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# *Toxic Potency Measurement for Fire Hazard Analysis*

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*Vytenis Babrauskas, Barbara C. Levin, Richard G. Gann, Maya Paabo,  
Richard H. Harris, Jr., Richard D. Peacock, and Shyuitsu Yusa*



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# Toxic Potency Measurement for Fire Hazard Analysis

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## Executive Summary

This report is the principal product of a long-term research program to provide a technically sound methodology for obtaining and using smoke toxicity data for hazard analysis. It establishes:

- (a) an improved bench-scale toxic potency<sup>1</sup> measurement, one which represents the important combustion conditions of real fires; and
- (b) a design and analysis framework which will allow the toxic potency data to be used in a rational, consistent, appropriate, and adequate way.

This establishment of proper bench-scale test conditions, validation of the output against real-scale fire measurements, and development of a consistent framework for the inclusion of toxic potency in fire hazard<sup>2</sup> analysis is unique and represents a successful, usable implementation of the state of the art.

This method focuses on post-flashover fires. The U.S. fire statistics show that 69% of all fire deaths are associated with post-flashover fires, with the preponderance of deaths due to smoke inhalation and occurring outside the room of fire origin. These fires are characterized by:

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<sup>1</sup> *Toxic potency*: toxicity of the smoke from a specimen of material or product, taken on a per-unit-specimen-mass basis. At present, for fire research, the dominant biological end point adopted is death; and the measured quantity is the LC<sub>50</sub>, which is the concentration (g·m<sup>-3</sup>) of smoke which is lethal to 50% of the exposed specified test animals in a specified time period. The LC<sub>50</sub> notation must include the exposure time, generally 30 minutes (along with a 14-day post-exposure observation period). **Toxic potency is not an inherent property of a material.**

<sup>2</sup> *Fire hazard*: the seriousness of the exposure conditions which threaten the physical well-being of the occupant. The hazard may come from various sources, for example, smoke inhalation, direct flame burn, injuries due to trauma (e.g., ceiling collapse), high temperatures, or inability to escape due to lack of visibility or the presence of acid gases which affect the eyes.

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- primarily radiant heating, with heat fluxes from about 20 to 150 kW/m<sup>2</sup> throughout the room;
- many items simultaneously on fire; and
- vitiated combustion air for some, but not all, burning items.

The toxic potency measurement method is also applicable to pre-flashover fires. However, deaths from these fires generally occur within the room of fire origin; and both computer modeling and full-scale simulation show that these deaths are far more likely to be due to heat and burns than smoke toxicity.

The importance of toxic fire hazard<sup>3</sup> (relative to heat, burns, generalized trauma from falling debris or leaping from a window, etc.) in the overall threat to life safety in fires varies with the type of fire, the location of the people relative to the fire, and the time they are exposed to the fire and its products. There is thus an inherent flaw in making materials selection decisions based solely on a single characterization (e.g., toxic potency) of the smoke or even a simple index containing toxic potency and other fire variables.

It is now possible to perform computations of fire hazard leading to assessments of the degree of threat to life safety. These range from:

- simple, closed-form equations ("hand calculations") generally not requiring a computer for solving, to
- computer simulations of a fire where a large number of differential equations are being solved simultaneously.

Either mode of calculation requires valid toxic potency (LC<sub>50</sub>) input data.

This study recommends that this data be obtained using a radiant apparatus. This device is the first to be validated against data from real-scale fires. It is a descendant of the cup furnace and the Weyerhaeuser radiant apparatus, and is an advanced version of the apparatus developed by the Southwest Research Institute for the National Institute of Building Sciences.

In this radiant apparatus, materials, products, composites, and assemblies are exposed to 50 kW/m<sup>2</sup> radiant heat under likely end-use conditions. The sample surface area may be as large as 7.6 cm (3") x 12.7 cm (5"), with a maximum thickness of 5.1 cm (2"). Six rats are exposed to the smoke collected in an approximately 200 L rectangular box located above the furnace. Changes in the concentration of smoke are achieved by variation of the surface area of the sample.

The number of animal tests is minimized by estimating the toxic potency of the smoke based on established toxicological interactions of the smoke components. Thus, a small fraction of the chamber

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<sup>3</sup> *Toxic fire hazard*: a subset of "fire hazard," where the threat is inhalation of toxic combustion products.



atmosphere is removed for chemical analysis of CO, CO<sub>2</sub>, O<sub>2</sub>, HCN, HCl, HBr, and NO<sub>x</sub>. An N-Gas Model had been previously developed to enable the use of these data to obtain approximate LC<sub>50</sub> values, based on the calculation of a Fractional effective Exposure Dose (FED) of mixtures of these gases. The FED value is approximately 1.1 at the LC<sub>50</sub>.

The determination of the approximate LC<sub>50</sub> is a 2- or 3-step process:

1. **Determine an estimated LC<sub>50</sub> (30-minute exposure plus 14-day post-exposure observation period) using the N-Gas Model.** This entails two experiments, neither involving animals. The specimen size for the first is obtained using existing data from similar products. The consumed sample mass and the concentrations of gases in the N-Gas Model are measured, and an FED is calculated. Based on this result, a similar second experiment is performed for a specimen that should produce an FED of about 1.1. The LC<sub>50</sub> for a test is estimated by dividing the volatilized sample mass by the product of the FED for that test and the apparatus volume.
2. **Check the estimated LC<sub>50</sub> (30-minute exposure plus 14-day post-exposure observation period) using animals.** Again two experiments are needed: one where the specimen surface area (and mass) is chosen to produce an FED of about 0.8, and one to produce an FED of about 1.4. In each, 6 rats are exposed to the smoke for 30 minutes, and the mass loss and standard gas concentrations are measured. The measurements are to assure that the sample decomposition indeed provided the desired FED. If the LC<sub>50</sub> estimate is accurate, the exposure at FED = 0.8 should result in 0 or 1 animal death and the exposure at FED = 1.4 should result in 5 or 6 animal deaths. If the animal deaths are as predicted, then the chemical data from the 4 experiments are used to calculate an approximate LC<sub>50</sub>, and no further measurement is needed. The calculation includes a correction for the generation of less-than-post-flashover amounts of CO in bench-scale devices. Post-flashover fires produce CO yields higher than any bench-scale device (or pre-flashover fires).
3. **If such results are not seen, then determine a more precise value for the LC<sub>50</sub>.** For a proper statistical determination, 3 experiments are needed in which some, but not all, of the rats die. The selection of sample sizes is guided by the prior 4 tests. After determining the LC<sub>50</sub>, it should be reported to 1 significant figure.

The LC<sub>50</sub> of CO in the presence of CO<sub>2</sub> is about 5 g/m<sup>3</sup>, and one-fifth of the smoke in post-flashover fires is CO. Therefore, the LC<sub>50</sub> of post-flashover smoke (based only on CO<sub>2</sub> and CO) is about 25 g/m<sup>3</sup>. The previous work on validation of this bench-scale apparatus showed that the results could be used to predict real-scale toxic potency to about a factor of 3.<sup>4</sup> Therefore, post-flashover smokes with LC<sub>50</sub> values greater than 8 g/m<sup>3</sup> [(25 g/m<sup>3</sup>)/3] are indistinguishable from each other.

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<sup>4</sup> A prior risk analysis had demonstrated that this level of uncertainty would not affect the prediction of loss from the most common fire loss scenario: furniture fires in residences.

A measured  $LC_{50}$  value greater than  $8 \text{ g/m}^3$  should be recorded only as "greater than  $8 \text{ g/m}^3$ ." A hazard analysis would then use this value for the toxic potency of the smoke. A measured  $LC_{50}$  value less than  $8 \text{ g/m}^3$  would be recorded to one significant figure. These products could well be grouped, reflecting the factor-of-3 accuracy of the bench-scale test. A hazard analysis would then use values of  $8 \text{ g/m}^3$ ,  $3 \text{ g/m}^3$ ,  $1 \text{ g/m}^3$ ,  $0.3 \text{ g/m}^3$ , etc.

Most common building and furnishing materials have  $LC_{50}$  values substantially higher than  $8 \text{ g/m}^3$  prior to the CO correction. Thus, the toxicity of the smoke will most often be determined by the fire ventilation, rather than the specific products burning.

Further simplification of step 2 is possible. One could perform a single animal test at an FED that corresponds to an  $LC_{50}$  of  $8 \text{ g/m}^3$ . An observation of no deaths would confirm the suggestion. If any animals were to die, then step 3 would be performed.

When the fire community has sufficient experience with  $LC_{50}$  measurements using this approach, some groupings of products could be exempted from further determinations by inspection and placed in the " $LC_{50}$  value greater than  $8 \text{ g/m}^3$ " category. Some possible examples are:

- wood and other cellulose, since all species would be expected to show similar  $LC_{50}$  values;
- synthetic materials containing only C, H, and O;
- polymer/additive mixtures that have been shown to follow the N-Gas Equation (i.e., produce no additional toxicants) and have  $LC_{50}$  values greater than  $8 \text{ g/m}^3$ ;
- products that are only used in small quantities (for this case, a procedure is presented in this report for determining the fractional contributions of concurrently-burning combustibles to the total toxic potency of the smoke); and
- products that would not be expected to become fuel for a flashed-over fire, such as those items only installed behind a sufficiently protective barrier.

Based on an overview of reported toxic potency values, this process could result in an extremely small fraction of commercial products needing to be measured. Note that this statement applies to post-flashover scenarios only.

There will be some cases where it is important to have toxic potency data useful for analysis of pre-flashover fires. For these, the combustion conditions in the radiant apparatus are directly applicable. One would determine the  $LC_{50}$  as above, but not correct it for post-flashover CO. The irradiance of  $50 \text{ kW/m}^2$  for a pre-flashover test is somewhat high, but should have little effect on the  $LC_{50}$ . Lower fluxes can be accommodated if necessary.

The computations in a hazard analysis must account for the fact that the oxygen concentration in post-flashover smoke is significantly depleted, with the amount of depletion depending on the entrainment (outside the fire room) of fresh air into the smoke. This effect could not be simulated in a bench-scale apparatus. By contrast, in the pre-flashover fire, such shortage of oxygen is small.

This study consolidates a number of investigations conducted by NIST over the last several years. Portions have been funded by The Society of the Plastics Industry, Inc., by BFGoodrich, and by The Industry Coalition. There were significant technical contributions from Dr. Arthur F. Grand of the Southwest Research Institute; the Smoke Toxicity Working Group of the National Institute of Building Sciences, chaired by Mr. Henry Roux; and Dr. Jack E. Snell, Ms. Magdalena Navarro, Mr. William H. Twilley, Mr. Emil Braun, and Mr. Ronald McCombs of NIST.





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## Abstract

A comprehensive methodology has been developed for obtaining and using smoke toxicity data for fire hazard analysis. This description of the methodology comprises: determination that the post-flashover fire is the proper focus of smoke inhalation deaths; criteria for a useful bench-scale toxic potency (LC<sub>50</sub>) measurement method; a method which meets these criteria, especially validation against real-scale fires; a computational procedure for correcting the results for the CO levels observed in real-scale post-flashover fires; procedures for reducing the usage of animals and broadening the applicability of data by interpreting gas measurement data using the N-Gas Model; and a procedure for identifying whether a product produces smoke within the ordinary range of toxic potency for post-flashover fires.

**Keywords:** building fires; combustion products; computer fire models; fire deaths; fire hazard analysis; N-gas model; radiant heating; smoke toxicity; toxicity test methods.

## 1 Introduction

The fire statistics of the United States reveal that the majority of persons who die in fires perish due to toxic gas inhalation and not due to burns, generalized trauma (from falling debris or leaping from a window), or other causes [1]. This was not generally recognized until well into this century. Even then, recognition came gradually. In 1933, Ferguson [2] noted that "It has been observed and commented upon that many of these victims are not burned but succumb to the effects of "smoke" and gases. When deaths from this source are reported it is notable that almost never has it been found, specifically, what poisonous gas or gases caused the fatality." He then proceeded to review the literature on chemical measurements of fire gases and attempt to reach some conclusions of general applicability. In 1940, the National Fire Protection Association (NFPA) constituted a committee to investigate this concern in more detail. Their 1952 report [3] examined causes of fire deaths in more detail, and presented data on toxicity of some important fire gases and on which products show tendencies to evolve

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<sup>1</sup> Retired

<sup>2</sup> Guest worker from Building Research Institute, Tsukuba, Japan

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which gases. Such a task was again attempted later at Underwriters Laboratories, where in 1963 Dufour [4] reviewed the by-then much more copious literature. What is striking is that during all this time there was no attempt made to devise a test for the toxicity of fire gases, even though numerous fire tests were being devised for other fire properties.

During the 1970s, there was a very distinct jump in the fire research effort being expended in the United States. One of its first manifestations was a number of proposals for various tests for fire toxicity. Initially, various aspects of toxicity were being examined, such as incapacitation preventing an animal from performing a simple motion. The spectrum of ill effects from toxic substances is large, however, ranging from discomfort or impairment of judgement at one end to lethality at the other. For assessing combustion products, it was eventually agreed that lethality is an unambiguous endpoint which can be examined without undue subjectivity. Thus, combustion toxicity tests have generally focused on measuring *toxic potency* as defined by the  $LC_{50}$ , which is the mass of combustion products needed to cause lethality to 50% of a set of test animals exposed to the smoke for a specified time.

While quite a few tests for combustion toxicity were developed, publicized, and proposed for usage, it is noteworthy that none became adopted by any U.S. standards organizations. Even though such a consensus was not reached, concern became raised in legislative bodies, to the extent that both New York State and New York City, separately, established fire toxicity requirements for building products in the 1980s. Such legislative activity caused significant concern among many in the fire engineering profession, who felt that the groundwork had not been laid for properly interpreting or utilizing the data which were mandated to be collected.

One of the groups showing this concern was the National Institute of Building Sciences (NIBS). NIBS concluded that existing toxicity tests failed to measure properties of products which were needed to competently assess their toxicity behavior in fires. NIBS also affirmed the value of fire hazard assessment, but concluded that an interim methodology was needed while full hazard methods were being developed. Their proposed solution was to be a single, simple bench-scale test, where the results would be an index directly reflecting the toxic fire hazard of the tested product.

At the National Institute of Standards and Technology (NIST), meanwhile, research on this topic had been progressing since 1974 when The Fire Prevention and Control Act established the Fire Research program with the mandate to conduct basic and applied *“research on all aspects of fire with the aim of providing scientific and technical knowledge applicable to the prevention and control of fires.”* The toxicity aspects of this research have been an integral part of the whole NIST program of fire safety research. However, for convenience, we can point to three areas of work.

- (1) The earliest task undertaken was to develop a standard toxic potency test method. A method, commonly referred to as the “cup furnace smoke toxicity method,” was developed at NIST (then called the National Bureau of Standards or NBS) with the help of an ad hoc committee consisting of representatives from government, academia and industry. That work was partially sponsored by the Product Research Committee, which administered a trust fund established in a consent order between the Federal Trade Commission and 25 firms involved in the manufacture and sale of cellular plastics or their components [5]. The final report on this method was published in 1982 [6],[7], and an interlaboratory evaluation of this method by seven laboratories indicating good repeatability and reproducibility was published the following year [8].



- (2) A program was undertaken to assay correctly the toxic potency of a mixture of combustion gases, based on the physiological interactions of a small number of individual gas components. This became known as the N-Gas Model [9],[10],[11].
- (3) The development of computer programs for calculating fire behavior and human response to fire [12] has resulted in a prototype methodology for estimating the hazards to occupants involved in a building fire. The method and available computer software, called HAZARD I, can predict the time-varying environment within a building resulting from a specified fire; the locations and actions of occupants; and the impact of the exposure of each of the occupants to the fire products in terms of whether the occupants successfully escape, are incapacitated, or are killed.

Like other proposed toxic potency tests, the original cup furnace method did not win standards organization approval. Partly this was because the combustion conditions created in the test method were not considered sufficiently representative of conditions occurring in real fires. Partly this was because insufficient evidence was available to show that results of real fires are successfully being predicted. A major reason, however, why no bench-scale test methods were advanced to standards status was because of a significant discomfort within the profession on how their data were to be used.

The present study is the culmination of an effort to (a) provide an improved bench-scale measurement for toxic potency which adequately represents the important combustion conditions of real fires; and (b) provide a design and analysis framework which will allow for the test data to be used in a rational, consistent, appropriate, and adequate way.

## 2 Computations of fire hazard

This chapter addresses definitions of fire hazard, the role of several engineering variables in fire hazard, and methods to predict fire hazard. This background will set the stage for what kinds of data are needed for successful computations of fire hazard and, therefore, will provide guidance for test design.

### 2.1 Quantifying hazard in fires

During the 1970s, knowledge about the toxicity of materials was considered a “missing link” in understanding fire hazard. Thus, a number of tests were developed and proposed in this area, although none have yet been accepted by U.S. standards organizations or by ISO. Nonetheless, methods for measuring the toxic potency of materials started being widely used in the 1980s; their history is reviewed in the next chapter. Use of a number of these tests became common for product evaluation, and one was even adopted for regulatory use [13]. Yet, the data from them could not be treated in a useful engineering way, since a suitably comprehensive analysis methodology was lacking.

One of the earliest milestones in the search for methods to evaluate quantitatively the fire hazard in buildings was a two-day workshop on “Practical Approaches for Smoke Toxicity Hazard Assessment” [14], sponsored by the National Fire Protection Association in February 1984. This workshop convened groups of leading toxicologists, fire protection engineers, fire scientists, fire modelers, and code and fire service representatives to study the problem. Later in 1984, the Toxicity Advisory Committee of NFPA proposed a simple four-step procedure [15] derived from the workshop’s efforts. As the project progressed, papers were published which discussed the evolving philosophy and structure of the hazard assessment methodology [16],[17]. These papers, and the growing questions regarding combustion product toxicity, stimulated some early hazard analyses using both hand-calculated estimates and some of the available computer fire models.

#### 2.1.1 Hand calculations

In May of 1984, the Toxicity Advisory Committee of the National Fire Protection Association published a procedure for providing “order of magnitude estimates” of the toxic hazards of smoke for specified situations [18]. In this report, Bukowski based the estimating procedure on a series of algebraic equations, which could be solved on a hand calculator. Individual equations were provided to estimate steady-state values for such parameters as upper-layer temperature, smoke density, and toxicity; and graphical solutions were provided for room filling time. This work was followed by the more extensive compilation of such equations for use by the U.S. Navy in assessing fire hazards on ships [19]. Subsequently, the Toxicity Advisory Committee was asked by the National Electrical Code Committee for assistance in addressing a toxicity hazard question regarding polytetrafluoroethylene (PTFE) plenum cables. In providing that help, a hand-calculated analysis was performed [20]. This paper concluded for a single, specified scenario, that the size of room fire needed to cause the decomposition of the cable insulation would itself cause a toxicity hazard in an adjacent space before the cable would become involved.



In general, such algebraic equations are constructed for single fire types and conditions. They are also limited to steady-state analyses and cannot deal consistently with the transient aspects of fire behavior. To obtain a complete answer of broad applicability, then, requires a computer to solve the differential equations which describe these transient phenomena. This is the role of computer fire models.

### 2.1.2 Computer models

The models of building fires that are currently available vary considerably in scope, complexity, and purpose. Simple “room filling” models such as the Available Safe Egress Time (ASET) model [21] run quickly on almost any computer, and provide good estimates of a limited number of parameters of interest for a fire in a single compartment. A special-purpose model can provide a single function; *e.g.*, COMPF2 [22] calculates post-flashover room temperatures. And, very detailed models like the HARVARD 5 code [23] predict the burning behavior of multiple items in a room, along with the time-dependent conditions therein.

In addition to the single-room models mentioned above, there are a smaller number of multi-room models which have been developed. These include the BRI (or Tanaka) transport model [24] which is similar to the FAST model [25], and the HARVARD 6 code [26], a multi-room version of HARVARD 5. All of these models are of the zone (or control volume) type. They assume that the buoyancy of the hot gases causes them to stratify into two layers: a hot, smokey upper layer and a cooler lower layer. With limitations, experiments have shown this to be an appropriate approximation [27], [28].

Other types of models include *network models* and *field models*. The former use one control volume per room and are used to predict conditions far removed from the fire room, in spaces where temperatures are near ambient and this layering does not take place. The field model goes to the other extreme, dividing the room into hundreds or even thousands of control volumes. Such models can predict the variation in conditions within the layers, but require long run times on supercomputers to do so. Thus, they are used sparingly, when highly detailed calculations are essential.

Thus, we can immediately see that two alternative methods for assessing fire hazard have emerged:

- simple, closed-form equations, generally not requiring a computer for solving (hand calculations); and
- numerical computations of a fire where a large number of equations, often differential equations, are being solved simultaneously (computer fire models).

For a reference to all of the commonly used computer fire models, the reader is referred to Friedman’s recent compilation [29]. Reference [30] reviews several of both the simple computational methods and the computer fire models and gives further references to example hazard analyses which have been conducted by using these tools.

## 2.2 Definitions of terms

At this point it is appropriate to define the most important terms used throughout this study.

*Toxic potency* — toxicity of the smoke from a specimen of material or product, taken on a per-unit-specimen-mass basis. A typical biological end point adopted is the “LC<sub>50</sub>,” which stands for the concentration which will be lethal to 50% of the exposed specified test animals in a specified time period. The units are in concentration, i.e., g·m<sup>-3</sup>. It must be emphasized, however, that **toxic potency is not an inherent property of a material** [31]. The LC<sub>50</sub> variable must be accompanied by an indication of the exposure time but, by itself, does not specify the test apparatus nor the specimen heating conditions. The length of the test animal exposure time is particularly important, since for many substances the value of the LC<sub>50</sub> is inversely dependent on the exposure time. For fire toxicity, a 30-minute exposure time (along with a 14-day post-exposure observation period) has often been specified. Note that a lower LC<sub>50</sub> value indicates a higher toxic potency.

*Fire hazard* — generally, this is taken to be a measure of the seriousness of the exposure conditions which threaten the physical well-being of the occupant. The hazard may come from various sources, for example, smoke inhalation, direct flame burn, injuries due to trauma (*e.g.*, ceiling collapse) or high temperatures, or inability to escape due to lack of visibility or the presence of acid gases which affect the eyes.

*Toxic fire hazard* — this term is a subset of “fire hazard,” occurring when the hazard being considered is due to inhalation of toxic combustion products alone.

The physical well-being of the occupants can be threatened in a number of ways, but two are severe: lethality and incapacitation. Fire hazard models incorporate values for incapacitation based on toxicity, temperature rise, heat flux, or other criteria. These can be very useful in trying to understand certain fire phenomena. However, incapacitation of humans is very difficult to predict based on animal exposure results. This is due both to the physiological differences between humans and test animals and the use of simple animal measurements to represent a diversity of human activities during a fire (*e.g.*, sleeping, running). Clearly, we also lack a *laboratory* model for the incapacitation of humans. Human lethality, on the other hand, while in some cases is still difficult to assess from animal data, is less ambiguous. Thus, while acknowledging the significant importance of incapacitation of occupants, it will not be explicitly quantified in this study; instead, we shall quantify only lethality.

## 2.3 Fire scenarios and toxic potency data

To compute the course of a fire and its impact on occupants, it is necessary to specify the details of the combustibles, the environment, and the type of ignition. This information, collectively, is known as the *fire scenario*. In many cases, fire histories are discussed where the scenario is tacitly implicit and is not spelled out. Such implicitness can be very misleading. To reach agreement on how products perform or how building occupants are or are not threatened by fire, the fire scenario being examined must be explicitly described.



Today's computer-based fire models [29] have the ability to incorporate full definition of the fire scenario. This can lead to determination of the course of a variety of enclosure fires, including the resulting thermal and (in some cases) toxicological environment. HAZARD I [12] additionally includes the response of occupants to residential fires. This enables the additional determination of the impact of the fire on people.

Fire hazard modeling, then, allows for differentiating among the complex, but realistic, performance of competitive products [32]. Consider, for example, the following situation. Product A produces smoke of moderate toxic potency throughout a fire. A hazard analysis of the chosen scenario shows that deaths occur only late in the fire. The alternative product B produces smoke of high toxic potency mainly at the beginning of the fire, resulting in fewer total deaths, but ones that occur soon after ignition. The computation can identify the early warning times and prompt evacuation rates that would make product A the better choice. For further examples of the use of fire hazard modeling, the reader is referred to the example cases in [33].

While the time-varying characteristic of a fire can only be computed by the use of a computer, one can use a simpler fire representation to obtain insight into toxic fire hazard and the needed product data for its computation. Babrauskas has developed such an expression for a fire where the spread of flame is symmetrically away from an ignition point, with all room boundaries being very far away [34]. He derived the following expression for use in estimating the relative contributions to toxic hazard for two products used in the same application:

$$FED \propto \frac{MLR}{t_{ig} \times LC_{50}} \quad (1)$$

MLR is the *averaged mass loss rate* from the time that 10% of the mass loss occurred to the time that 90% has occurred.

$t_{ig}$  is an indicator of the rate at which the product's burning area is increasing. If one conceives flame spread as a continuous series of ignitions, then the shorter the ignition delay time, the faster the flame spread rate.

FED is the *Fractional effective Exposure Dose* [35],[36]. It is the time-integrated concentration (C) of smoke encountered by the occupant during the course of the fire, normalized by the product of the  $LC_{50}$  and the exposure time used in its determination. This dimensionless number equals 0 at the start of the fire and 1 at the time that the occupant has received a lethal dose of the smoke.

A similar analysis can be performed for the situation where the fire is no longer spreading, i.e., when all the combustibles in the room are already fully burning. This is the situation after the fire has passed the point of flashover. The resulting equation is:

$$FED \propto \frac{[\dot{m}_1''] [A_1]}{LC_{50}(1)} + \frac{[\dot{m}_2''] [A_2]}{LC_{50}(2)} + \dots \quad (2)$$

where:

the  $A_n$  values are the areas of the combustibles covered by flames,

the  $\dot{m}''$  values are the mass loss rates per unit area of the combustibles, and

the  $LC_{50}$  values are as defined before.

The latter equation has the additional value of enabling an estimate of the relative contributions of multiple products to the FED and thus the toxic hazard.

This simplified presentation indicates the types of data needed for including smoke toxicity in a fire hazard analysis. The model accepts a dimensional definition of the combustibles and accepts or generates their burning history. The time-dependent mass loss data give the quantity of smoke formed. The toxic potency figure, characterized by the  $LC_{50}$  and integrated as the FED, indicates the severity of that smoke.

### 3 Types of fires

An apparatus for fire property measurement, such as the toxic potency device to be described in Chapter 8, has some degree of versatility. Proper selection of combustion conditions can replicate more than one fire type. This Chapter considers the relative importance of smoke toxicity in the hazard from different fire types. This will lead to optimal use of the ensuing methodology.

The characteristics of unwanted fires can be almost endlessly diverse. Yet, while various fire types can occur, they are not at all equally represented in national fire death statistics. Based on these statistics, we can identify the real fires in which smoke toxicity is most critical.

There are various ways in which fire types can be grouped. The important consideration is to start with a list which includes **all** potential fire scenarios; the way that this entirety is then subdivided becomes of lesser concern. The British Standards Institution (BSI) has developed a combustion-characterized outline, which has gained a substantial degree of international acceptance. In their Code of Practice for the Assessment of Toxic Hazards in Fire in Buildings and Transport [37] they itemize the following six types of fires:

- I. Self-sustained smoldering decomposition (i.e., a cigarette on upholstered furniture or bedding).
- II. Non-flaming oxidative decomposition.
- III. Non-flaming pyrolytic decomposition.
- IV. Developing fires, flaming (pre-flashover fires).
- V. Fully-developed fires, high ventilation (post-flashover fuel-controlled fires).
- VI. Fully-developed fires, low ventilation (post-flashover ventilation-controlled fires).

The BSI Code is limited to evaluation of buildings and transport vehicles. While the problems may be similar to certain industrial or other protection needs, we will here likewise limit ourselves to the same scope. Thus, explosions, fires in mines, and similar issues will not be addressed.

In the United States, the largest single cause of fire deaths (27% of the annual total) is due to cigarettes and other smoking materials ignitions [38]. Such a fire starts out as type I, although it may proceed to the flaming stages. That the bulk of the smoke is produced *after* the furniture item bursts into flames has been reinforced by results of animal exposures in large-scale fire tests [39],[40]. These indicate that only after the smoldering goes to flaming do animal deaths tend to occur. Thus, simulation of smoldering combustion is not a priority for a toxic potency measurement method.

Fire types II and III are considered rare in terms of fire fatalities and generally not of top importance as far as public concern goes. The scenarios which could include such a fire type would be overheated electric wiring (but without accompanying flaming) or overheated combustibles placed near heating appliances (again, without flaming). The statistics do not lend themselves readily to identifying these fire types, but part of the reason is that the systems themselves which are used for collecting fire statistics were set up primarily to obtain details on the more likely scenarios. In some applications, possibly



industrial occupancies, concerns with type II and III fires may become important, but this is not likely to be true for general applications.

What remains to consider are types IV, V, and VI. For considering these open-flame fire types, we first wish to clarify the terms used.

Pre-flashover fires generally show:

- primarily radiant heating, with heat fluxes ranging from 20 to 50 kW·m<sup>-2</sup> near the ignition source, to being negligible further away
- only one item or a small number of items on fire
- combustion air not vitiated.

Post-flashover fires are distinguished by, among other things:

- primarily radiant heating, with heat fluxes from about 20 to 150 kW·m<sup>-2</sup> all over the room
- many items simultaneously on fire
- vitiated combustion air for some, but not all, burning items.

The transition between fire type V and VI occurs when the amount of fuel being gasified becomes great enough that all of the pyrolysate cannot burn within the room of fire origin. Thus, in a type VI fire, considerable burning also occurs outside, at doors, windows, or other openings. The distinction between types V and VI may be made as a simple either/or choice for certain types of fire models. For examining the toxicity aspects, however, as we shall see later, the exact fuel/oxygen ratio needs to be known, not just a bipartite split. Thus, we will consider V + VI as the post-flashover fires of interest.

What is still important to decide, however, is whether pre-flashover (IV) or post-flashover (V/VI) fires should be where the focus of standard toxic potency measurement lies. In this case, U.S. fire death statistics can be consulted. The statistics are not tabulated according to “pre-flashover” or “post-flashover,” but they do include an equivalent concept. The factor analyzed for is “flame damage beyond the room.” Such flame damage does not occur if the fire does not progress beyond the pre-flashover stage, but does occur if flashover is reached and burning continues. The U.S. fire statistics [41],[42] show that 69% of all fire deaths are associated with post-flashover fires, with the vast majority of deaths occurring outside the room of fire origin.

Clearly, the post-flashover fires are the most important problem to be addressed. One question remains, however. For pre-flashover fires, is toxicity an important issue? From the fire statistics, we see that for pre-flashover fires, most of the deaths occur in the room of fire origin. Moreover, computer-based hazard modeling can provide an estimate of the importance of toxicity relative to thermal effects for this scenario.

Figure 1. shows a comparison of the estimated toxicity and thermal hazards in the lower layer of the room of fire origin for a range of fire growth rates. (The results are similar for the upper layer.) To understand the impact of the comparison, a definition of tenability limits for temperature, radiant flux, and toxic gases must first be introduced:

- The effects of temperature as an exposure limit under fire conditions have not been well studied. Industrial hygiene literature primarily gives data for heat stress under conditions of prolonged, typically 8-hour, exposures. The older literature, as it relates to fire, has been reviewed by

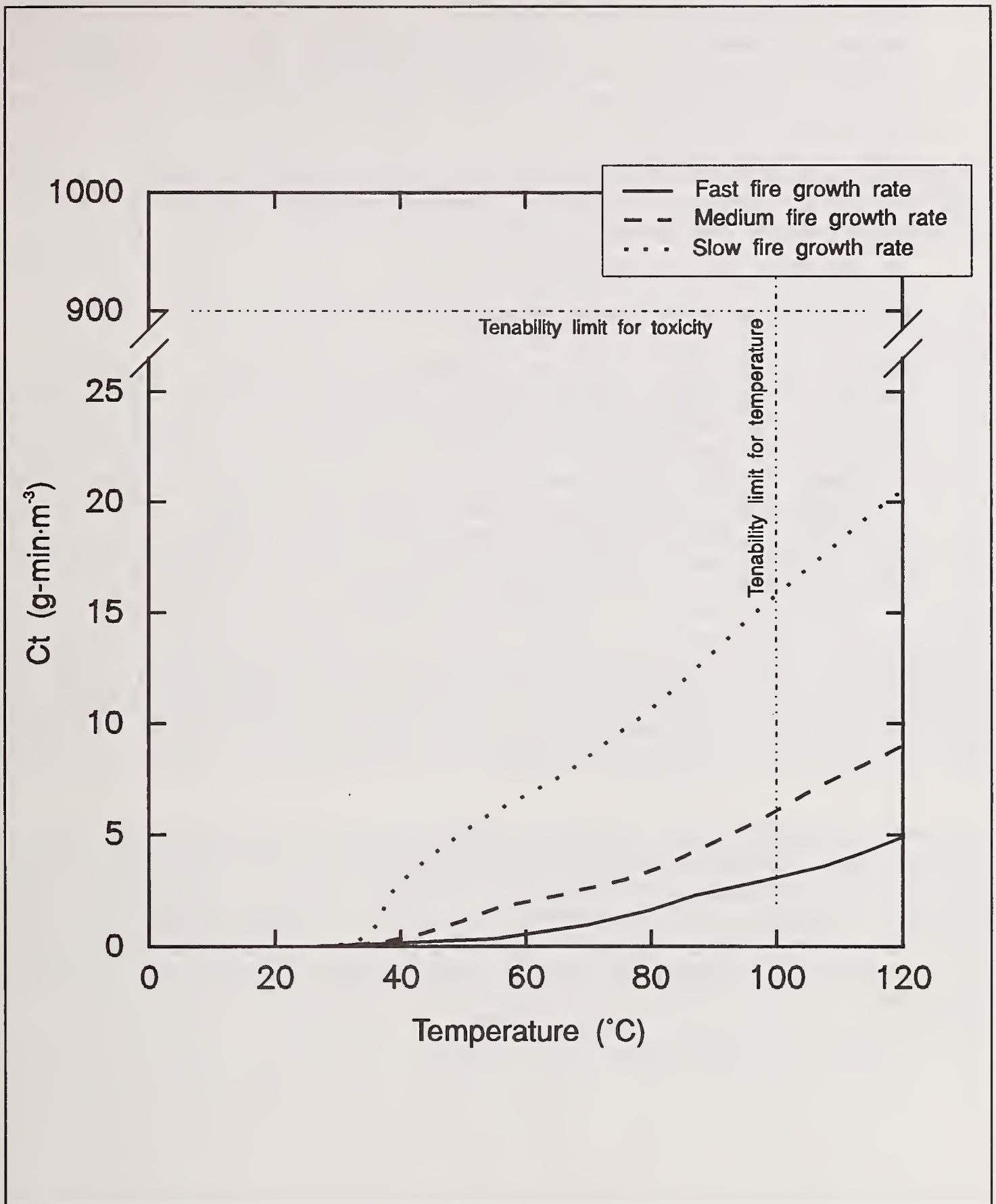


Figure 1. Relative impact of toxicity and thermal effects in the room of fire origin for a range of fire growth rates.

Simms and Hinkley [43], although, based on that review, they could not make any recommendations of tenability values. Criteria for temperature are, in fact, especially difficult to set, since the temperature at which adverse effects are noted depends not only on the exposure time, but also on additional factors such as the relative humidity and the interactions of heat and toxic gases. Experimental data from studies with pigs have shown no injuries at 120°C for 2 min, 100°C for 5 min, and 90°C for 10 min [44], [45]. Some experimental data for humans have been reported which show that temperatures of 100°C could be withstood by a clothed, inactive adult male for about 30 min before intolerable discomfort is reached; a 75°C exposure could be withstood for about 60 min [46]. These experimental values seem high. Zapp [47] has stated that "...air temperatures as high as 100°C can be tolerated only under very special conditions (i.e., still air) for more than a few min, and that some people are incapacitated by breathing air at 65°C..." The following comparisons are based on the 100°C limit.

As noted earlier, the effects of toxic gases have been studied in depth by many researchers. One indicator of toxicity that has been used in hazard calculations is the parameter  $Ct$ . This parameter represents the time-integrated exposure to the mass concentration of all of the mass of fuel lost within the structure and is thus a concentration-time product (hence the name  $Ct$ ). The units are gram-minutes per cubic meter. A value of  $900 \text{ g}\cdot\text{min}\cdot\text{m}^{-3}$  has been proposed as a reference value for the lethality of smoke from most common building materials [7],[33]. The value for  $Ct$  of  $900 \text{ g}\cdot\text{min}\cdot\text{m}^{-3}$  comes from a constant 30-min exposure to smoke of typical toxicity (in these studies,  $LC_{50}$  concentrations of approximately  $30 \text{ g}\cdot\text{m}^{-3}$ ). Where materials more or less toxic are considered, this reference value can be varied accordingly (e.g., by factors of 10).

As Figure 1. shows, the tenability limit for temperature (100°C) is reached long before the typical limit for toxicity ( $900 \text{ g}\cdot\text{min}\cdot\text{m}^{-3}$ ) is approached. At the temperature limit,  $Ct$  levels below  $20 \text{ g}\cdot\text{min}\cdot\text{m}^{-3}$  are seen for a broad range of fire growth rates. To put these in perspective, one must consider the concentrations necessary to cause lethal effects at such low  $Ct$  levels. Presuming, for the moment, the same linear relationship for  $Ct$ , then the  $LC_{50}$  concentrations necessary to cause lethality are simply the  $Ct$  value divided by the time to reach that value.

Over the time period necessary to reach the lethal level from temperature, an equal life safety threat due to smoke toxicity would require an  $LC_{50}$  value of approximately  $0.1 \text{ g}\cdot\text{m}^{-3}$ . This is over 100 times smaller than those found for typical building materials; virtually no commercial products exposed under realistic fire conditions have  $LC_{50}$  values so low. Thus, for well-mixed smoke within the room of fire origin, toxic hazard is much less frequently a threat than is thermal hazard. In this context, the value of a toxic potency measurement method is the identification of those products that produce smoke of extreme toxic potency.



## 4 Toxic potency measurements

In this chapter we examine briefly the history of development of bench-scale measurement methods for toxic potency. We then consider the current needs of the voluntary standards organizations. We find, in examining this situation, that a test development effort is warranted, despite many years of earlier such efforts.

The impelling need for a small-scale laboratory procedure to ascertain the toxic potency of the combustion products from materials was revealed by a scientific paper in *Science* in 1975 [48]. This research by Petajan et al. showed that the combustion products from an experimental fire-retarded rigid polyurethane foam caused grand mal seizures and death in rats, while the same foam without the fire-retardant did not produce any abnormal neurological effects. The toxicity of the combustion products from the fire-retarded foam was attributed to the formation of a bicyclic phosphate ester, namely, 4-ethyl-1-phospha-2,6,7-trioxabicyclo[2,2,2]octane-1-oxide, in the smoke. This result raised an alarm about the possible presence of “supertoxicants” in smoke from burning or smoldering materials. Since the presence of this bicyclic phosphate ester would not have been detected by ordinary chemical analysis of the smoke, this paper also emphasized the need for animals as the measurement “instruments.” Many laboratories had pursued the chemical approach and had published extensive lists of chemical compounds found in the combustion atmospheres of materials thermally decomposed under different conditions. A summary of the literature on the combustion products and smoke toxicity from seven plastics indicated over 400 detected compounds [49]. Since the toxicity of all of those compounds was not known nor was the toxicity of the mixed atmospheres known, the need for a combined biological and chemical approach was obvious. The observation of adverse effects in rodents would indicate the presence of unusual toxicants or synergistic effects of combined toxicants that might not be discovered by routine chemical analysis alone.

World-wide concern about the toxicity of combustion products was indicated by the many laboratories which developed smoke toxicity test methods in the next decade. At least 20 such methods were described in 1983 [50]. At about the same time, 13 published methods were evaluated by Arthur D. Little, Inc., to assess the feasibility of incorporating combustion toxicity requirements into building material and furnishing codes of New York State [51]. On the basis of seven different criteria, only two methods — the closed-system cup furnace smoke toxicity method developed at the National Bureau of Standards and the flow-through smoke toxicity method developed at the University of Pittsburgh — were found acceptable. The state of New York decided to use the method (“UPitt”) developed at the University of Pittsburgh [13]. Since it was unclear how to use the results of toxicity testing in regulation, the state of New York only requires that materials be examined with the UPitt protocol and that the results be filed with the state.

In separate regulation, New York City has also adopted toxicity requirements as part of their building code. They require that products not be more toxic than wood<sup>3</sup>. Since wood is not a product of specific composition or fire behavior, New York City uses an “average” wood, corresponding to the LC<sub>50</sub>s of several different species tested in the UPitt method and then averaged. A number of other states also announced their intentions to regulate in this area; however, this has not yet come about.

Four smoke toxicity measurement procedures were eventually proposed to the American Society for Testing and Materials (ASTM). These included the cup furnace method, the UPitt method, and two others which were somewhat less commonly used. These were the University of San Francisco “Dome Chamber” test [52] and the original radiant heat test, developed at Weyerhaeuser [53]. None of the four proposed methods were accepted; and, at present, there is no ASTM standard smoke toxicity test method.

The latter two were not accepted because they enjoyed only limited use by laboratories. With the Dome Chamber, serious toxicological reservations were raised about a method which only measures *time* to various incapacitation effects (such as collapse) or to death, and does not evaluate actual product toxic potency. The Weyerhaeuser test was, simply, rarely used, largely because certain mechanical aspects were felt lacking in robustness.

Both the cup furnace and the UPitt methods had achieved rather widespread use in the United States, yet certain reservations remained. Primary issues were that neither method was felt to represent adequately the combustion environment occurring in actual building fires. Also, it was felt that data validating the results of these tests against real-scale fires were scant. Although these test methods were never standardized, it is said that a number of products were changed or withdrawn because of their test results. As more materials were examined in these systems, it became evident that the number of products generating “supertoxics” was small. Indeed, most of the toxicity of combustion atmospheres could be explained by the main toxic combustion gases (e.g., CO, CO<sub>2</sub>, HCN, reduced O<sub>2</sub>, HCl), and that one rarely had to worry about minor or obscure components [11],[36],[54],[55].

There has also been significant discussion concerning the potential misuse of toxicological data. The concern was that if **any** method for obtaining toxic potency data alone were approved, it might become a new determinant for the acceptability of products. As a result, a view emerged that a method worthy of approval should have the following attributes:

- the combustion conditions would appropriately represent real-scale fires, and the method could be validated to demonstrate its success in predicting the real-scale fire; and
- a technique was in place, as part of the proposed method or separately, for assembling enough needed data so that a credible fire hazard assessment could be made.

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<sup>3</sup> Chapter 7 examines some details of wood combustion. It will be seen that wood is about the least desirable choice for a reference material. The two main reasons for this are: (1) wood toxicity is dominated by CO production, which is extremely test- and protocol-dependent and is badly represented in all bench-scale methods unless special computational corrective measures are taken. (2) The repeatability of tests on wood, due to natural lumber variability, is much poorer than for most other materials.



To satisfy these two criteria above, development of three new methods was pursued. Professor Alarie at the University of Pittsburgh undertook to design “UPitt II,” which would use the well-validated combustion system of the Cone Calorimeter, instead of the box furnace used in the older UPitt test. The resulting method has been recently published [56]. The method is costly and difficult to install and has some of the same operational difficulties encountered as were earlier encountered by NIST in an exploratory study on an attempted coupling of a conical-heater type of combustion system to the animal exposure system used with the cup furnace method [57]. Partly because of these reasons, the fire safety community has not shown interest in this development.

The second solution was proposed by NIBS when it established a project on combustion toxicity in 1982. After a 1986 conference [58] suggested the need for a “performance test method” for combustion toxicity, NIBS commissioned test development work to be conducted by the Southwest Research Institute (SwRI). The fundamental principles of the method were described in the 1988 NIBS conference [59], [60]; and, after some further development work and public comment, the method was submitted to ballot at ASTM in March 1991. Since the NIBS method formed the starting point for the development work at NIST on a new measurement method, some features of this apparatus are examined more closely in Chapter 8.

The third solution is the protocol being described in the present study.

## 5 Criteria for bench-scale toxic potency measurement

A rational measurement method development program must be guided by sound, comprehensive, and well-accepted criteria for what is a satisfactory measurement method. This methodology is built on what was learned through numerous discussions on such occasions as ASTM, NFPA, or NIBS committee meetings.

The measurement method presented here addresses the concerns noted above. In particular, the method is directed at:

- (a) obtaining LC<sub>50</sub> values where combustion condition realism, representation of real-scale fire, and similar requirements could be met; and
- (b) being an integral part of a fire hazard assessment method. Thus, means had to be in place for supplying **all** of the needed information, not just the LC<sub>50</sub> value alone.

The method development followed the guidelines that have become generally accepted by the profession. At this point presenting them serves to outline why the development progressed in the manner it did.

As reasoned in Chapter 3, the community (as evidenced in the NIBS meetings) considers that the primary application of toxicity data is for assessing the smoke hazard in post-flashover fires. However, since pre-flashover fires are also of interest, it was desirable to explore the same physical apparatus for representing those fires, provided this did not compromise the primary use.

### 5.1 Types of data to be obtained

Various fire hazard assessments can have differing toxicological data needs, depending on the exact situation that is being examined. The following is a minimum data set (see Chapter 2) for representing even the simplest fire conditions.

- A. Toxic potency should be measured, reportable in correct concentration (e.g., g·m<sup>-3</sup>) units.
- B. The chemical data necessary for the N-Gas Model should be properly obtainable from the measurement method. We shall review the N-Gas Model in the next chapter and demonstrate how its use produces a measurement method which is simple to conduct and which minimizes the usage of experimental animals.

## 5.2 General measurement method requirements

The following is a list of the general requirements which are expected of any viable engineering measurement method. These are presented for background and need not be elaborated.

- A. Adequate repeatability.
- B. Adequate reproducibility.
- C. Adequate validity.
- D. Safety to operator.
- E. Safety to environment, i.e., no excessive pollution.
- F. Affordable apparatus costs.
- G. Tests conductible reasonably quickly and efficiently.
- H. Sample preparation not excessively difficult.
- I. Ease of cleaning and maintaining of the apparatus.

## 5.3 Specific measurement method requirements

A more direct set of requirements combines the above with state-of-the art understanding of fire physics and chemistry and of inhalation toxicology.

- A. The measurement method should represent the chosen full-scale combustion scenario correctly.

Adequate repeatability and reproducibility can be tested by standard statistical methods and require no elaboration. Validity, however, is a different issue. While it can be tested statistically, the hopes of success are small, unless accurate knowledge of physics, chemistry and toxicology are incorporated into its design. For this reason, there are some important corollaries:

1. Composite specimens should be testable as composites.  
Normally, the burning behavior of composites cannot be predicted from information about the constituents alone, and neither can the toxic potency. In a few cases, efforts have been made to compute the toxic potency of the composite from measurements on the components [61],[62]; however, this is not viable in the general case.
2. Since in the post-flashover fire, radiant heating predominates, the specimen should receive uniform, well-controlled radiant heating.  
In practice it has proven to be impossible to relate adequately the results from combustion under conditions of predominantly convective, conductive, or thermal immersion heating to those from radiant heating tests. Face uniformity is needed since calculations or modeling on the basis of the average radiant heat flux are not possible if there are wide variations in heating over the face of the specimen.



3. Specimens should be burned to their natural conclusion in much the same way they would in real-scale fires; i.e., a specimen should not artificially be stopped from burning before all the combustibles that can burn do burn.

At most times after ignition in the real-scale fire, some surface elements will be barely ignited, others will be half-burned-through, while others yet will be nearly burned through. This situation holds true in the pre-flashover fire relatively soon after ignition is established, and remains true until the late stages of the post-flashover fire. Any bench-scale measurement method procedure which captures products of combustion only from the surface layer of the specimen, or the back layer, etc., will not represent post-flashover fire scenarios, nor most other useful fire scenarios.
4. For establishing the correlation of the bench-scale result to the full scale by chemical analysis, the specimen's yields of various toxic gases species must be measurable.

This is essential, since it is one of the primary ways of comparing the full-scale to the bench-scale result.
5. For establishing the correlation of the bench-scale result to the full scale by bioassay, both the LC<sub>50</sub>s (or an approximation thereto) and the causes of animal deaths need to be measured and recorded.

Again, if this information is not available, validation efforts become compromised.
6. There must be a minimum loss of gases and particulates.

It will be impossible to represent desired real-scale fire scenarios if the losses in the bench-scale measurement method are high, erratic, or subject to anomalies.
7. Specimens should be tested without crushing, powdering, etc.

To achieve a good representation of the real-scale behavior, specimens should be presented with similar thermal boundary conditions and internal heat/mass transfer conditions. Both of those will not be possible if the form of the specimen is altered.
8. Specimens of a wide range of densities, thicknesses and toxicities which may occur in the real world should be testable without needing to be excluded or "beating" the test.

A practical measurement method will, perforce, have limits for these variables, but they should be as broad as possible and include the range of interest for hazard assessment. Due to the nature of commercial composites, products of at least 25 mm thickness (or 50 mm, in the case of upholstered furniture specimens) must be accommodated.
9. Protective outer layers should be realistically represented in the measurement method procedure.

Some composites are designed whereby skin layers will protect internal layers which may be more flammable or more toxic. These systems should be subjected to realistic testing; the outer layers should be allowed to give about as much protective value as they do in real-scale performance.

10. Edge effects should not influence the results disproportionately.  
This issue is closely related to the previous one. The specimen testing arrangement should be such that products which burn primarily from the top-down in real-scale should not burn from the sides-in in the bench-scale measurement method.
11. Samples should be tested in the horizontal, face-up orientation.  
Samples tested in any other orientation are prone to melt and drip or to fall out of the holder during testing.
12. The combustion environment to which the specimen is subjected in the measurement method should correspond to that in the design scenario. This includes correct oxygen levels, and also the absence of such phenomena as re-circulation or re-combustion of combustion products, catalytic conversion, etc., unless they are also present in the design fire scenario.  
As will be seen in Chapter 7, this condition will generally be impossible for any bench-scale measurement method to meet in its entirety. In such a case, alternative provisions need to be made to correct the results for known biases.
13. Since the measurement method is to be designed for, at least, post-flashover fires, it is important that the test data be in such a format so that the prediction of several items simultaneously burning in a room could be done.  
This was discussed in Chapter 2.

B. The measurement method should provide for a well-characterized, toxicologically sound exposure of animals.

1. There is a very broad consensus pointing to the wisdom of providing animals with a 30-min exposure period, followed by a 14-day post-exposure observation period [6]. The 14-day post-exposure period is necessary to identify those materials that produce combustion products which cause metabolic or physiologic effects following the exposure (e.g., HCl and other irritant gases).
2. The rat has been chosen in the largest number of combustion toxicity measurement methods. There are cogent reasons (reasonable cost and availability; extensive amount of historical data; large enough to provide for blood samples; closest overall model to human response among animals of similar cost) for continuing with this practice.
3. The gases to which the animals are exposed should consist of the total combustion products from the specimen's burning history.
4. As close to a square-wave exposure as possible is desired.  
The evaluation of LC<sub>50</sub>s is accompanied by increased uncertainty if the gas concentrations are significantly changing during the course of the exposure. The time during which the combustion gases in the animal chamber are present at a

level near their average integrated level should be as close as possible to the average value during the entire 30-min period.

5. The biological effects on the animals' condition during the measurement method should be adversely affected as little as possible by causes other than specimen toxicity.

This includes: providing a sufficient size of animal exposure chamber so that the animals' exhaled CO<sub>2</sub> does not affect them adversely; making sure that heating conditions from specimen heaters do not create an excessive heat burden to the animals; providing a restraint system that does not cause undue physical injury.

6. The usage of animals should be minimized, consistent with obtaining data of acceptable quality.

There are two reasons for this: (a) humanitarian concerns; and (b) the costliness and time-consuming nature of animal experiments.

The objective of the measurement method development work was then to ensure that all of the requirements were met as well as possible. Before examining the results of the development program, we shall next look at the details of the N-Gas Model, then consider in some detail the issues associated with CO toxicity.



## 6 The N-Gas Model

To minimize the cost and time for conducting tests, while at the same time providing the maximum amount of information valuable in fire hazard computations, NIST has been developing a concept which has come to be known as the N-Gas Model. In this chapter we explain how this model was developed and how it is applied in testing.

Experience with earlier toxicity tests indicated that the cost of doing them was invariably high. The main reason for this was that the toxic potency had to be determined by a trial-and-error process, where each of 4-10 trials involved using a number of animals (often 6). Over the last 8 years, a significant effort was mounted at NIST to develop an alternative approach whereby emphasis would be placed on toxicological interactions of mixtures of gases. Thus, usage of animals, while not entirely eliminated, could be substantially reduced. This became known as the N-Gas Model. Its use forms an integral part of the new procedures which we present in a later chapter. Thus, at this point, as prerequisite background, we will describe the N-Gas Model and discuss the quality of predictive results that can be obtained by its use.

The N-Gas Model has been developed using rats. As such, its use in hazard modeling depends on the quality of its correlation with human data, and verifiable lethal inhalation data for humans are not available. Nonetheless, an effort to approximate this relationship is underway. Figure 2. shows that the equilibrium carboxyhemoglobin (COHb) values for humans and rats are quite comparable over a considerable range of exposures to carbon monoxide [63]. As will be demonstrated in Chapter 7, carbon monoxide is the principal toxicant in most smokes, and COHb formation is the mechanism by which the body is deprived of oxygen, leading to death. Thus, this similarity is encouraging, and the rat toxicity data can be used to simulate human toxicity, albeit with caution. Further work on the kinetics of the uptake and unloading of toxicants is also underway and will be described in a separate publication.

### 6.1 Basis of the model

The N-Gas Model is based on the now well-established hypothesis that a small number ("N") of gases in the smoke accounts for a large percentage of the observed toxic potency [9], [10], [11], [36], [54], [64], [65], [66], [67], [68]. The lethality of each of these gases is determined for laboratory animals, e.g., rats. Similar measurements for combinations of these gases tell whether the gases are additive, synergistic, or antagonistic. The results of these mixed gas tests have been reduced to an algebraic equation which has been empirically determined for the exposure of rats to mixtures of CO, CO<sub>2</sub>, HCN, reduced O<sub>2</sub> and HCl. Examination of these results in both bench-scale and larger-scale tests have shown that the predictability of the N-Gas Model is good [69].

The concept that simple additivity may be sufficient to explain the toxicity of mixtures of fire gases was originally proposed by Tsuchiya and Sumi [70]. Significant work was also done in this area by Hartzell [35], who proposed that the term "Fractional Effective Dose" is suitable for naming the variable which quantifies what fraction of a lethal dose the animal has received. Huggett [71], however,

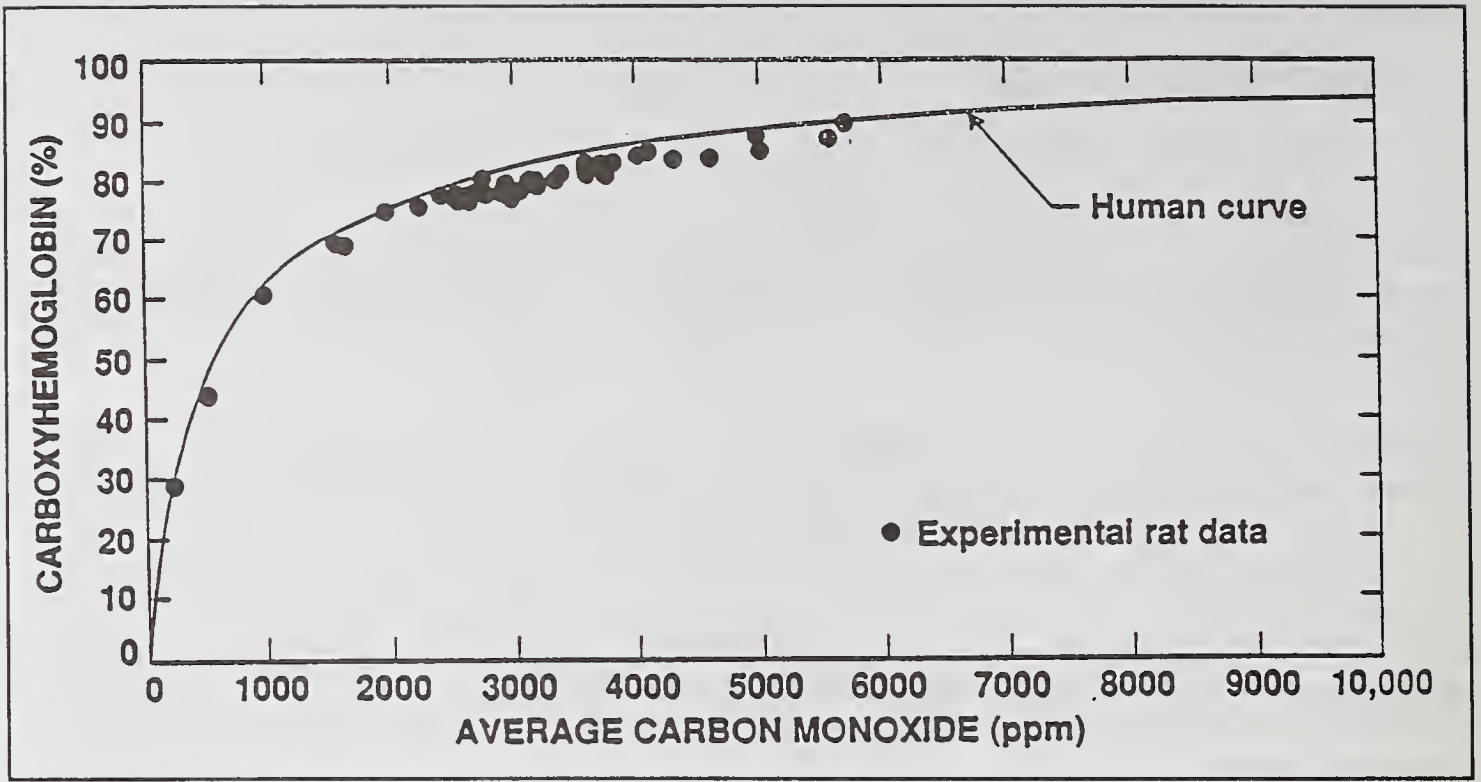


Figure 2. Comparison of human curve and rat data for carboxyhemoglobin equilibrium levels for different CO exposures.

pointed out that the *actual* dose delivered to an animal via inhalation cannot normally be quantified; instead, we may consider an *exposure* dose, which is defined as the product of the gas concentration in the atmosphere, multiplied by the time of the exposure. Thus, we more appropriately refer to the Fractional effective Exposure Dose (FED). Thus, for the case of simple additivity, the FED can be defined as

$$FED = \sum_i \frac{\int_0^t C_i dt}{LCt_{50}(i)} \quad (3)$$

where  $C_i$  is the concentration of the  $i^{\text{th}}$  gas species, and  $LCt_{50}(i)$  is the lethal concentration  $\times$  time product for that gas species. In quite a few practical cases the time of exposure is fixed and uniform, while the concentrations vary slightly or not at all. In such a case, the simplification can be made to

$$FED = \sum_i \frac{C_i}{LC_{50}(i)} \quad (4)$$



Experimental work has borne out that mixtures of the important toxic gases follow the above relationship generally, but with some modifications. The most current version of the equation is as follows:

$$FED = \frac{m[CO]}{[CO_2]^{-b}} + \frac{[HCN]}{LC_{50}(HCN)} + \frac{21 - [O_2]}{21 - LC_{50}(O_2)} + \frac{[HCl]}{LC_{50}(HCl)} + \frac{[HBr]}{LC_{50}(HBr)} \quad (5)$$

where the numbers in brackets indicate the actual atmospheric concentrations of the gases, and the constants to be discussed below are for deaths within the 30-minute exposure + 14-day post-exposure period. Note that two terms differ from the form of equation 12.

The first term reflects the potentiation of CO by CO<sub>2</sub> [66]. Studies at NIST have shown that while CO<sub>2</sub> is of very low toxicity by itself (30 minute LC<sub>50</sub> = 470,000 ppm with 95% confidence limits from 430,000 to 510,000 ppm [72]), its effect on mixtures is not as slight as linear additivity would suggest. As the concentration of CO<sub>2</sub> increases (up to 5%), the toxicity of CO increases. Above 5%, the toxicity of CO starts to decrease again. The empirically-determined values of m and b are -18 and 122000 if the CO<sub>2</sub> concentrations are 5% or less and 23 and -38600 when the CO<sub>2</sub> concentration is above 5%. Carbon dioxide also increases the toxicity of other gases currently included in the model as well as that of NO<sub>2</sub> [36],[72]. However, for simplicity, the effect of the CO<sub>2</sub> is added into this equation once. Since CO is generally the dominant toxicant in nearly all real fires, the CO<sub>2</sub> effect is merged into the CO factor. As more information becomes available, the N-Gas equation will be changed to indicate the effect of CO<sub>2</sub> on the other gases as well.

The form of the third term arises because oxygen itself is not a toxicant; instead, its *lack* is what is toxic. Thus, the form for O<sub>2</sub> in the above equation will follow as (21 - O<sub>2</sub>). The 30 minute LC<sub>50</sub> of O<sub>2</sub> is 5.4% which is subtracted from the normal concentration of O<sub>2</sub> in air, *i.e.*, 21%.

Even with these non-linearities there is still some systematic deviation from the ideal. 50% of the animals should die at an FED = 1.0, plus-or-minus a confidence interval. Instead, as shown in Table 1. through Table 3. [54], due to small non-linearities, the 50% lethality level corresponds to FED = 1.1 (95% confidence interval of ± 0.2). Since the concentration-response curves for animal lethality from smoke are very steep, the experimental loading is close to the predicted LC<sub>50</sub> value if some percentage (other than 0 or 100%) of animals die. The data in Table 1. through Table 3. indicate the high value of this equation. (These tables separate the within-exposure deaths from the post-exposure deaths to provide additional information for the reader regarding validation hypothesis #5 in Chapter 9.)

The LC<sub>50</sub> values for 30-minute exposures plus post-exposure deaths for the linear terms are as follows: HCN: 150 ppm [67]; HCl: 3800 ppm [73]; HBr: 3000 ppm from the literature [74].

The model is now considered well enough established to be offered for engineering use, as explained below. Further refinements to the 7-gas model are not precluded, however. There are several areas for potential future study. For example, HCl concentrations below 1000 ppm may not have any lethal effect even in the post-exposure period. Nitrogen oxides, especially NO<sub>2</sub>, need consideration, and the NO<sub>x</sub> factor is not strictly additive. As noted earlier, NO<sub>2</sub> and CO<sub>2</sub> are synergistic; NO<sub>2</sub> plus HCN show antagonistic effects [75].



Table 1. Predictability of N-Gas Model using CO, CO<sub>2</sub>, and reduced O<sub>2</sub>

FED Value	Deaths Within Exposure	Deaths Post Exposure	Day of Death
0.84	0/6	0/4 <sup>a</sup>	-
0.89	0/6	0/4 <sup>a</sup>	-
0.93	0/6	1/4 <sup>a</sup>	0
0.96	0/5	0/4 <sup>b</sup>	-
1.01	3/6	0/3	-
1.06	4/6	1/2	3
1.07	3/6	1/2	0
1.12	4/6	0/1 <sup>b</sup>	-
1.22	5/6	0/1	-
<sup>a</sup> two animals cannulated for blood analysis, then sacrificed. <sup>b</sup> one animal cannulated for blood analysis, then sacrificed.			

Table 2. Predictability of N-Gas Model using CO, CO<sub>2</sub>, and HCN

FED Value Post Exposure <sup>a</sup>	Deaths Within Exposure	Deaths Post Exposure	Day of Death
0.95	0/6	1/6	0
1.03	0/6	1/6	1
1.01	0/6	0/6	-
1.11	0/6	2/6	1,3
<sup>a</sup> calculated for post-exposure deaths since no animals died within the 30-min. exposure.			

Table 3. Predictability of N-Gas Model using CO, CO<sub>2</sub>, HCN, and reduced O<sub>2</sub>

FED Value Within Exposure <sup>a</sup>	FED Value Post Exposure	Deaths Within Exposure	Deaths Post Exposure	Day of Death
0.77	-	0/6	0/6	-
0.91	-	0/6	0/6	-
1.06	1.23	1/6	1/5	1
1.08	-	0/6	0/6	-
1.22	-	4/6	0/2	-
<sup>a</sup> calculated primarily for within-exposure since only one animal died during the post-exposure period. In these cases, the difference in FED between within- and post-exposure is that the post exposure LC <sub>50</sub> for HCN is 50 ppm lower than that used for within-exposure deaths.				

## 6.2 Validation and application

The model was developed on the basis of experiments with gas mixtures which could be very well controlled and analyzed. The model should certainly perform successfully under those conditions, and it did, as documented by the data presented above. The model would have little applicability, however, if it were not also predictive of the lethalties from burning solid materials.

A number of diverse materials had been studied at bench scale, mostly using the cup furnace. These are shown in Table 4. Table 5. illustrates data from the radiant apparatus. The data show the degree of the accuracy of the model.

The model was also tested against smoke from real-scale fires [39],[40],[76],[69] with similar accuracy.

By using the N-Gas Model with the radiant toxicity apparatus, both the time necessary to evaluate a material and the number of animal tests needed for the toxic potency determination are reduced. It also helps establish whether the toxicity is usual (i.e., the toxicity can be explained by the measured gases) or is unusual (additional gases are needed to explain the toxicity). The procedures used will be described in detail in Chapter 8.

Table 4. Data from materials decomposed in the flaming mode in the cup furnace smoke toxicity method

Generic Material	Reference	LC <sub>50</sub> <sup>a</sup> (g·m <sup>-3</sup> )	FED at LC <sub>50</sub>	Comments
ABS	8	19	1.59	NO <sub>x</sub> antagonism with HCN ?
ABS	77	23	1.51	NO <sub>x</sub> antagonism with HCN ?
ABS	77	23 24	1.13 1.33	NO <sub>x</sub> antagonism with HCN ?
Douglas fir	8	40	1.03	
Douglas fir	69	46	0.96	
FPU/FR	39	26	1.15	
Modacrylic	8	4	1.80	NO <sub>x</sub> antagonism with HCN ?
Polyester fiber	61	31	0.79	Unusual toxicity
Polyester fabric	62	36	0.74	Unusual toxicity
Polyester + FPU #13	62	38	1.17	
Polyphenylsulfone	8	20	0.88	
Polystyrene	8	39	0.42	Unusual toxicity
PVC	69	18	1.12	
Red oak	8	57	0.92	
Rigid PU	8	13	1.56	NO <sub>x</sub> antagonism with HCN ?
Rigid PU	69	11	1.13	
Wool	8	28	1.58	NO <sub>x</sub> antagonism with HCN ?
<sup>a</sup> based on mass consumed.				



Table 5. Validation of the N-Gas Model against various combustibles, using the radiant test method

Material	Integrated gas concentrations (30 min)							LC <sub>50</sub> (g/m <sup>3</sup> )	FED at LC <sub>50</sub>
	CO (ppm)	CO <sub>2</sub> (ppm)	O <sub>2</sub> (%)	HCN (ppm)	HCl (ppm)	HBr (ppm)	NO <sub>x</sub> (ppm)		
Douglas fir	3100	36000	17.2	NM	NM	NM	NM	56	0.9
Rigid PU foam	1800	21000	18.5	130	NM	NM	NM	22	1.3
PVC	2000	13000	19.2	NM	2300	NM	NM	26	1.1
Flexible PU foam #43644-1	1400	53000	13.2	44	NM	NM	NM	52	1.1
Melamine PU foam	380	9600	19.7	150	NM	NM	48	12	1.2
Vinyl fabric	3000	18000	18.6	NM	650	NM	12	32	0.9
Melamine PU foam + vinyl fabric	1800	18000	18.6	92	170	NM	57	26	1.1
NM not measured									

## 7 The CO problem in fires

High carboxyhemoglobin (COHb) in most fire victims indicates that CO inhalation is the primary cause of most fire deaths [78]. Thus, it is extremely important that CO should be treated correctly in fire hazard analysis. There are some special testing difficulties involved. These are explored in this chapter, which provides the necessary solutions.

### 7.1 The bench-scale CO problem

In a previous comprehensive hazard analysis of fire-retardant-treated versus unretarded products [79] we were unable "to predict accurately the production of CO from less-than-room-sized tests." The problem has not been solved in its entirety. Nonetheless, there is now available an empirical solution. This section explains the implications of this problem and then describes the solution.

By examining the data contained in the above-mentioned hazard study [79], in earlier studies on the fire toxicity of upholstered furniture [39],[40],[76], and in the recent toxicity validation study [69], certain general trends can be seen. The yields of certain combustion products ( $\text{CO}_2$ , HCl, HBr, HCN) are seen to be roughly independent of whether the measurement was taken in a closed-box bench-scale test (cup furnace smoke toxicity method; also the current radiant method), a flow-through bench-scale test (Cone Calorimeter), an open burning environment (furniture calorimeter), or in an actual room fire. The number of combustion gases examined in this manner was not large, and some (*e.g.*, HCl) are difficult to measure to high precision. Nonetheless, the yields in the various scales and environments are similar, at least to the resolution of our measuring capability. This was emphatically not seen for one gas: CO. Table 6. shows the collected CO results.

Since CO toxicity is the major factor in fire deaths, the ability to treat it in a realistic, quantitative way is a top priority. NIST has recently started an effort geared towards understanding and quantifying the production of CO in fires. Sound, theoretically-based models are still some ways off in this area. Empirical methods and some degree of understanding, however, are currently emerging. The state of the art in understanding CO production has recently been reviewed [80]. This document may be consulted for a detailed understanding of the issues; here, some of the pertinent conclusions are summarized.

The production of CO from burning fuels is intimately associated with the supply of oxygen for combustion. In general, there are two paths by which the supply of oxygen to the fire can be limited:

- (1) By lowering the oxygen concentration in the incoming air stream from 21% to a lower value.
- (2) By maintaining the oxygen concentration in the incoming air stream at 21%, but reducing the volume flow of air into the fire.

Table 6. Yields of CO, as measured in various test programs (kg/kg fuel consumed)

Method	Test conditions	DF	RPU	PVC	NFR <sup>a</sup>	FR <sub>b</sub>	Chair NFR	Chair FR
Reference		[69]	[69]	[69]	[79]	[79]	[76]	[76]
Cup furnace	flaming	0.2	0.2	0.1	0.074	0.155	0.02	0.05
Radiant apparatus	50 kW/m <sup>2</sup>	0.03 — 0.04	0.09 — 0.12	0.09	—	—	—	—
Cone Calorimeter	25 kW/m <sup>2</sup>	—	—	—	—	—	0.01	0.05
	30 kW/m <sup>2</sup>	—	—	—	0.02	0.06	—	—
	35 kW/m <sup>2</sup>	0.005	0.06	0.08	—	—	—	—
	50 kW/m <sup>2</sup>	0.003	0.08	0.08	—	—	—	—
	75 kW/m <sup>2</sup>	0.003	0.04	0.07	—	—	—	—
Furniture Calorimeter	average	0.013	0.08	—	0.09	—	0.04	0.05
	steady-state	0.012	0.06	—	—	—	—	—
Real-scale	flaming	0.07 — 0.12	0.10 — 0.14	0.2 — 0.5	0.18	0.23	0.04 — 0.11	0.06 — 0.12
DF Douglas fir RPU Rigid polyurethane foam PVC Polyvinyl chloride a Mixed commodities, all without fire retardants. b Mixed commodities, all containing fire retardants.								

The effects on CO of limiting oxygen availability via Path #1 was recently examined by Mulholland and co-workers [81]. Their results are indicated in Figure 3. The left-most point for each fuel corresponds to the minimum oxygen concentration at which combustion can be sustained. It can be seen that this is, typically, about 13%. It can also be seen that there is a very regular dependence of the CO yield on the oxygen concentration. The important thing to note, however, is that the increase in CO, as one goes from a 21% air stream down to an air stream having 14% oxygen, is only by a factor of 2.5. This increase is consistent and repeatable, but is only of modest importance in establishing fire hazard.



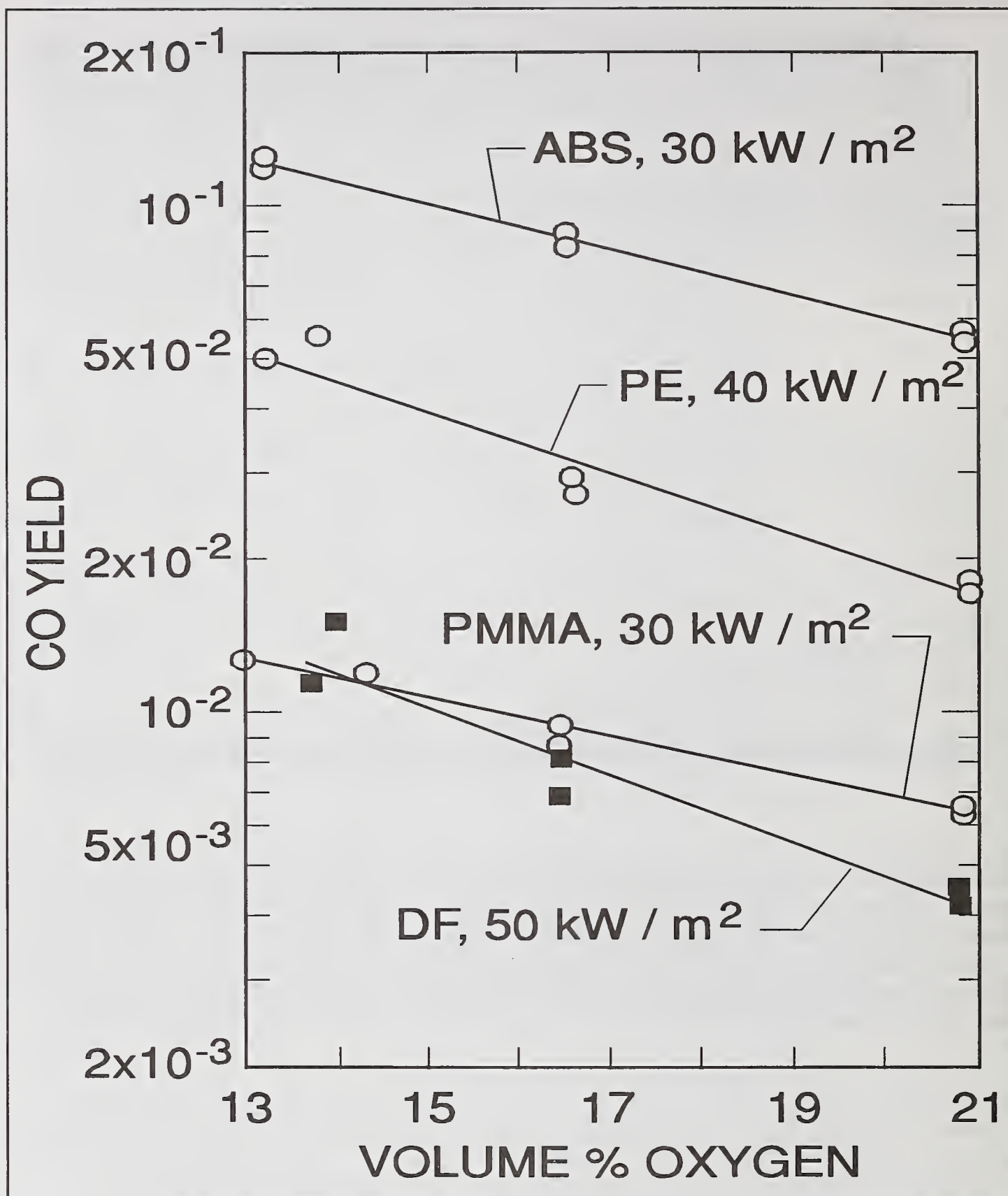


Figure 3. The dependence of CO yield (kg/kg fuel consumed) on oxygen concentration in the incoming air stream (ABS: acrylonitrile butadiene styrene; PE: polyethylene; PMMA: polymethylmethacrylate; DF: Douglas fir).

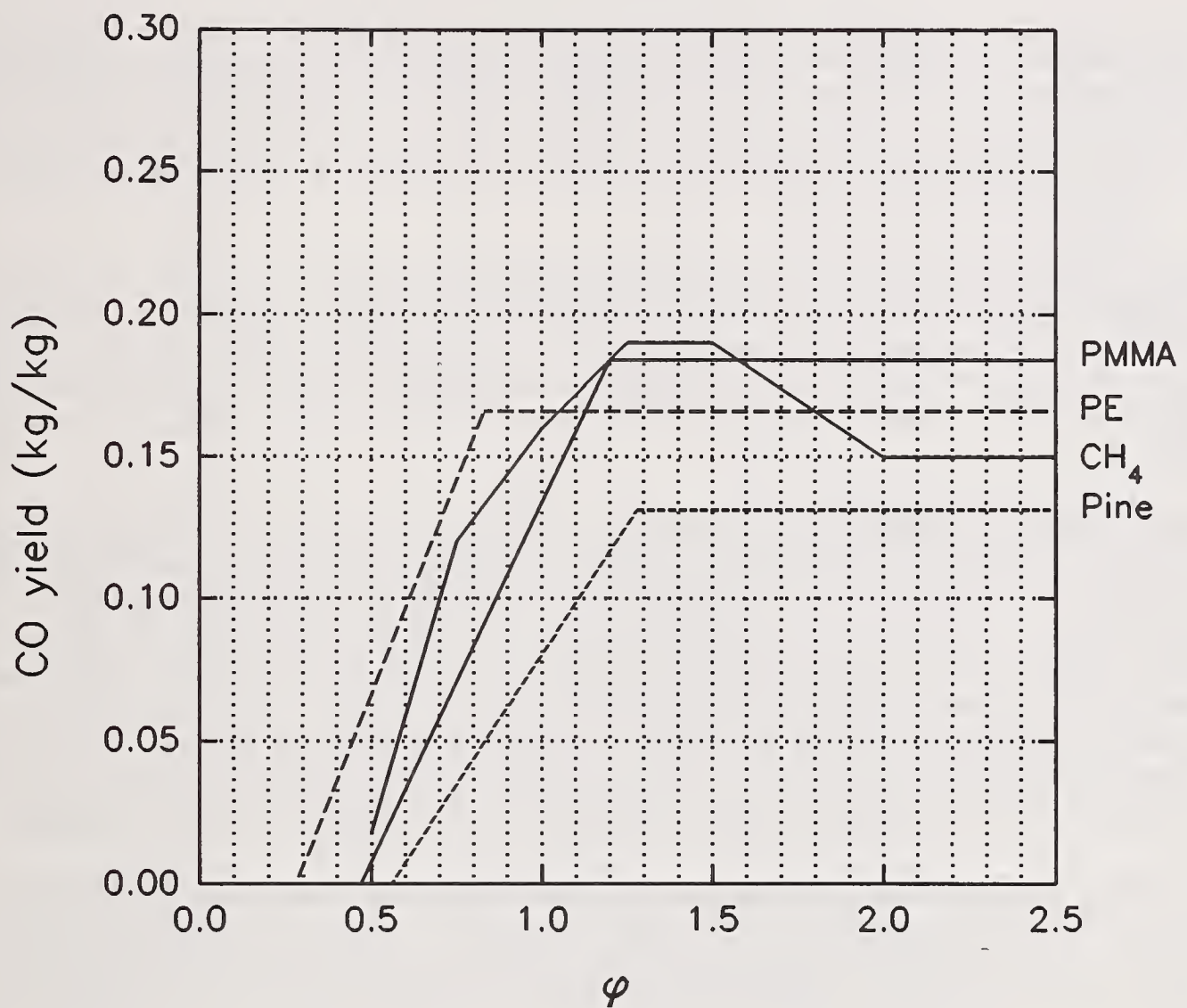


Figure 4. CO yields associated with fires where the volume of air supply is restricted (indicated trends are from catcher hood experiments).

The CO increase associated with Path #2 can be assessed from the data of Beyler [82] and also of Morehart, Zukoski, and Kubota [83]. Figure 4. shows the CO yield results, based on both sets of data<sup>4</sup> and somewhat simplified to portray only the major trends. The yields of CO,  $f_{CO}$ , are plotted as a function of  $\varphi$ , the equivalence ratio, which is defined as

$$\varphi = \frac{(kg \text{ fuel}/kg \text{ air})}{(kg \text{ fuel}/kg \text{ air})_{stoich}} \quad (6)$$

where “stoich” denotes conditions at which the ratio between fuel and oxygen is in the amount required to yield complete combustion, with no excess oxygen. The plateau seen in this figure is consistent with the recommendation of Mulholland [84], who surveyed data from a large number of room fire tests and recommends that  $f_{CO} = 0.2$  (g CO)/(g fuel burned) be taken as characteristic for flashed-over room fires. It is remarkable how small an effect of fuel type is seen in Figure 4., despite the wide variation in the chemical structure of these fuels.

Figure 5. shows these data replotted in a slightly different variable, the CO/CO<sub>2</sub> ratio. This makes the rising portion of the curves fall on a single line, while differentiating two plateaus. The lower plateau, at CO/CO<sub>2</sub> = 0.05, corresponds to fuels which have no oxygen atoms in the fuel molecule. The higher plateau, at CO/CO<sub>2</sub> = 0.08, corresponds to oxygen-containing fuels. Thus, at the present time, a fuel chemistry effect can be seen with Path #2 fires, but it is small and only refers to a fairly gross description of the fuel.

In neither Figure 4. nor Figure 5. do we show data for low values of  $\varphi$ . This is because, in the regime where there is a copious amount of excess air, there is no dependence of CO on  $\varphi$ , there being, instead, another plateau. The value of that low plateau (unlike the upper plateau) is highly dependent on fuel type. Furthermore, it appears to be also dependent on the scale of the combustion and possibly on other variables. Table 7. shows some detailed results from [79]. In some, but not all, cases, the FR material shows CO yields about a factor of 10 higher than the non-FR control. (This, of course, does **not** translate into increased fire hazard, since real-scale burning rates of the FR commodities were seen to be greatly less than the non-FR one.) Thus, we can generalize that while the  $f_{CO}$  drops to very low values for non-FR fuels in oxygen-rich mixtures, the  $f_{CO}$  for FR commodities drops only slightly.

The above generalizations are based on the rather limited experimental evidence available to date. Very recent unpublished studies in this area [85] suggest that while the shape and the plateaus of the curves shown may be correct, there is some discussion as to whether the rising part of the curve begins at the correct value of  $\varphi$ . The recent studies are finding that the rise begins at  $\varphi = 1.0$ , rather than  $\varphi = 0.5$ .

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<sup>4</sup> Experimental work in this area continues at this time. Preliminary indications from studies being conducted both at NIST and at the Virginia Polytechnic Institute and State University indicate that actual room fires may exhibit slightly different results from the ones shown for ‘catcher hood’ experiments. The differences are attributed to the fact that the temperatures in post-flashover room fires are substantially higher than in the catcher hood experiments. As a consequence, in the current unpublished findings, curves are seen which intersect the x-axis at about  $\varphi = 1.0$ , instead of  $\varphi = 0.5$ . Otherwise, the indications are that for these fires with more realistic temperatures both the slopes and the plateau levels of CO yield are similar to the catcher hood results shown here.

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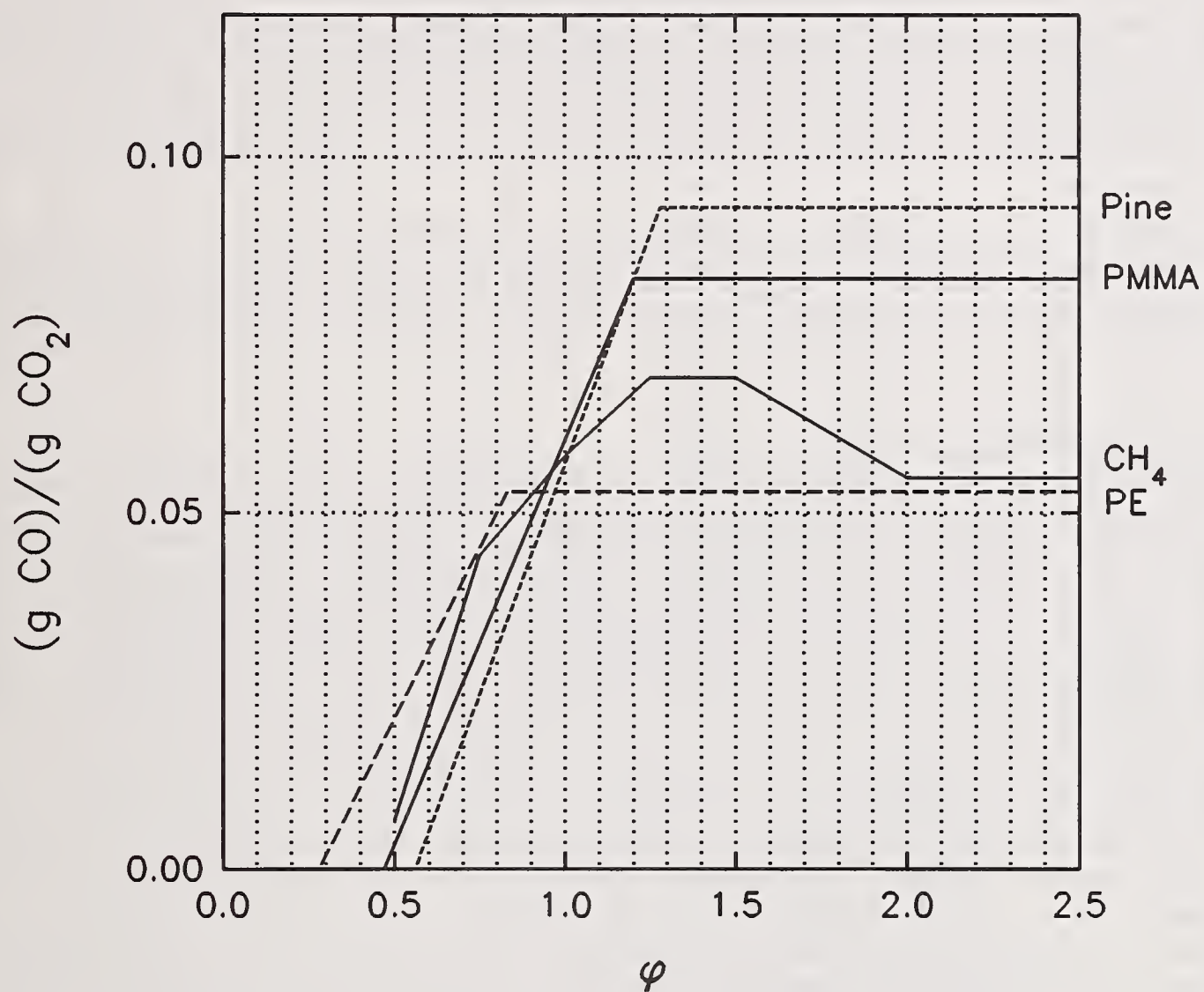


Figure 5. The results from the preceding figure, replotted as a CO/CO<sub>2</sub> ratio.

Table 7. Additional details on CO yields for FR and non-FR materials, from [79]

Specimen	NFR /FR	CO (kg/kg)			CO <sub>2</sub> (kg/kg)					
		Cone Cal.	Furn. Cal.	Cup Furn.	Cone Cal.	Furn. Cal.	Cup Furn.			
TV Cabinet H	NFR	0.015	0.12	0.084	2.28	1.39	2.09			
TV Cabinet G	FR	0.109	0.37	0.18	0.67	0.74	0.78			
Bus. Machine F	NFR	0.037	0.13	0.17	2.21	1.61	1.98			
Bus. Machine A	FR	0.055	0.29	0.30	1.60	1.45	1.53			
Chair T—whole	NFR	0.020	0.01	—	1.62	1.89	—			
Chair S—whole	FR	0.051	—	—	0.964	—	—			
Chair T—foam	NFR	0.016	—	0.025	1.71	—	2.05			
Chair S—foam	FR	0.055	—	0.15	0.81	—	1.19			
Cable D—whole	NFR	0.041	0.12	—	1.77	1.61	—			
Cable K—whole	FR	0.060	0.10	—	1.34	1.04	—			
Cable D—wire ins.	NFR	0.029	—	0.050	2.19	—	2.38			
Cable K—wire ins.	FR	0.135	—	0.13	1.00	—	1.26			
Circuit Bd. C	NFR	0.014	0.10	0.075	2.07	1.71	2.13			
Circuit Bd. L	FR	0.103	0.10	0.15	1.87	1.36	1.24			
Specimen	NFR /FR	HCN (kg/kg)			HBr (kg/kg)			HCl (kg/kg)		
		Cone Cal.	Furn. Cal.	Cup Furn.	Cone Cal.	Furn. Cal.	Cup Furn.	Cone Cal.	Furn. Cal.	Cup Furn.
TV Cabinet H	NFR	—	—	—	—	—	—	—	—	—
TV Cabinet G	FR	—	—	—	0.069	0.082	0.017	—	—	—
Bus. Machine F	NFR	—	—	—	—	—	—	—	—	—
Bus. Machine A	FR	—	—	—	—	—	—	—	—	—
Chair T—whole	NFR	0.002	0.001	—	—	—	—	—	—	—
Chair S—whole	FR	0.005	—	—	—	—	—	0.023	—	—
Chair T—foam only	NFR	0.002	—	0.0007	—	—	—	—	—	—
Chair S—foam only	FR	0.0023	—	0.0032	—	—	—	0.022	—	—
Cable D—whole	NFR	—	—	—	—	—	—	0.112	0.121	—
Cable K—whole	FR	—	—	—	—	—	—	0.131	0.133	—
Cable D—wire ins.	NFR	—	—	—	—	—	—	ND	—	—
Cable K—wire ins.	FR	—	—	—	—	—	—	0.093	—	—
Circuit Bd. C	NFR	—	—	—	—	—	—	—	—	—
Circuit Bd. L	FR	—	—	—	0.022	—	0.0043	—	—	—

This observation does not change the implications for fire hazard analysis, since the upper plateau values for  $\varphi$  are similar in the new studies to those found previously.

The implication of these results for bench-scale testing are profound. Since the objective is to represent post-flashover fires, it would be appropriate if bench-scale test conditions could be created so that the sample is always burned at a fixed  $\varphi$ . This is not possible, however. Bench-scale toxic potency devices may be configured in two ways: as closed boxes, or as flow-through systems. A recent investigation at the University of Pittsburgh [56] revealed that flow-through systems can be designed for combustion toxicity bioassay tests; however, they are extremely cumbersome and cannot be recommended for routine work. Even with a system of such complexity, practical control to achieve a desired, constant  $\varphi$  during the test was not found to be feasible. There are additional problems with a flow-through geometry of this sort. One of them is that, in trying to achieve adequately high values of  $\varphi$ , the heater ends up being submersed in a stagnant gas pool. This, in turn, will cause an unanticipated secondary combustion on the face of the heater.

In a closed-box test, by contrast, there is no means of adjusting the oxygen supply, short of adding some during the test. In both geometries, if  $\varphi$  were to be high, the animals would be presented with a very low oxygen environment in which they could not survive. Thus, it can be seen that a bench-scale bioassay test where the mixture is maintained at a desired, post-flashover value of  $\varphi$  ( $\varphi > 0.5$ ) is not feasible.

Since it is not feasible to set  $\varphi$  to the desired value in a bench-scale test, how much of an error is being committed? The CO yield in the Cone Calorimeter (Table 6.) is from a factor of 2 to an order of magnitude lower than in the room fire. The results in the radiant toxicity apparatus were not as low, but were still about a factor of 2 lower than in the room fire. The cup furnace method results, however, were extremely variable, ranging from a factor of 5 lower to a factor of 2 higher than in the room fire.

For the purposes of comparison, it is especially important to realize that the furniture calorimeter results did not track the results in the room fires, being lower by a factor of 2 to 10. This piece of information is crucial to understanding the behavior of CO generation. The distinction being created here is between “large-scale” testing (*i.e.*, the end-use object, but tested in the open, not in the room) and “real-scale” testing, where an actual fire test room is used. For heat release rate, the distinction between the two is, in many cases, unimportant [86]. For CO production, however, the distinction is crucial. The furniture calorimeter is normally operated in the high excess air regime, with  $\varphi \rightarrow 0$ . The exact value of  $\varphi$  at which room flashover will be reached depends somewhat on the details of room construction. Nonetheless, for rooms roughly the size of the ASTM fire test room [87], flashover is reached generally at the time  $\varphi$  reaches a value of 0.5. By examining Figure 4. or Figure 5., we can note that  $\varphi = 0.5$  is exactly the point at which rapid rise of CO yields begins. Thus, the occurrence of flashover and a sharp increase in CO *yield* occur essentially simultaneously. Here, we emphasize that CO yield is defined as (kg CO)/(kg fuel loss). The fire hazard, however, is controlled either by the *production rate* of CO (for nearby victims) or by the *production* (for victims far away), and not by the yield. The production rate of CO is (kg CO)/s, while the production of CO is (kg CO) for the total fire. The production rate of CO can be factored as



$$CO \text{ production rate} = f_{CO} \times \dot{m}_f \quad (7)$$

At flashover, if the CO yield rises, the CO production rate is ever more so increased, since the flashover event is marked by a very rapid rise in  $\dot{m}_f$ , the mass loss rate of the fuel ( $\text{kg}\cdot\text{s}^{-1}$ ). Thus, there are two entirely different reasons why a very rapid rise in CO production rate occurs at flashover.

## 7.2 Adjusting $LC_{50}$ s for expected CO yield in real-scale fires

Since it is not possible to design a bench-scale test to simulate the flashover conditions occurring in a room fire, it becomes important to establish an alternative means by which correct CO data could be utilized. The technique to do this was developed in [69]; here the basic steps are summarized.

The basis of the N-gas or FED approach to assessing the toxicity of multiple gases is primarily an additive one, as shown in Eq. (4). If in the toxic potency test the amount of CO generated is such that the yield is  $< 0.2$  g/g fuel burned, it is possible to add a term into the FED equation which represents this “missing CO.” The concentration to be used is the difference between the CO concentration at  $f_{CO} = 0.2$  and the actual measured concentration. The  $LC_{50}$  should be the value for CO. Since this is only a correction term, a simplification is warranted to avoid the non-linearity of CO/ $CO_2$  interaction; thus  $LC_{50}\text{-CO}$  (volumetric) = 4000 ppm is assumed. (This is its value at the  $CO_2 = 5\%$  condition). Converted into concentration units, this becomes  $LC_{50}\text{-CO}$  (conc.) =  $4.58 \text{ g}\cdot\text{m}^{-3}$ . Giving,

$$LC_{50}(\text{corr}) = \frac{1}{\frac{1}{LC_{50}} + 44 \times 10^{-3} - 5.0 \times 10^{-5} \frac{[CO]}{m_{100}}} \quad (8)$$

where the variables, along with their units are:

- $LC_{50}(\text{corr})$  = the corrected value of the  $LC_{50}$ , in concentration units ( $\text{g}\cdot\text{m}^{-3}$ )
- $LC_{50}$  = the measured or approximate value of the  $LC_{50}$ , in concentration units ( $\text{g}\cdot\text{m}^{-3}$ )
- $[CO]$  = the average measured CO concentration in the box (ppm)
- $m_{100}$  = total specimen mass lost during test (g).

Thus, the solution to handling data from bench-scale toxic potency tests is to correct the measured  $LC_{50}$  by the equation given above. The equation is valid under all circumstances where the present FED computational scheme is valid, even if the measured  $f_{CO}$  should be  $> 0.2$ , and, thus, the correction would decrease the toxicity of the specimen.

## 7.3 CO production in the radiant apparatus and in the cup furnace method

For the same materials, the CO produced in the cup burner method is generally larger (and closer to post-flashover levels) than in the radiant apparatus. The disparity is the strongest for wood (Table 6.), but is true for the other materials examined also. This is not due to a more fuel-rich (higher  $\varphi$ ) combustion

mixture in the cup; in fact the opposite is true. For wood, the dominant toxicant is CO. Since the CO yield is lower for the radiant furnace, more fuel must be combusted to produce the same amount of CO as with the cup furnace. Since the air volume is nominally the same in both devices, the fuel/air mixture is richer in the radiant apparatus. Some years ago, opinions had been expressed that the specimen in the cup furnace method is, in fact, seeing a high value of “local  $\phi$ ,” due to poor mixing in the system. Experiments were conducted specifically to examine this point by flowing air directly into the specimen zone [57]. The results showed that localized vitiation did not exist, and that forced aeration did not increase the yield of CO.

If *localized* vitiation is not part of the answer, it still becomes important to consider the effect of *overall* oxygen concentrations on production of CO. During the experiments conducted by SwRI leading to the development of the NIBS method, an initial opportunity arose to examine the effects of oxygen concentration on  $f_{CO}$ . This was followed by more detailed studies, conducted at the request of NIST. As explained above, it is not feasible to create oxygen conditions in a bench-scale toxicity test which mimic the situation in the real-scale fire. Fortunately, a numerical correction method can be used and corrects for this effect. This eliminates much of the direct concern [88] with CO yields obtained in the test. Nonetheless, it was desired to investigate the CO environment in the apparatus in some detail, to make certain that no anomalies in combustion are occurring. This work was done at SwRI and was done in two stages.

In the first stage, some preliminary data on Douglas fir (DF), rigid polyurethane foam (RPU), and Polyvinyl chloride (PVC) were gathered. The data suggested that CO yields for RPU and PVC were insensitive to oxygen levels, but that DF was. Clearly the issue needed further exploration. This was done in a follow-up study at SwRI [89], where many more tests were run using these same three materials. To be able to better study and control conditions, these exploratory tests (*unlike* the recommended method which was eventually developed and is presented in Chapter 8) involved controlling the oxygen conditions within the apparatus by introduction of supplementary  $O_2$  from a gas bottle. The initial analysis of the new data indicated results similar to those from the first study. For RPU and PVC, the data shown in Table 8, again suggest no special CO dependence on  $O_2$  levels. The data, as first analyzed from this series for Douglas fir, are shown in Figure 6. The data points appear to show a consistent increase of  $f_{CO}$  with dropping oxygen concentration. As an interesting point of reference, this trend is compared with the predictive relation of Mulholland et al. [81], which is shown as a solid line.

Upon further analysis, the suggestive trend of the points in Figure 6 was seen to be misleading. The data plotted in this figure were test-average  $f_{CO}$  values. Yet, in most of the tests, the burning would switch from flaming to non-flaming some time before the end of the 15-min period. While care was taken to exclude from the data analyzed those tests where a large fraction of the time was in the non-flaming mode, nonetheless the  $f_{CO}$  values actually represented a mixture of flaming and non-flaming CO yields. We now see the following explanation. Some materials exhibit a near-constant CO yield throughout their burning history. Others, of which woods are a prime example, show very little CO yield during flaming combustion and a great deal once flaming has gone out (Figure 7). The results for RPU (Figure 8) are similar with regard to change in yield levels. Unlike DF, the RPU box concentrations do not increase in slope at the flame extinction point, since the flaming values for RPU  $f_{CO}$  are not as low as for DF.



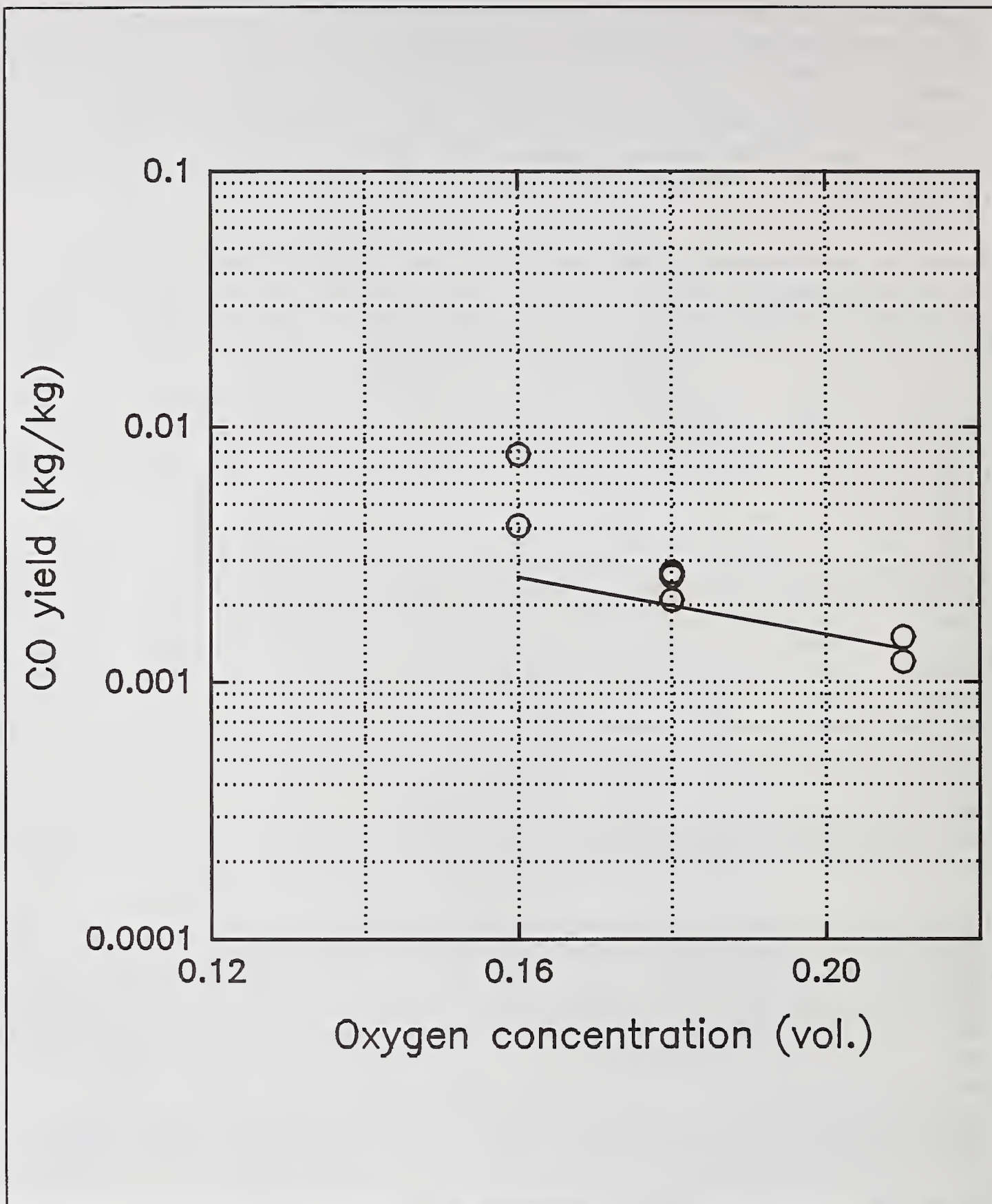


Figure 6. Preliminary analysis of SwRI results of Douglas fir (○); also shown is a trend line (—) from the results of Mulholland et al. [81].



# CO AND CO YIELD - DOUGLAS FIR #5

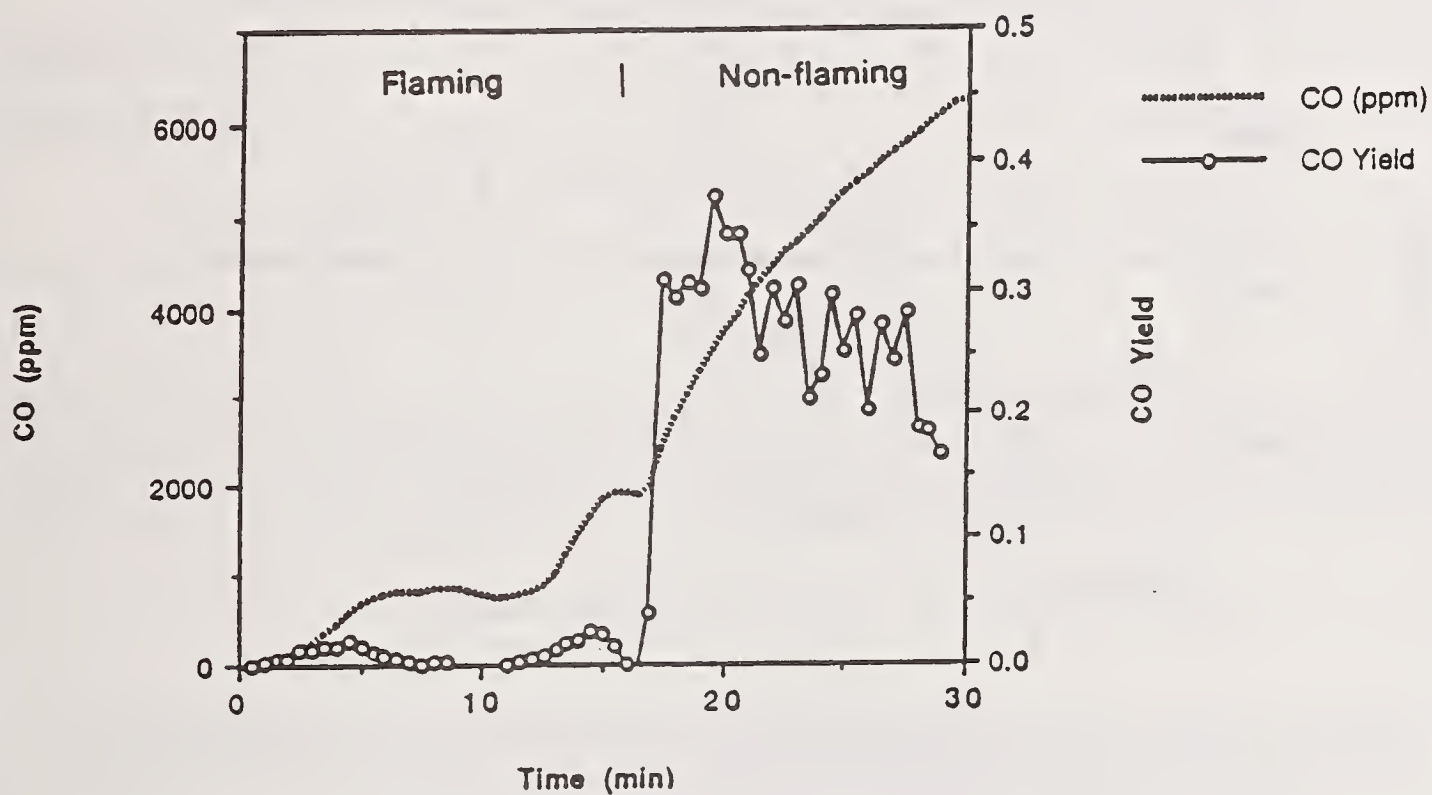


Figure 7. Results obtained at SwRI for Douglas fir, demonstrating how the CO yield changes with mode (flaming vs. non-flaming) of burning.

### CO CONCENTRATION AND YIELD - RPU #1

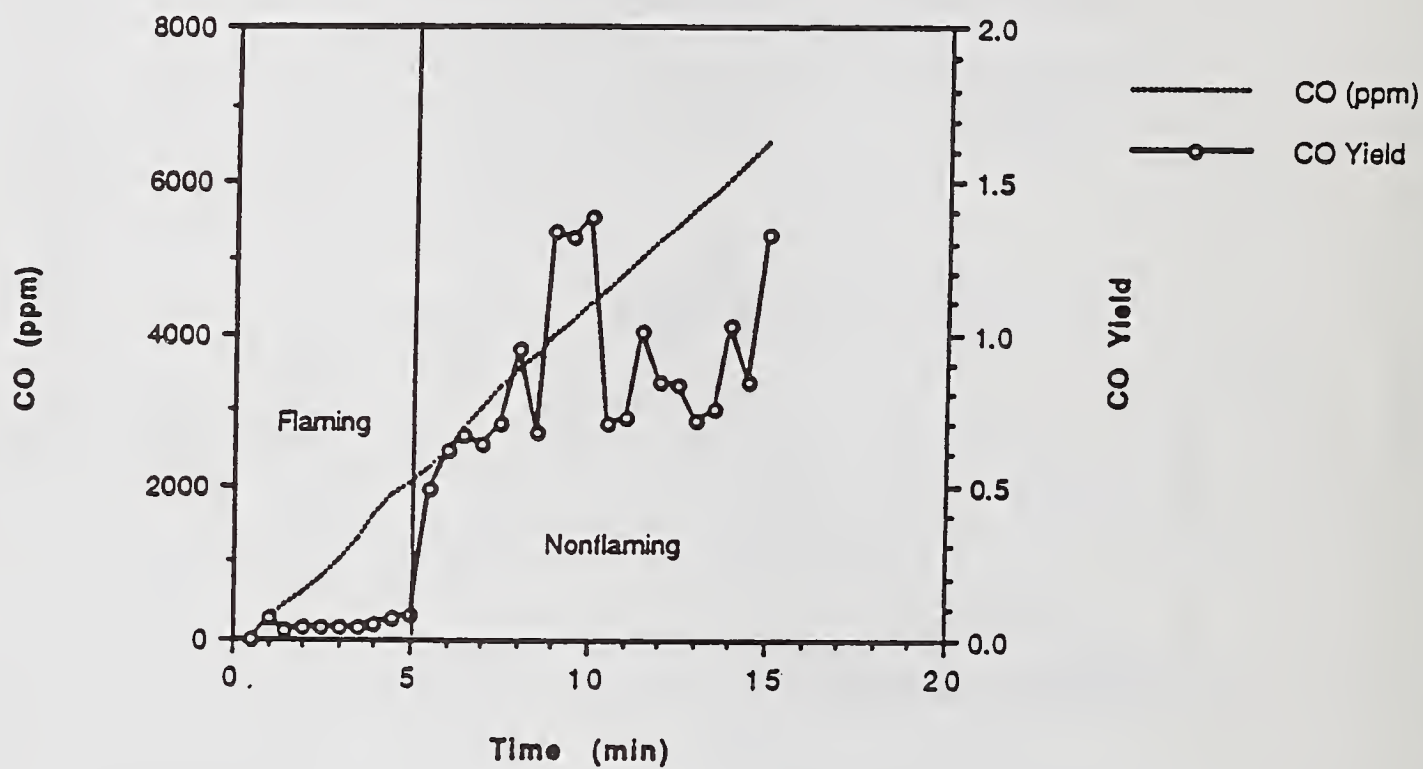


Figure 8. Results obtained at SwRI for rigid polyurethane foam, demonstrating the change in CO yield with mode of burning.

Table 8. CO yield measurements on various combustibles made in the radiant apparatus at SwRI

Material	Specimen size (cm <sup>2</sup> )	Oxygen concentration <sup>a</sup> (%)	CO yield (kg/kg)
Rigid polyurethane foam	25.9	18	0.062
	25.9	16	0.089
	25.9	14	0.084
	50.6	18	0.084
	50.6	16	0.080
Rigid PVC	25.9	21	0.074
	25.9	18	0.065
	25.9	16	0.061
<sup>a</sup> Only data for specimens burning primarily in flaming mode are shown.			

The level of O<sub>2</sub> in the apparatus does not directly affect the yield of CO for these materials. Instead, lower levels of O<sub>2</sub> correspond to shorter times for burning to change from flaming to non-flaming. The data were, thus, re-analyzed. Yields of CO were examined for time periods which were strictly confined to all-flaming or all-non-flaming. This analysis indicated the following:

Table 9. Approximate CO yield ratios in SwRI tests

Material	Ratio f <sub>CO</sub> (non-flaming)/f <sub>CO</sub> (flaming)
Douglas fir	70 - 240
Rigid polyurethane foam	20
PVC	< 1

The fact that a broad range, as opposed to a constant value, for Douglas fir is seen might be attributed to the following effect: the char combustion is expected to be significantly sensitive to the history of



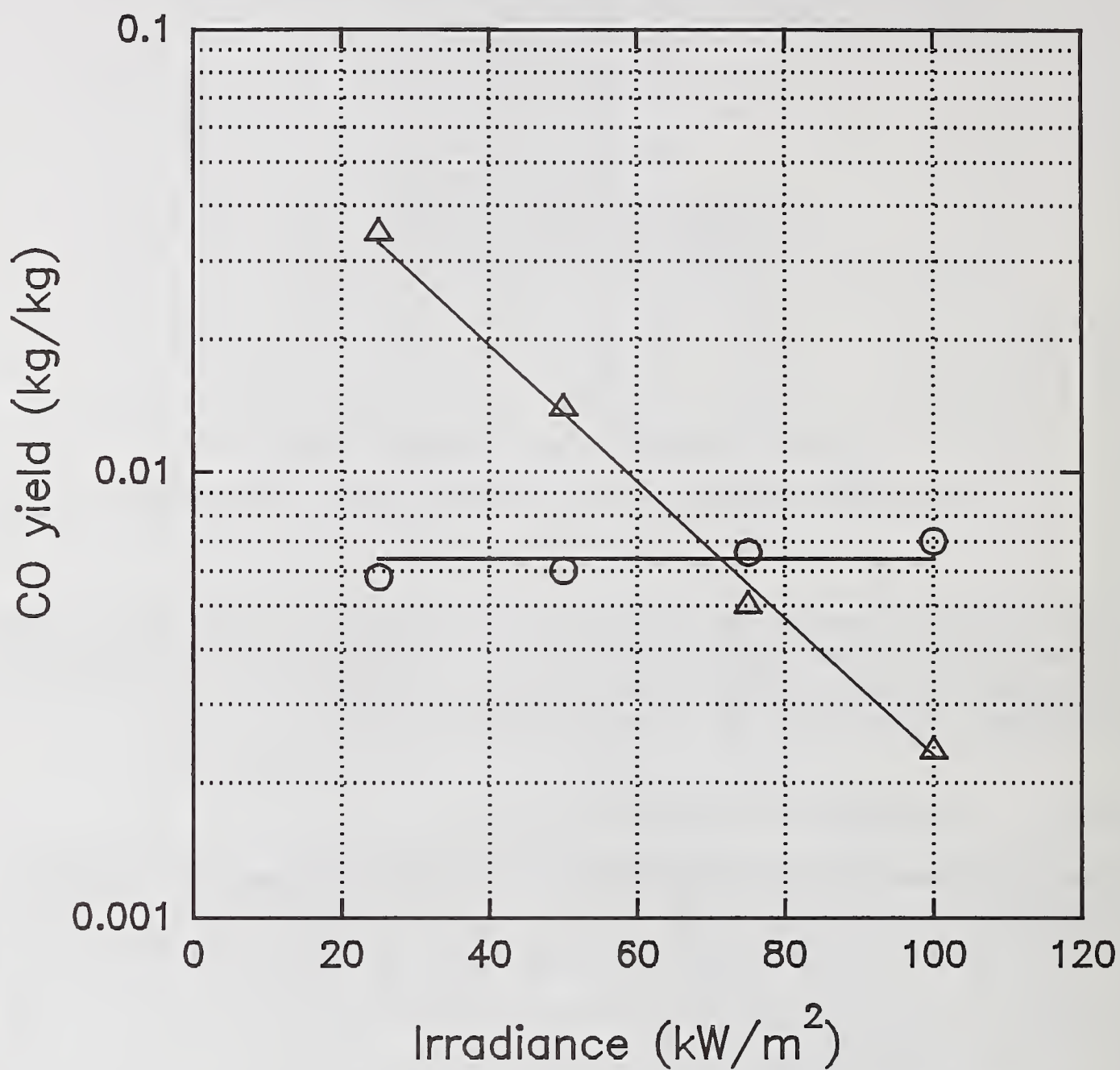


Figure 9. Comparison between CO yields measured in the Cone Calorimeter for a cellulosic material (Douglas fir,  $\Delta$ ) and a thermoplastic (PMMA,  $\circ$ ).

burning; how the char layer was built up, and to what thickness, might be expected to influence the results. This burning history will, in turn, be affected by how soon a dropping oxygen level causes switchover to a non-flaming burning regime.

As a point of further interest, levels of HCN and HCl were also monitored in the SwRI experiments. Neither of these was affected by O<sub>2</sub> levels in any systematic way at the levels tested.

Another factor which might be effecting the difference between the two apparatuses is the heating flux. The specimens in the radiant heat apparatus are exposed to an irradiance of 50 kW·m<sup>-2</sup>. A Douglas fir specimen in the cup furnace, by comparison, is exposed to a furnace temperature of around 525 °C, which corresponds to a black body flux of slightly less than 25 kW·m<sup>-2</sup>. This may comprise part of the answer, but would not be judged to be a large factor. The Cone Calorimeter data in Table 6 suggest some increase in CO yield for Douglas fir, with inconsistent results for rigid polyurethane foam and no effect for PVC. Further results from other Cone Calorimeter tests are presented in Figure 9. For the thermoplastic material (PMMA), it is clear that there is no significant dependence of  $f_{CO}$  on the irradiance. For the cellulosic material (a different stock of Douglas fir), however, a dependence is seen as follows:

$$f_{CO} = \exp(-0.036 \dot{q}'' - 2.5) \quad (9)$$

where  $\dot{q}''$  denotes the test irradiance. In quite a few other cases, the results would not be as clear-cut as the examples cited, due to difficulties in measuring very low CO concentrations. Nonetheless, for Douglas fir, Eq. (9) suggests that  $f_{CO}$  rises by a factor of 2.5 as the test irradiance is lowered from 50 to 25 kW·m<sup>-2</sup>. This does not, however, explain the findings of Table 6, where it is seen that  $f_{CO}$  for Douglas fir is nearly 6 times greater in the cup furnace than in the radiant apparatus. Similarly, it does not explain the data in that Table, where at the same irradiance of 50 kW·m<sup>-2</sup>, the CO yield in the Cone Calorimeter is nearly an order of magnitude lower than in the radiant apparatus. Further complicating understanding of the apparatus-dependence of CO generation are the values seen from the next two materials shown—rigid polyurethane foam and PVC. For those materials, the Cone Calorimeter and the radiant apparatus results are nearly identical.

To summarize, at the present time, strong differences in CO production among the different bench-scale apparatuses occur for some charring materials, but not for others, nor for melting materials. It appears to be a phenomenon associated with the heating conditions to which char is subjected. The focus on char is offered since materials which show none (or a smaller amount of charring) seem to behave more similarly in the different environments. In addition, the specimen in the cup furnace, as contrasted to the one in the radiant apparatus,

- is much smaller,
- is heated from all sides nearly uniformly, and not just from the top down, and
- can also pyrolyze.

It would be desirable, in the longer term, to develop an improved capability to predict CO evolution in bench-scale apparatuses. Nonetheless, using the CO correction method, outlined in this chapter, enables widely varying bench-scale results to be adjusted to predict the real-scale generation of CO adequately.



## 8 The radiant toxicity method

This chapter begins with a historical overview of earlier designs, then describes the main features of the radiant test method, discusses some of the reasons for specific features of the method, and gives results obtained with a number of materials.

### 8.1 Basic design and test apparatus

A method to assess the acute inhalation toxic potency of combustion products requires three main components: an animal exposure system, a chemical analysis system, and a combustion system.

#### 8.1.1 Animal exposure system

The animal exposure system in the present test method is similar to that designed for use in the cup furnace smoke toxicity method [7] developed at NIST. This animal exposure system was, subsequently, adapted for use in an earlier radiant lamp smoke toxicity method developed at Weyerhaeuser Company [53] and in the NIBS method [58], [59], [60]. The animals are exposed in an approximately 200 L polymethylmethacrylate or polycarbonate rectangular box. The furnace is located below the left side and six portholes are positioned across the front to hold the test animals. The portholes are designed such that only the heads of the animals, which are held in restrainers, are exposed to the smoke. The head-only exposure reduces the problem of overheating the animals, and eliminates the problems of animals huddling together (and possibly breathing smoke that has been filtered through the fur of another animal) and ingesting smoke particulates deposited on the fur during the exposure when grooming after the exposure. The current exposure chamber is changed in only one minor aspect from the earlier version. Instead of a blowout panel on the top of the chamber, an expansion bag of approximately 49 L capacity is attached to a porthole located in the far right wall (Figure 10 ). This expansion bag provides for safety in case of an explosion and also minimizes leaks which otherwise would occur into or out of the system due to pressure differences between the air inside and outside the chamber.

#### 8.1.2 Chemical analysis system

The chemical analysis system is the same used with the cup furnace smoke toxicity method and is in compliance with the principles outlined in ASTM E 800 [90]. Since this is a closed system, the atmosphere which is removed for nondestructive chemical analysis and which can be recirculated is returned to the animal exposure chamber. The atmosphere which can be returned is that analyzed for CO, CO<sub>2</sub>, and O<sub>2</sub>. Some analyzers are destructive (i.e., the atmosphere after analysis is different from that taken prior to analysis) and therefore do not permit the return of the samples to the exposure chamber. These include those for HCN, HCl, HBr, and NO<sub>x</sub>. Care should be taken to minimize the amount of air taken from the chamber to monitor these non-returnable gases. Further details on the gas analysis systems are provided in the description of the standard test procedure (see the Annex).



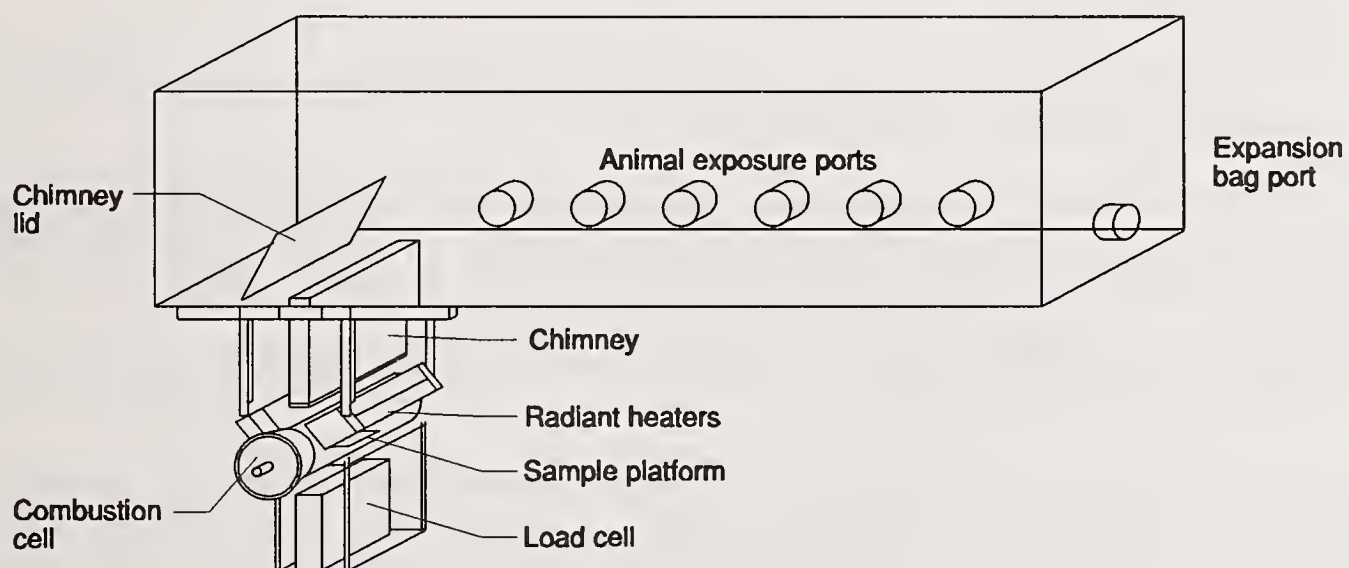


Figure 10. General view of radiant toxicity apparatus.

### 8.1.3 Combustion system

Most fire researchers have accepted the animal exposure system and the chemical analysis systems described in the previous two sections. The main issue with regard to smoke toxicity test methods has been the combustion systems. Certainly no one test procedure can simulate all possible fire scenarios. The cup furnace was used to decompose materials under two severe conditions, namely, 25°C above (flaming conditions) and 25°C below (nonflaming conditions) each material's autoignition temperature. The heating conditions provided by the cup furnace could best be described as "thermal immersion." This means heating of small specimens under conditions where the temperature gradients across the specimen are small, the temperatures on all specimen faces are rather similar, and a substantial amount of the heat transfer occurs by conduction. These are conditions which are commonly seen, for example, in thermo-gravimetric analysis (TGA) instruments. The cup furnace, however, does not readily represent the fire conditions occurring in a room fire. Most fire scientists now agree that:

1. The combustion system should thermally decompose materials under more realistic conditions, namely radiant heat,
2. The furnace should allow for the decomposition of materials, products, composites, and assemblies under likely end-use conditions,
3. The system should allow for the testing of larger sample sizes than previously acceptable in the cup furnace (*e.g.*, the cup furnace test procedure recommended sample sizes no larger than 8 grams although larger sizes were successfully tested) and in some tube furnaces and
4. The fire scenario should simulate the conditions under which the greatest number of human lives are lost, namely post-flashover.

Thus, various investigators have sought to find a better combustion system. In 1984, Alexeeff and Packham proposed using the radiant heater system (Figure 11) developed by H. W. Stacy at Weyerhaeuser Company [53],[50]. This method did not achieve wide use because of problems with the test hardware. It did, however, offer the possibility of testing composite materials realistically exposed to radiant heating fluxes. The new University of Pittsburgh II radiant test procedure [56] couples the Cone Calorimeter combustor with the flow-through animal exposure system previously used with the UPitt I smoke toxicity method [91].

In 1986, the National Institute of Building Sciences (NIBS) formed a working group to develop a performance test method for evaluating toxic hazard of materials and products. After the determination of their required criteria, they asked SwRI to design, build, and test the new system. SwRI used the animal exposure system and chemical analytical system from the cup furnace smoke toxicity method and started with the Weyerhaeuser radiant heating system. In this development work, while SwRI kept the basic principles of the Weyerhaeuser design, all of the individual components were re-designed. The major improvements were:

- The weighing system was designed for increased robustness and sensitivity.
- The combustion cell was built to a new design, allowing for easy disassembling, cleaning, and eventual replacement, as needed.

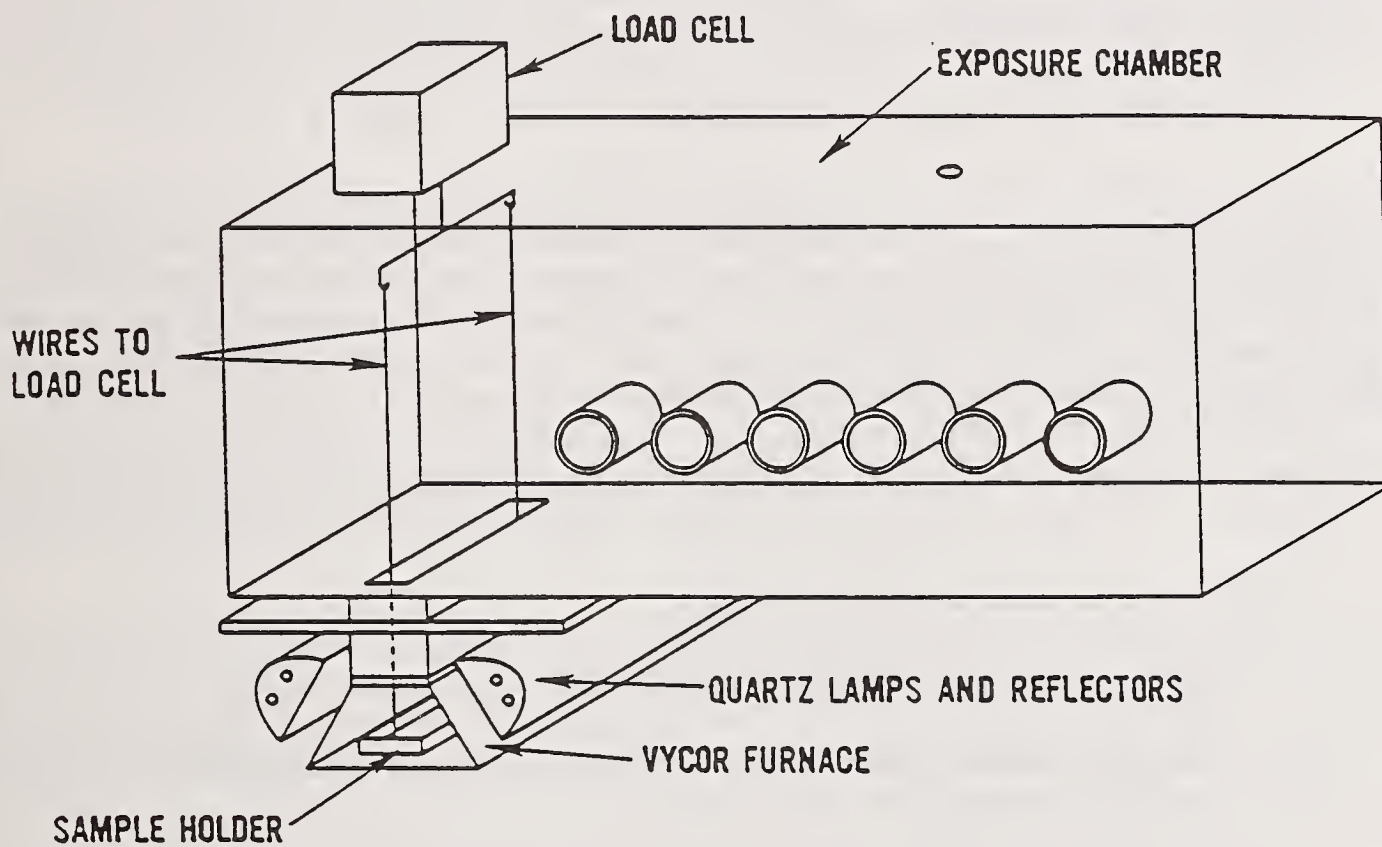


Figure 11. The radiant exposure system designed at Weyerhaeuser.



- The circulation of gases was changed to avoid cyclic flow behavior. The earlier system had a single chimney connecting the combustion chamber to the animal exposure chamber. Natural convection-driven gas movements were erratic in such a system, showing sporadic “gulping” behavior. It was found that by segregating the chimney with two vertical septa into three flow channels this gulping could be eliminated.
- The materials usage for the chimney was examined. A construction made of calcium silicate board was found to be unacceptable, but one made of stainless steel was found to be suitable.
- An expansion bag was provided to reduce gas leakage due to pressure differentials.
- A shutter was provided to close off the combustion cell from the animal exposure chamber at the appropriate time.
- A new lamp design was used, together with improved calibration and control procedures for the lamps.
- A special exhaust line was provided for carrying away any combustion products that might leak out of the load cell hole.
- A spark ignition system, similar to the one used on the Cone Calorimeter, was added.

As development work at SwRI progressed, a joint activity between SwRI and NIST began developing the apparatus for measuring toxic potency of materials and products. The purpose was to gather data for engineering analyses of fire hazard, rather than an index of toxic hazard as pursued by NIBS. This cooperation resulted in additional areas where the combustion system was re-designed to provide ruggedness, ease of operation, and safety. These were:

- Stainless steel foil was installed to cover the insulating refractory blanket which acts as the trap door seal.
- The shutter was modified to avoid free falling.
- The load cell was covered with a calcium silicate board to act as a heat shield.
- A precise locating bracket was developed for the heat flux meter to allow consistent repositioning in the combustion chamber.
- The spark ignitor was fitted with a standard tapered joint in the quartz combustion cell in order to eliminate gas leakage.
- Provision was made to wrap specimens with stainless steel foil instead of aluminum foil in those cases where the aluminum was being melted or was reacting with the specimen.
- The specimen was backed up by a layer of low-density refractory blanket, placed on top of an impervious stainless steel plate as the means of supporting it on the load cell.
- A temperature controller circuit was developed for maintaining constant irradiance from the heat lamps.
- A removable, tinted polycarbonate safety shield was installed in front of the combustion chamber.

The apparatus as implemented for the NIBS test has been documented by SwRI [92]. The preliminary results of the SwRI/NIST collaboration were published in a NIST publication [69]. The Annex contains step-by-step details in ASTM format of the method as developed. This chapter summarizes the basic operating principles, illustrated with sample data.

## 8.2 Operating the method

The steps to measure the toxic potency of the smoke from a product are straightforward. All samples are exposed to an irradiance of  $50 \text{ kW}\cdot\text{m}^{-2}$ . Changes in the concentration of smoke in the animal exposure chamber are achieved by variation of the surface area of the sample. The number of animal tests is minimized by using chemical information on the smoke. The results to be reported are the values of  $\text{LC}_{50}$  and  $\text{LC}_{50}(\text{corr})$ . A number of other variables should be reported to enable a complete and useful documentation of the procedures. These are detailed in the Annex.

**Procedure A:** *Determine an estimated  $\text{LC}_{50}$  (30-minute exposure plus 14-day post-exposure observation period) using the N-Gas Model.* This entails two experiments, neither involving animals. The specimen size for the first is not critical, but is normally of less-than-maximum dimension and is guided by prior data for similar products. The sample's lost mass and the concentrations of gases in the N-Gas Model are measured, and an FED is calculated using Equation (5). Based on this result, a similar second experiment is performed for a specimen that should produce an FED of about 1.1. This is verified by inserting values of the gas concentrations from the second test into the N-Gas Model. The  $\text{LC}_{50}$  is then estimated by dividing the volatilized (2nd) sample mass by the apparatus volume. For the present, this should be recorded to two significant figures for use in the following procedures.

**Procedure B:** *Check the estimated  $\text{LC}_{50}$  (30-minute exposure plus 14-day post-exposure observation period) using animals.* Again two experiments are needed: one where the specimen surface area (and mass) is chosen to produce an FED of about 0.8, and one to produce an FED of about 1.4. In each, the mass loss and standard gas concentrations are measured and 6 rats are exposed to the smoke for 30 minutes. The measurements are to assure that the sample decomposition indeed provided the desired FED. (The presence of the animals will influence these values in that their respiration will generate some  $\text{CO}_2$  and deplete some of the other gases.)

The range of FED values here is wider than the entire rising portion of all dose-response curves previously measured for fire smokes. Therefore, if the  $\text{LC}_{50}$  estimate is accurate, the exposure at  $\text{FED} = 0.8$  should result in 0 or 1 animal deaths and the exposure at  $\text{FED} = 1.4$  should result in 5 or 6 animal deaths. (These could occur either during the 30-minute exposure or during the 14-day post-exposure period.)

If the animal deaths are as predicted, then no further measurement is needed. The chemical data for the 4 experiments in Procedures A and B are used to calculate the best approximate value of the  $\text{LC}_{50}$ . This should be reported to 1 significant figure (because the method reproduces real-scale results within a factor of 3).



If such results are not seen, then Procedure C, below, must be used.

The CO concentration corresponding to the  $LC_{50}$  is needed so that the  $LC_{50}$  (corr) can be determined. This concentration is determined by plotting the CO concentrations vs. the mass consumed for the 4 experiments. The best value of  $[CO]/m_{100}$  is determined by a least squares linear regression analysis which is forced through zero. (At zero mass loss, the CO concentration should also be zero.) The result is inserted into Equation (8).

**Procedure C:** *Determine a more accurate value for the  $LC_{50}$ . For a proper statistical determination, 3 experiments are needed in which some, but not all, of the rats die.* The idea is to bracket the  $LC_{50}$  and then converge. The selection of sample sizes is guided by the prior 4 tests, but some trial-and-error will occur. One need not always wait for the end of the 14-day post-exposure period to determine the next sample size. Unless all of the deaths occur late in the 14 days, the physiological status of the animals at the end of the 30-minute exposure and after 24 hours should provide some indication of how close one is to the lethal concentration. After determining the  $LC_{50}$ , it should be reported to 1 significant figure.

A total of 7 materials were examined experimentally with this method; the results are given in Tables 10 through 16. The statistical analysis of  $LC_{50}$  values was done according to the method of Litchfield and Wilcoxon [93].

## 8.3 Simplifying the method

**Post-flashover limit.** As noted in Chapter 7, the  $LC_{50}$  of  $CO_2$ -potentiated CO is about  $5\text{ g}\cdot\text{m}^{-3}$ , and the yield of CO is about 0.2 g/g of fuel burned. Therefore, the  $LC_{50}$  of post-flashover smoke is about  $25\text{ g}\cdot\text{m}^{-3}$ . The previous work on validation of this radiant apparatus showed that the results could be used to predict real-scale toxic potency to about a factor of 3 [76]. Therefore, post-flashover smokes with  $LC_{50}$  (corr) values greater than  $8\text{ g}\cdot\text{m}^{-3}$  are indistinguishable from each other. Most common building and furnishing materials have  $LC_{50}$  values substantially higher than this. (Note the  $LC_{50}$  data throughout this report.) Thus, the toxicity of the smoke will most often be determined by the fire ventilation, rather than the specific products burning.

If the results in Procedure A suggest a specimen  $LC_{50}$  (corr) higher than this value, then a precise determination is unnecessary for post-flashover scenarios. Rather, Procedure B could then be modified to a single test at an FED that corresponds to an  $LC_{50}$  (corr) of  $8\text{ g}\cdot\text{m}^{-3}$ . An observation of no animal deaths would confirm the suggestion. The  $LC_{50}$  (corr) would then be recorded as “greater than  $8\text{ g}\cdot\text{m}^{-3}$ ,” and one would use the  $8\text{ g}\cdot\text{m}^{-3}$  value in a hazard analysis.



Table 10. Test results for Douglas fir

Test	Irrad. Time (min.)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc. <sup>a</sup>		
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)
DF-1	15	19.4	12.26	11.01	90	32	350	39300	2860	16.7
DF-2	15	24.2	12.43	11.04	89	37	405	37400	3200	16.9
DF-3	15	21.3	13.03	11.60	89	40	380	39800	3150	16.6
DF-4	15	24.2	13.64	12.20	89	42	405	42700	3700	16.3
DF-5	15	38.7	19.78	16.89	85	20	475	55600	5150	14.9
DF-6	15	67.7	25.50	21.36	84	30	510	64400	6220	13.7

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE			WE	WE + PE
DF-1	55.1	2/6	2/6	0.91	0.91	1.45	.088		
DF-2	55.2	4/6	4/6	0.96	0.96	1.40	.103		
DF-3	58.0	4/6	4/6	0.99	0.99	1.42	.096	56	56
DF-4	61.0	6/6	6/6	1.16	1.16	1.44	.106	(54-57)	(54-57)
DF-5	84.4	6/6	6/6	1.63	1.63	1.37	.110		
DF-6	106.8	6/6	6/6	1.83	1.83	1.28	.104		

<sup>a</sup> Calculated from the time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass loaded per chamber volume times average percent mass loss according to the method of Litchfield and Wilcoxon.

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.

Table 11. Results for rigid polyurethane foam

Test	Irrad. Time (min.)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc. <sup>a</sup>			
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCN (ppm)
RP-1	15	19.4	4.70	4.22	90	15	285	N.M.	N.M.	N.M.	140
RP-2	15	19.4	4.71	N.M.	N.M.	15	< 180	22500	1300	18.3	50
RP-3	15	19.4	4.80	4.29	89	20	< 360	20300	1600	18.4	110
RP-4	15	19.4	4.83	4.62	96	20	265	20100	2200	18.5	200
RP-5	15	19.4	4.86	4.42	91	20	270	20600	1700	18.5	130
RP-6	15	19.4	4.97	4.64	93	29	265	21400	2300	18.6	150
RP-7	15	19.4	5.21	4.65	89	15	285	21200	1740	18.4	150

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCN (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE				WE	WE + PE
RP-1	21.1	1/6	1/6	N.A.	N.A.	N.M.	N.M.	.0094		
RP-2	23.3	-	-	0.67	0.75	1.91	0.077	.0029		
RP-3	21.4	1/6	2/6	0.99	1.17	1.84	0.120	.0075		22
RP-4	23.1	6/6	6/6	1.54	1.86	1.68	0.146	.0134		(21.6 - 22.2)
RP-5	22.1	5/6	5/6	1.10	1.30	1.80	0.123	.0088		
RP-6	23.2	5/6	6/6	1.33	1.58	1.84	0.155	.0101		
RP-7	23.2	6/6	6/6	1.22	1.46	1.78	0.117	.0090		

<sup>a</sup> Calculated from the time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

N.A. Not applicable

N.M. Not measured

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.

- No animals exposed

Table 12. Results for PVC

Test	Irrad. Time (min.)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc. <sup>a</sup>			
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCl (ppm)
PV-1	15	2.4	4.43	3.98	90	50	205	10400	1300	19.6	1600
PV-2	15	2.4	5.17	4.65	90	40	230	13000	1700	19.2	2500
PV-3	15	2.4	6.01	5.41	90	51	210	13300	1900	19.1	2800
PV-4	15	2.4	6.80	6.15	90	45	212	14600	2300	18.9	2900
PV-5	15	2.4	7.39	6.63	90	41	235	16500	2800	18.7	2200

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCl (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE				WE	WE + PE
PV-1	19.9	0/6	1/6	0.30	0.73	1.04	.0837	.116		
PV-2	23.2	1/6	4/6	0.39	1.07	1.21	.0935	.156		
PV-3	27.0	0/6	3/6	0.44	1.19	1.06	.0913	.148		
PV-4	30.8	1/6	4/6	0.54	1.33	0.98	.103	.138		
PV-5	33.2	0/6	5/6	0.63	1.22	1.02	.110	.093		

<sup>a</sup> Calculated from the time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.



Table 13. Results for flexible PU foam

Test	Irrad. Time (min.)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to Ign. (s)	Flame out (s)	Average conc. <sup>a</sup>			
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCN (ppm)
FP-1	15	96.8	8.22	8.22	100	15	115	42800	660	15.7	22
FP-2	15	96.8	10.09	10.08	100	15	120	N.A.	N.A.	N.A.	24
FP-3	15	96.8	10.39	10.38	100	20	140	53900	1800	14.1	44
FP-4	15	96.8	11.00	11.00	100	19	150	54400	1300	13.8	50
FP-5	15	96.8	11.40	11.39	100	20	145	57300	1950	13.7	74
FP-6	15	96.8	12.16	12.12	100	10	165	62600	1400	13.3	40

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCN (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE				WE	WE + PE
FP-1	41.1	0/6	0/6	0.63	0.64	2.04	0.223	.0010		
FP-2	50.4	0/6	0/6	N.A.	N.A.	N.M.	N.M.	.0011		
FP-3	52.0	1/6	3/6	1.09	1.16	1.95	.0424	.0014	58	52
FP-4	55.0	4/6	4/6	1.02	1.11	1.95	.0279	.0015	(53-63)	(46-59)
FP-5	57.0	4/6	4/6	1.29	1.42	1.94	.0425	.0019		
FP-6	60.6	3/6	4/6	1.01	1.08	2.11	.0299	.0011		

<sup>a</sup> Calculated from the time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

N.A. Not applicable

N.M. Not measured

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.

Table 14. Results for melamine type PU foam

Test	Irrad. Time (min.)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc. <sup>a</sup>				
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCN (ppm)	NO <sub>x</sub> (ppm)
MF-1	15	19.5	2.60	2.49	96	40	195	12800	330	19.4	130	110
MF-2	15	19.5	2.80	2.68	96	20	290	9180	430	19.8	220	25
MF-3	15	19.5	3.00	2.86	95	30	150	10800	520	19.6	170	50
MF-4	15	19.5	3.40	3.22	95	35	275	12800	440	19.2	N.M.	80
MF-5	15	19.5	3.40	3.27	96	30	285	12800	520	19.2	N.M.	80
MF-6	15	19.5	3.40	3.27	96	30	165	11500	620	19.6	180	40
MF-7	15	19.5	4.01	3.89	97	45	190	15400	590	18.9	260	60

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCN (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE				WE	WE + PE
MF-1	12.5	2/6	3/6	0.81	1.03	2.13	.041	.017	14.8 (12.6-17.3)	12.5 (9.7-16.1)
MF-2	13.4	4/6	5/6	1.26	1.63	1.44	.051	.026		
MF-3	14.3	2/6	2/6	1.01	1.29	1.64	.059	.018		
MF-4	16.1	-	-	-	-	1.58	.044	N.M.		
MF-5	16.4	-	-	-	-	1.61	.051	N.M.		
MF-6	16.4	4/6	6/6	1.11	1.41	1.52	.061	.017		
MF-7	19.5	6/6	6/6	1.52	1.94	1.59	.044	.019		

<sup>a</sup> Calculated from the time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

N.M. Not measured

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.

Table 15. Results for vinyl fabric

Test	Irrad Time (min)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g)	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc. <sup>a</sup>					
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCN (ppm)	HCl (ppm)	NO <sub>x</sub> (ppm)
1	15	22.6	7.0	5.7	81	7	130	16500	2700	18.7	N.M.	642	N.M.
2	15	22.6	8.0	6.5	81	7	140	19100	3200	18.3	5	569	13
3 <sup>c</sup>	15	22.6	8.0	6.3	79	7	150	14800	2300	19.4	6	575	10
4	15	22.6	8.4	6.7	80	7	150	19300	3300	18.4	8	237	12
5	15	22.6	9.0	7.3	81	7	155	20800	3600	18.1	13	1214	16

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCN (g/g)	Yield HCl	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE					WE	WE + PE
1	28.6	0/6	2/6	0.62	0.81	1.20	0.115	N.M.	0.033	ND	32 (28-37)
2	32.5	0/5	2/5	0.78	0.94	1.18	0.120	0.0002	0.025		
3	31.4	0/6	3/6	0.53	0.69	0.95	0.103	0.0002	0.027		
4	32.8	3/6	4/6	0.79	0.87	1.20	0.122	0.0003	0.010		
5	36.6	3/6	6/6	0.90	1.25	1.15	0.119	0.0005	0.048		

<sup>a</sup> Calculated from time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

<sup>c</sup> This experiment had a small leak in the pressure relief bag during the first 10 min. of the test

N.M. Not measured

ND Not determined

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.



Table 16. Results for melamine type PU foam and vinyl fabric composite

Test <sup>c</sup>	Irrad Time (min)	Spec. Area (cm <sup>2</sup> )	Init. Mass (g) <sup>a</sup>	Mass Loss		Time to ign. (s)	Flame out (s)	Average conc.					
				(g)	(%)			CO <sub>2</sub> (ppm)	CO (ppm)	O <sub>2</sub> (%)	HCN (ppm)	HCl (ppm)	NO <sub>x</sub> (ppm)
1	15	19.3	5.5	4.7	86	6	140	14700	1400	19.1	60	137	48
2	15	16.1	5.7	5.0	88	7	145	18300	1800	18.6	94	190	53
3	15	19.3	6.1	5.2	85	7	135	19600	1900	18.4	83	189	64
4	15	19.3	6.6	5.6	85	7	140	20000	2000	18.3	114	222	61
5	15	19.3	7.0	6.0	86	6	> 170	21400	2000	18.2	120	155	68

Test	Smoke Conc. (g·m <sup>-3</sup> )	Animal lethality # dead/# tested		FED Value		Yield CO <sub>2</sub> (g/g)	Yield CO (g/g)	Yield HCN <sup>b</sup> (g/g)	Yield HCl (g/g)	LC <sub>50</sub> <sup>b</sup> (g·m <sup>-3</sup> )	
		WE	WE + PE	WE	WE + PE					WE	WE + PE
1	23.6	1/6*	1/6*	0.65	0.79	1.24	0.075	0.004	0.008	27 (25-29)	26 (24-28)
2	25.0	0/6	1/6	0.95	1.16	1.50	0.091	0.006	0.011		
3	26.0	2/6	5/6	0.92	1.11	1.55	0.090	0.005	0.011		
4	28.1	5/6	5/6	1.11	1.36	1.47	0.090	0.006	0.011		
5	30.0	5/6	6/6	1.19	1.43	1.47	0.092	0.006	0.007		

<sup>a</sup> Calculated from time-integrated concentration under the instrument response curve.

<sup>b</sup> Calculated based on mass consumed per chamber volume according to the method of Litchfield and Wilcoxon.

<sup>c</sup> In these experiments, the foam was 27.3% and the fabric was 72.7% of the mass.

WE Animals died within the 30-min exposure.

WE+PE Animals died either within the 30-min exposure or during the 14-day post-exposure period.

\* Some question as to whether animal died as a result of toxic exposure.

Table 17. The values obtained for  $LC_{50}$  —raw and corrected

Material	Raw $LC_{50}$ ( $g \cdot m^{-3}$ )		$LC_{50}$ (corr) ( $g \cdot m^{-3}$ )	
	Value	95% confidence interval	Value	95% confidence interval
Douglas fir	56	54 - 57	21	20.6 - 21.1
Rigid polyurethane foam	22	21.6 - 22.2	14	14.3 - 14.5
PVC	26	21 - 31	16	13.7 - 17.5
Flexible polyurethane foam	52	46 - 59	18	16.9 - 18.4
Melamine type PU foam	13	10 - 16	8	7.2 - 10.4
Vinyl fabric	32	28 - 37	19	17.7 - 20.9
Melamine type PU foam and vinyl fabric composite	26	24 - 28	15	14.7 - 16.2

**Visual Inspection.** When the fire community has sufficient experience with  $LC_{50}$  measurements using this approach, some groupings of products could be exempted from further determinations **by inspection** and be described as “having an  $LC_{50}(\text{corr})$  greater than  $8 \text{ g} \cdot \text{m}^{-3}$ .” Some possible examples follow.

- Wood and other celluloseics, since all species would be expected to show  $LC_{50}$  values similar to the Douglas fir value cited here.
- Synthetic materials containing only C, H, and O.
- Polymer/additive mixtures that have been shown to follow the N-Gas Equation (i.e., produce no additional toxicants) and have  $LC_{50}$  values greater than  $8 \text{ g} \cdot \text{m}^{-3}$ .
- Products that are only present in small quantities, perhaps demonstrated using equation (9) in Chapter 2.
- Products that would not be expected to become fuel for a flashed-over fire, such as those items only installed behind a sufficiently protective barrier.



Based on an overview of reported toxic potency values, this process could result in an extremely small fraction of commercial products needing to be measured. Note that this applies to post-flashover scenarios only.

## 8.4 Use of the $LC_{50}$ data in hazard analyses

In the previous section, the conservative value of  $8 \text{ g}\cdot\text{m}^{-3}$  was offered as representing the toxic exposures. However, people are likely to inhale smoke from real fires for a range of times. It is therefore pertinent to discuss how one might obtain appropriate  $LC_{50}$  values for other exposure times without having to perform additional measurements.

It has often been convenient to presume that the product of the  $LC_{50}$  and the exposure time is a constant. Thus, the  $LC_{50}$  for, e.g., a 5-minute exposure would be  $30/5 = 6$  times the  $LC_{50}$  for a 30-minute exposure. Reference [67] contains data for exposures ranging from 5 to 60 minutes, enabling a test of this convention. As was shown in the previous chapter, most of the smoke toxicity results from inhalation of CO and  $\text{CO}_2$ . Figure 12. shows (a) the product of exposure time and  $LC_{50}$  value for CO in the presence of 5%  $\text{CO}_2$  and (b) the  $LC_{50}$  value alone, both plotted as a function of exposure time. The first curve has a positive slope of about  $3 \text{ g}\cdot\text{m}^{-3}$ ; i.e., the above convention is not conservative at shorter exposure times.

From this analysis, a better approximation than the constant value for the  $LC_{50}\cdot t$  product is warranted. Empirically, the values of  $LC_{50}\cdot t^{1/2}$  (for the CO/ $\text{CO}_2$  mixtures above) are a nearly constant  $27 \pm 2 \text{ g(CO)}\cdot\text{m}^{-3}\cdot\text{min}^{1/2}$ . (Others have found similar relationships for this type of data [94],[95].) Following the procedure in Section 8.3, this value is divided by  $0.2 \text{ g(CO)}/\text{g(fuel burned)}$  and adjusted for the factor of 3 uncertainty in the bench-scale test data. One then obtains an approximation for the toxic potency of post-flashover smoke of  $LC_{50} \approx 45 \text{ g}\cdot\text{m}^{-3}\cdot\text{min}^{1/2}\cdot\text{t}^{-1/2}$ . This approximation is offered for hazard calculations for exposure times greatly different from 30 minutes.

## 8.5 Use with pre-flashover fires

Chapter 3 showed that for pre-flashover, flaming fires, smoke toxicity is usually less life threatening than are thermal effects. There will, however, be some products whose smoke is of much higher than ordinary toxic potency. It is thus important to be able to measure these low  $LC_{50}$  values, and it is desirable to use the same apparatus as for post-flashover toxic potency data.

$LC_{50}$  values for products burning pre-flashover can be determined simultaneously with the post-flashover values, using the same procedure described above, but without the correction for post-flashover CO. The rationale for doing so follows.

For a small, developing fire, the bench-scale specimen in the radiant apparatus is, in fact, a fair representation of the full-scale fire. The thermal boundary conditions are appropriate: they are radiative, and they are from one face only. A small fire will impose about  $35 \text{ kW}/\text{m}^2$  on an adjacent unburned



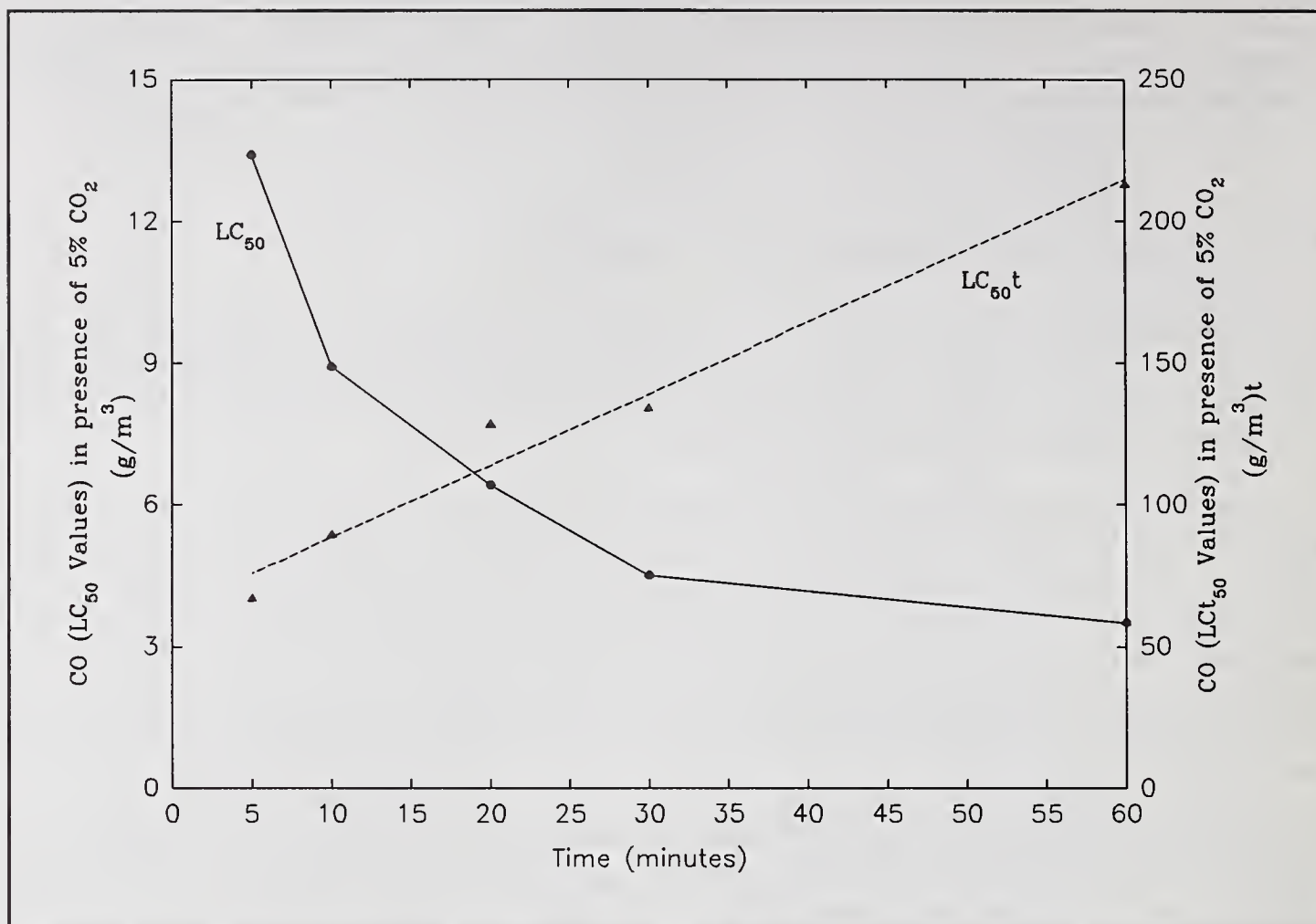


Figure 12. Toxic potency of CO<sub>2</sub> potentiated CO as a function of exposure time.

surface [96],[97], although values around 48 kW/m<sup>2</sup> are common, and values over 100 kW/m<sup>2</sup> can be measured. Thus, an irradiance of 50 kW/m<sup>2</sup> for a pre-flashover test is somewhat high, but is by no means out of line. (If a specific scenario involves a heating flux lower than 50 kW/m<sup>2</sup>, it can be accommodated readily in the procedure.)

The other difference between pre- and post-flashover fires concerns the atmospheres. For post-flashover applications, the LC<sub>50</sub> was corrected to account for the fact that the oxygen supply in a post-flashover fire is limited. This effect could not be simulated in a bench-scale apparatus and needed to be put in by a computational procedure. In the pre-flashover fire, however, such a shortage of oxygen does not occur.

## 8.6 Fulfillment of criteria for a good test

Chapter 5 provided a list of the criteria against which a toxicity test method should be examined to determine if it satisfies basic expectations. The proposed method is now reviewed against these criteria.

### 8.6.1 Types of data to be obtained

- a. Toxic potency should be measured, and should be measurable and reportable in correct ( $\text{g}\cdot\text{m}^{-3}$ ) units.  
This is done.
- b. The chemical data necessary for the N-Gas Model should be properly obtainable from the test. We reviewed the N-Gas Model and demonstrated why it should be used in order to produce a test which is as simple to conduct as possible and which minimizes the usage of animals.  
This is done by using Procedures A, B, and C.

### 8.6.2 General test method requirements

- a. Adequate repeatability.  
The statistical confidence limits obtained are shown in Table 17. The average deviation for the 95% confidence limits is  $\pm 12\%$  of the mean. This is certainly acceptable for a bioassay test.
- b. Adequate reproducibility.  
This issue cannot be completely answered until an inter-laboratory trial (a “round-robin”) is conducted. However, a comparison of SwRI and NIST data is encouraging. The results for raw  $\text{LC}_{50}$  were discussed in the previous section; differences here indicate differences in protocols used, not lack of reproducibility problems. These results do not show the kind of data scatter for wood materials as was reported by Hirschler [98]; however, natural cellulosic materials may be prone to variability problems and should be avoided for use as reference materials.
- c. Adequate validity.  
Validation of the method is described in the next chapter.
- d. Safety to operator.  
The apparatus has been in use in two laboratories for over one year now and has a good safety record.
- e. Safety to environment, *i.e.*, no excessive pollution.  
The procedures here are typical of any small-scale fire test. In most jurisdictions, only a competent venting system will be required. In some locales, extra measures such as afterburners could be mandated.
- f. Affordable apparatus costs.  
The cost of the apparatus is estimated to be comparable to the cup furnace apparatus.
- g. Tests to be conductible reasonably quickly and efficiently.  
The procedures, because of reliance on the N-Gas Model, take substantially less time than with the cup furnace method. A labor savings of up to a factor of 3 can be expected.

- h. Sample preparation not to be excessively difficult.  
Sample preparation is routine for any fire test laboratory.
- i. Ease of cleaning and maintaining of the apparatus.  
The cleaning procedures for the exposure chamber are similar to those for the cup furnace method. Cleaning the quartz combustion chamber and the connecting chimney are more difficult, but not unduly so.

### 8.6.3 Specific test method requirements

- a. The test should represent the chosen full-scale combustion scenario correctly.  
We consider this according to each of the specific items below.
  - 1. Composite specimens should be testable as composites.  
The design of the specimen holder system and the procedures developed have been specifically geared towards the capability to test composites. The depth of 50 mm allowed has been found adequate to address almost all product needs, as verified by experience in testing to this depth with the Cone Calorimeter.
  - 2. Since in the post-flashover fire radiant heating predominates, the specimen should receive uniform, well-controlled radiant heating.  
The design of the heating lamp assembly and the calibration procedures allows for this to be done satisfactorily. Each lampholder is separately adjustable for height, lateral location, and angle of inclination. The verification of flux uniformity is done with a special calibration jig, described in the Annex.
  - 3. The test must be set up so that specimens are burned to their natural conclusion in much the same way they would in real-scale fires; i.e., a specimen should not artificially be stopped from burning before all the combustibles that can burn do burn up.  
Specimens are burned for 15 min in the combustion cell. This allows products of reasonable thickness to be tested in a process simulating natural combustion. The specimen thickness can be chosen to obtain an accurate measure of LC<sub>50</sub>, but to avoid specimens so thick that burning is highly incomplete.
  - 4. For establishing the correlation of the bench-scale result to the full scale by chemical analysis, the specimen's yields of various toxic gases species must be measurable.  
The method does this successfully and follows the guidance for good practice laid down in ASTM E 800.
  - 5. For establishing the correlation of the bench-scale result to the full scale by bioassay, both the LC<sub>50</sub>s and the causes of animal deaths need to be measured and recorded.  
This is done by examining during- and post-exposure lethality and analyzing the contributions of the gases with the use of the N-Gas Model.
  - 6. There must be a minimum loss of gases and particulates.  
The best indicator of this phenomenon is HCl loss, since this combustion product is the most readily depositable of all the toxicologically important combustion



products. Experience with the real-scale test program previously conducted [69] has suggested that yields of HCl in the cup furnace method, in the radiant apparatus, and in the Cone Calorimeter are very similar. Interestingly enough, it was found in that program that the yields in the real-scale tests were smaller. This was attributable both to the travel distance for gases between the burn room and the target room and due to losses in the sampling system from the target room. On the whole, those findings confirmed that HCl is very easy to lose and that the closed box type of test, as represented by either the radiant apparatus or the cup furnace method, is one of the systems *least* prone to losses.

These findings are further confirmed in additional studies at SwRI. When four different polyvinyl chlorides were tested in the radiant apparatus and in the cup furnace method, it was found that, within the limits of error of the measurements (ranging from 15% to 57%), there was seen to be no difference in the yield of HCl in these two bench-scale toxicity test methods [99].

7. Specimens should be tested without crushing, powdering, etc.  
Specimens are prepared from the finished product without changing its basic shape or nature.
8. Specimens of a wide range of densities, thicknesses and toxicities which may occur in the real world should be testable without needing to be excluded or “beating” the test.  
A maximum specimen volume of  $480 \text{ cm}^3$  is accommodated. The maximum  $\text{LC}_{50}$  which can be measured is  $= 2.4 \rho$ , where  $\rho$  = specimen density ( $\text{kg}\cdot\text{m}^{-3}$ ). For example, for a wood product having  $\rho = 500$ , a maximum  $\text{LC}_{50}$  of  $1200 \text{ g}\cdot\text{m}^{-3}$  can be measured. For a foam having a density of  $30 \text{ g}\cdot\text{m}^{-3}$  this would be a maximum  $\text{LC}_{50}$  of  $72 \text{ g}\cdot\text{m}^{-3}$ . This is well above the  $\text{LC}_{50}$  value of  $8 \text{ g}\cdot\text{m}^{-3}$  derived in Section 8.2. On the low end of the  $\text{LC}_{50}$ , there is no unique lower limit. Infinitesimally small specimens can be tested; this, however, presents practical difficulties if they are composites. More uncertainty, however, can be accepted at small- $\text{LC}_{50}$  end of the scale, since high precision would not usually be required in computing situations where “supertoxic” products with large exposed areas and large mass loss rates would occur.
9. Protective outer layers should be realistically treated by the test procedure.  
The layers are tested exactly as occurring in the end-use product. For products showing special flammability problems around the edges, well-known procedures [100] can be used by the testing laboratory to ensure that a representative test has been made.
10. Edge effects should not be disproportionately influencing the results.  
Specimens are exposed on a single face to radiation, and side heating is avoided.
11. Samples should be tested in the horizontal, face-up orientation.  
This was successfully implemented in the test method.

12. The combustion environment to which the specimen is subjected in the test should correspond to that in the design scenario. This includes correct oxygen levels, and also the absence of such phenomena as re-circulation or re-combustion of combustion products, catalytic conversion, etc., unless they are also present in the design fire scenario.

Chapter 7 examined this requirement with regard to CO production, and showed that this condition is generally impossible for any bench-scale test to meet in its entirety. Thus, alternative provisions need to be made to correct the results for known biases. Such a procedure was evolved for computing  $LC_{50}(\text{corr})$ . Re-circulation occurs in the radiant test apparatus, but is limited due to the large 200 L box used and can correspond to re-circulation occurring in post-flashover fires. Catalytic conversion and re-combustion are avoided by not submerging a heater into the combustion product volume.

13. Since the test is to be used for, at least, post-flashover fires, it is important that the test data be in such a format so that the prediction of several items simultaneously burning in a room could be done.

Good hazard models include multiple burning objects. This provides either an indication that the  $LC_{50}$  is indistinguishable from  $8 \text{ g}\cdot\text{m}^{-3}$  or an actual  $LC_{50}$  for each. The model then integrates.

- b. The test should provide for a well-characterized, toxicologically sound exposure to animals.

1. There is a very broad consensus pointing to the wisdom of providing animals with a 30-min exposure period, followed by a 14-day post-exposure observation period.

Done.

2. The rat has been chosen in the largest number of combustion toxicity tests. There are persuasive reasons (reasonable cost and availability; closest overall model to human response among animals of similar cost) for continuing with this practice.

Done.

3. The gases to which the animals are exposed should consist of the total combustion products from the specimen's burning history.

The animal exposure chamber collects gases from the entire burning history of the specimen.

4. As close to a square-wave exposure as possible is desired.

The gases are collected in a closed system and reasonable leak-tightness is maintained. The exposure is nearly constant during 15 through 30 mins and is a rapidly rising function during the first 15 min.

5. Animal condition during the test should be adversely affected as little as possible by causes other than specimen toxicity.

The 200 L size of the animal chamber is adequate to prevent toxic buildup of  $\text{CO}_2$  or of lower levels of  $\text{O}_2$  due to animal respiration.

The animals are exposed in a head-only configuration, which reduces undue stress from heat to the body.

The restraint system is well-proved and does not cause undue physical injury as long as the rats used are in the correct weight range.

6. The usage of animals should be minimized, consistent with obtaining data of acceptable quality.

This is accomplished by implementing the N-Gas Model as an integral part of the test method. Roughly a factor of 3 savings in animal use is seen, compared to full LC<sub>50</sub> bioassays normally performed in fire toxicity studies.



## 9 Validation results

This chapter considers the general requirements for validating a bench-scale combustion toxicity test method, then examines the data available for the present method.

### 9.1 Background

Attempts to validate bench-scale toxicity tests have generally been limited and not entirely convincing. Thus, during the course of 1989-1990, a pilot-scale validation project was conducted at NIST. The project had several objectives. Foremost was to establish quantitative, plausible validation hypotheses by which future exercises of validating bench-scale toxicity tests could be accomplished. Another objective was to provide illustrative results by subjecting two bench-scale tests to the procedures developed. One of the two methods examined was the radiant apparatus method. The data used for the method were based on the earliest available data from SwRI. In real scale, three materials were tested in a burn room/corridor/target room configuration. All of the test details were reported in the Technical Note [69]; here, some of the salient findings are summarized.

### 9.2 The validation hypotheses

The following five validation hypotheses were established as pertinent.

1. The equal  $LC_{50}$  hypothesis

*$LC_{50}$  values, as measured in the bench-scale test and in the real-scale one, agree to within the acceptable uncertainty.*

2. The primary toxic gases hypothesis

*The bench-scale test shows the same primary toxic gases as the real-scale test.*

3. The equal yields hypothesis

*The yields of the measured toxic gases (except CO) are the same, to within the acceptable uncertainty, in the bench-scale and in the real-scale tests. Yields of CO from bench-scale tests are not compared directly against real-scale yields; rather, a post-correction method is used for CO values from bench-scale tests.*

4. The N-Gas hypothesis

*The real-scale and the bench-scale results agree, to within the acceptable uncertainty, with predictions based on measured gas concentrations and computations made according to the N-Gas Model.*

5. The type of death hypothesis

*The type of death (within- or post-exposure) is similar for the bench-scale and for the real-scale tests.*

To be usable, the hypotheses must be accompanied by a statement as to what factor of agreement is expected between the bench-scale and the real-scale results. The study showed that a factor of 3 was useful and achievable.

### 9.3 Performance of the radiant test method

The validation study mentioned [69] not only established the basis for validation procedures, but also provided validation data for the present radiant apparatus method. Three materials were used for this validation: Douglas fir, rigid polyurethane foam, and PVC. These are, in fact, the first 3 of the 7 products examined in detail in the present study. While a choice of only three materials is, of course, limited, the materials were chosen with significant care. The objective of any proof-by-enumeration validation exercise is to challenge the method with as diverse a set of test cases as possible. The materials chosen were indeed diverse. Natural cellulose and man-made plastics were included. Solid and foam plastics were represented. Materials where CO (along with CO<sub>2</sub> and low O<sub>2</sub>) is the only toxicant were compared against ones which produced significant amounts of HCl and HCN.

The results showed the following for the radiant apparatus method:

- The equal-LC<sub>50</sub> hypothesis was proved.
- The yields of non-CO species showed agreement.
- The N-Gas hypothesis was proved.
- The primary toxic gas hypothesis was proved. This hypothesis carries a *caveat* regarding oxygen levels and sorbable gases. Oxygen levels encountered in the real-scale fire will depend largely on ventilation conditions within the environment. This will, in general, only fortuitously be reproduced by any bench-scale condition. Thus, mismatch of O<sub>2</sub> on the lists of primary gases from the bench-scale vs. the real-scale test will indicate nothing more than actual ventilation conditions. Similarly, the results showed recovery of more of the fully sorbable species (e.g., HCl) in the bench-scale radiant apparatus (and also in the cup furnace apparatus) than in the real-scale environment. This, again, has very plausible reasons. The flow path lengths are normally significantly longer in the real-scale fire, thus making losses more likely there than in the small closed-box tests.
- The type of death hypothesis could not be checked out due to lack of sufficient data. However, if there were problems in this area they would equally show up as an inability to get acceptable N-Gas predictions. Since the latter were well-behaved, there should be no major conflicts with this hypothesis.

Thus, even with the small number of materials used to challenge the method, we conclude that a successful demonstration of validity has been made. As opportunities arise to subject the protocol to additional validation, we will pursue them, but we do not anticipate that these conclusions would undergo serious revision.

## 10 Conclusions

A complete package has been assembled for the engineering analysis of fire toxicity within the context of fire hazard. The package comprises:

- A determination that attention should be focused on the post-flashover fire, due to the preponderant fraction of U.S. fire deaths under these conditions.
- A determination that the endpoint sought should be lethality.
- A detailed examination of the requirements that a useful bench-scale toxic potency measurement method has to meet and the data it should produce.
- A bench-scale toxic potency measurement method which meets these requirements.
- A computational procedure for correcting the results obtained so as to indicate CO levels to be expected from real-scale post-flashover fires.
- Procedures for reducing the usage of animals and broadening the applicability of data by interpreting gas measurement data from the method in the context of the N-Gas Model.
- A procedure for identifying whether the product produces smoke within the ordinary range for post-flashover fires.
- Validation results against real-scale fires, demonstrating that the bench-scale results can successfully predict such fires.

The package is based on a careful analysis of fire death statistics and fire modeling results which indicate that the major concern is with post-flashover fires. A method is also provided for utilizing the data from the bench-scale method for determining smoke toxic potency from flaming, pre-flashover fires. Other fire types are considered highly specialized and have not been treated within the scope of this study.



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# ANNEX — The Measurement Method

## STANDARD MEASUREMENT METHOD FOR THE DEVELOPMENT OF DATA FOR USE IN TOXIC FIRE HAZARD MODELING

### 1. Introduction

1.1 The pyrolysis or combustion of every combustible material produces smoke which is toxic. It is, therefore, desirable to establish a standard test method for the development of data for use in toxic hazard modeling. Such data include quantification of the toxicity of the smoke, along with the fire parameters of time to ignition and mass burning rate. It is also desirable to ascertain whether or not the observed toxicity is attributable to the major common toxicants.

### 2. Scope

2.1 This laboratory procedure is designed to assess the acute inhalation toxicity of combustion products generated under post-flashover conditions. It also provides for a determination of the time to ignition and mass burning rate.

2.2 The procedure entails exposing full-thickness specimens to radiant heaters and allowing them to combust completely. The amount of combustion products generated is adjusted by changing the exposed area of the specimen.

2.3 Specimens are exposed to a radiant heating flux of 50 kW/m<sup>2</sup> for 15 min. An electric spark is used for ignition.

2.4 Lethal toxic potency values are estimated using calculations which employ combustion atmosphere analytical data for carbon monoxide, carbon dioxide, oxygen (vitiation) and, if present, hydrogen cyanide, hydrogen chloride, and hydrogen bromide.

2.5 Estimated toxic potency values are verified with two tests in which the test animals (rats) are exposed. Verification is considered successful if exposure to a combustion product concentration equal to 70% of that needed for the estimated LC<sub>50</sub> produces no deaths, while exposure to a concentration equal to 130% of that needed for the estimated LC<sub>50</sub> produces 100% deaths.

2.6 The method is limited to test specimens no larger than 76 mm x 127 mm (3 in x 5 in), with a thickness no greater than 50 mm (2 in). Specimens are intended to be representative of finished products, including composite and combination systems.

2.7 This method has been designed to generate data for fire hazard analysis, to provide a means for material and product evaluations (including composites), and to assist in the research and development of materials and products.

2.8 The values stated in SI units are to be regarded as the standard.

2.9 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used alone to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a product in a particular end use.*

2.10 *This standard does not purport to address all of the safety problems 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 (especially with regard to the institutional care and*

use of experimental test animals) prior to use. For specific hazard statements see Section 8.

### 3. Referenced Documents

#### 3.1 *ASTM Standards*.<sup>1</sup>

E 176 Terminology Relating to Fire Standards.

E 800 Standard Guide for Measurement of Gases Present or Generated During Fires.

#### 3.2 *International Standards Organization Standard*.<sup>2</sup>

ISO/TR 9122 Toxicity Testing of Fire Effluents.

### 4. Terminology

4.1 *Definitions*—For definitions of *general* terms used in this test method, refer to ASTM Standard E 176.

#### 4.2 *Descriptions of Terms Specific to This Measurement Method*:

4.2.1 *concentration-time curve*—a plot of the concentration of a gaseous toxicant (ppm) or smoke (mass of material consumed per chamber volume,  $\text{g}\cdot\text{m}^{-3}$ ) as a function of time.

4.2.2 *Ct product*—the concentration-time product in  $\text{ppm}\cdot\text{min}$  obtained by integration of the area under a concentration-time curve.

4.2.3 *time-integrated concentration*—the concentration-time product in  $\text{ppm}\cdot\text{min}$  obtained by integration of the area under a concentration-time curve and dividing by the time (ppm).

4.2.4 *Fractional effective Exposure Dose (FED)*—the ratio of the Ct product or the time-integrated concentration for a gaseous toxicant produced in a given test to that Ct product or time-integrated concentration of the toxicant which has been statistically determined from independent experimental data to produce lethality in 50% of test animals within a specified exposure (deaths only during the exposure) or within a specified exposure plus post-exposure observation time.

4.2.5 *mass loss concentration*—the mass loss of a test specimen per unit exposure chamber volume ( $\text{g}\cdot\text{m}^{-3}$ ).

4.2.6 *post-flashover*—the stage of a room fire when the average air temperature in the upper half of the room exceeds 600 °C.

4.2.7 *LC<sub>50</sub>*—the specimen mass loss per chamber volume ( $\text{g}\cdot\text{m}^{-3}$ ) or time-integrated gas concentration (ppm) which causes 50% of the animals to die during or following a specified time exposure. In this test procedure, LC<sub>50</sub> values are estimated based on 30-minute exposures and a 14-day post-exposure observation period.

### 5. Summary of Test Method

5.1 This method uses an apparatus wherein a test specimen is subjected to ignition while exposed to 50 kW/m<sup>2</sup> of radiant heat for 15 minutes. The smoke is held within a 200 L chamber which is joined to the combustion cell through a connecting chimney. Three procedures are used in the test. Under Procedure A, concentrations of the major gaseous toxicants are monitored over a 30-minute period, with

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<sup>1</sup> *Annual Book of ASTM Standards*, Vol. 04.07.

<sup>2</sup> Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.



Ct products for each being determined from integration of the areas under the respective concentration-time plots. The Fractional effective Exposure Dose (FED) is computed from these results. The predicted value of the  $LC_{50}$  is taken as corresponding to  $FED = 1$ . Under Procedure B, the estimated  $LC_{50}$  is checked in verification tests by exposing six rats, restrained for head-only exposure, for 30 minutes to the smoke produced under two conditions: for  $FED = 0.7$  and for  $FED = 1.3$ . If no rats die during the 30-minute exposure, or within 14-days post-exposure to the mass loss concentration corresponding to  $FED = 0.7$  and all six rats die during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to  $FED = 1.3$ , the estimated  $LC_{50}$  is assumed to be confirmed. If such results are not obtained, then in Procedure C a complete experimental and statistical determination of the  $LC_{50}$  value is performed.

## 6. Significance and Use

6.1 This test method is used to estimate on the basis of specific toxic gas concentrations, and subsequently confirm with animal exposures, the lethal toxic potency of smoke produced upon exposure of a product to fire. The confirmation determines whether certain major gaseous toxicants account for most of the observed toxic effects and the lethal toxic potency. If the estimated  $LC_{50}$  value is not adequately confirmed, indicating the potential for unusual or unexplained toxicity, the estimated value obtained from this test method needs to be examined more thoroughly. In that event, more precise  $LC_{50}$  values need to be determined.

6.2 This radiant heat flux of  $50 \text{ kW/m}^2$  is chosen to represent a post-flashover fire condition.

6.3 The time history of the oxygen/fuel ratio occurring in a real fire is important in determining the CO produced in the fire. Bench-scale tests do not have the capability to simulate this variable; instead, a method is used to correct the raw  $LC_{50}$  values obtained in the test to reflect the CO which has been shown to be produced in actual, post-flashover fires.

6.4 Tests are conducted on small-size specimens that are representative of materials, products, or composites in their intended end use. This test method is not specified for end-use products that do not have planar, or nearly planar, external surfaces.

6.5 This test method provides a means for additional analytical and physiological measurements which provide more detailed information on the nature of the toxic effect.

6.6 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.

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

6.8 This test method does not assess incapacitation. In most cases, it can be assumed that incapacitation will occur at levels lower than the lethal toxic potency values. However, it has been observed that exposure levels sufficient to cause a post-exposure death have not been sufficient to produce incapacitation during the exposure.

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

## 7. Apparatus

### 7.1 *Animal Exposure Chamber:*

7.1.1 The animal exposure chamber, shown in Figures A1 and A2, can be constructed of clear polycarbonate or polymethylmethacrylate with a 200 L nominal volume. Its inside dimensions are 1220 mm x 370 mm x 450 mm (48 in. x 14-1/2 in. x 17-3/4 in.). The six animal ports are located in a



horizontal row, approximately half way from the bottom to the top of the chamber, in the front wall. The ports are intended to allow exposure of the heads of the rats. Thermocouples are placed at animal ports No. 1, 3, and 6 to monitor the temperature to which the animals are exposed. A plastic bag with at least a 49 L volume is attached to a special port at the end of the chamber to provide for gas expansion and to serve as a blow-out panel. 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. There are two doors in the animal 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, the chimney, the smoke shutter, and other components. Opening of the doors keeps the exposure chamber from overheating during the calibration of the heat lamps and provides fresh air to the animals immediately prior to testing.

## *7.2 Chimney:*

7.2.1 The chimney (Figure A3) is a stainless steel assembly approximately 30 × 300 mm (1-1/4 × 11-3/4 in.), inside dimensions, and 300 mm (11-3/4 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 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<sup>3</sup>). 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.

## *7.3 Smoke Shutter:*

7.3.1 The smoke shutter is made of stainless steel plate and is 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/m<sup>3</sup>), which is further covered with 0.1 mm (0.005 in) thick stainless steel foil.

## *7.4 Combustion Cell:*

7.4.1 The combustion cell (Figures A4, A5, and A6) is a horizontal quartz tube with a 127 mm (5 in.) inside diameter and approximately 320 mm (12-1/2 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 as a removable plug for the open end (see Figure A6). The combustion cell has a rectangular opening on the top parallel to the axis of the cylinder with a “collar” which allows it to fit securely into the chimney. The bottom of the cell has a hole for the rod connecting the specimen support platform and the load cell. The sealed end of the combustion cell is fitted with a standard tapered glass joint for the electric sparker.

7.4.2 The combustion cell is supported by a metal frame that also holds the load cell which monitors mass loss rate. This entire frame is supported by a laboratory jack which holds the combustion cell tightly to the chimney during experimentation, and allows the combustion cell to be lowered for removal and cleaning. The load cell is always at a fixed distance from the combustion cell.

## *7.5 Radiant Heaters:*

7.5.1 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 (Figure A4) are attached to adjustable metal frames, which allow the lamps to be moved vertically, laterally, and rotated, in such a way as to give a uniform flux field across the sample surface.

7.5.2 To keep the lamps from overheating, cooling water must be circulated through their respective holders. Each pair of lamps requires a minimum flow of 600 ml per minute (0.16 GPM). A rotameter is installed onto each coolant line to give a visual check of coolant flow during the test. To prevent accidentally operating the lamps without the required cooling water, a reverse-acting pressure switch located in the cooling water line ensures that cooling water is flowing before allowing the lamps to operate.

7.5.3 The irradiance of the lamps must be held at a preset level corresponding to the required radiant flux. This can be accomplished by various control methods; the procedure described here uses a temperature controller and two thermocouples (Type K) placed between the lamps and the combustion cell and wired in parallel.

7.5.4 The irradiance from the lamps is to be uniform within the central area of the sample holder to within  $\pm 10\%$ . Figure A7 shows the calibration holder used to determine the uniformity of the radiant field from the lamps. If the field is found not to be adequately uniform, the lamp holders must be repositioned, as necessary.

#### *7.6 Temperature Controller:*

7.6.1 When a temperature controller is used for maintaining the required radiant flux, the quartz lamps output is controlled by a thermocouple signal to the temperature controller. The outputs from the two Type K 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, if needed, the power controller from being driven wide-open. The power controller is selected to be compatible with the radiant heat lamps used.

#### *7.7 Heat Flux Meter:*

7.7.1 The total heat flux meter shall be of the Schmidt-Boelter (thermopile) type, or equivalent, with a design range of at least  $75 \text{ kW/m}^2$ . 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  $\pm 3\%$  and a repeatability within 0.5%.

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

7.7.3 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. Figure A7 indicates a calibration bracket suitable for this purpose.

#### *7.8 Ignitor:*

7.8.1 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 degrees and flattened on the end and positioned to give the appearance of the tip of an automotive-type spark plug. The gap between the two rods shall be about  $2 \text{ mm} \pm 0.5 \text{ mm}$ . These rods pass through a 29/42 male ground glass stopper, forming a gas-tight seal with a mating joint found on the combustion cell (Figure A6). These two rods are connected to the high-voltage spark system which uses a 10 kV transformer (Figure A8). To reduce the propagation of radio frequency interference into the instrumentation, a 20,000  $\Omega$ , 5 watt resistor is connected in series with one of the electrodes. The spark gap is positioned approximately 25 mm (1 in.) above the center of the top surface of the specimen,

inside the combustion cell.

#### **7.9 Specimen Holder:**

7.9.1 The specimen holder is a stainless steel assembly approximately 76 mm x 127 mm (3 x 5 in.), inside dimensions, and 50 mm (2 in.) deep (Figure A9). The specimen is backed by a layer of ceramic fiber insulating blanket on a stainless steel plate. It is positioned for testing on the specimen platform, inside the combustion cell.

#### **7.10 Load Cell:**

7.10.1 The general arrangement of the load cell and specimen holder is illustrated in Figure A4. The load cell is installed under the combustion cell. 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. The load cell shall be well-insulated against the heating effects of the radiant heaters.

#### **7.11 Gas Sampling:**

7.11.1 The gas sampling system shall be designed using the requirements specified in ASTM E 800. Since this is a closed system, gases which are removed for chemical analysis and which can be recirculated to the animal exposure chamber are returned. A suitable gas sampling arrangement is shown in Figure A10. It includes a pump, a glass wool filter at the sampling port, a cold trap to remove soot and moisture, and a pressure relief valve which returns all flow not required by the CO, CO<sub>2</sub>, and O<sub>2</sub> 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.

##### **7.11.2 Gas Analyzers:**

7.11.2.1 The oxygen analyzer shall have a range from 0 to at least 21%.

7.11.2.2 The carbon dioxide analyzer shall have a range encompassing at least 0 to 10%.

7.11.2.3 The carbon monoxide analyzer shall have a range encompassing at least 0 to 10,000 ppm.

7.11.2.4 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 in E 800. For any gases where analysis methods are used which involve chemical reaction, such products are not returned to the animal exposure chamber but, rather, disposed in an environmentally correct manner.

#### **7.12 Digital Data Collection:**

7.12.1 The data collection system must have facilities for the recording of the output from the carbon monoxide, carbon dioxide, and oxygen analyzers; any gas analyzers used for optionally-monitored gases; the thermocouples at the animal's noses; and the load cell. The data system shall have an accuracy corresponding to 0.01 % of full-scale instrument output.

#### **7.13 Animal Restraint:**

7.13.1 Animal restrainers designed to permit exposures of only the animals' heads shall be used. A detailed illustration of one animal restrainer meeting this requirement is given in Figure A11. Openings in the animal restrainer are necessary to allow for dissipation of body heat.

## **8. Hazards**

8.1 The test procedure involves high temperatures, bright lights, and combustion processes. Therefore, precautions must 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 is 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 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 specimen holder. The combustion cell, while hot, must only be touched with protective gloves.

8.2 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.

8.3 To guard against bodily or eye injury to the operator, a tinted safety shield constructed out of polymethylmethacrylate or polycarbonate in front of the combustion chamber is recommended.

## 9. Test Specimens

9.1 Test specimens shall be cut to appropriate area and thickness, no larger than 76 x 127 mm (3 x 5 in.), representing the end-use product. Raw materials (e.g., paints, adhesives, wall coverings, etc.) shall be tested on the substrate to which they are normally applied. For testing, the sides and the bottom of the specimens shall be wrapped in aluminum foil. Specimens shall be backed by at least a 6 mm thick layer of ceramic fiber insulating blanket in the specimen holder.

Note 1. Specimens which, upon testing, show loss of or reactivity with aluminum foil cannot be tested by covering the sides and bottom with aluminum foil. Such specimens must be re-tested using stainless steel foil.

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

## 10. Animals

10.1 The test animals shall be inbred 3-to 4-month-old male rats, weighing between 225 and 350 grams (also, see Section 14.3). Larger rats may undergo undue stress if forced into the restrainer. The rats shall be obtained from a reputable supplier that certifies its animals to be pathogen-free.

10.2 Maintenance and care of animals shall be performed by qualified trained personnel in accordance with guidelines of the National Institutes of Health Guide for the Care and Use of Laboratory Animals [1]. The animal housing facilities shall be inspected and the experimental plan approved by the animal care and use committee of the institution where the experiments are to be conducted.

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

10.4 Animals are to be weighed daily from the day of arrival to the end of the 14-day post-exposure observation period. Normally, 1 rat in 5 is to be used as a control.

10.5 Prior to exposure, the animals shall be weighed and secured into individual restrainers for placement into the portholes of the animal exposure chamber.

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

## 11. Calibration of Apparatus

11.1 The following parts of the test apparatus require calibration: the temperature controller, the radiant heaters, the gas analyzers, and the load cell.

### 11.2 *Calibration of the Temperature Controller:*

11.2.1 To set up the controller, first insert the flux meter in its holder 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 must already be completed. 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 over-shoot, which can occur if the controller is not tuned optimally. It is also important to avoid using settings which 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 done by either using the "load line out" function of the temperature controller, if so equipped, or else, 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  $\pm 5\%$  for the rest of the test.

### 11.3 Heater Flux Calibration:

11.3.1 For heat flux calibration, install the calibration bracket (Figure A12) and insert the heat flux meter into the proper opening. If necessary, verify that the sensing surface of the flux meter is centered at the location equivalent to that of the top of the specimen when the specimen holder is in place on the specimen platform. The ignitor shall be removed from its position during this procedure. Set the temperature controller to the desired flux temperature. Turn on the radiant heat lamps and adjust the temperature controller until the desired irradiance ( $50 \text{ kW/m}^2 \pm 10\%$ ) is achieved. Recheck the established flux level prior to the test (be certain that the combustion cell has cooled to ambient temperature before rechecking).

11.3.2 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 Figure A7. The top face of the calibration jig is to 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 (e.g.,  $50 \text{ kW/m}^2$ ) at the center hole. Adjust the power and allow at least five minutes for equilibration. Adjust the orientation of the radiant heat lamps so that no measurement at seven locations across the face of the specimen (see Figure A7) deviates more than 10% from the average.

### 11.4 Gas Analyzer Calibration:

11.4.1 At the beginning of each test, the  $\text{O}_2$ ,  $\text{CO}_2$ , and CO analyzers are calibrated by using nitrogen gas for "zeroing" and an appropriate gas mixture near to, but less than, the analyzer full-scale reading for "spanning." For zeroing,  $\text{N}_2$  flowing at the same rate and pressure as the sample gas is used. For spanning the  $\text{O}_2$  analyzer, ambient air (20.9%  $\text{O}_2$ ) is used, while the  $\text{CO}_2$  and CO analyzers are spanned with a gas cylinder containing  $\text{CO}_2$  and CO at known concentrations. Either separate gas cylinders or a single mixture containing both CO and  $\text{CO}_2$  may be used for spanning both the CO and the  $\text{CO}_2$  analyzers. *During the calibration procedure the gas return lines must be diverted into the exhaust and not into the exposure chamber to prevent inadvertent accumulation of CO and  $\text{CO}_2$ .*

11.4.2 Calibration of apparatus for analysis of optional gases (e.g., HCN,  $\text{HCl}$ , HBr, and  $\text{NO}_2$ ) shall be performed using guidance given in ASTM E 800.

### 11.5 Load Cell Calibration:

11.5.1 The load cell shall be calibrated with standard weights in the range of test specimen initially when first setting up or after making adjustments for sensitivity and range.

11.5.2 Before each test, the load cell is routinely checked with a reference weight. 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.

## 12. Procedure

12.1 Three different procedures are described in this method. Procedures A and B are performed, in order, in all cases. Procedure C is contingent upon the results obtained in Procedure B. The steps for preparation are described first, followed by the instructions for conducting the three procedures.

### 12.2 Preparation (Applicable to all Procedures, Except as Noted):

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

12.2.2 Verify that the spark ignition circuit is operational.

12.2.3 Perform the required calibration procedures specified in Section 11.

12.2.4 Weigh the specimen on a laboratory balance capable of  $\pm 0.01$  g. Prepare the test specimen as described in Section 9. Verify that the load cell readout corresponds to the appropriate weight of the specimen plus holder.

### 12.3 Running the Test—Procedure A:

12.3.1 Select a specimen exposed area for this procedure. In the absence of information from tests of similar products, select an area equal to 1/4 of the maximum 96.5 cm<sup>2</sup> area.

12.3.2 Insert the prepared specimen in the holder into the combustion chamber and close the chamber with the standard taper plug. Secure the plug with springs. Close the front door, all animal ports and access doors to the animal chamber. Ascertain that the shutter is open.

12.3.3 Activate the power to the radiant heat lamps and start the data collection.

12.3.4 Turn on the sparker and note the time ignition occurs. Turn off the sparker. Note the time of flameout. For samples which have the tendency to self-extinguish immediately (e.g., containing certain fire retardants) the sparker is to be left on until flaming ceases.

Note 2: Constant use of the sparker increases the concentration of NO<sub>x</sub> in the combustion atmosphere.

12.3.5 Close the shutter and switch off the heat lamps at 15 min. Collect the data until 30 min have elapsed.

12.3.6 Cool to ambient temperature and re-weigh the specimen still wrapped in the stainless steel foil.

12.3.7 Determine the FED estimate observed in this procedure as:

$$FED = \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{21 - LC_{50} O_2} + \frac{[HCN]}{LC_{50} HCN} + \frac{[HCl]}{LC_{50} HCl} + \frac{[HBr]}{LC_{50} HBr} \quad (1)$$

$$= \frac{m[CO]}{[CO_2] - b} + \frac{21 - [O_2]}{21 - 5.4\%} + \frac{[HCN]}{150 \text{ ppm}} + \frac{[HCl]}{3700 \text{ ppm}} + \frac{[HBr]}{3000 \text{ ppm}} \quad (2)$$

where the values of all gas concentrations are the integrated values under the concentration-time curve taken over the 30-minute test period and divided by 30. All the values are in ppm except O<sub>2</sub> which is in %. The values of *m* and *b* depend on the concentration of CO<sub>2</sub>. If [CO<sub>2</sub>] ≤ 5%, *m* = -18 and *b* = 122,000. If [CO<sub>2</sub>] > 5%, *m* = 23 and *b* = -38,600.

12.3.8 Determine the specimen mass (mass loaded) needed for the FED = 1 condition by dividing the



initial mass of the specimen used in this procedure by the FED value derived in the previous step.

12.3.9 Determine the estimated  $LC_{50}$  by dividing the specimen mass that is estimated to be consumed at the  $FED = 1$  condition by the chamber volume, as indicated in eq (3), below. Record this estimated  $LC_{50}$  to 2 significant figures.

$$LC_{50} = \frac{\text{Specimen mass loss}}{\text{Total FED} \times \text{Chamber volume}} \quad (3)$$

where the  $LC_{50}$  is expressed in  $g/m^3$ , the specimen mass loss is in grams, and the chamber volume is in  $m^3$ .

Note 3. The values of  $LC_{50}$  are derived on the basis of mass consumed. To determine the amount of specimen which will need to be used in Procedure B, however, requires that the initial mass (mass loaded) also be known for the same conditions.

12.3.10 From the FED estimate in 12.3.7 and the specimen exposed area, determine a new specimen exposed area for a sample that would produce an FED of 1.1.

12.3.11 Repeat steps 12.3.2 through 12.3.9 using the new sample size. Verify that the FED for this sample is near 1.1.

#### 12.4 *Running the Test—Procedure B:*

12.4.1 Procedure B consists of two tests, the first done at  $FED = 0.8$ , the second at  $FED = 1.4$ .

12.4.2 Determine the specimen mass for the  $FED = 0.8$  test by selecting mass to be 70% of the mass needed for  $FED = 1.1$ . Adjust the area of the exposed specimen face in the same ratio as for the mass required.

12.4.3 When ready to start the test, weigh the animals to be tested and place them in their restrainers.

12.4.4 Insert the specimen in the appropriate holder into the combustion system and close the chamber with the standard taper plug (use no grease or sealant on the ground glass). Secure the plug with springs. Immediately prior to beginning the test, place the animals into the ports in the animal exposure chamber and close the doors to the box.

12.4.5 Activate the power to the radiant heat lamps and start the data collection.

12.4.6 Turn on the sparker. Record the time when ignition of the specimen occurs and turn off the sparker. Record the time of flameout. For samples which have the tendency to self-extinguish immediately (e.g., containing fire retardants) the sparker is to be left on until flaming ceases (see Note 2).

12.4.7 Switch off the power to the radiant heat lamps and close the smoke shutter when 15 minutes have elapsed after the start of the test.

12.4.8 Collect data for 30 minutes after the start of test. Note the behavior of the animals and the time and number of animals that died during the exposure.

12.4.9 At the end of 30 minutes, stop collecting data. Remove the animals from the exposure chamber. Vent the exposure chamber with a high-capacity exhaust system.

12.4.10 Check the animals for any signs of toxic effects (e.g., difficulty in breathing, convulsions), posture, exploratory behavior, eye opacity, discharge from nose and mouth, and eye and righting reflex. The status and weights of the animals are to be followed for at least a 14-day post-exposure period. If test animals are still losing weight at the end of the 14-day period, they should be kept until they die or recover, as indicated by 3 successive days of weight gain.

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

12.4.12 Determine an estimated  $LC_{50}$  value from the data of this test run by using eq (3), above.

12.4.13 Clean the combustion chamber. Clean the exposure chamber before each different test material (or as often as it seems necessary after visual inspection). Ethyl alcohol is a suitable solvent.

12.4.14 Repeat the above testing steps for a specimen size corresponding to  $FED = 1.4$ .

12.4.15 If no animals die during the 30-minute exposure, or within 14 days post-exposure to the mass loss concentration corresponding to  $FED = 0.8$  and all six rats die during the 30-minute exposure, or within 14 days post-exposure, to the mass loss concentration corresponding to  $FED = 1.4$ , the testing is then complete. Determine a final approximate  $LC_{50}$  using the animal data and N-Gas equation results from all 4 tests. Otherwise, Procedure C is used, wherein a complete experimental and statistical determination of the  $LC_{50}$  value is performed.

12.5 *Running the Test—Procedure C:*

12.5.1 Procedure C consists of a complete experimental and statistical determination of the  $LC_{50}$  value of the sample.

12.5.2 Depending on whether the estimated  $LC_{50}$  was shown by the tests in Procedure B to be too low or too high, select a smaller or larger specimen area to be tested.

12.5.3 Perform the testing steps as described in 12.4.3 to 12.4.12 for the selected specimen size.

12.5.4 Repeat the testing steps for additional specimen areas, until 3 tests are obtained where animal deaths are neither 0 nor 6.

12.5.5 Make a statistical determination of the  $LC_{50}$  value using the method of Litchfield and Wilcoxon [2].

## 13. Report

13.1 *Report the Following General Information.*

13.1.1 Responsible laboratory and person.

13.1.2 Test dates and identification.

13.1.3 Specimen description, manufacturer or submitter, generic components (if available), and any identification known from the manufacturer.

13.1.4 Irradiation time and heat flux conditions.

13.1.5 Strain of rat and identity of the commercial supplier.

13.2 *Report the Following Information From Procedures A and B.*

13.2.1 Mass of the specimen (g) before the test and the amount consumed ( $g/m^3$ ) during the test.<sup>3</sup>

13.2.2 Time to ignition and flameout (s).<sup>3</sup>

13.2.3 Other observations, such as melting, char formation, spalling, unusually vigorous burning, or re-ignition.<sup>3</sup>

13.2.4 Time-integrated concentration of gases (ppm or %) measured for the 30-minute exposure period.<sup>3</sup>

13.2.5 Lowest concentration of oxygen (%) observed during the exposure.<sup>3</sup>

13.2.6 Average and maximum animal chamber temperatures for the 30-minute exposure period.<sup>3</sup>

13.2.7 Weight of each animal (g) when received, prior to test, and during post-exposure observation period.<sup>3</sup>

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<sup>3</sup> Report this information for each test conducted.



13.2.8 Time of animal deaths during the exposure and post-exposure period. Report day of deaths with day 0 being the day of exposure.<sup>3</sup>

13.2.9 Observations made about the condition of animals immediately after the exposure and unusual observations during the post-exposure period (Procedure B and C only).<sup>3</sup>

13.2.10 The gas concentrations and the computed FED value, as determined in 12.4.7.

13.2.11 The estimated LC<sub>50</sub> values, using eq (3), for the four test specimens in Procedures A and B. Determine the final estimated value for LC<sub>50</sub>(raw) as follows:

For the four tests, plot the sample mass lost vs. the FED values.

Perform a linear least squares analysis of the plot and use the resulting line to determine a best approximate LC<sub>50</sub> using eq (3).

13.2.12 Compute the value of LC<sub>50</sub> corrected for the expected post-flashover yield of CO according to:

$$LC_{50}(corr) = \frac{1}{\frac{1}{LC_{50}(raw)} + 44 \times 10^{-3} - 5.0 \times 10^{-5} \frac{[CO]}{m}} \quad (4)$$

where LC<sub>50</sub>(corr) is the corrected value of LC<sub>50</sub> (g·m<sup>-3</sup>); LC<sub>50</sub>(raw) is the raw value of LC<sub>50</sub> (g·m<sup>-3</sup>), obtained above; m is the mass of specimen lost during test at the FED = 1.1 condition (g); and [CO] is the concentration of CO at the FED = 1.1 condition (ppm).

13.2.13 Whether or not the estimated LC<sub>50</sub> was verified by the tests of Procedure B.

13.3 *Report the Following Information From Procedure C.*

13.3.1 The same test information as specified under 13.3.1 through 13.3.9, above.

13.3.2 The concentration-response curve (on the appropriate graph paper: probit or log-probability) and the statistically computed LC<sub>50</sub>. (This includes a concentration-response curve from which a statistically determined LC<sub>50</sub> value and 95% confidence limits on the LC<sub>50</sub> are calculated according the method of Litchfield and Wilcoxon [2].

## 14. Precision and bias

14.1 *Repeatability (Within-Laboratory Precision)*—The repeatability for this method has not yet been determined.

14.2 *Reproducibility (Between-Laboratory Precision)*—The reproducibility for this method has not yet been determined.

14.3 *Bias*—This test method uses rats as the test animals. The degree of bias reflected in the results from this test, compared to toxic effects on humans, has not been quantified. The numerical values cited in eq (2) have been determined under laboratory conditions for one type of test animal (Fischer 344 male rats) only. It is possible that use of other strains of rats or animals from other suppliers could give different LC<sub>50</sub> values. The numbers used are to be those applicable to the rat strain and laboratory in which the research is being conducted.



## APPENDIX (Nonmandatory information)

### X1. COMMENTARY

#### X1.1 Introduction

X1.1.1 The purpose of this commentary is to provide information on the development of a radiant heat smoke toxicity test method and to describe certain uses for the data.

#### X1.2 Development of the method

X1.2.1 Many smoke toxicity test procedures have been developed and tested since the publication of "America Burning" by The National Commission on Fire Prevention and Control [3] in 1973 noted that most fire victims die from inhaling smoke and toxic gases. At least 20 such methods were described in 1983 [4]. At about the same time, 13 published methods were evaluated by Arthur D. Little, Inc., to assess the feasibility of incorporating combustion toxicity requirements into building material and furnishing codes of New York State [5]. On the basis of seven different criteria, only two methods—the closed-system cup furnace smoke toxicity method [6] developed at the National Institute of Standards and Technology (NIST) [previously known as the National Bureau of Standards (NBS)] and the flow-through smoke toxicity method developed at the University of Pittsburgh [7]—were found acceptable. The state of New York decided to use the method developed at the University of Pittsburgh. Since it was unclear how to use the results of this toxicity method in regulation, the state of New York only requires that materials be examined with the University of Pittsburgh's protocol and that the results be filed with the state. There are no criteria to judge whether a material needs to be regulated or not. Smoke toxicity test methods continue to be developed and evaluated; the three methods most recently developed are the radiant furnace smoke toxicity protocol (described in the present standard), the National Institute of Building Sciences (NIBS) toxic hazard test method [8],[9] [which was developed at Southwest Research Institute (SwRI)], and the University of Pittsburgh II radiant furnace method [10]. Although these methods differ in numerous characteristics, all three use radiant heat to thermally decompose materials.

X1.2.2 A test method to assess the acute inhalation toxicity of products of combustion requires three main components: a combustion system, a chemical analysis system, and an animal exposure system. The chemical analysis system and the animal exposure system of the radiant heat smoke toxicity method described herein are the same as that used in the cup furnace smoke toxicity method. This animal exposure system and chemical analysis system were also adapted from those used in an earlier radiant heat toxicity method [11]. The combustion furnace used in the present method was developed at SwRI by A.F. Grand and is an improvement of a furnace designed by H.W. Stacey in the laboratories of the Weyerhaeuser Company and which was used in the earlier radiant heat toxicity method [4], [11].

X1.2.3 Since the animal exposure system and the chemical analysis system achieved widespread professional acceptance, the development issue of most concern was the combustion system. No one test method can simulate all possible fire scenarios. The cup furnace decomposed materials under two severe conditions—25°C above (flaming conditions) and 25°C below (nonflaming conditions) the material's autoignition temperature and provided thermal immersion heating. The cup furnace does not readily represent the fire conditions occurring in a room fire. Instead, it was realized that:

1. the combustion system should thermally decompose materials under more realistic conditions, namely radiant heat;
2. the furnace should allow for the decomposition of materials, products, composites, and assemblies under likely end-use conditions;
3. the system should allow for the testing of larger sample sizes than previously acceptable in the cup furnace (e.g., the cup furnace test procedure recommended sample sizes no larger than 8 grams although larger sizes were tested) and in some tube furnaces; and
4. the fire scenario should simulate the conditions under which the greatest number of human lives are lost, namely post-flashover fires.

X1.2.4 Thus, various investigators have sought to find a better combustion system. In 1984, Alexeeff and Packham proposed using the radiant heater system developed at Weyerhaeuser Company [11]. This method did not achieve wide use because of problems with the test hardware. It did, however, offer the possibility of testing composite materials realistically exposed to radiant heating fluxes. In 1985, Levin and co-workers [12] explored the use of the well-established, robust combustion system of the Cone Calorimeter [13]. The intrinsically flow-through Cone Calorimeter combustion system was coupled to the 200 L closed-animal exposure system in this investigation. The results proved feasible, but not practicable: coupling closed-box and flow-through systems resulted in a very difficult test procedure. The University of Pittsburgh II radiant test procedure [10] couples the Cone Calorimeter combustor with the flow-through animal exposure system previously used with the University of Pittsburgh I smoke toxicity method [7].

X1.2.5 In 1986, the National Institute of Building Sciences (NIBS) formed a working group to develop a performance test method for evaluating toxic hazard of materials and products. After the determination of the criteria, they asked SwRI to design, build, and test the new system. SwRI used the animal exposure system and chemical analytical system from the cup furnace smoke toxicity method and modified the radiant heating system described in reference [11]. As this work was in progress, the National Institute of Standards and Technology (NIST) noted the hardware being evolved by SwRI and initiated a joint activity between SwRI and NIST. The task here was to develop an improved test for measuring toxic potency of materials and products; unlike the NIBS effort, the test procedure was not intended to yield an index of toxic hazard. The radiant furnace smoke toxicity test method developed at NIST with the help of SwRI uses the same design for the combustion system and animal exposure system as the NIBS and cup furnace methods, respectively. However, in a number of areas, the actual hardware of the combustion system was re-designed to provide for ruggedness and ease of operation.

X1.2.6 The preliminary results of the SwRI/NIST collaboration were published in a NIST publication [14]; this publication also contained documentation examining the validity of the method compared to real-scale fires. The test apparatus as implemented for the NIBS test has also been documented by SwRI [15].

### **X1.3 Presentation of the data in terms of the N-Gas Model**

X1.3.1 The N-Gas Model developed by Levin and co-workers [16], [17], [18], [19],



[20] is based on the hypothesis that a small number ("N") of gases in the smoke accounts for a large percentage of the observed toxic potency. The lethality of each of these gases was determined for laboratory animals, e.g., rats. Similar measurements for combinations of these gases tell us whether the effects of these gases are additive, synergistic, or antagonistic. The results of these mixed gas tests to date have been reduced to an algebraic equation which has been empirically determined for the exposure of rats to mixtures of CO, CO<sub>2</sub>, HCN, and reduced O<sub>2</sub> [18], [19]. Data for the addition of HCl into the equation came from the work of Hartzell et al. [21] and was tested at NIST with the material thermal decomposition of vinyl materials in both small and full-scale tests [22],[23],[24]. The equation used for evaluation (eq (1), above) can be viewed as performing a summation of various gaseous toxicants, adding up to a Fractional effective Exposure Dose (FED). If FED = 1.1, then it is expected that the gas mixture will be lethal to 50% of the exposed animals. Values of FED > 1.1 represent mixtures of greater yet toxicity, while FED < 1.1 denotes a lesser toxicity. The validation of the N-Gas Model indicates that, for the preponderance of conditions examined, the toxicity of gaseous combustion products can be estimated by measuring the gases named above (plus, HBr in systems where Br is present in the specimen [22]).

X1.3.2 If the N-Gas Model were always sufficient to describe the actual product toxicity, then only Procedure A would be needed for testing. It does happen, however, that products can be found where the preponderance of the toxic effects generated comes from more unusual gaseous components. To guard against erroneous estimates in such cases, Procedures B and C are required.

X1.3.3 Traditional biological testing for combustion product toxicity was formulated on the basis of solely conducting procedures analogous to Procedure C. Procedure C requires that a complete concentration-response curve be generated by animal testing. The present method reduces testing costs, animal usage and time by developing Procedures A and B; by incorporating these procedures, the typical usage of animals per test product can be significantly reduced.

## **X1.4 Representation of post-flashover fires**

X1.4.1 Recent studies at NIST [14] have demonstrated that a bench-scale toxic potency test can adequately represent most aspects of a post-flashover fire. The one crucial exception is the generation of carbon monoxide. This is governed primarily by the available air supply in the actual full-scale fire, and cannot be simulated in a practical bench-scale test method. It was also found, however, that actual post-flashover fires exhibit a yield of CO of approximately 0.2. The yield of CO is defined as the mass of CO evolved, per mass of specimen lost. Based on this finding, it is possible to develop an equation whereby the raw LC<sub>50</sub> value is corrected to correspond to the condition which would prevail in a post-flashover fire. This equation is given above as eq (4). In any cases where a pre-flashover fire representation is desired, the raw, rather than the corrected, LC<sub>50</sub> values, would be used.



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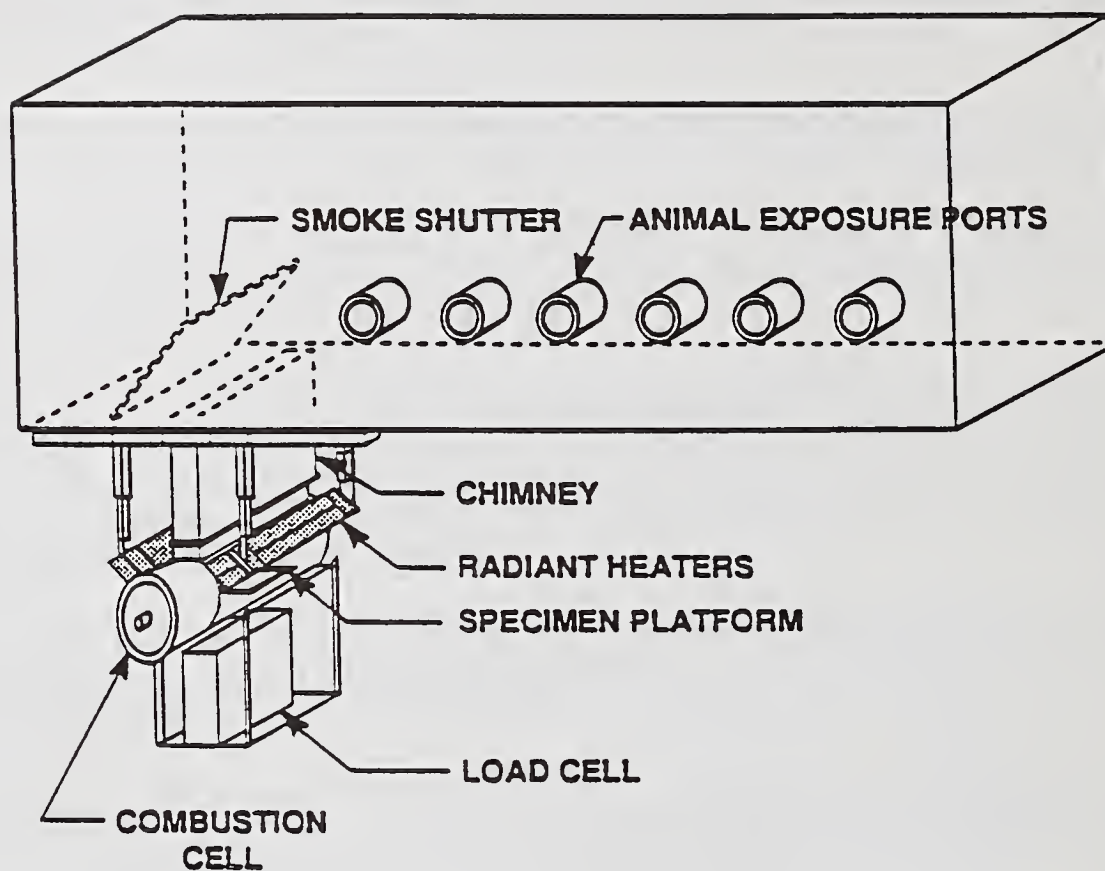


Figure A1. General view of radiant heat apparatus.

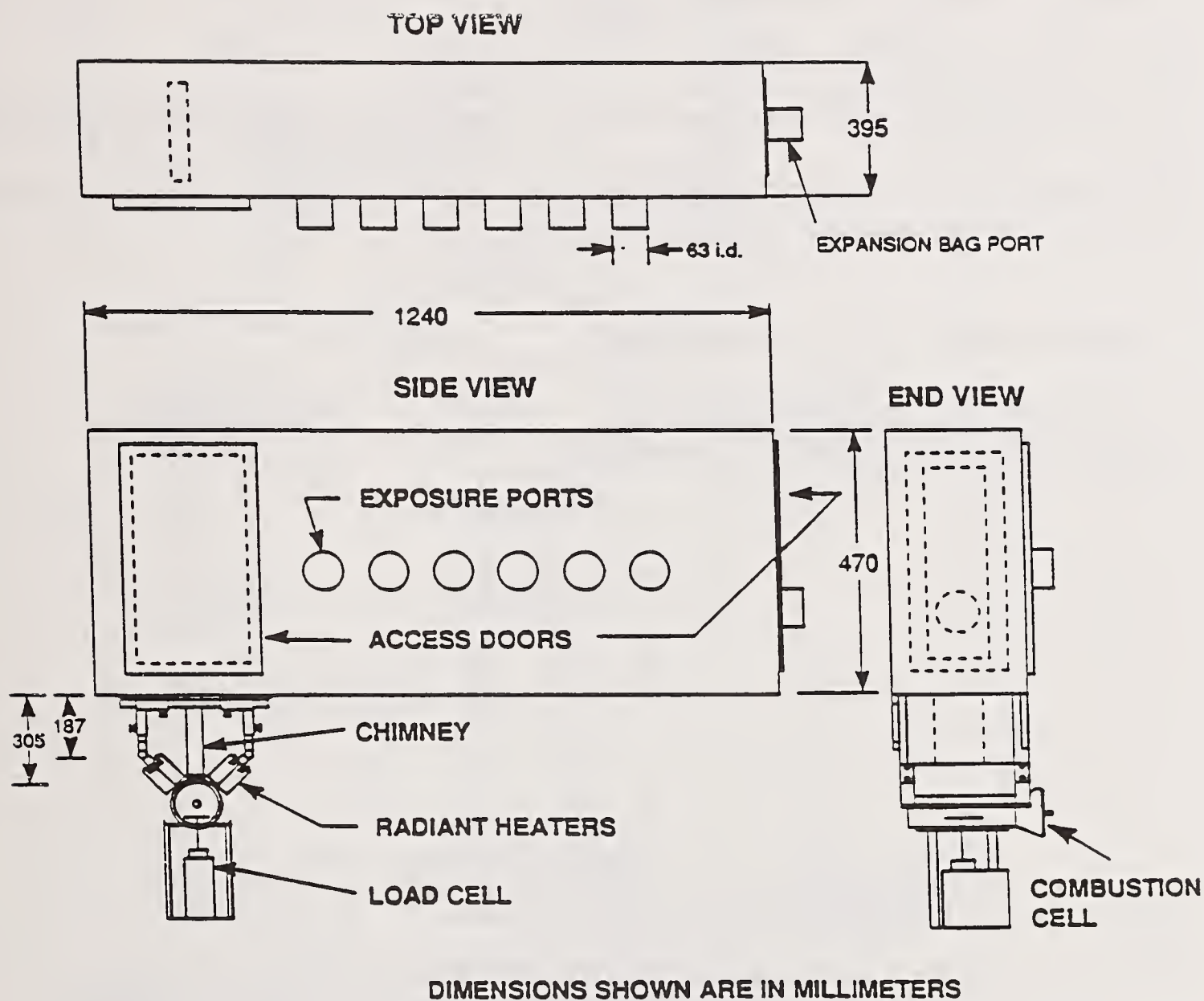
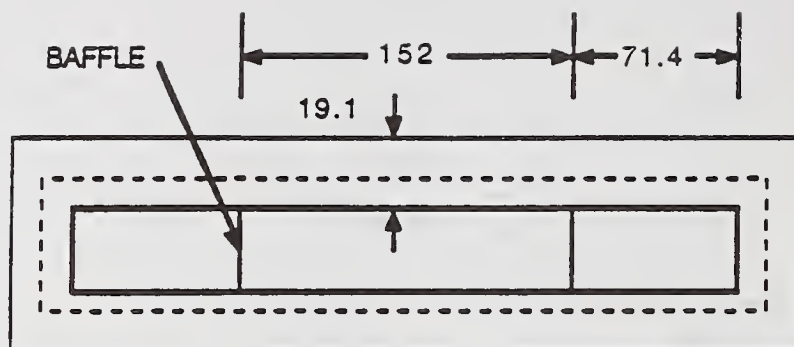


Figure A2. Main dimensions of the apparatus.



**NOTES:**

DIMENSIONS IN MILLIMETERS, INSIDE MEASURE  
WHERE APPLICABLE

MATERIAL: 1.6 MM (1/16") STAINLESS STEEL

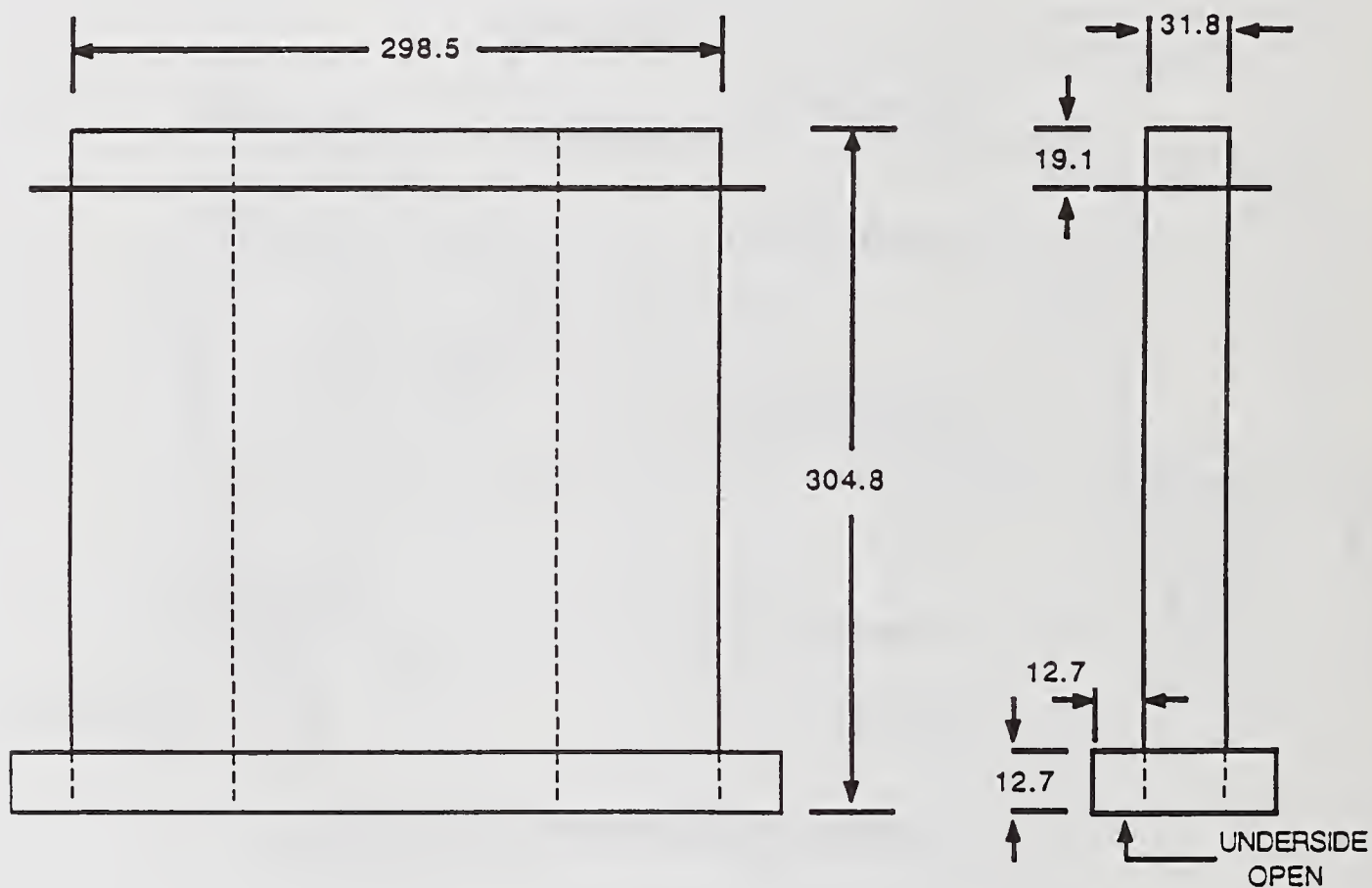


Figure A3. Chimney.



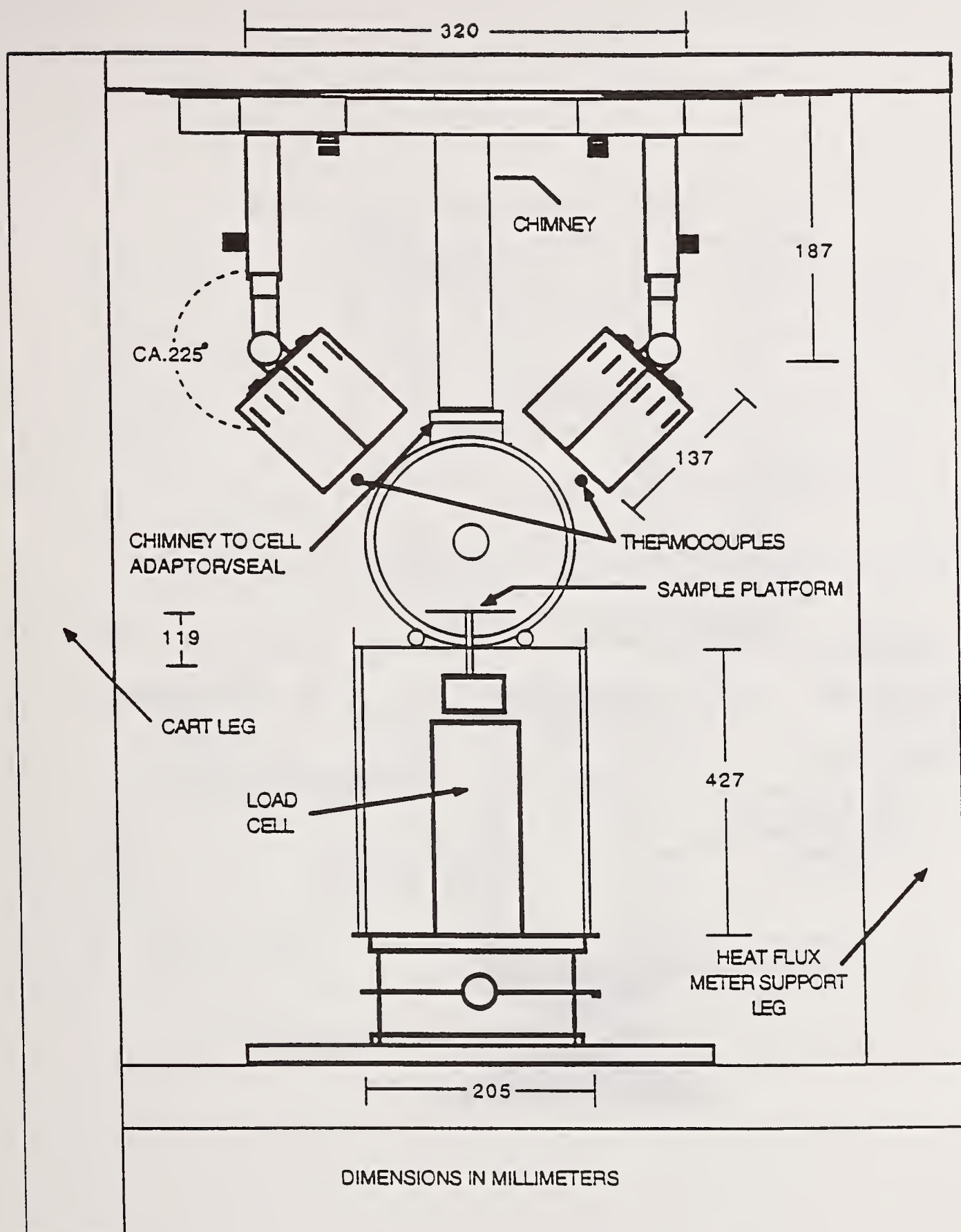


Figure A4. End view showing dimensions pertinent to combustion cell and heaters.

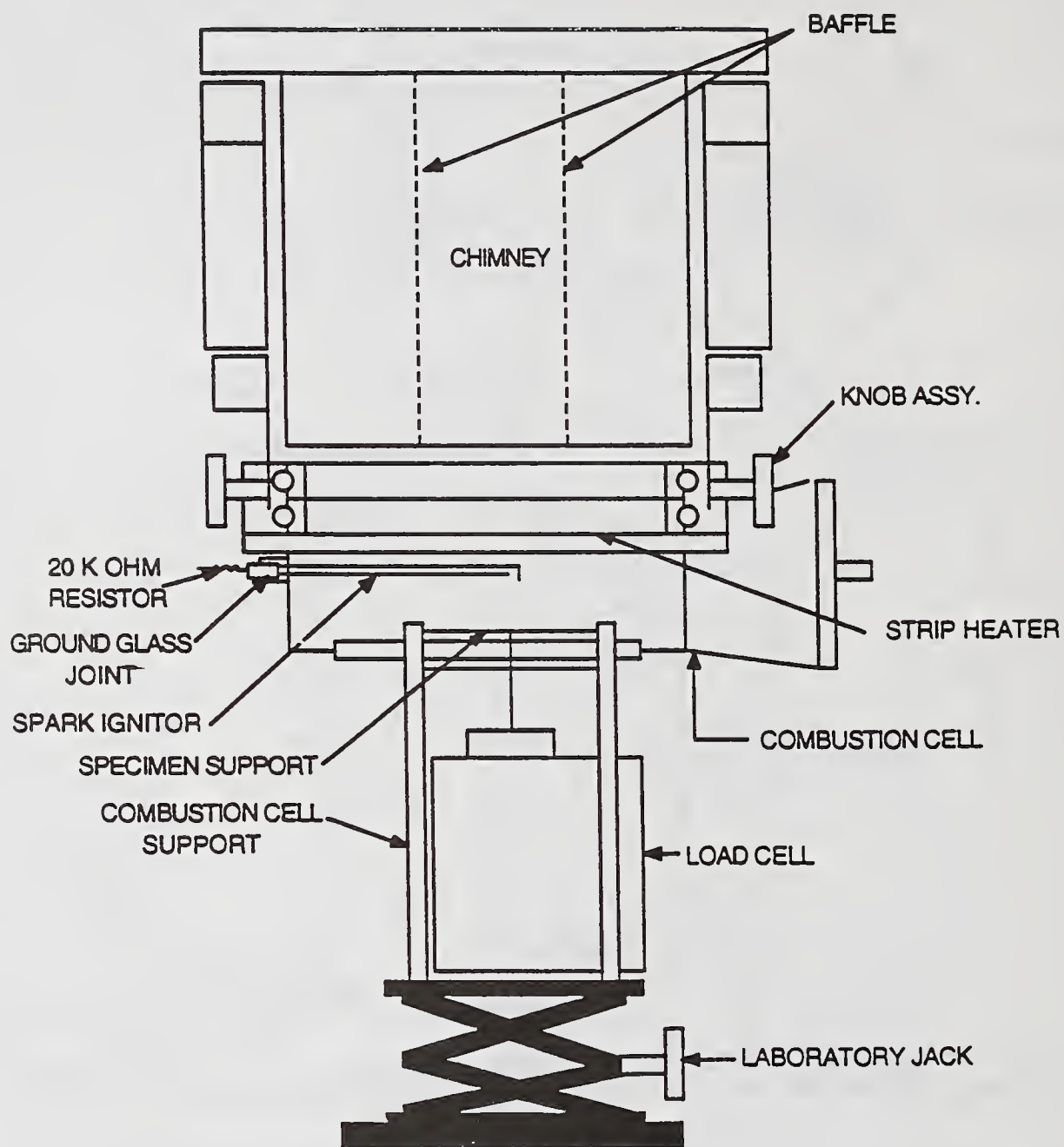


Figure A5. Side view of combustion cell.

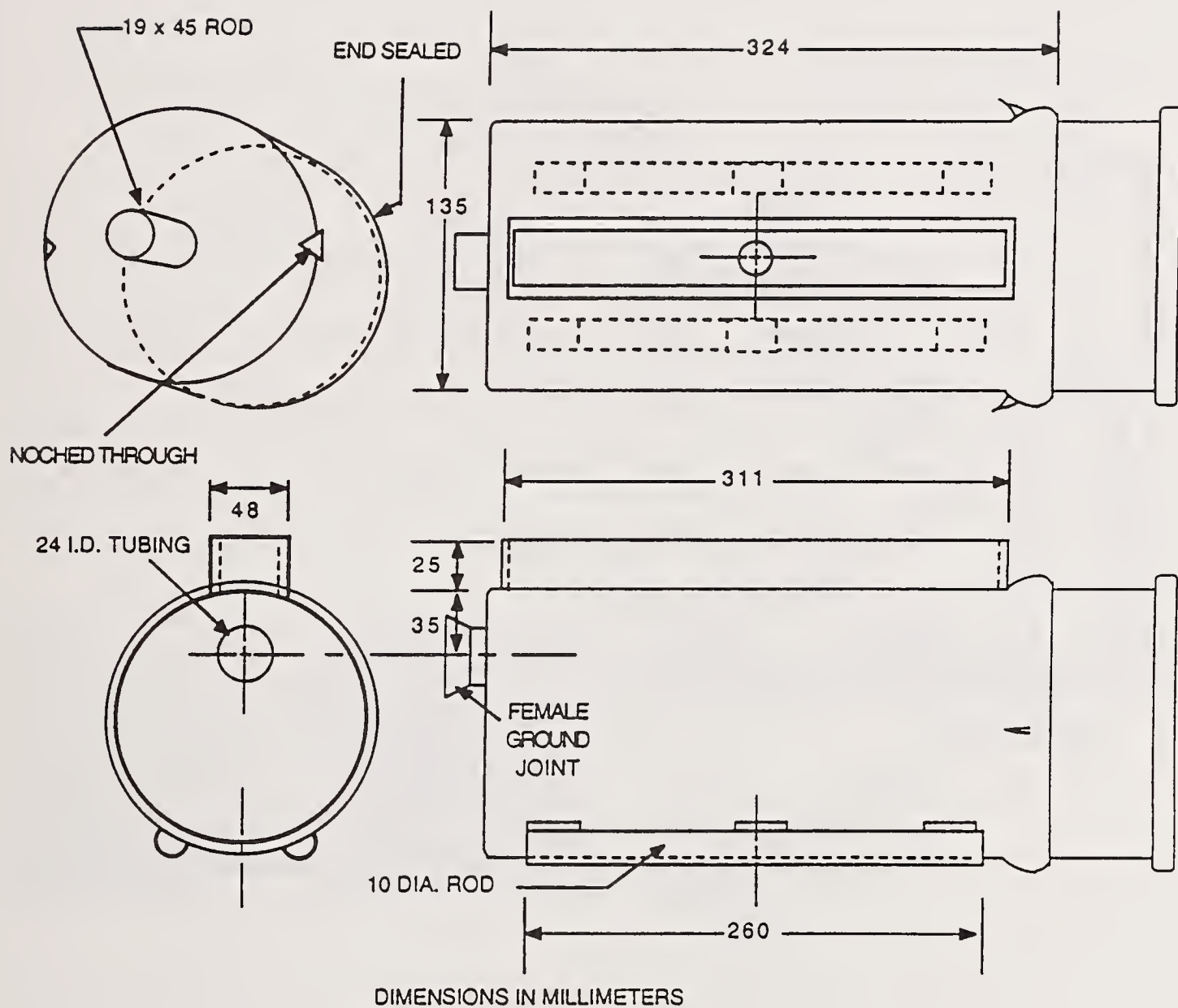


Figure A6. Construction details for the combustion cell.



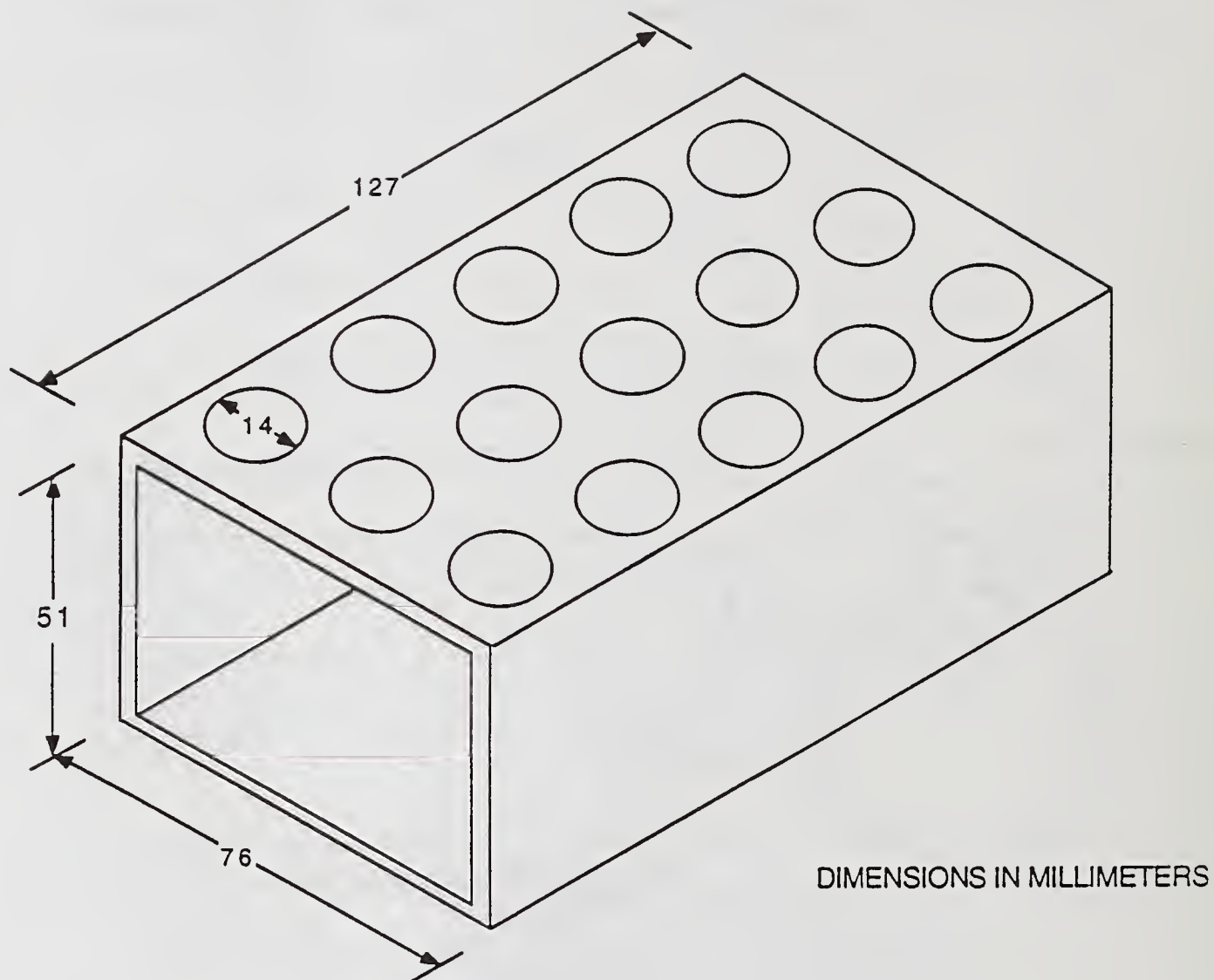


Figure A7. Calibration jig used for checking the uniformity of irradiance.

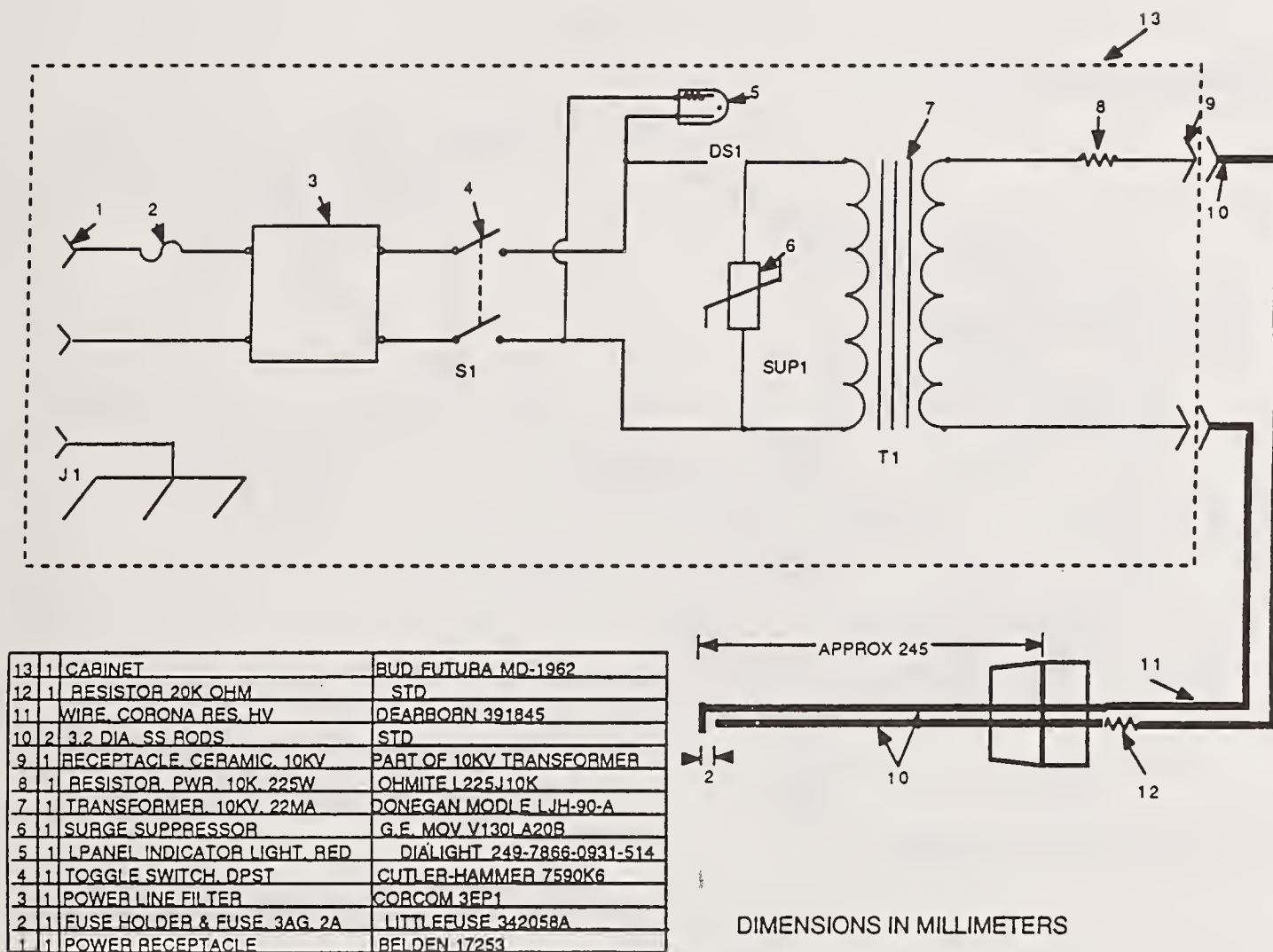


Figure A8. Power supply for spark ignition.

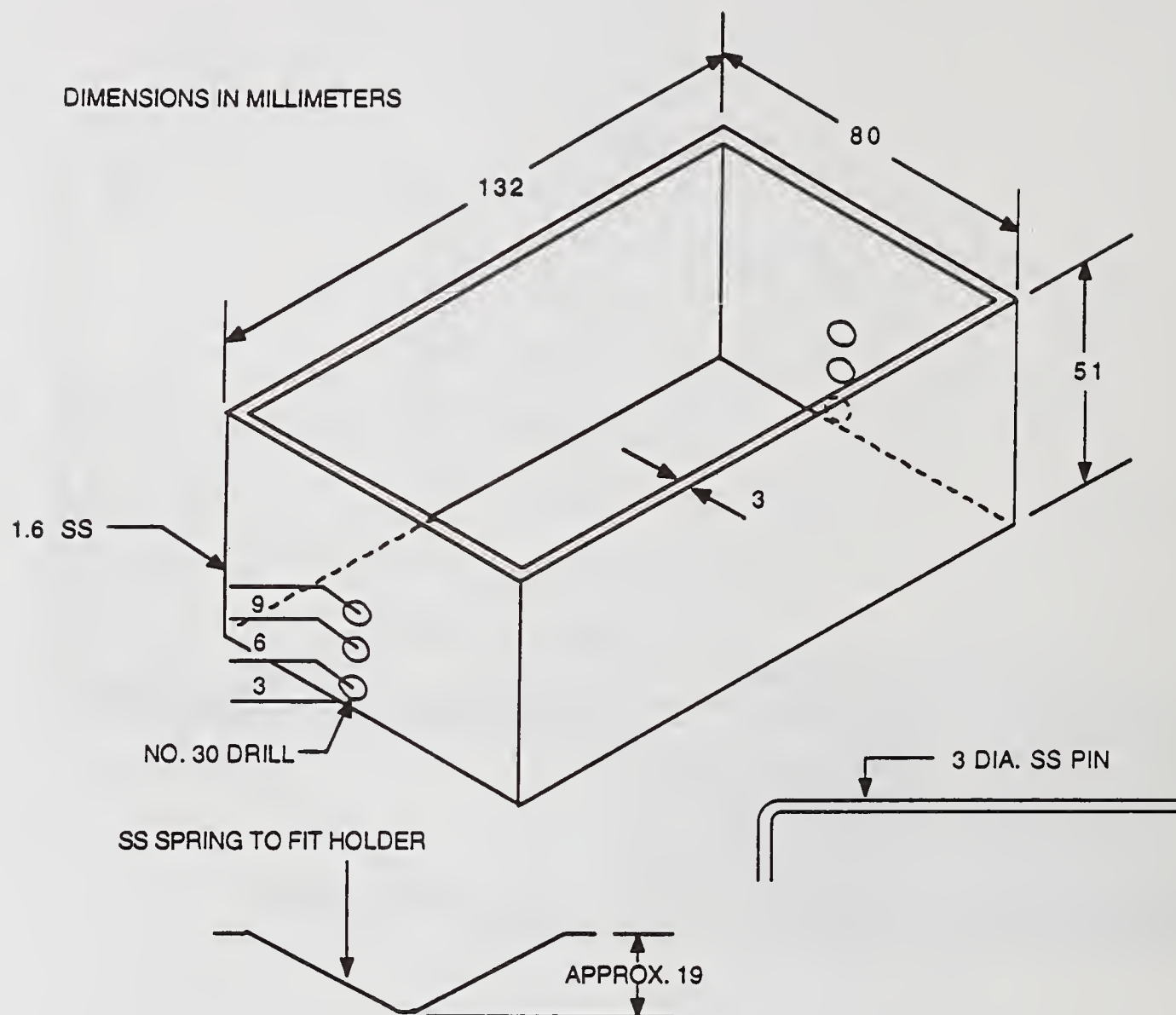


Figure A9. Specimen holder.



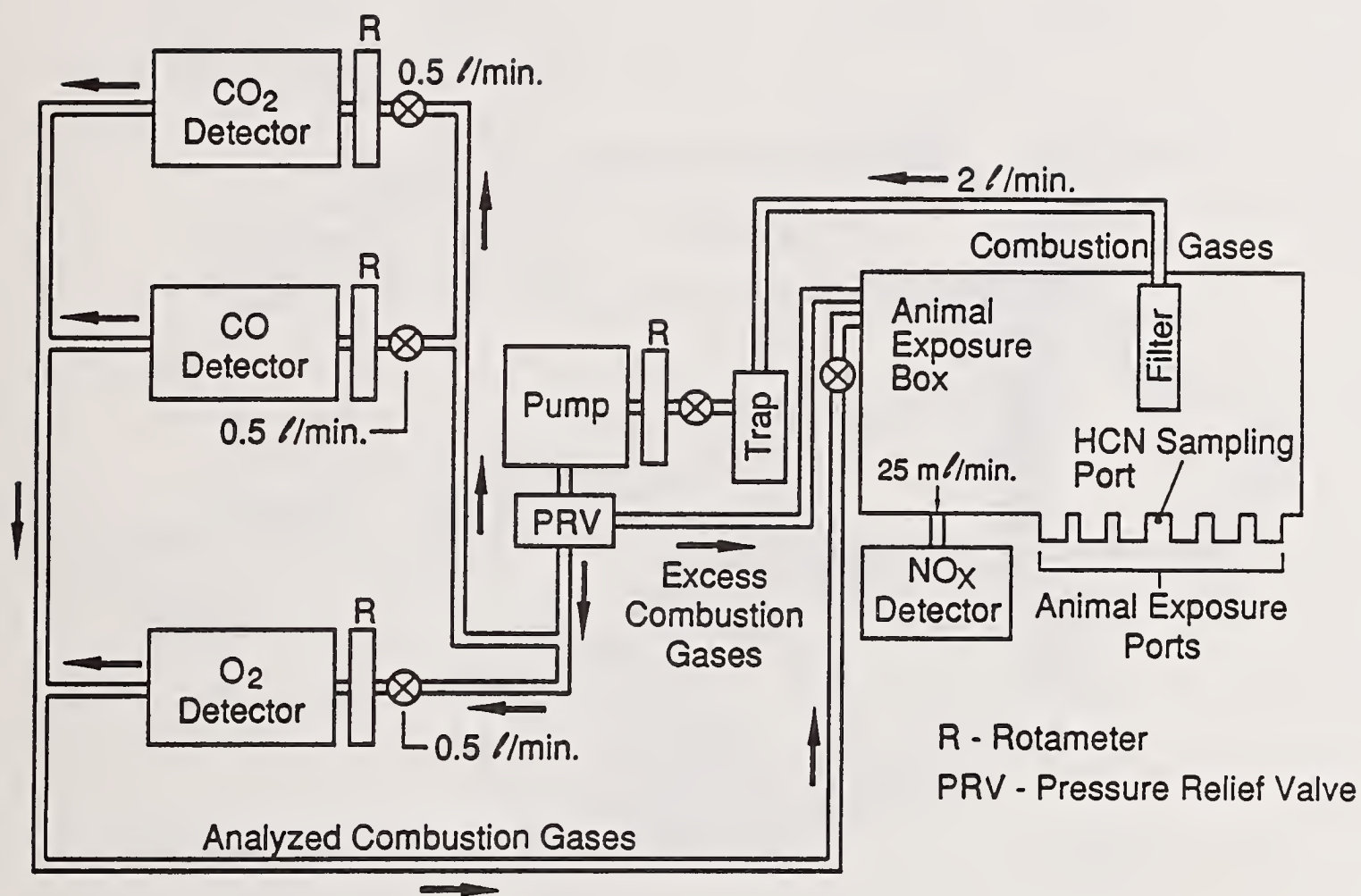


Figure A10. Gas sampling system.

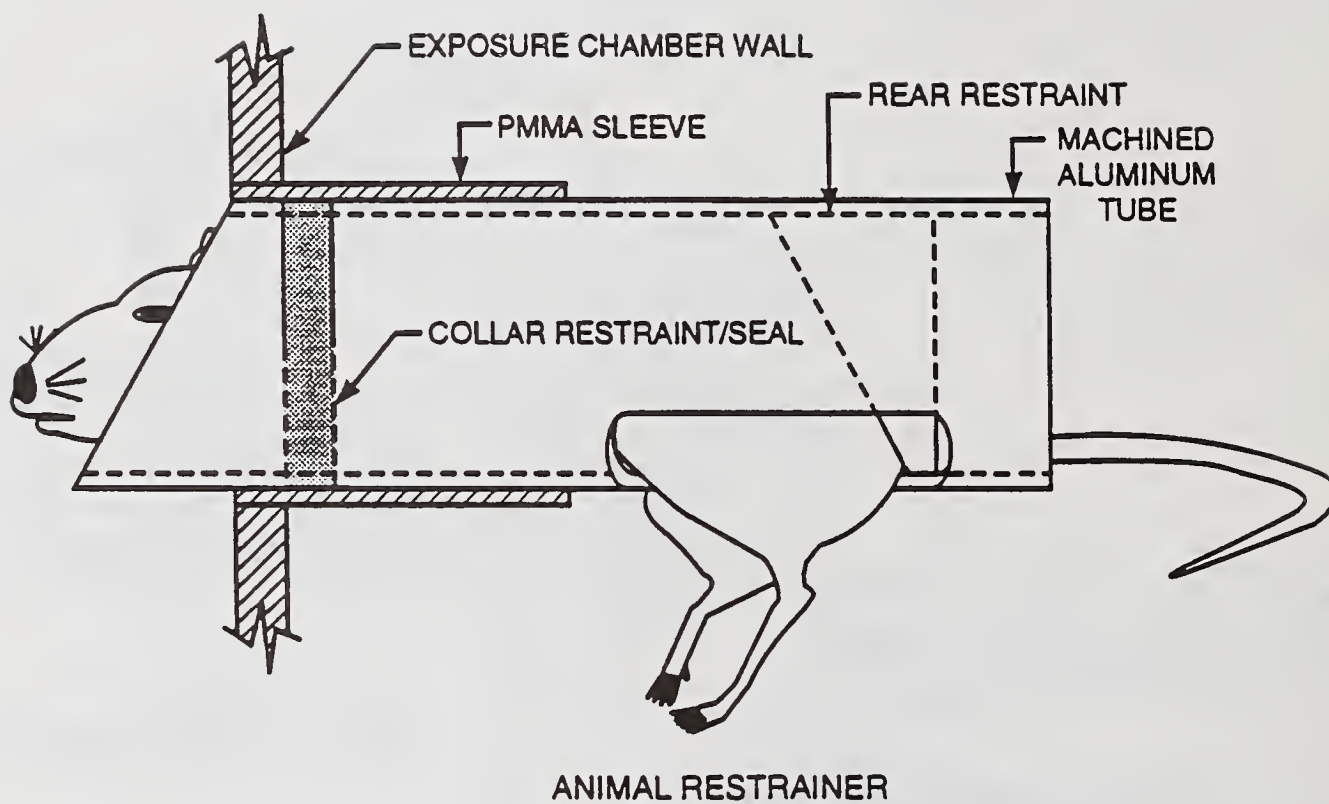
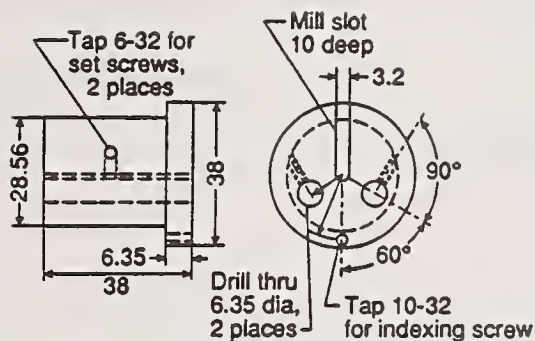
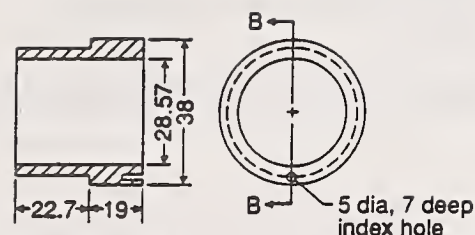


Figure A11. Animal restrainer.

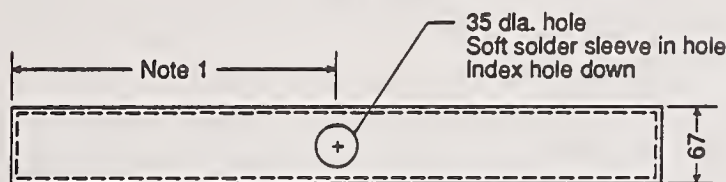
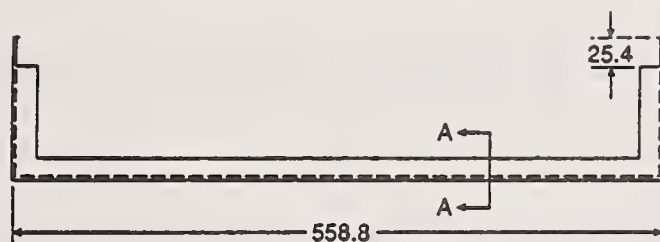


**FLUX METER HOLDER**



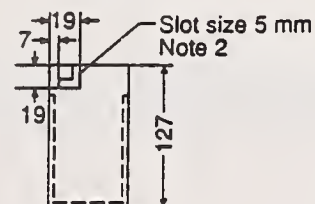
**SECTION B-B**

**SLEEVE**



**FLUX METER SUPPORT BRACKET**

(Made of 0.15 mm steel sheet)



**SECTION A-A**



- Note 1: Adjust length to get flux meter to center in combustion cell  
 Note 2: Position bracket on cart support legs and center hole in combustion cell  
 Mark and drill and tap 10-32 to support bracket

All dimensions in meters  
 Not to scale

Figure A12. Holder used for positioning flux meter for pre-test calibration.



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11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

A comprehensive methodology has been developed for obtaining and using smoke toxicity data for fire hazard analysis. This description of the methodology comprises: determination that the post-flashover fire is the proper focus of smoke inhalation deaths; criteria for a useful bench-scale toxic potency (LC<sub>50</sub>) measurement method; a method which meets these criteria, especially validation against real-scale fires; a computational procedure for correcting the results for the CO levels observed in real-scale post-flashover fires; procedures for reducing the usage of animals and broadening the applicability of data by interpreting gas measurement data using the N-Gas Model; and a procedure for identifying whether a product produces smoke within the ordinary range of toxic potency for post-flashover fires.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

Keywords: building fires; combustion products; computer fire models; fire deaths; fire hazard analysis; N-gas model; radiant heating; smoke toxicity; toxicity test methods.

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