hood. After the specimen has cooled, the specimen holder shall be disassembled and the weight of the aluminum foil and the residue shall be determined.

11-3.11 The combustion chamber and the chimney shall be removed and cleaned after each test. The exposure chamber

shall be cleaned after each test. Ethyl alcohol shall be considered a satisfactory solvent. There shall be no residue on the inside of any of the pieces of the apparatus.

Chapter 12 Calculation

12-1 General. The lethal toxic potency (LC_{50}) of the test specimen shall be predicted from the combustion atmosphere analytical data for CO, CO_2, O_2, and, if present, HCN, HC1, and HBr (*see B-2.7*). This shall be determined for a given specimen mass loss by first calculating the total FED for the test. The LC_{50} then shall be calculated as that specimen mass loss that would yield a total FED = 1.1 within a chamber volume of 1 m^3. Although the theoretical value of the FED associated with 50 percent lethality is 1.0, a median value of 1.1 has been determined experimentally.

12-2 Equations.

12-2.1 The total 30-minute FED for a given specimen mass loss shall be determined from the following equation:

$$\text{FED} = \frac{m[CO]}{[CO_2]-b} + \frac{21-[O_2]}{21-LC_{50}O_2} + \frac{[HCN]}{LC_{50}HCN} + \frac{[HC1]}{LC_{50}HC1} + \frac{[HBr]}{LC_{50}HBr}$$

$$= \frac{m[CO]}{[CO_2]-b} + \frac{21-[O_2]}{21-5.4\%} + \frac{[HCN]}{150\ ppm} + \frac{[HC1]}{3700\ ppm} + \frac{[HB_r]}{3000\ ppm}$$

Where the values of all gas concentrations are the integrated Ct product values under their respective concentration–time curves taken over the 30-minute test period divided by 30. All values are in ppm, except O_2, which is expressed as a percentage. The values for m and b depend on the concentration of CO_2. If $[CO_2] < 5\%$, m = –18 and b = 122,000. If $[CO_2] > 5\%$, m = 23 and b = –38,600. For each individual toxicant, the LC_{50} values shown have been statistically determined from independent experimental data to produce lethality in 50 percent of test animals (rats) within a 30-minute exposure plus 14 days post-exposure.

12-2.2 The 30-minute LC_{50} for the test specimen shall be determined from the following equation:

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

Where the specimen mass loss is in g and the chamber volume is 0.2 m^3, the resulting LC_{50} = g·m^{-3}.

12-2.3* To calculate toxic hazard data for smoke from a post-flashover fire, a value of the LC_{50} corrected for the expected post-flashover yield of CO is needed. The adjusted value shall be determined from the following formula:

$$LC_{50}(corr) = \frac{1}{\dfrac{1}{LC_{50}(raw)} + 44 \times 10^{-3} - 5.0 \times 10^{-5}\dfrac{[CO]}{m}}$$

where $LC_{50}(raw)$ is the value of the LC_{50} determined from the equation in 12-2.1, m is the mass of specimen loss during the test at the FED = 1.1 condition, and [CO] is the concentration of CO at the FED = 1.1 condition (ppm).

Chapter 13 Analysis

13-1 Report. The report shall include the following information for all tests:

(a) Laboratory identification;

(b) Test identification and date;

(c) Laboratory ambient conditions (temperature and humidity);

(d) Description of specimen;

(e) Specimen dimensions;

(f) Irradiation time and heat flux conditions;

(g) Maximum exposure chamber temperature and time when attained (*see B-2.6*);

(h) Initial specimen mass and mass loss during test in $g \bullet m^{-3}$ of chamber volume (*see B-3.3*);

(i) Time to ignition and flameout;

(j) Observations of specimen;

(k) Observations that shall be required include the times to smoke evolution, ignition, and flameout. Other observations shall include melting, char formation, spalling, unusually vigorous burning, and reignition.

13-2 Gas Analysis Data. The required exposure chamber data shall include integrated Ct product values during the 30-minute test for CO, O_2, HCN, HCl, and HBr; minimum O_2 concentration and maximum CO_2 concentration; and times to reach minimum O_2 and maximum CO_2. The methods used for analyses shall be identified.

13-3 Calculations. The following data shall be calculated for all tests:

(a) Ct product for each analyzed toxicant;

(b) FED;

(c) Predicted LC_{50}.

13-4 Best Overall Predicted LC_{50} Value. A least squares regression analysis of FED versus mass loss values for all tests shall be used to determine the best overall predicted LC_{50} value.

13-4.1 The LC_{50} value shall be corrected for high post-flashover CO yield.

13-4.2 Plots of individual toxicant concentrations, specimen mass loss, and temperature as functions of time shall be considered optional.

13-5 Report. The report shall include the following information for each test using the exposure of animals:

(a) Strain of rat and identity of supplier;

(b) Weight of each animal when received, prior to test, and of surviving animals at 7 and 14 days post-exposure;

(c) Number of animals that die during the test (including up to 10 minutes post-test), and number of animals that die up to 14 days post-test;

(d) Blood carboxyhemoglobin saturation values for animals that die during the test;

(e) Animal observations, (e.g., unusual behavior during test); immediate post-test observations of live animals, such as tremors, convulsions, difficulty in breathing, and severe eye irritation.

Chapter 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 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.

14-2.1 Comparison of the LC_{50} (corrected) values derived using this method (*see B-4.2*) have been shown to reproduce the LC_{50} values from real-scale, post-flashover fires to within a factor of three. Therefore, product LC_{50} values differing by less than a factor of three are indistinguishable from each other.

14-2.2 The accuracy of the bench-scale data for pre-flashover fires has not been established experimentally. However, the combustion conditions in the apparatus are quite similar to real pre-flashover fires, although the mass burning rate is higher at the 50 kW/m^2 irradiance.

Appendix A Explanatory Material

This Appendix is not a part of the requirements of this NFPA document but is included for informational purposes only.

A-12-2.3 If real-scale, post-flashover fire test data are available for a particular product/occupancy combination, the measured CO yield should be used in the equation in 12-2.1, and the correction in the equation in 12-2.3 should not be made.

Appendix B Commentary

This Appendix is not a part of the requirements of this NFPA document but is included for informational purposes only.

B-1 Introduction. This appendix provides insight into the development of the test method, to describe the rationale for the unique features of the method, and to describe the proper use of the resulting data. For a more comprehensive treatment, along with presentation of data and results obtained on typical materials, see NIST Special Publication 827.[1]

B-2 Development of the Method. A test method to assess the acute inhalation toxicity of combustion products has three basic components: a combustion system, a chemical analysis system and an animal exposure system.[2] Additionally, there should be a rational and accepted strategy for the incorporation of raw experimental data into a quantified expression for toxic potency.

B-2.1 This test method employs the combustion system that was developed at Southwest Research Institute (SwRI) for the National Institute of Building Sciences (NIBS).[3] Representing a significant improvement over an earlier radiant heat device first used at the Weyerhaeuser Company,[2] the combustion system was adopted jointly by the National Institute of Standards and Technology (NIST) and SwRI for the development of this test method.[4] Its main feature is that of providing for combustion of a test specimen under the realistic conditions of radiant heat within an apparatus especially designed for ruggedness and ease of operation.

B-2.2 For a small, developing fire, the bench-scale specimen in the radiant apparatus provides a reasonable representation of a full-scale fire. The thermal boundary conditions are appropriate, being radiative and emanating from only one face. A small fire imposes about 35 kW/m² on an adjacent unburned surface, although a value of approximately 48 kW/m² is common, and values over 100 kW/m² can be measured.[5,6] Thus, while an irradiance of 50 kW/m² for a pre-flashover test might be somewhat high, it is by no means unusual. If a specific scenario involves a heating flux other than 50 kW/m², it can be readily accommodated in the test method.

B-2.3 In a real-scale fire, the combustion products generally contain contributions from portions of the burning product that are burning near the front surface, those that are partially burned through, and those produced from nearly burned out portions of the burning product. 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 minutes. The actual thickness of nonlayered products should be selected so that thermal decomposition is complete when 15 minutes has elapsed and the shutter is closed. This should be determined by examination of the generation of CO, which should have ceased before the shutter is closed. A preliminary trial run where neither animals nor gas analyzers need to be used should suffice. Homogeneous products then can be prepared for actual testing by cutting to the appropriate thickness. Layered composites also generally might be reduced in thickness, as necessary. If the two face materials are not identical, however, then separate tests should be performed, with each face being exposed in separate testing.

B-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 ASTM E 800, *Standard Guide for Measurement of Gases Present or Generated During Fires* and in ISO/TR 9122, *Toxicity Testing of Fire Effluents*, Part 3.

B-2.5 The use of rats as an acceptable model for human exposure has, within the scope and significance of this test method, been well documented in ISO/TR 9122, *Toxicity Testing of Fire Effluents*, Part 5.

B-2.6 The animal exposure system is that employed in the NBS Cup Furnace method.[7] It has been widely used in a number of laboratories and has been found to be highly satisfactory. An important consideration in conducting animal exposures is that the biological effects on the animals' condition during a test should be adversely affected as little as possible by causes other than specimen toxicity. Such adverse affects can be minimized by:

(a) Providing an animal exposure chamber of sufficient size so that the animals' exhaled CO_2 does not affect them adversely;

(b) Making certain that heating conditions from specimen heaters do not create an excessive heat burden to the animals; and

(c) Providing a restraint system that does not cause undue physical stress. Judgment regarding exposure chamber temperatures excursions above 40°C should be exercised. Gener-

ally, such excursions have not been regarded as detrimental to the resulting test data. However, there could be exceptions in extreme cases.

B-2.7 The strategy employed in this method for quantification of smoke toxicity represents utilization 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, *Toxicity Testing of Fire Effluents*, Part 5. It employs a methodology for the calculation of toxic potencies from combustion product analysis 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 fractional exposure doses (FEDs) of the individual toxicants.[8-18] Expressed mathematically, the principle is shown in the following equation:

$$FED = \sum_{i=1}^{n} \int_{0}^{t} \frac{C_i}{(Ct)_i} dt$$

where C_i is the concentration of the toxic component, i, and $(Ct)_i$ is the specific exposure dose required to produce the toxicological effect.[19] Where the FED = 1, it is expected that the mixture of gaseous toxicants would be lethal to 50 percent 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 CO_2 on the toxicity of CO, as expressed empirically from studies conducted at NIST.[8] Examination of a series of pure gaseous toxicant experiments in which various percentages of animals die indicates that the mean FED value using the N-Gas calculation was 1.07, with 95 percent ± 0.20 percent confidence limits.[11]

B-3 Limitations on Materials and Products.

B-3.1 All products should be tested using the calculation method described. However, the lethal toxic potency of the smoke from certain products cannot be attributed to the common toxic gases analyzed. These products might need to be tested according to conventional animal exposure methodology that requires the experimental determination of an LC_{50} value. Otherwise, further research is necessary in order to explain why the lethal toxic potency cannot be estimated from the concentrations of the common toxic gases. For certain materials or products containing perfluorinated polymers, this test method could be inappropriate, because a number of highly specialized combustion and toxicological phenomena can arise that make the creation of proper bench-scale conditions that represent real-scale toxicity difficult.[20]

B-3.2 The research for the N-gas Model did not include the combustion gases likely to be produced when materials or products that contain elements such as sulfur, bromine, or fluorine are burned. Therefore, the calculations might not fully account for the smoke toxicity in such cases.

B-3.3 Certain ash-producing materials (e.g., silicones) necessitate the use of appropriate procedures to measure the quantity of sample burned, since the load cell readings are compromised by the deposit of ash resulting from the burning of the material.