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A methodology for obtaining and using toxic potency data for fire hazard analysis

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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 (1) determination that the post-flashover fire is the proper focus of smoke inhalation deaths; criteria for a useful bench-scale toxic potency (LC_{50}) measurement method; (2) a method which meets these criteria, especially validation against real-scale fires; (3) a computational procedure for correcting the results from the bench-scale test for the CO levels observed in real-scale post-flashover fires; (4) procedures for reducing the usage of animals and broadening the applicability of data by interpreting gas measurement data using the N-Gas Model; and (5) a procedure for identifying whether a product produces smoke within the ordinary range of toxic potency for post-flashover fires. Published by Elsevier Science Ltd.

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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

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falling debris, leaping from windows, etc.), or other causes [1]. This was not generally recognized until well into this century. In an early study, Ferguson [2] noted in 1933 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". Several other such studies were completed in the ensuing decades.

During the 1970s, there was a distinct increase in fire research activities, including for the first time a number of proposals for various tests to measure fire toxicity. Initially, various manifestations of inhalation toxicity were examined, such as incapacitation preventing an animal from performing a simple motion. The spectrum of ill effects from fire smoke is large, however, ranging from discomfort or impairment of judgement at one end to lethality at the other. The primary focus that emerged was on lethality, an unambiguous endpoint which could be examined without undue subjectivity. The general metric for measuring *lethal toxic potency* was 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.

This activity was especially pronounced in the United States. Yet, while a number of tests for combustion toxicity were developed, publicized, and proposed for usage, none became adopted by any US standards organizations. Even though such a consensus was not reached, concern in legislative bodies has led to both New York State and New York City, separately, establishing fire toxicity requirements for building products. 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 the existing toxicity tests failed to capture the complex properties of fires and *commercial products* that were needed to 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 (sic) of the tested product.

At the National Institute of Standards and Technology (NIST), meanwhile, research on this topic had been progressing since 1974. A test apparatus was developed in 1982 commonly referred to as the 'cup furnace smoke toxicity method', [3, 4]. This was followed by a program to assay 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 [5–7]. A context for utilizing the toxicity data was created with the development of a computer-assisted methodology for calculating fire behavior and human response to fire, named HAZARD I [8].

Like other proposed toxic potency tests, the original cup furnace method did not win standards organization approval. In part this was because the combustion conditions created in the test method were not seen as sufficiently representative of conditions occurring in real fires. It was also felt there was a lack of evidence relating

these data to the results of real-scale fires. In other words, it was deemed necessary to demonstrate that the smoke produced under the bench-scale conditions was acceptably similar to that produced in real-scale fires. A third critical reason why no bench-scale test methods were advanced to standards status was because of a significant discomfort within the profession on how their output data were to be used.

The present study is the culmination of an effort to:

(a) provide an improved bench-scale apparatus for toxic potency measurement 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. Fires to be studied

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 fire death statistics. Based on these statistics, we can identify the real fires in which smoke toxicity is most critical.

The British Standards Institution (BSI) has developed a scheme [9] whereby they itemized six different types of fires. For the NIST study it was most important to examine the US fire death statistics. These revealed [10] that about 70% of all fire deaths are associated with post-flashover conditions, with the vast majority of deaths occurring outside the room of fire origin. Thus, the major focus of the fire toxicity studies has to be on post-flashover fires. As will be shown below, however, the physical measurement procedures developed can equally well be used for flaming, pre-flashover fires. Other fire types (e.g., glowing fires or those due to flameless overheating) are rare enough in the fire death statistics not to warrant use of a general test procedure.

3. Criteria for bench-scale toxic potency measurement

The two most important criteria for a successful test method are:

(1) LC_{50} values and toxic gas yields must be obtained under realistic combustion conditions, representative of real-scale fires, and
(2) the data must be usable as an integral part of a fire hazard assessment method.

Thus, means have to be in place for supplying *all* of the needed information, not just the LC_{50} value alone. The list of other criteria a suitable test should meet was extensive:

- Toxic potency should be measured, reportable in correct concentration (e.g., $g\,m^{-3}$) units.
- The yields of the principal toxic gases should be measured and reported, e.g., as grams of toxicant per gram of product burned. This is used both in fire hazard

calculations and for establishing the correlation of the bench-scale result to the full scale by chemical analysis.

- The chemical data necessary for the N-Gas Model should be properly obtainable from the measurement method. The N-Gas Model is discussed in the next section. There, we demonstrate how its use produces a measurement method which is simple to conduct and which minimizes the usage of experimental animals.
- Adequate repeatability.
- Adequate reproducibility.
- Adequate validity.
- Safety to operator.
- Safety to environment, i.e., no excessive pollution.
- Affordable apparatus costs.
- Tests conductible reasonably quickly and efficiently.
- Sample preparation not excessively difficult.
- Ease of cleaning and maintaining of the apparatus.
- The measurement method should represent the chosen full-scale combustion scenario correctly.
- Composite specimens should be testable as composites.
- Since in the post-flashover fire, radiant heating predominates, the specimen should receive uniform, well-controlled radiant heating.
- 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.
- For establishing the correlation of the bench-scale result to the full scale by bioassay, both the LC_{50}s (or an approximation thereto) and the causes of animal deaths need to be measured and recorded.
- Minimum loss of gases and particulates.
- Specimens testing without crushing, powdering, etc.
- 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.
- Protective outer layers should be realistically represented in the measurement method procedure.
- Edge effects should not influence the results disproportionately.
- Samples should be tested in the horizontal, face-up orientation.
- The combustion environment to which the specimen is subjected in the measurement method should correspond as closely as possible to that in the design scenario.
- 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.
- The measurement method should provide for a well-characterized, toxicologically sound exposure of animals. There is a broad consensus for using rats at the test animals and for providing a 30 min exposure period, followed by a 14 d post-exposure observation period.

- The gases to which the animals are exposed should consist of the total combustion products from the specimen's burning history.
- As close to a square-wave exposure as possible is desired.
- The biological effects on the animals' condition during the measurement method should be adversely affected as little as possible by causes other than smoke toxicity.
- The usage of animals should be minimized, consistent with obtaining data of acceptable quality.

The objective of the development work was then to ensure that all of these conditions were met as well as possible.

4. 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 developed a concept which has come to be known as the N-Gas Model. Use of this Model was seen crucial to minimize both testing costs and the usage of animals.

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 [5–7, 11–16]. This is based on a concept of the fractional effective exposure dose (FED) [17, 18]. In the simple case of linear additivity, the FED can be defined as:

$$\mathrm{FED} = \sum_i \frac{\int_0^t C_i \,\mathrm{dt}}{LCt_{50}(i)} \tag{1}$$

where C_i is the concentration of the ith gas species, and $LCt_{50}(i)$ is the lethal concentration × time product for that gas species. In the laboratory experiments, the time of exposure is fixed and uniform, while the concentrations vary only modestly. In such a case, the simplification can be made to

$$\mathrm{FED} = \sum_i \frac{C_i}{LC_{50}(i)} \tag{2}$$

Based on research at NIST of toxicologically important gases and their interactions, the form of the N-Gas Model used is based on the equation

$$\mathrm{FED} = \frac{m\,[\mathrm{CO}]}{[\mathrm{CO_2}] - b} + \frac{[\mathrm{HCN}]}{LC_{50}(\mathrm{HCN})} + \frac{21 - [\mathrm{O_2}]}{21 - LC_{50}(\mathrm{O_2})} + \frac{[\mathrm{HCl}]}{LC_{50}(\mathrm{HCl})} + \frac{[\mathrm{HBr}]}{LC_{50}(\mathrm{HBr})} \tag{3}$$

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 min exposure + 14 d post-exposure period. Other gases can be added as warranted by findings from fire investigations or laboratory tests.

In the current equation, CO and CO_2 interact in a non-linear way. Empirically determined values of m and b are -18 and 122,000 if the CO_2 concentrations are $\leqslant 5\%$, and 23 and $-38{,}600$ when the $CO_2 > 5\%$. With oxygen, the toxicity results from its depletion; thus, the form for O_2 in the above equation involves $(21 - O_2)$. [The 30 min LC_{50} of O_2 is 5.4% which is subtracted from the normal concentration of O_2 in air, i.e. 21%.] The LC_{50} values for the linear terms are HCN: 150 ppm; HCl: 3800 ppm; HBr: 3000 ppm.

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, 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 lethalities from smoke are very steep, the experimental loading is close to the predicted LC_{50} value if *some* percentage (other than 0 or 100%) of animals die.

The N-Gas Model was developed on the basis of experiments with gas mixtures which could be very well controlled and analyzed. It was then validated against the burning of real, solid materials. Extensive data are provided in a NIST report [19]. 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).

5. The radiant toxicity method

The criteria necessary for a successful test method could not be met directly by simply adopting any of the two dozen or so toxicity tests which had been proposed. Yet, many of the necessary features were developed to quite a useful degree in earlier studies.

5.1. The animal exposure system

This first basic feature of the present test method (Fig. 1) is similar to that designed for use in the cup furnace smoke toxicity method [3] developed at NIST. This animal exposure system was, subsequently, adapted for use in an earlier radiant lamp smoke toxicity method developed at Weyerhaeuser Company [20] and in the NIBS method [21–23]. The animals are exposed in an approximately 200 L, clear 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

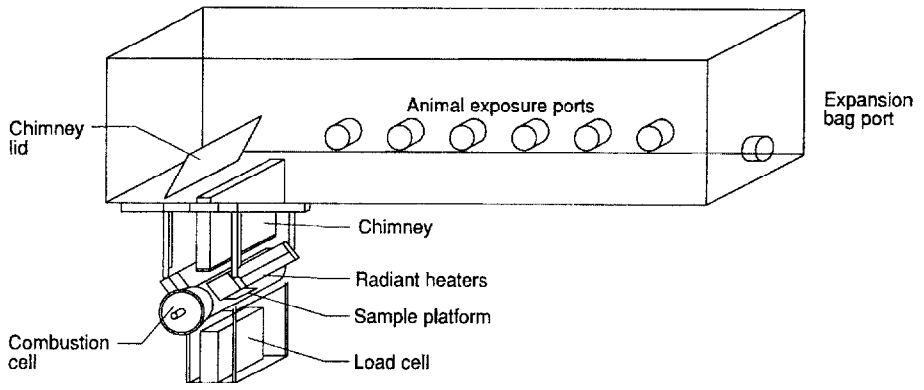

Fig. 1. General view of radiant toxicity test apparatus.

Table 1
Values obtained for the LC_{50} — raw and corrected

Material	Raw LC_{50} ($g\,m^{-3}$)		LC_{50} (corr) ($g\,m^{-3}$)	
	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

when grooming after the exposure. The current exposure chamber is changed in only a 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. 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.

5.2. *The chemical analysis system*

The chemical analysis system is the same as used with the cup furnace smoke toxicity method and is in compliance with the principles outlined in ASTM E 800 [24]. 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.

5.3. The combustion system

Earlier devices, including the cup furnace method, used combustion environments quite different from the radiant heating found in normal building fires. Thus, various investigators have sought a closer experimental replication of the fire. In 1984, Alexceff and Packham proposed using the radiant heater system developed by H. W. Stacy at Weyerhaeuser Company [20, 25]. This method did not achieve wide use. It did, however, offer the possibility of testing composite materials realistically exposed to radiant heating fluxes. In 1986, NIBS funded the Southwest Research Institute (SwRI) to develop an apparatus for the toxic hazard performance test method mentioned earlier. SwRI used the radiant heat geometry envisioned by Weyerhaeuser, but made major hardware improvements. Further hardware design efforts were funded by NIST and conducted both at NIST and at SwRI. The major new design features included:

- 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.
- 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.
- 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 spark ignition system, similar to the one used on the Cone Calorimeter, was added.

These were accompanied by numerous hardware improvements. The details of the apparatus are given in Ref. [19].

6. Test protocol

The basic steps to measure the toxic potency of the smoke from a product are straightforward. A sample consists of a rectilinear cutting of the commercial product. The normally-outward surface is exposed to an irradiance of 50 $kW\,m^{-2}$. 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 LC_{50} and LC_{50}(corr), as well as the yields of the individual principal toxic gases. A corrected value of the LC_{50} is necessary due to CO considerations. Example data collected at NIST are given in Table 1.

7. CO in fires

Carbon monoxide (CO) is the dominant gas in fire smoke toxicity; in many cases, CO plus CO_2 and the depletion of oxygen are the *only* toxic agents of consequence. Thus, its handling must be quantitatively correct for a test method to be successful. The CO problem has been studied at NIST intensively for several years now [26]; analyses of the engineering issues and the phenomenology have been published [27, 28]. The relevant conclusions are:

- The production of CO in post-flashover fires is almost totally dependent on the fuel/air ratio and only modestly dependent on the chemical composition of the fuel being burned.
- Approximately 0.2 g of CO is produced in post-flashover fires for each gram of fuel burned, as shown by the data in Refs. [29–32]. (Note that recent research shows that there are some circumstances in which much larger CO yields are produced [28, 33]. These high concentrations appear to be related to specific uses of products or conditions in the burn room, and should be treated separately in the full hazard analysis.)
- It is possible to measure the LC_{50} values in a bench-scale test under different ventilation conditions, then correct the results obtained mathematically to produce that LC_{50} which would have been measured had the fuel/air ratio been such as to produce 0.2 g of CO per gram of fuel.

Reference [27] above provides the details of this correction process. The LC_{50}(corr) can be obtained knowing the values of the measured LC_{50}, the specimen mass burned during the test, and the average CO concentration measured in the animal exposure box during the test:

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

8. Data for pre-flashover fires

The same measurement of the LC_{50} is appropriate for flaming, pre-flashover fires. The combustion conditions in both the bench-scale test apparatus and the pre-flashover fire are fuel-lean. Therefore, the uncorrected LC_{50} value can be used for those cases where a value representative of pre-flashover fires is needed.

9. Details of test

The test is divided into several procedures which are conducted sequentially.

Procedure A: Determine an estimated LC_{50} using the N-Gas Model. This entails two experiments, neither involving animals. In the first experiment one uses a sample

exposed area of any convenient size, guided by any 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. Based on this result, a second experiment is performed using an exposed surface, scaled to produce an expected FED of about 1.1. The LC_{50} is then estimated by dividing the volatilized sample mass (from the second experiment) by the apparatus volume.

Procedure B: Check the estimated LC_{50} 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 min. Because of the characteristically steep slope of the dose-response curves in these tests, if the LC_{50} estimate is accurate, the exposure at FED = 0.8 should result in 0 or 1 animal deaths and the exposure at FED = 1.4 should result in 5 or 6 animal deaths. (These could occur either during the 30 min exposure or during the 14 d 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 LC_{50}. If such results are not seen, then Procedure C, below, must be used.

The CO concentration corresponding to the LC_{50} is also 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.

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 experimental approach 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. The statistical analysis of LC_{50} values is done according to the method of Litchfield and Wilcoxon [34].

9. Validation results

NIST staff have conducted validation tests to determine the degree to which the smoke from the bench-scale method reproduces that from room fires [35]. In each case, the test product was installed as a wall lining in a room 2.4 m wide × 3.7 m long × 2.4 m high. Both animal exposure data and chemical species data were collected in the fire room and in an external compartment. Most of the data were obtained after flashover, although limited pre-flashover results were possible. The products tested were:

- wood planking,
- a rigid poly(vinylchloride) sheet, and
- a rigid polyurethane foam.

Describing the degree of validation was not simple. To capture the various facets comprising 'agreement', five different criteria were used:

- The hypothesis of equal values of LC_{50} in small and large scale.
- The hypothesis that yields of all non-CO gases should be similar in both scales.
- The N-Gas Model should be able to predict the results equally well at both scales.
- The primary toxic gases, as measured in both scales, should be similar.
- The type of death (within- or post-exposure) should be similar at both scales.

The results from the first set of products showed that for three materials of very different chemistry, burning characteristic, and smoke composition, the validation was successfully proven to within a factor of 3. The results from the second series are comparable.

10. Simplifying the method

10.1. Post-flashover limit

The LC_{50} of CO_2-potentiated CO is about 5 $g\,m^{-3}$, and the yield of CO is about 0.2 $g\,g^{-1}$ of fuel burned. Therefore, the LC_{50} of post-flashover smoke is about 25 $g\,m^{-3}$. 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. Therefore, post-flashover smokes with LC_{50}(corr) values greater than 8 $g\,m^{-3}$ are indistinguishable from each other. Most common building and furnishing materials have LC_{50} values substantially higher than this. 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 $g\,m^{-3}$. An observation of no animal deaths would confirm the suggestion. The LC_{50}(corr) would then be recorded as 'greater than 8 $g\,m^{-3}$', and one would use the 8 $g\,m^{-3}$ value in a hazard analysis.

10.2. 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}(corr) greater than 8 $g\,m^{-3}$'. Some possible examples follow:

- Wood and other cellulosics, 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 g m^{-3}.
- Products that are only present in small quantities.
- 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.

11. Conclusions

A complete package has been assembled for the engineering analysis of smoke 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 US 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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