Toxicology Letters, 64/65 (1992) 257–264

© 1992 Elsevier Science Publishers B.V., All rights reserved 0378-4274/92/$ 5.00

The development of a new small-scale smoke toxicity test method and its comparison with real-scale fire tests

B.C. Levin

National Institute of Standards and Technology, Gaithersburg, MD (USA)

Key words: Smoke toxicity tests; Real-scale fire tests; N-Gas Model; Fire hazard; Post-flashover; Radiant heat

SUMMARY

A comprehensive methodology has been developed for obtaining and using smoke toxicity data for fire hazard analysis. This bench-scale method can simulate diverse fire conditions and identify extremely toxic smoke under both pre- and post-flashover conditions. However, incidence data show that most of the fire deaths in the U.S. occur outside the room of fire origin from smoke and toxic gases that are generated from a fire under post-flashover conditions. Therefore, the most relevant real-scale combustion conditions to simulate in the bench-scale apparatus would be the post-flashover conditions which are achieved by using radiant heat, a high heat flux, and correcting the bench-scale carbon monoxide (CO) results to agree with CO yields observed in real-scale post-flashover fires. The number of test animals (Fischer 344 male rats) is minimized by using the N-Gas Model to estimate the LC_{50} value from the chemical analysis of the smoke. The current N-Gas Model predicts the toxicity of complex fire gas mixtures based on a large data base of experimental results of individual and mixed gases that include CO, CO_2, reduced O_2, HCN, HCl, HBr, and NO_x. The prediction is checked with a small number of animal tests and an approximate LC_{50} value is determined. The bench-scale results have been validated with full-scale room wall burns of a limited number of materials of widely differing characteristics chosen to challenge the system. The toxic potency values are assessed to determine if the smoke from a material or product is unusually or extremely toxic and can then be used in computations of fire hazard.

INTRODUCTION

Fire in the United States continues to cause deaths and injuries at an unacceptable level. In 1990, the number of fire deaths was approximately 5200 and the number of reported injuries was about 29 000 [1]. Although

Correspondence to: B.C. Levin, National Institute of Standards and Technology, 612 Smallwood Road, Rockville, MD 20850, USA.

large fires that claim many lives receive the most media attention, the majority of fire deaths actually occur in two's and three's in residences. According to a 5-year study of 500 residential deaths in the state of Maryland, 80% of the deaths were due to smoke and toxic gas inhalation and not to direct burns [2]. The single fire scenario that causes the most deaths in the U.S. is one in which a cigarette is dropped inadvertently in a piece of upholstered furniture [3]. The furniture smolders for some undetermined amount of time (real-scale room tests of upholstered furniture at the National Institute of Standards and Technology (NIST) have indicated this time may be approximately 60 min). Then, the chair bursts into flames. Shortly thereafter, the room may experience flashover. At this time, smoke and toxic gases erupt from the room and can affect individuals who are located some distance away. Many of the victims are found in or near their beds indicating little or no attempt to escape.

Many countries in the industrialized world have developed one or more small-scale smoke toxicity test procedures to determine the toxic potency or toxic hazard of the combustion atmosphere from materials and products [4]. To date, none of these methods have been adopted as a standard by either the International Standards Organization (ISO) or any of the organizations that set standards in the U.S. (e.g., American Society for Testing and Materials (ASTM) or The National Fire Protection Association (NFPA)).

In this paper, we will describe a new small-scale smoke toxicity method designed to address many of the problems of previous test procedures and to simulate the fire scenario (post-flashover) that causes the most fire deaths in the U.S. [5]. This apparatus can also be used to simulate pre-flashover conditions. We will also describe the use of a method (called the N-Gas Model) to predict the LC_{50} value from the chemical analysis of the smoke and to minimize the number of test animals (rats) needed to determine toxic potency and unusual toxicity [6–15]. In addition, the outcome of validation studies comparing the small-scale results with those of real-scale room wall burns [16,17] will be discussed. Finally, we will show how to assess the toxic potency values to determine if the smoke from a material or product is extremely toxic (based on mass of material) or unusually toxic (based on gases deemed responsible) and how to incorporate these values in computations of fire hazard.

THE RADIANT HEAT SMOKE TOXICITY APPARATUS

This method to assess the acute inhalation toxicity of combustion products consists of three main components: an animal exposure system, a chemical analysis system, and a combustion system. The radiant heat smoke toxicity method is a closed design in which all the combustion

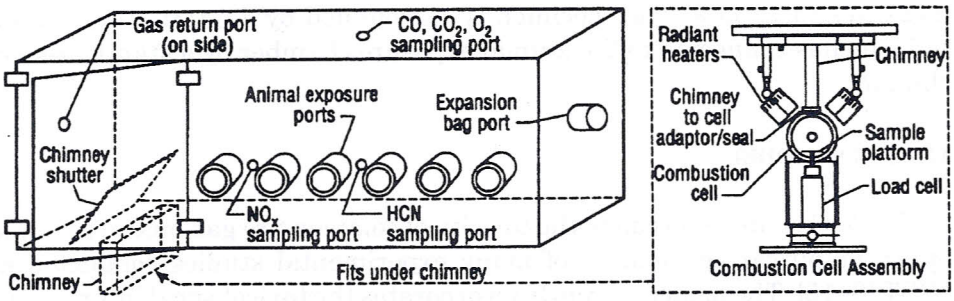

Fig. 1. US radiant smoke toxicity apparatus and radiant furnace.

products (except those samples removed for destructive chemical analysis, i.e., HCN, HCl, HBr, and NO_x) are kept within the system. The atmosphere which is removed for nondestructive chemical analysis (i.e., CO, CO_2, and O_2) is recirculated within the system. The smoke particulates are filtered out before analysis.

The animal exposure system is composed of a 200 l polymethylmethacrylate rectangular box (Fig. 1). The radiant furnace is located below one end and six portholes are positioned across the front to hold the test animals who are restrained such that only their heads are exposed to the smoke and particulates.

The chemical analysis system has been described previously [5]. In all experiments, chemical analyses are conducted to determine the concentrations of CO, CO_2, and O_2. In some experiments, HCN, and NO_x are measured if the material contains nitrogen; HCl, HF, and HBr are measured if the material is suspected of containing these halogens.

The main issue regarding smoke toxicity test methods has been the combustion systems. The problem is that no one test procedure will simulate all possible fire scenarios. The fire scenario chosen for this system is post-flashover. These conditions are achieved in the small-scale apparatus by using radiant heat, a high heat flux and correcting the CO yields obtained under small-scale conditions to agree with those yields observed in real-scale post-flashover fires [5]. In addition, the furnace is designed to permit the decomposition of materials, products, composites, and assemblies under likely end-use conditions and the testing of sample sizes up to 127 mm long, 76 mm wide and 50 mm deep (5 x 3 x 2 in).

The radiant heating combustion system consists of two quartz lamp heaters which provide an irradiance of 50 kW/m^2 to the test specimen. The specimen is tested in the horizontal, face up, orientation, and is exposed to the radiant heat for 15 min while a spark ignitor located above the center-point of the specimen insures immediate flaming. A load cell continuously

measures mass loss. The specimen is surrounded by a quartz combustion cell which is connected to the animal exposure chamber by a stainless-steel chimney.

THE N-GAS MODEL

The N-Gas Model predicts the toxicity of complex fire gas mixtures based on an empirical assessment of many experimental studies conducted at NIST [6–15]. The model currently incorporates the toxicological interactions of six gases, CO, CO_2, O_2, HCN, HCl, and HBr; NO_2 will be added this year. The prediction is checked with a small number of animal tests and the biological endpoint, an approximate LC_{50} value, is determined. The LC_{50} in these cases is defined as the mass of material loaded in the furnace or consumed by the exposure divided by the animal exposure chamber volume (g/m^3) which causes 50% of the animals to die during the 30-min exposure (within exposure deaths) or the 30-min exposure plus a 14-day post-exposure observation period (within exposure plus post-exposure deaths). If other exposure times are of concern, the model can be modified by changing the values in the denominator of the factors. The model prediction is based on the following empirical mathematical relationship:

$$\text{N-GasValue} = \frac{m[\text{CO}]}{[\text{CO}_2]-b} + \frac{[\text{HCN}]}{\text{LC}_{50}\text{HCN}} + \frac{21-[\text{O}_2]}{21-\text{LC}_{50}\text{O}_2} + \frac{[\text{HCl}]}{\text{LC}_{50}\text{HCl}} + \frac{[\text{HBr}]}{\text{LC}_{50}\text{HBr}}$$

where the numbers in brackets are the time-integrated average atmospheric concentrations during a 30-min exposure period ((ppm x min)/min or for O_2 (% x min)/min). We have found that CO_2 acts synergistically with all toxic gases tested to date. However, we have found empirically that the CO_2 term can be used in the equation only once. Therefore, since CO is found in all fires and we have the most data on the CO and CO_2 synergism [11], the CO_2 effect is utilized with the CO factor. As the concentration of CO_2 increases up to 50 000 ppm (5%), the toxicity of CO increases. Above 50 000 ppm of CO_2, the toxicity of CO starts to decrease again. The terms m and b define this synergistic interaction and equal –18 and 122 000, respectively, if the CO_2 concentrations are 50 000 ppm or less. For studies in which the CO_2 concentrations are above 50 000 ppm, m and b equal 23 and –38 600, respectively. The LC_{50} concentration of HCN is 200 ppm for 30-min exposures or 150 ppm for 30-min exposures plus 14-day post-exposure deaths. The 30-min exposure with or without the 14-day post-exposure LC_{50} value for O_2 is 5.4%. Ideally, when the N-Gas Value equals one, 50% of the animals should die. Examination of our animal lethality data for the three and four gas combinations indicate that the mean N-Gas Value where animal deaths

occur is 1.1 with a standard deviation of ±0.1. We have found in the pure gas work that one half of the animals are likely to die when the N-Gas Value is approximately 1.1, no animals usually die below 0.9 and all the animals usually die above 1.3.

Use of the N-Gas Model reduces the time necessary to evaluate a material and the number of test animals needed for the toxic potency determination. It also indicates whether the toxicity is usual (i.e., the toxicity can be explained by the measured gases) or is unusual (i.e., additional gases are needed to explain the toxicity). The N-Gas approach has been shown to work well under different combustion systems (radiant as well as convective heat sources; bench-scale as well as full-scale room tests) [16–17].

THE RADIANT HEAT SMOKE TOXICITY METHOD

To measure the toxic potency of a given material with this method, two analytical chemical and two animal exposure experiments are conducted for each material. In analytical experiment 1, a sample approximately 1/4th the largest sample permissible or 10 g (whichever is smaller) is exposed to the radiant heat flux for 15 min with the ignitor on. Concentrations of the principal gaseous components (CO, CO_2, HCN, reduced O_2, HCl and HBr) of the smoke are measured for 30 min. Based on the concentrations of the measured gases and the N-Gas Model, the mass of material necessary to produce an N-Gas Value of 1.1 is calculated. Analytical experiment 2 is conducted at this mass loading. Animal exposure experiment 1 is based on the results of analytical experiments 1 and 2. The mass loading necessary to generate an N-Gas Value of 0.8 is calculated and six rats are exposed to the combustion atmosphere for 30 min (the radiant lamps are turned off after 15 min). In this experiment, one would expect no animal deaths either during the exposure or during the 14-day post-exposure observation period (one death may be acceptable). In the second animal exposure experiment, the mass loading (calculated based on all previous experiments) necessary to generate an N-Gas Value of 1.4 is placed in the combustion chamber. In this experiment, one would expect all the test animals to die (5 animals out of the six tested would also be acceptable). If no animals die at an N-Gas Value of 0.8 and all the animals die at an N-Gas Value of 1.4, then the predicted LC_{50} value which is equivalent to an N-Gas Value of 1.1 is considered the approximate LC_{50} value. This value can be calculated for deaths that occur during the 30-min exposure and for the 30-min plus 14-day post-exposure observation period for both the mass of the material loaded into the furnace or the mass consumed.

The deaths of more than one of the animals at an N-Gas Value of 0.8 indicates the presence of one or more unknown toxicants, an unknown

synergism or the presence of other unknown factors. Fewer than 5 animal deaths at an N-Gas Value at or above 1.4 suggests that a toxicological antagonism is occurring. If more accuracy is needed, a detailed LC_{50} can be determined. In this case, the percentage of animals dying at each fire effluent concentration is plotted to produce a concentration-response curve from which LC_{50} values can be statistically calculated.

VALIDATION OF SMALL-SCALE RESULTS WITH REAL-SCALE ROOM BURNS

Five criteria were established to compare the small-scale and real-scale results and determine if the small-scale methodology could predict the outcome of room size fires.

1. LC_{50} values measured in the small-scale test should agree with those obtained in the real-scale one within the acceptable uncertainty.
2. The primary toxic gases found in the small-scale test should be the same as those in the real-scale test.
3. The yields of the measured toxic gases in the small-scale tests should agree with those found in the real-scale tests within the acceptable uncertainty.
4. The N-Gas Model predictions based on the small-scale results should agree with those found in the real-scale tests to within the acceptable uncertainty.
5. The type of death (whether the deaths occurred within- or post-exposure) should be similar for the small-scale and the real-scale tests.

To be usable, the criteria must be accompanied by a statement as to what factor of agreement is expected between the small-scale and the real-scale results. The study showed that a factor of 3 was useful and achievable. Although only a small number of materials has been examined under both small-scale and real-scale conditions, validation was achieved for all criteria except number 5 for which there was insufficient data and for the CO gas yield in number 3 where we found that the small-scale underpredicted the yield (the other measured gas yields showed good agreement).

USE OF THE TOXIC POTENCY DATA IN HAZARD ANALYSIS

The LC_{50} of CO_2-potentiated CO for 30-min exposures is about 5 g/m^3 and the yield of CO during flashover is about 0.2 g/g of fuel burned. Therefore, the LC_{50} of post-flashover smoke (assuming the primary toxicants are CO and CO_2) is about 25 g/m^3. Previous work on validation of this radiant system showed that the results could be used to predict real-scale toxic potency to about a factor of 3 [16]. Therefore, since all combustion produces CO and CO_2, post-flashover smoke with LC_{50} values greater than 8 g/m^3 are

indistinguishable from each other. In small-scale tests, most common building and furnishing materials have LC_{50} values (corrected for CO yields from real-scale fires) substantially higher than this. If the LC_{50} value is 8 g/m^3 or greater, the value of 8 would be used in a hazard assessment. If the LC_{50} value is less than 8 g/m^3, then the actual value should be used.

To deal with exposure times other than 30 min, we have empirically found that an approximation for the toxic potency of post-flashover smoke is $LC_{50} \approx 45$ g m^{-3} min$^{1/2}$. This approximation is offered for hazard calculations for exposure times greatly different from 30 min.

CONCLUSIONS

A new radiant smoke toxicity method has been developed to:

1. simulate a fire scenario (post-flashover) known to cause the majority of U.S. deaths from combustion gases,
2. evaluate toxicity of products, composites and assemblies,
3. determine extreme toxic potency and unusual toxicity,
4. use a bioanalytical approach (the N-Gas Model) to predict the toxicity and minimize the use of experimental animals (rats),
5. predict lethal atmospheres in real fires; for this, a CO correction is necessary and
6. generate data to be used in fire hazard computer models such as HAZARD I.

This new toxicity procedure provides information for properly including fire smoke toxicity in safety decisions.

REFERENCES

1 Karter, M.J. Jr. (1991) Fire loss in the United States during 1990. NFPA J. 85, 36–48.
2 Birky, M.M., Halpin, B.M., Caplan, Y.H., Fisher, R.S., McAllister, J.M. and Dixon, A.M. (1979) Fire fatality study. Fire Materials 3, 211–217.
3 Miller, A. (1991) What's burning in home fires. NFPA J. 85, 72–78.
4 Kaplan, H.L., Grand, A.F. and Hartzell, G.E. (1983) In: Combustion Toxicology: Principles and Test Methods. Technomic Publishing, Lancaster, PA.
5 Babrauskas, V., Levin, B.C., Gann, R.G., Paabo, M., Harris, R.H. Jr., Peacock, R.D. and Yusa, S. (1991) Toxic Potency Measurement for Fire Hazard Analysis. NIST Special Publication 827, National Institute of Standards and Technology, Gaithersburg, MD.
6 Levin, B.C., Paabo, M., Bailey, C., Harris, S.E. and Gurman, J.L. (1985) Toxicological effects of the interactions of fire gases and their use in a toxic hazard assessment computer model. Toxicologist 5, 127.
7 Levin, B.C. and Gann, R.G. (1990) Toxic potency of fire smoke; measurement and use. In: G.L. Nelson (Ed.), Fire and Polymers: Hazards Identification and Prevention. ACS Symposium Series 425, American Chemical Society, Washington, DC. pp. 3-11.
8 Levin, B.C., Paabo, M., Gurman, J.L. and Harris, S.E. (1987) Effects of exposure to single or multiple combinations of the predominant toxic gases and low oxygen atmospheres produced in fires. Fundam. Appl. Toxicol. 9, 236–250.

9 Levin, B.C., Paabo, M., Gurman, J.L., Clark, H.M. and Yoklavich, M.F. (1988) Further studies of the toxicological effects of different time exposures to the individual and combined fire gases: Carbon monoxide, hydrogen cyanide, carbon dioxide, and reduced oxygen. In: Polyurethane '88, Proceedings of the 315th Society of Plastics Meeting. Technomic Publishing, Lancaster, PA. pp. 249–252.

10 Levin, B.C., Paabo, M., Highbarger, L. and Eller, N. (1990) Toxicity of complex mixtures of fire gases. Toxicologist 10, 84.

11 Levin, B.C., Paabo, M., Gurman, J.L., Harris, S.E. and Braun, E. (1987) Toxicological interactions between carbon monoxide and carbon dioxide. Toxicology 47, 135–164.

12 Levin, B.C., Gurman, J.L., Paabo, M., Baier, L. and Holt, T. (1988) Toxicological effects of different time exposures to the fire gases: Carbon monoxide or hydrogen cyanide or to carbon monoxide combined with hydrogen cyanide or carbon dioxide. In: Proceedings of the Ninth Joint Panel Meeting of the US-Japan (UJNR) Panel on Fire Research and Safety. NBSIR 88-3753, National Institute of Standards and Technology, Gaithersburg, MD.

13 Levin, B.C., Paabo, M., Highbarger, L. and Eller, N. (1989) Synergistic Effects of Nitrogen Dioxide and Carbon Dioxide Following Acute Inhalation Exposures in Rats. NISTIR 89-4105, National Institute of Standards and Technology, Gaithersburg, MD.

14 Hartzell, G.E., Grand, A.F. and Switzer, W.G. (1990) Toxicity of smoke containing hydrogen chloride. In: G.L. Nelson (Ed.), Fire and Polymers: Hazards Identification and Prevention. ACS Symposium Series 425, American Chemical Society, Washington, DC. pp. 12–20.

15 Levin, B.C., Paabo, M. and Navarro, M. (1991) Toxic interactions of binary mixtures of NO_2 and CO, NO_2 and HCN, NO_2 and reduced O_2, and NO_2 and CO_2. Toxicologist 11, 222.

16 Babrauskas, V., Harris, R.H. Jr., Braun, E., Levin, B.C., Paabo, M. and Gann, R.G. (1991) The Role of Bench-Scale Test Data in Assessing Real-Scale Fire Toxicity. NIST Technical Note 1284, National Institute of Standards and Technology, Gaithersburg, MD.

17 Braun, E., Levin, B.C., Harris, R.H. Jr., Paabo, M., Navarro, M. and Gann, R.G. (1992) Real-Scale Test Data for Verification of Bench-Scale Toxicity Tests. National Institute of Standards and Technology, Gaithersburg, MD. (NISTIR, manuscript in preparation).