TECHNICAL REPORT

ISO/TR 16312-2

First edition
2007-03-01

Guidance for assessing the validity of physical fire models for obtaining fire effluent toxicity data for fire hazard and risk assessment —

Part 2: Evaluation of individual physical fire models

Lignes directrices pour évaluer la validité des modèles de feu physiques pour l'obtention de données sur les effluents du feu en vue de l'évaluation des risques et dangers —

Partie 2: Évaluation des différents modèles de feu physiques

Reference number
ISO/TR 16312-2:2007(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 16312-2 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

ISO 16312 consists of the following parts, under the general title *Guidance for assessing the validity of physical fire models for obtaining fire effluent toxicity data for fire hazard and risk assessment*:

— *Part 1: Criteria*

— *Part 2: Evaluation of individual physical fire models* [Technical Report]

Introduction

Providing the desired degree of life safety for an occupancy increasingly involves an explicit fire hazard or risk assessment. This assessment includes such components as information on the room/building properties, the nature of the occupancy, the nature of the occupants, the types of potential fires, the outcomes to be avoided, etc.

This type of determination also requires information on the potential for harm to people due to the effluent produced in the fire. Because of the prohibitive cost of real-scale product testing under the wide range of fire conditions, most estimates of the potential harm from the fire effluent depend on data generated from a physical fire model, a reduced-scale test apparatus and procedure for its use.

The role of a physical fire model for generating accurate toxic effluent composition is to simulate the essential features of the complex thermal and reactive chemical environment in full-scale fires. These environments vary with the physical characteristics of the fire scenario and with time during the course of the fire, and close representation of some phenomena occurring in full-scale fires can be difficult or even not possible at the small scale. The accuracy of the physical fire model, then, depends on two features:

a) degree to which the combustion conditions in the bench-scale apparatus mirror those in the fire stage being simulated;

b) degree to which the yields of the important combustion products obtained from burning of the commercial product at full scale are matched by the yields from burning specimens of the product in the small-scale model. This measure is generally performed for a small set of products, and the derived accuracy is then presumed to extend to other test subjects. At least one methodology for effecting this comparison has been developed.[1]

This part of ISO 16312 provides a set of technical criteria for evaluating physical fire models used to obtain composition and toxic potency data on the effluent from products and materials under fire conditions relevant to life safety. This Technical Report comprises the application by experts of these criteria to currently used test methods that are used for generating data on smoke effluent from burning materials and commercial products.

There are 12 physical fire models discussed in this part of ISO 16312. Additional apparatus can be added as they are developed or adapted with the intent of generating information regarding the toxic potency of smoke.

For the 12 models in this part of ISO 16312, the first five are closed systems. In these, no external air is introduced and the combustion (or pyrolysis) products remain within the apparatus except for the fraction removed for chemical analysis. The second seven are open apparatus, with air continuously flowing past the combusting sample and exiting the apparatus, along with the combustion products.

To make use of this part of ISO 16312, it is necessary for the user to have present a copy of ISO 16312-1, which contains much of the context and definitions for the present document. It is also necessary to make reference to ISO 19701[33], ISO 19702[34], ISO 19703, ISO 13344[31], and ISO 13571[32] for discussions of analytical methods, bioassay procedures, and prediction of the toxic effects of fire effluents.

Guidance for assessing the validity of physical fire models for obtaining fire effluent toxicity data for fire hazard and risk assessment —

Part 2: Evaluation of individual physical fire models

1 Scope

This part of ISO 16312 assesses the utility of physical fire models that have been standardized, are commonly used and/or are cited in national or international standards, for generating fire effluent toxicity data of known accuracy. It does so using the criteria established in ISO 16312-1 and the guidelines established in ISO 19706. The aspects of the models that are considered are the intended application of the model, the combustion principles it manifests, the fire stage(s) that the model attempts to replicate, the types of data generated, the nature and appropriateness of the combustion conditions to which test specimens are exposed and the degree of validity established for the model.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 13943, *Fire safety — Vocabulary*

ISO 16312-1, *Guidelines for assessing the validity of physical fire models for obtaining fire effluent toxicity data for fire hazard and risk assessment — Part 1: Criteria*

ISO 19703, *Generation and analysis of toxic gases in fire — Calculation of species yields, equivalence ratios and combustion efficiency in experimental fires*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943 and in ISO 19703 apply.

4 General principles

4.1 Physical fire model

A physical fire model is characterized by the requirements placed on the form of the test specimen, the operational combustion conditions and the capability of analysing the products of combustion.

4.2 Model validity

For use in providing data for effluent toxicity assessment, the validity of a physical fire model is determined by the degree of accuracy with which it reproduces the yields of the principal toxic components in real-scale fires.

4.3 Test specimens

Fire safety engineering requires data on commercial products or product components. In a reduced-scale test, the manner in which a specimen of the product is composed can affect the nature and yields of the combustion products. This is especially the case for products of non-uniform composition, such as those consisting of layered materials.

4.4 Combustion conditions

The yields of combustion products depend on such apparatus conditions as the fuel/air equivalence ratio, whether the decomposition is flaming or non-flaming, the persistence of flaming of the sample, the temperature of the specimen and the effluent produced, the thermal radiation incident on the specimen, the stability of the decomposition conditions and the interaction of the apparatus with the decomposition process, with the effluent and the flames.

4.5 Effluent characterization

4.5.1 For the effluent from most common materials, the major acute toxic effects have been shown to depend upon a small number of major asphyxiant gases and a somewhat wider range of inorganic and organic irritants. In ISO 13571[32], a base set of combustion products has been identified for routine analysis. Novel materials can evolve previously unidentified toxic products. Thus, a more detailed chemical analysis can be needed in order to provide a full assessment of acute effects and to assess chronic or environmental toxicants. A bioassay can provide guidance on the importance of toxicants not included in the base set. ISO 19706[35] contains a fuller discussion of the utility of bioassays.

4.5.2 It is essential that the physical fire model enable accurate determinations of chemical effluent composition.

4.5.3 It is desirable that the physical fire model accommodate a bioassay method.

4.5.4 The use of laboratory animals as test subjects is the only means of insuring inclusion of the impact of all combustion gases. However, it is recognized that the adoption and use of protocols using laboratory animals can be prohibited in some jurisdictions. An animal-free protocol captures the effects of known combustion gases but misses the impact of any uncommon and highly toxic species, those smoke components that are most in need of identification. Laboratory studies to date have shown that lethality from smoke inhalation results from the combined effects of a small number of gases and that none of the missing gases is “supertoxic.” There are also data that indicate incapacitation results from half the lethal exposure for a wide range of today's materials, indicating that exotic gases do not affect incapacitation without affecting lethality as well. The decision to base hazard and risk assessments on analytical or animal-based measurements resides with the authority having jurisdiction.

5 Significance and use

5.1 Most computational models of fire hazard and risk require information regarding the potential of fire effluent (gases, heat and smoke) to cause harm to people and to affect their ability to escape or to seek refuge.

5.2 The quality of the data on fire effluent has a profound effect on the accuracy of the prediction of the degree of life safety offered by an occupancy design. Uncertainty in such predictions commonly leads to the use of safety factors that can compromise functionality and increase cost.

5.3 Fire safety engineering requires data on commercial products. Real-scale tests of such products generally provide accurate fire effluent data. However, due to the large number of available products, the high cost of performing real-scale tests of products and the small number of large-scale test facilities, information on effluent toxicity is most often obtained from physical fire models.

5.4 There are numerous physical fire models cited in national regulations. These apparatus vary in design and operation, as well as in their degree of characterization. The assessments of these models in this part of ISO 16312 provide product manufacturers, regulators and fire safety professionals with insight into appropriate and inappropriate sources of fire effluent data for their defined purposes.

5.5 None of the models in this part of ISO 16312 is appropriate for simulation of smouldering combustion.

5.6 The assessments of physical fire models in this part of ISO 16312 do not address means for combining the effluent component yields to estimate the effects on laboratory animals (see ISO 13344[31]) or for extrapolating the test results to people (see ISO 13571[32]).

6 Physical fire models

6.1 Cup-furnace smoke-toxicity test method

6.1.1 Application

This method[2] is designed to generate toxic potency data for materials and, perhaps, end products. It is not a national or international standard.

6.1.2 Principle

A schematic of the apparatus is shown in Figure 1. The furnace is open to an 0,2 m^3 closed reservoir from which (air) oxygen is supplied by natural buoyancy. Vitiation in the reservoir is measured. The sample (approximately 10 g) is cut into pieces and heated conductively, convectively and (at higher temperatures) radiatively to just below or just above its auto-ignition temperature.

6.1.3 Fire stage(s)

The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis;

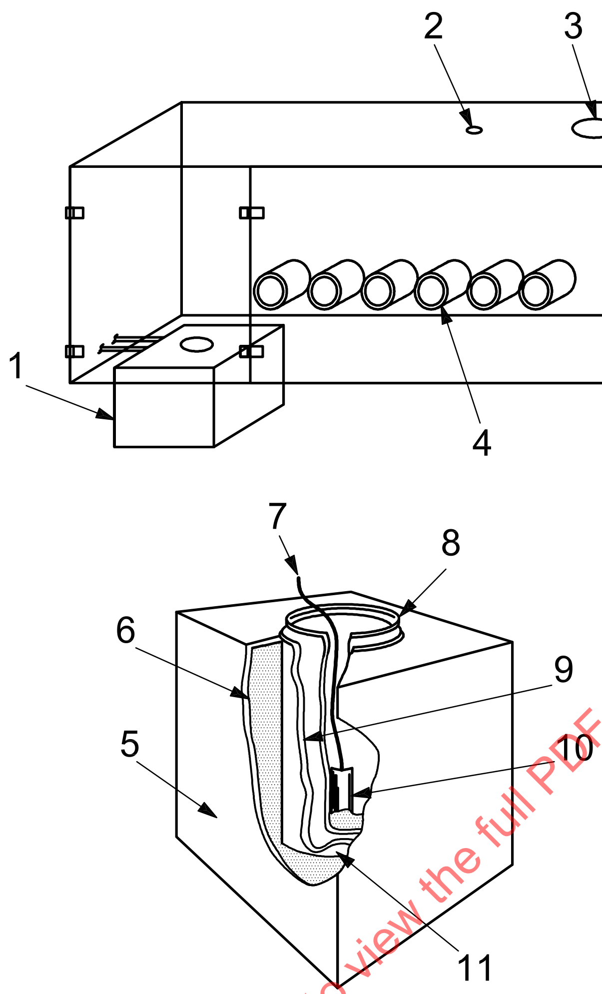
— 2, well-ventilated flaming.

6.1.4 Types of data

The standard procedure includes measurement of total mass loss, averaged mass consumed and mass charged concentrations, gas concentrations and gas yields. The gases to be measured are: CO_2, CO, O_2, HCN, HCl and HBr. In addition, the procedure includes measurement of the incapacitation (by hind-leg flexion or immobilization) and mortality of six rats, the times to these effects and documentation of any physiological harm, determined post-mortem. Blood samples are taken during and after exposure for subsequent analysis.

6.1.5 Presentation of results

Sufficient tests are performed, at different mass loadings, to determine LC_{50} and IC_{50} values and their confidence limits for within exposure and within-plus-post-exposure periods.



Key

1 furnace
2 gas-sampling port
3 pressure-relief panel
4 animal ports
5 galvanized sheet
6 insulation
7 thermocouple
8 1 000 ml quartz beaker
9 ceramic
10 thermocouple well
11 heating element in bottom

Figure 1 — Schematic of the cup-furnace smoke-toxicity apparatus

6.1.6 Apparatus assessment

6.1.6.1 Advantages

Each test uses a small sample. The apparatus is inexpensive and easy to operate. Data for a wide range of materials and products have been published. There is a close similarity to the oxidative pyrolysis conditions in real-scale fires.

6.1.6.2 Disadvantages

The realism of sample exposure is questionable due to the cutting up of the sample, especially for non-homogeneous products. For well-ventilated combustion, the simulation of real-scale heating, which is primarily radiative, is poor. Mixing by natural buoyancy makes values of the global equivalence ratio somewhat uncertain. In common with many physical fire models, no indication is given about the rate of burning;

therefore, additional data input on burning rates at different fire stages are needed for fire safety engineering calculations.

6.1.6.3 Repeatability and reproducibility

A successful inter-laboratory evaluation of this method has been performed[3].

6.1.7 Toxicological results

6.1.7.1 Advantages

The method produces true measures of smoke lethality and incapacitation and identifies instances of extreme and unusual smoke toxic potency. It also produces data enabling calculation of the yields of measured toxicants. It can identify cases where unusual toxicity occurs as a result of constituents not identified by the analytical procedures applied.

6.1.7.2 Disadvantages

The relationship between data for a finished product and data for its component materials has not been determined. The concentration of combustion products is not truly uniform over the entire animal-exposure period, introducing some reduction in the precision of the lethality and incapacitation measures.

6.1.8 Miscellaneous

This is primarily an animal-exposure test with chemical instrumentation to quantify the expected major toxicants. Additional analytical instrumentation can be added with little interference with the standard method. The apparatus can be used without test animals, but it then loses the ability to identify the principal cases of real interest.

6.1.9 Validation

The toxic potency and gas yield data did not replicate real-scale post-flashover test data well[1]. The method has not been assessed against real-scale test data for oxidative pyrolysis or well-ventilated flaming.

6.1.10 Conclusion

This method is potentially a useful test for screening the toxic potency of materials and homogeneous products. However, cutting the specimen into pieces makes it unlikely that the test results relate to the real fire exposure of heterogeneous end products. Thus, with validation, it can produce useful information for hazard models for oxidative pyrolysis and well-ventilated flaming of homogeneous materials, but not of complex commercial products.

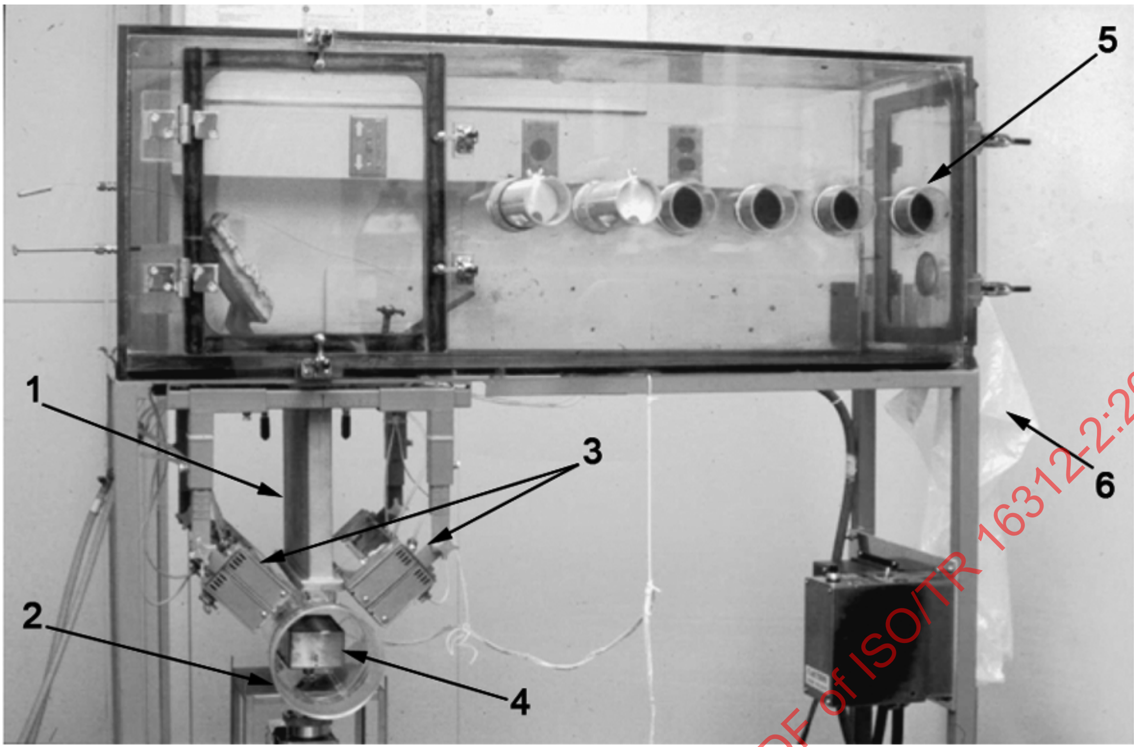
6.2 Radiant furnace toxicity test method (United States)

6.2.1 Application

This apparatus, used in NFPA 269[4] and ASTM E 1678[5], was designed to generate toxic potency data for building and furnishing materials and end products for use in fire and hazard analyses.

6.2.2 Principle

A photograph of the apparatus is shown in Figure 2. A sample, up to 76 mm x 127 mm in area and up to 50 mm in thickness and representative of the end-use configuration of the finished product, is exposed to thermal radiation. Buoyancy from the burning sample entrains air from a closed reservoir similar to that described in 6.1.



Key

1 chimney
2 combustion cell
3 radiant heaters
4 specimen holder
5 animal ports
6 expansion bag

Figure 2 — Photograph of the NFPA 269 apparatus

6.2.3 Fire stage(s)

The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 2, well-ventilated flaming combustion;

— 3, under-ventilated flaming (using a post-flashover correction for the yield of CO);

— 1.b, oxidative pyrolysis, if the sample does not auto-ignite.

6.2.4 Types of data

The standard procedure includes continuous measurement of mass loss and gas concentrations, gas yields, and atmosphere vitiation. In addition, the procedure includes measurement of the mortality of six rats and documentation of any physiological harm, determined post-mortem.

6.2.5 Presentation of results

Sufficient tests are performed, at different sample surface areas, to determine LC_{50} values and their confidence limits for within exposure and within-plus-post-exposure periods.

6.2.6 Apparatus assessment

6.2.6.1 Advantages

The representation and exposure of finished products is accurate. The mass burning rate is recorded, enabling direct use of the yield data in engineering calculations.

6.2.6.2 Disadvantages

Mixing by natural buoyancy makes values of global equivalence ratio somewhat uncertain. Limited sample intumescence can be tolerated.

6.2.6.3 Repeatability and reproducibility

No inter-laboratory evaluation of this method has been performed.

6.2.7 Toxicological results

6.2.7.1 Advantages

The method produces a true measure of smoke lethality and identifies instances of extreme and unusual smoke toxic potency. It also produces data enabling calculation of the yields of measured toxicants. The method can be adapted to measure incapacitation (hind-leg flexion or immobilization).

6.2.7.2 Disadvantages

The use of an empirically derived correction for CO introduces some uncertainty into the LC_{50} values. Furthermore, altered yields of other product gases are not included. However, these factors are included in the comparison of LC_{50} values with room-scale test data in Reference [1]. This correction limits the value of this method for other sublethal effects in which the uncorrected gas yields play a prominent role.

6.2.8 Miscellaneous

This is primarily an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation can be added with little interference with the standard method. The apparatus can be used without test animals, but it then loses the ability to identify the principal cases of real interest.

Using a generic, experimentally observed carbon monoxide yield correction, accurate post-flashover LC_{50} values have been obtained relative to real-scale fire tests of the same combustibles[1].

6.2.9 Validation

The output of this method for three products has been compared to room-scale data for the same products (wall lining configuration)[1]. The post-flashover LC_{50} values were well within a factor of 2. No data were taken for pre-flashover combustion.

6.2.10 Conclusion

This is a useful test for obtaining quantitative toxic potency information for materials and end products for input to fire hazard models.

6.3 Closed cabinet toxicity test (international)

6.3.1 Application

This physical fire model is used in ISO 5659-2[6] and ASTM E 1995[7]. It was designed to generate smoke optical density data. The International Maritime Organization (IMO) also requires use of this apparatus for toxic gas concentration data for qualification of materials.

6.3.2 Principle

A schematic of this closed cabinet test is shown in Figure 3. A horizontally mounted specimen, 75 mm square and up to 25 mm thick, is exposed to a radiant heater for a minimum of 10 min. A test is conducted at 25 kW/m^2 with and without pilot flame and at 50 kW/m^2 without pilot flame. The gases are sampled through probes positioned at the geometrical centre of the smoke box.

6.3.3 Fire stage(s)

The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis;

— 3.a, well-ventilated flaming combustion; see 6.3.6.2.

6.3.4 Types of data

The standard procedure includes measurement of total mass loss, smoke obscuration and specific effluent gas concentrations (CO_2, CO, HCN, HCl, HF, HBr, NO_x, SO_2) at the time when the maximum smoke concentration is reached. In the two aircraft tests, the specific optical density of the smoke and the gas concentrations are determined at 90 s and 240 s. Fire effluent for cables may be determined after 20 min.

6.3.5 Presentation of results

The specific optical density of the smoke and the combustion fire gas concentrations are compared to specified values.

6.3.6 Apparatus assessment

6.3.6.1 Advantages

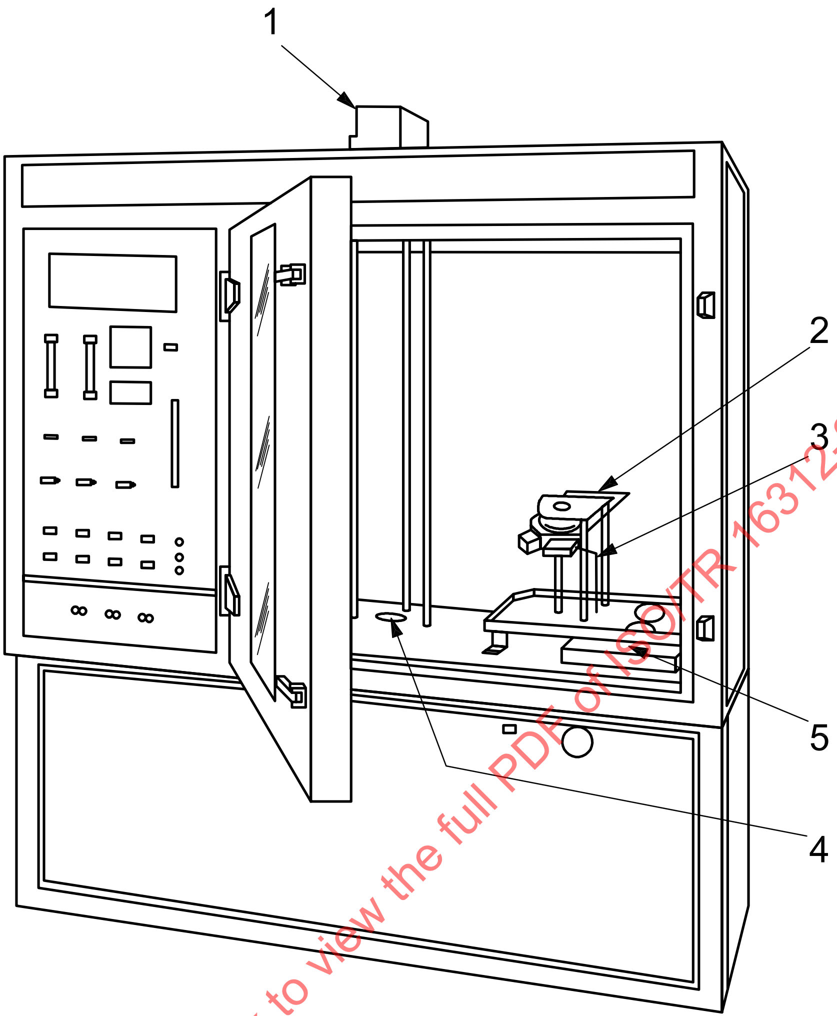
The apparatus is simple to use and widely available. The test specimen can be a reasonable representation of a finished product.

6.3.6.2 Disadvantages

The combustion conditions are fixed and not well characterized. The test specimen is vertical and melting materials can flow into the trough below the specimen holder or even onto the floor of the test chamber, thereby altering the combustion mode or even reducing the amount of specimen destroyed. The gases are mixed by natural convection and possible stratification can lead to non-representative sampling of the combustion gases. Vitiation can occur with thick or less thermally stable materials and affects the yields of combustion products.

6.3.6.3 Repeatability and reproducibility

Inter-laboratory evaluations have been performed for the smoke density test and gave satisfactory results for a range of materials. No inter-laboratory evaluation of toxic gas production has been reported.



Key

1 photomultiplier-tube housing
2 radiator cone
3 pilot burner
4 light source window
5 blow-out panel

Figure 3 — Schematic of the closed cabinet toxicity test apparatus

6.3.7 Toxicological results

6.3.7.1 Advantages

The initial conditions are few and well prescribed.

6.3.7.2 Disadvantages

Possible vitiation can lead to time-dependent generation of toxicants that are sampled only at a specified time. Condensation can occur on the wall of the chamber leading to removal of some gases from the sampled environment. The possibility of condensation on the wall of the chamber can lead to removal of some gases from the sampled environment. The prescribed set of gases to be measured can be insufficient to estimate lethal toxic potency.

6.3.8 Miscellaneous

No animals are exposed in the test, nor is the apparatus compatible with such an addition. The use of the chemical data is typically limited to a comparison with critical concentrations of listed toxic gases.

6.3.9 Validation

There are no reported comparisons of toxic gas generation with data from real-scale fire tests.

6.3.10 Conclusion

While relatively easy to perform, this method is of questionable value for generating smoke toxicity data for use in fire hazard analysis. It is also necessary to verify its use as a screening tool against real-scale fire test data. The absence of animal-exposure data means that smoke extreme or unusual toxic potency is not identified.

6.4 Closed flask test (Israel)

6.4.1 Application

This apparatus, used in the Israeli standard SI 755[8], is for classification of building materials. SI 755:1998[8], Section 2.6, addresses the total risk of gas toxicity.

6.4.2 Principle

As shown in Figure 4, this apparatus is a closed spherical bulb with a volume of at least 3 l. It has a tubular extension, about 10 % of the total bulb volume, that is inserted into an oven heated to either 250 °C or 550 °C. The former temperature is below nearly all auto-ignition temperatures; the latter is above the auto-ignition temperature for vapours from many materials. The test specimen is about 3×10^{-5} of the bulb volume. There is no ignition source. The bulb volume contains excess air. Several gas samples are extracted from the bulb to find the maximum concentration of each of six gases. The test that gives the highest gas concentration is used.

6.4.3 Fire stage(s)

The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis;

— 2, well-ventilated flaming;

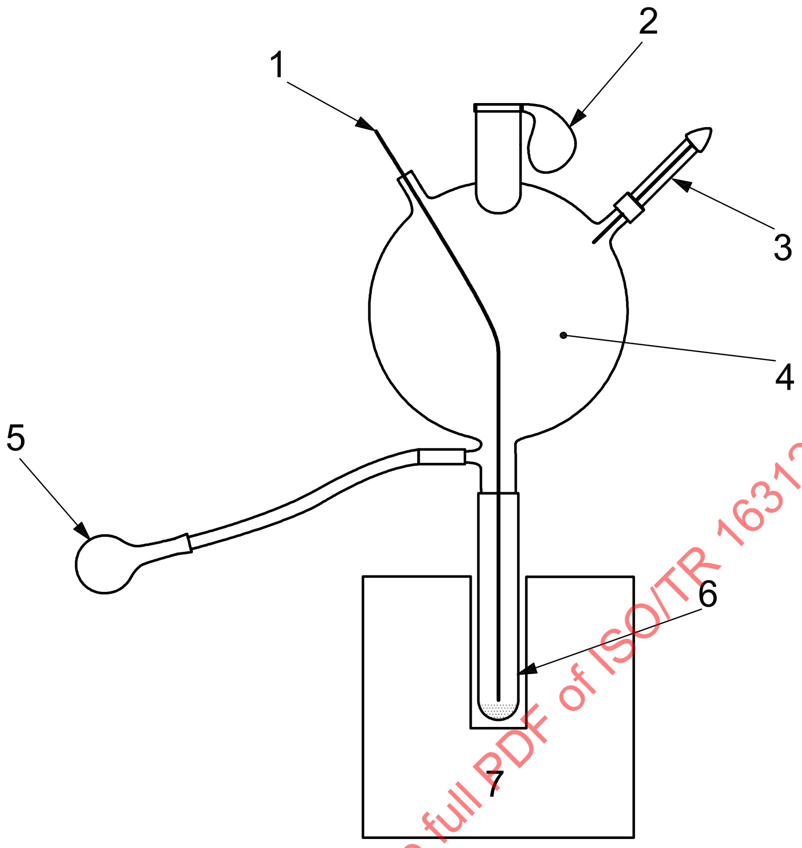
— 3a, under-ventilated flaming.

6.4.4 Types of data

The reported data are the maximum concentrations of CO, HCN, NO_x, HCl, SO_2, and formaldehyde.

6.4.5 Presentation of results

The output is a simplified toxicity index similar to a fractional effective dose (FED) for the six gases. This is presumably for incapacitation, since the coefficients for the gas concentrations are based on “critical concentrations for escape during fires.” The rationale for the equation to calculate the total risk of gas toxicity in a 300 cm high room is not given.



Key

1 thermocouple
2 expansion balloon
3 gas-sampling port
4 collection vessel for effluent
5 device for mixing gases
6 test specimen
7 oven

Figure 4 — Schematic of the SI-755 smoke toxicity apparatus

6.4.6 Apparatus assessment

6.4.6.1 Advantages

The method uses an inexpensive, easy-to-operate apparatus and a small sample.

6.4.6.2 Disadvantages

The higher temperature should produce the highest effluent concentrations. However, results for different materials might not be comparable since ignition is not controlled. If ignition occurs in the tubular extension, the combustion would become vitiated quickly. Manual mixing of air within the bulb is not well defined. Poor mixing can result in stratification, poor sampling, oxygen depletion and the condensation of certain gases. The small sample might not be representative of a non-homogeneous material or product. There is a small balloon, presumably to contain a pressure rise if auto-ignition occurs. It is not clear whether this is sufficient to prevent leakage. The concentrations of the gases depend on the bulb volume, which is not fully defined. In common with many physical fire models, no indication is given about the rate of burning, so highly fire-retarded materials can be forced to burn at the same rate as materials without any fire retardants. Therefore, additional data input on burning rates at different fire stages is required for fire safety engineering calculations.

6.4.6.3 Repeatability and reproducibility

There are no known published reports of an inter-laboratory evaluation.

6.4.7 Toxicological results

6.4.7.1 Advantages

The calculation of the index is simple and enables estimation of the relative importance of the six gases in affecting escape.

6.4.7.2 Disadvantages

The realism of sample exposure is questionable due to its small size and the shape of the tubular extension. The coefficients for the toxicity index calculation are not current. It can be necessary to include additional gases, as the prescribed set of gases to be measured might be insufficient to estimate lethal toxic potency. The basis for the risk equation is unclear and does not appear to be related to actual risk.

6.4.8 Miscellaneous

The test does not involve laboratory animals. The gases may be measured by any suitable method.

6.4.9 Validation

No data are provided.

6.4.10 Conclusion

The method has questionable value for screening the toxic potency of materials and products, since some test specimens flame and others pyrolyse. The pyrolysis or combustion gas concentrations are not likely to be representative of room-scale effluent.

6.5 NES 713 (United Kingdom)

6.5.1 Application

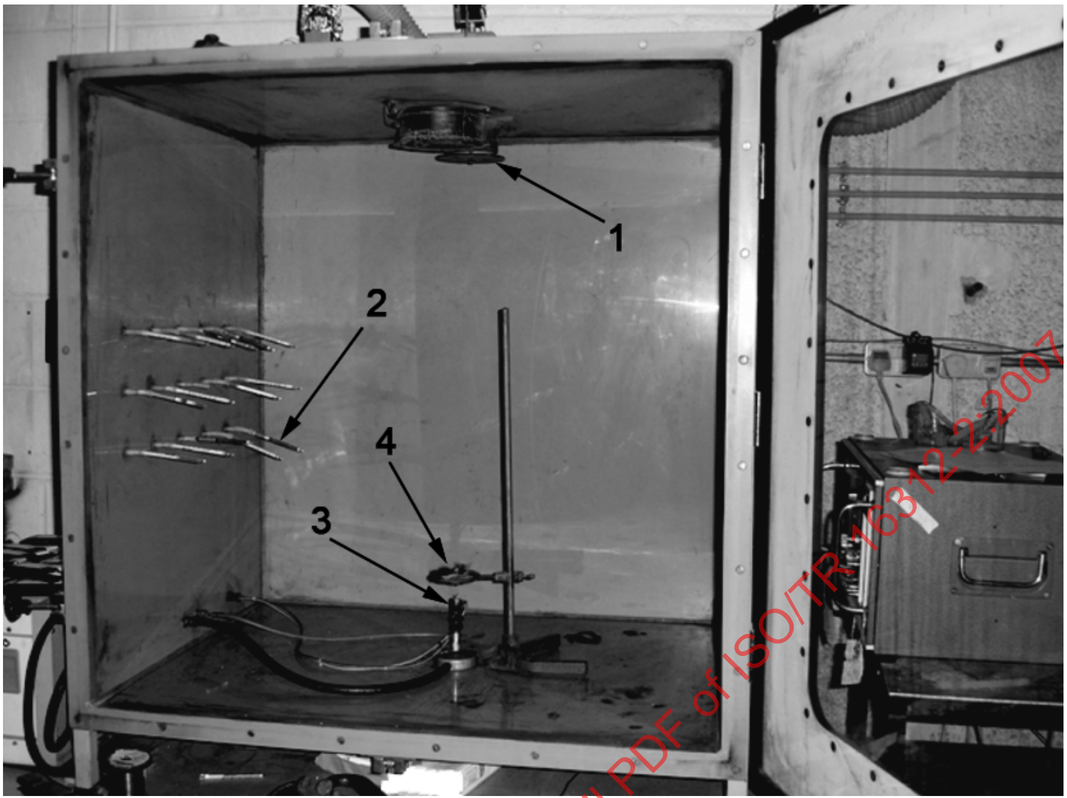
This apparatus described in NES 713[9] is designed to provide values of a toxicity index for use in short-listing materials and end products for warship marine use.

6.5.2 Principle

A photograph of this closed cabinet test is shown in Figure 5. A specimen of size chosen to provide optimal analytical precision (typically 1 g to 2 g) is exposed to a premixed Bunsen burner flame. The burner is turned off after the specimen has burned to completion, and the atmosphere is mixed with a fan before being sampled for gas measurement.

6.5.3 Fire stage(s)

The fire stage from ISO 19706:2007[35], Table 1, is 2, well-ventilated flaming. However, this might not relate to a real fire, as the burner is actually a premixed blow-torch-type flame at about 850 °C and not a free-burning fire.



Key

1 mixing fan
2 gas-sampling tube
3 gas burner
4 specimen support

Figure 5 — Photograph of the NES 713 apparatus

6.5.4 Types of data

The standard procedure includes measurement of CO, CO_2, formaldehyde, NO_x, HCN, acrylonitrile, phosgene, SO_2, H_2S, HCl, NH_3, HF, HBr and phenol. Corrections are applied for the concentrations of CO, CO_2 and NO_x produced by the gas flame alone burning for the same period as the test specimen.

6.5.5 Presentation of results

The output is an FED-like toxicity index for the 14 gases. The weightings of the gases are the concentrations considered nominally lethal to a man for a 30 min exposure. The index is calculated for 1 g of sample or for 1 m of wire or cable.

6.5.6 Apparatus assessment

6.5.6.1 Advantages

The apparatus is simple to use. The combustion period is short, so the combustion environment is stable throughout. The test specimen can be a reasonable representation of a finished product.

6.5.6.2 Disadvantages

Nearly all materials and end products are composed of multiple components. These can gasify at different times during burning. The test specimen is immersed in a pre-mixed gas flame and is burned to completion but this might not produce gases representative of the combustion of the sample in real fire conditions. The test specimen is small and is immersed in the test flame and combusted from all sides and to completion. In common with many physical fire models, no indication is given about the rate of burning, so highly fire-retarded materials can be forced to burn at the same rate as materials without any fire retardants. Therefore, additional data input on burning rates at different fire stages is required for fire safety engineering calculations. Colorimetric tubes are not a reliable measurement technique for combustion products due to possible interferences.

6.5.6.3 Repeatability and reproducibility

There are no reported results of an inter-laboratory evaluation. However, repeatability is reported to be reasonably good, since the specimen is relatively small, is completely immersed in the gas flame and is burned to completion.

6.5.7 Toxicological results

6.5.7.1 Advantages

The initial conditions are few and well prescribed.

6.5.7.2 Disadvantages

The gases selected are those considered by the originators of the standard to be a hazard in warship fires and the levels specified are considered to be relevant when the standard was reviewed in 2000. The coefficients for the toxicity index calculation are not current. The basis for the index equation is unclear.

6.5.8 Miscellaneous

No animals are exposed in the test, nor is the apparatus compatible with such an addition.

6.5.9 Validation

There are no reported comparisons of toxic gas generation with data from real-scale fire tests.

6.5.10 Conclusion

While relatively easy to perform, this method is of questionable value for generating smoke toxicity data for use in fire hazard analysis because of its unsatisfactory fire model. Its use as a screening tool has not been verified against real-scale fire test data as it is intended that short-listed materials would be retested with more relevant tests. The small sample size limits the use for evaluation of finished products. The absence of animal-exposure data means that smoke extreme or unusual toxic potency will not be identified.

6.6 Japanese toxicity test

6.6.1 Application

This apparatus described in Reference [10] is designed to obtain toxic potency data for slow burning building and furnishing materials. It is the basis for method 1231 of the Japanese Ministry of Construction.

6.6.2 Principle

This is a two-chamber apparatus (see Figure 6). There is a slow flow of air through the combustion chamber in order to keep the oxygen in the mouse-exposure chamber above 16 %. The samples, 220 mm square and not more than 15 mm thick, are exposed in moderately vitiated air to convective and radiative heating. The exhaust gas is introduced into an animal-exposure chamber in which there are 8 rotary cages, each containing a mouse. The movement of the mice is monitored and reflected to the evaluation of toxicity.

6.6.3 Fire stage(s)

The fire stage from ISO 19706:2007[35], Table 1, is 3.a, small, under-ventilated fire.

6.6.4 Types of data

The standard procedure is to measure the times to incapacitation of the mice. In addition, gas samples can be extracted for external analysis. Following the test, blood samples can be extracted for analysis.

6.6.5 Presentation of results

The reported information is the incapacitation times of the eight mice. Usually, 15 min is the maximum.

6.6.6 Apparatus assessment

6.6.6.1 Advantages

The single combustion test condition is well defined. The method could be adapted to produce data enabling calculation of yields of toxicants.

6.6.6.2 Disadvantages

The test is limited to a single fire stage.

6.6.6.3 Repeatability and reproducibility

In a 4-laboratory examination of the method for six materials[11], the inter-laboratory standard deviation of the times to incapacitation of the mice was under 15 %. The agreement of duplicate tests within each laboratory was within 5 %.

6.6.7 Toxicological results

6.6.7.1 Advantages

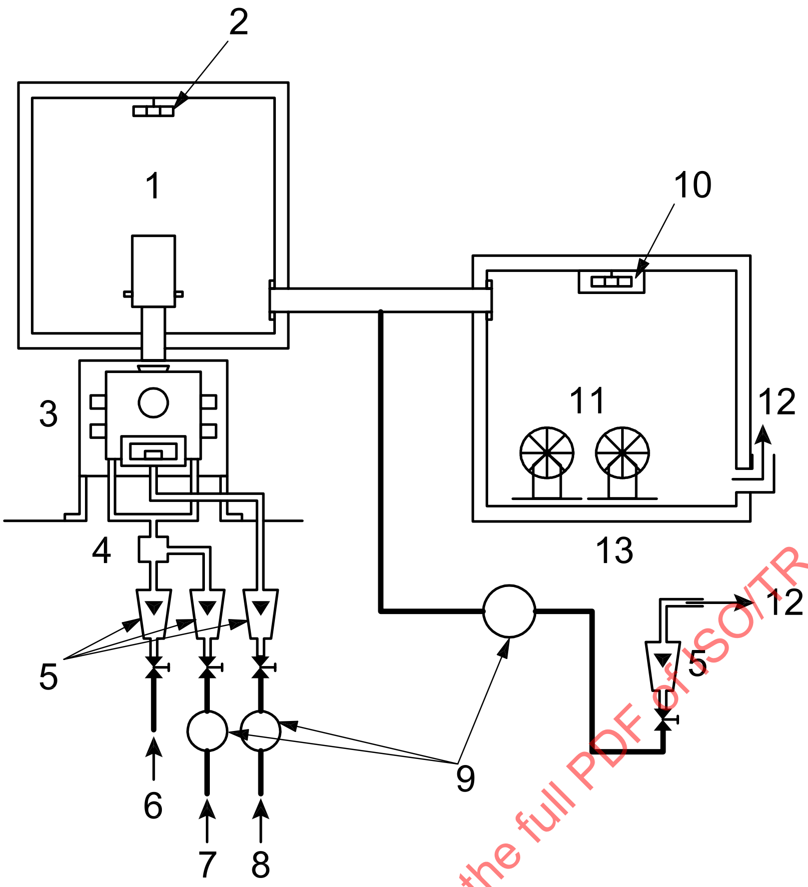
The test provides a direct measure of the incapacitation capability of the smoke. It can identify instances of extreme and unusual smoke toxic potency.

6.6.7.2 Disadvantages

The test requires specialized equipment for animal exposure.

6.6.8 Miscellaneous

This is primarily an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation can be added with little interference with the standard method.



Key

1 mixing box
2 stirrer
3 furnace
4 mixer
5 flowmeter(s)
6 LP gas
7 primary air
8 secondary air
9 pumps
10 heater and stirrer
11 rotary cages
12 exhaust
13 animal-exposure box

Figure 6 — Schematic of the Japanese smoke-toxicity apparatus

6.6.9 Validation

No comparison against real-scale fire tests has been published.

6.6.10 Conclusion

This method is useful for screening the incapacitation potency of smoke from various products, to the extent that the mouse response to the effluent is similar to human response. The combustion conditions simulate only a single fire stage. If the potency varies with the degree of vitiation, it is necessary to perform multiple tests.

6.7 Cone Calorimeter (International)

6.7.1 Application

The physical fire model in this apparatus is the one used in ISO 5660-1[12], ASTM E 1354[13], NFPA 271[14] and NFPA 272[15]. The apparatus is designed to generate measurement of the rate of heat release (RHR) and smoke from samples of materials and finished products. The data are used in fire-hazard analyses. Some workers have used the apparatus to measure other combustion products, especially with FTIR, and to calculate LC_{50} values, but it has not been accepted as a smoke toxicity measurement device.

6.7.2 Principle

A schematic of the apparatus is shown in Figure 7. A sample, up to 100 mm x 100 mm in area and up to 50 mm thick and representative of the end-use configuration of the finished product, is exposed to thermal radiation. The vapours can be ignited by a spark. They are drawn by a downstream fan through a hole in the radiation source into an instrumented duct. The calculation of rate of heat release is from oxygen consumption, smoke by light obscuration. The gases measured, O_2, CO_2, and CO, are those needed for the heat-release-rate calculation.

6.7.3 Fire stage(s)

The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis;

— 2, well-ventilated flaming.

6.7.4 Types of data

The standard procedure includes continuous measurement of mass loss and effluent gas concentrations, gas yields, smoke obscuration and exhaust gas vitiation. The exhaust duct flow is pre-set.

6.7.5 Presentation of results

There are calculation procedures for rate of heat release, effective heat of combustion, smoke generation and gas yields. No method for obtaining toxicity data is specified.

6.7.6 Apparatus assessment

6.7.6.1 Advantages

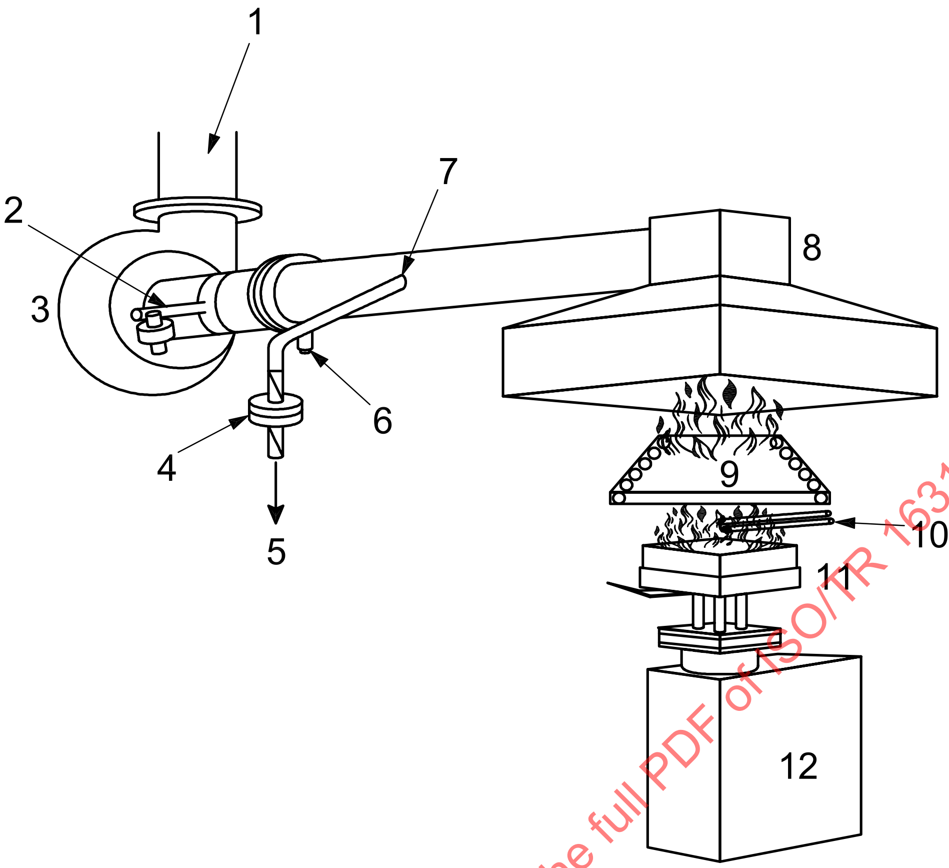
The representation and exposure of finished products is accurate. There are numerous apparatus worldwide. Obtaining toxic-potency data linked to heat-release rate enables linking the former to the fire development curve in the hazard analysis. The mass-burning rate is recorded, enabling direct use of the yield data in engineering calculations.

6.7.6.2 Disadvantages

The flames are highly over-ventilated, so the gas yields are not clearly linked to real-scale results, especially post-flashover fires[1]. The fraction of the air flow passing through the combustion zone is unknown, making values of the global equivalence ratio somewhat uncertain.

6.7.6.3 Repeatability and reproducibility

Multiple inter-laboratory evaluations for rate of heat release have been performed successfully. None have been performed for gas yields or effluent toxicity.



Key

1 temperature- and differential-pressure-measurement location
2 laser extinction beam
3 exhaust blower
4 soot-collection filter
5 exhaust
6 gas-sampling port
7 soot-sampling port
8 exhaust hood
9 cone heater
10 spark igniter
11 sample
12 load cell

Figure 7 — Schematic of the cone calorimeter

6.7.7 Toxicological results

6.7.7.1 Advantages

Since the heat-release rate and smoke density correlate with pre-flashover, flaming fires, it is possible that toxic gas yields also correlate, but this has not been confirmed.

6.7.7.2 Disadvantages

The product gases are highly diluted in the exhaust stream, making quantitative assessment of some toxic gases difficult. The gases pass through the conical heater, which can cause some chemical change. The prescribed set of gases measured might be insufficient to estimate lethal toxic potency.

6.7.8 Miscellaneous

The method does not include test animals and is not amenable to such an adaptation. Thus, smoke of extreme and/or unusually toxicity is not identified.

6.7.9 Validation

The toxic potency and gas yield data do not replicate real-scale post-flashover test data well[1]. The method has not been assessed against real-scale test data for oxidative pyrolysis or well-ventilated flaming.

6.7.10 Conclusion

With no animals, the method is not usable for direct measurement of LC_{50} or IC_{50} values. The flames are highly over-ventilated under normal operating conditions, so yields of organic gases other than CO_2 are likely to be low, perhaps even for replicating well-ventilated combustion. The apparatus can be used to generate yield data for oxidative pyrolysis with additional chemical instrumentation. Tests have been carried out using a reduced entry-air flow and/or oxygen concentration, which can enable vitiated fire conditions and effluents to be simulated. However, no validation experiments have been conducted.

6.8 Flame propagation apparatus (United States)

6.8.1 Application

The physical fire model in this apparatus is the one described in NFPA 287[16] and ASTM E 2058[17]. This method is designed to measure the heat release rate, fixed gases (CO, CO_2) and smoke from samples of materials and finished products for use in fire hazard models. It has been used by some workers to measure other combustion products, especially with FTIR. It has been shown that the apparatus can be used to measure other combustion products using either conventional on-line gas measuring equipment or FTIR spectroscopy. Calculating LC_{50} values lies also within the apparatus capacity, however, it has not been proposed or accepted as a smoke toxicity measurement device.

6.8.2 Principle

A schematic of the apparatus is shown in Figure 8. The test specimens are up to 102 mm x 102 mm in area and up to 25 mm thick (horizontal sample) or 102 mm wide x 300 mm high and up to 13 mm thick (vertical sample) and are representative of the end-use configuration of the finished product. The vapours from the sample, exposed to thermal radiation, can be ignited by a pilot flame. The sample and its holder are mounted within a vertical quartz tube, enabling control of the equivalence ratio. The vapours are collected in an instrumented duct. The calculation of rate of heat release relies primarily on CO_2 generation, but some laboratories have measured HRRs using both oxygen consumption and CO_2 generation techniques. Smoke is determined by optical obscuration. The gases measured, O_2, CO_2, and CO, are those needed for the heat release rate calculation.

6.8.3 Fire stage(s)

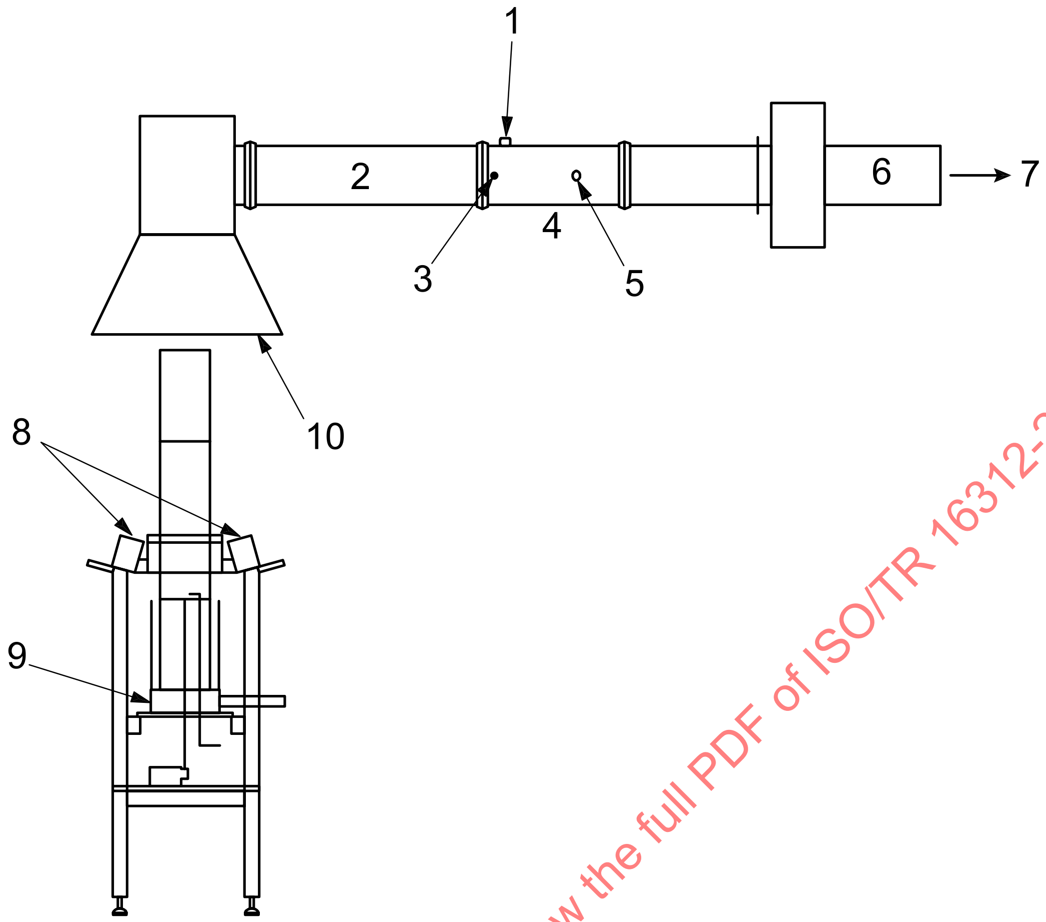
The fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis;

— 1.c, anaerobic pyrolysis;

— 2, well-ventilated flaming;

— 3, under-ventilated flaming.



Key

1 air-velocity port
2 mixing duct
3 thermocouple port
4 test-section duct
5 gas-sampling port
6 blower
7 exhaust
8 radiant heaters
9 test specimen
10 intake funnel

Figure 8 — Schematic of the flame propagation apparatus

6.8.4 Types of data

The standard procedure includes continuous measurement of mass loss and effluent gas concentrations, gas yields, smoke obscuration and exhaust gas vitiation.

6.8.5 Presentation of results

There are calculation procedures for rate of heat release, effective heat of combustion, smoke generation and gas yields. No method for obtaining toxicity data is specified.

6.8.6 Apparatus assessment

6.8.6.1 Advantages

The representation and exposure of finished products is accurate. There is over 25 years of experience with the device. Since the mass-loss rate is measured continuously and the air flow is controlled, it is possible to determine and control the fuel/air equivalence ratio and hence the combustion conditions. Measurement of the mass-loss rate enables direct use of the effluent data in engineering models. The fire effluent is contained within a vertical tube and does not contact heaters, etc. Obtaining toxic potency data linked to the heat-release rate enables linking the former to the fire-development curve in the hazard analysis.

6.8.6.2 Disadvantages

While the yield of CO has been related to real-scale results[18], this comparison has not been made for other components of fire effluent. There are few apparatus worldwide.

6.8.6.3 Repeatability and reproducibility

No formal inter-laboratory evaluation of this method has been performed. However, some inter-laboratory comparison tests have been performed, with satisfactory results[18].

6.8.7 Toxicological results

6.8.7.1 Advantages

The CO yield correlates with real-scale, flaming fires. Since the method enables defined combustion conditions to be reproduced and since CO yield data correlate well with those in full-scale fires under similar combustion conditions, the method can provide a good indication of the yields of major toxic products for comparable full-scale fire conditions. However, this has not been confirmed.

6.8.7.2 Disadvantages

There have been no reported attempts to obtain toxic potency data. Thus, there is no list of prescribed gas measurements.

6.8.8 Miscellaneous

The prescribed method does not include test animals. Thus, smoke of extreme and/or unusually toxicity is not identified.

6.8.9 Validation

CO yields have been related to real-scale results.

6.8.10 Conclusion

With no animals, the prescribed method is not usable for direct measurement of LC_{50} or IC_{50} values. The apparatus can be used to generate yield data for a variety of fire stages with additional instrumentation.

6.9 University of Pittsburgh tube furnace

6.9.1 Application

This apparatus described in Reference [20] is designed to generate toxic-potency data for building materials. The (U.S.) State of New York formerly required listing the output data for materials used in construction. It is not a national or international standard.

6.9.2 Principle

A schematic of the apparatus is shown in Figure 9. This is a flow-through system. The sample, approximately 5 g, is cut into small pieces, put in a tray and heated radiatively, conductively and convectively in a temperature-ramped furnace. Gas from the furnace is pumped past a chamber holding 4 mice. Auto-ignition to flaming occurs episodically. The ventilation rate is not tuned to the combustion rate.

6.9.3 Fire stage(s)

The method integrates several stages that are not sufficiently defined for isolation and evaluation:

— 1.b, oxidative pyrolysis (varying fuel-to-air ratio);

— 2, well-ventilated flaming;

— 3, low-ventilated flaming.

6.9.4 Types of data

The standard procedure includes measurement of mass loss, gas concentrations and yields (with additional instrumentation). However, no specific gases are specified for measurement. The respiratory rate changes and times of death (if any occur) of 4 mice are determined within the exposure period. The animals are observed for a 10 min post-exposure period. Any physiological effects are determined post-mortem.

6.9.5 Presentation of results

Sufficient tests are performed to determine the mass changed or consumed that results in animal mortality and incapacitation. It is technically possible to calculate an average LC_{50} and IC_{50}, but the strongly non-linear burning rate and resulting smoke concentration makes these values questionable.

6.9.6 Apparatus assessment

6.9.6.1 Advantages

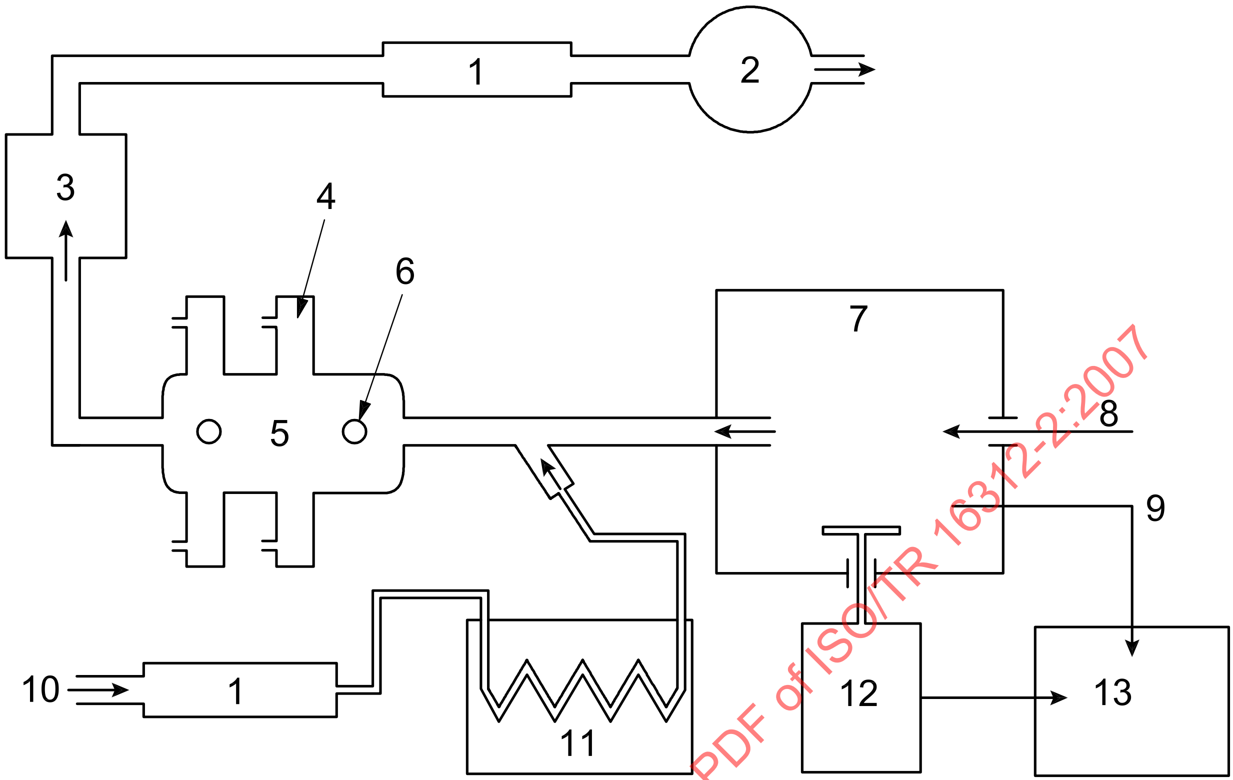
Each test uses a small sample. The apparatus is inexpensive and easy to operate. An extensive database of product performance classes (but not individual product results) has been generated for New York State.

6.9.6.2 Disadvantages

The relation of the combustion conditions in the apparatus to specific stages of fire is indeterminate. The realism of the sample exposure is questionable due to the cutting up of the sample, especially for non-homogeneous products. The simulation of the real-scale thermal exposure is poor. The lack of an igniter leads to unrepeatable flaming. It is not clear that the sample reaching the mice is representative of the combustion effluent due to indeterminate mixing in the furnace. In common with many physical fire models, no indication is given about the rate of burning, so highly fire-retarded materials can be forced to burn at the same rate as materials without any fire retardants. Therefore, additional data input on burning rates at different fire stages is required for fire safety engineering calculations.

6.9.6.3 Repeatability and reproducibility

A small inter-laboratory evaluation of this method was performed but not documented. The reproducibility within the three laboratories is said to be ± 30 %.



Key

1 flowmeter
2 pump
3 filter
4 animal chamber
5 exposure chamber
6 gas-sampling port
7 furnace
8 air
9 thermocouple
10 dilution air
11 ice bath
12 weight sensor
13 recorder

Figure 9 — Schematic of the University of Pittsburgh tube furnace

6.9.7 Toxicological results

6.9.7.1 Advantages

The method produces true measures of smoke lethality and incapacitation and identifies instances of extreme and unusual smoke toxic potency if the effect occurs within exposure. The data can be used to rank substances based on sample weights.

NOTE Mice, whose respiration rate is higher than that of rats, respond at lower concentrations, giving conservative results.

6.9.7.2 Disadvantages

The animal-exposure integrates over a rapidly changing fire effluent mixture. Post-exposure lethality (an issue with pulmonary irritants) cannot be assessed without a modification of the test procedure. The 30 min test begins at a temperature where 0,1 % of the sample weight is lost; thus, toxicity is evaluated under different thermal environments for each material tested. Temperature excursions in the exposure chamber can compromise interpretation of animal response.

6.9.8 Miscellaneous

This is primarily an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation can be added.

6.9.9 Validation

Efforts to validate the test results against real-scale fire data were unsuccessful due to the lack of a smoke accumulator for the duration of the test and the inability to relate the time-dependent test results to the real-scale burning[21].

6.9.10 Conclusion

This method is of questionable value for screening the toxic potency of materials and products. It does not replicate any single fire phase and thus does not produce usable input data for fire hazard models.

6.10 Tube furnace (Germany)

6.10.1 Application

This apparatus, Reference [22] and DIN 53436[23], is designed to determine the critical temperature at which the highest concentration of toxic fire effluents is produced. It is used to generate toxic potency data for liquid/solid test articles, building and furnishing materials and end products.

6.10.2 Principle

A schematic of the apparatus is shown in Figure 10. This is a flow-through system. The sample, approximately 10 ml, is cut from the end product and heated radiatively, conductively and convectively in a tube furnace. The furnace moves continuously countercurrent to the air-flow direction, maintaining a constant combustion rate over the duration of the 30 min test. Auto-ignition to flaming occurs episodically. The combustion effluent is diluted with air prior to exposing test animals.

6.10.3 Fire stage(s)

The method does not specify any particular fire stage(s). The fire conditions in any particular test depend on the specimen behaviour.

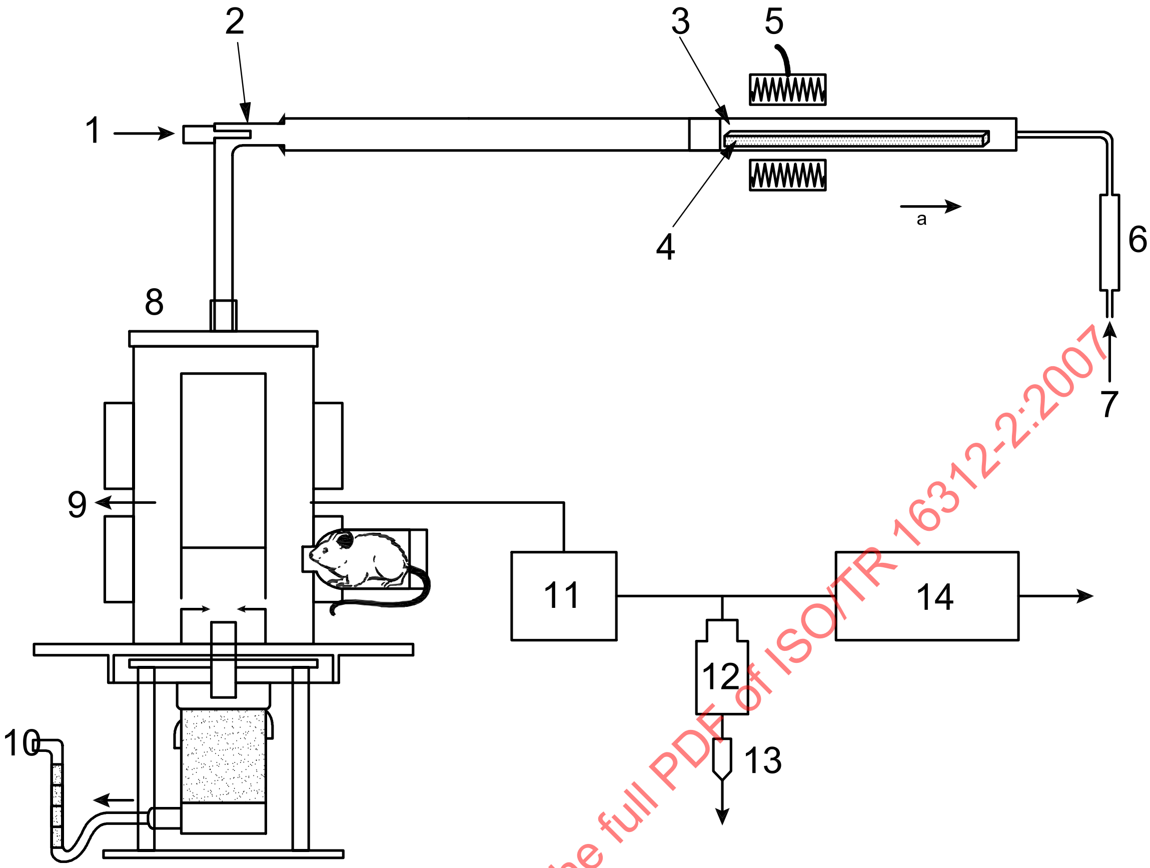
Appropriate fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis (low- and well-ventilated), if the sample does not auto-ignite;

— 3, under-ventilated flaming.

6.10.4 Types of data

The standard procedure includes measurement of mass loss, gas and particle concentrations, including yields, and exhaust gas vitiation. Gas analyses of CO, CO_2, NO_x, and total hydrocarbons have been reported [24], [25]. In addition, the procedure includes determination of lethal and sub-lethal effects of the effluent on 10 rats, enabling differentiation of different modes of toxic actions (asphyxia, irritation, unexpected toxicity) and effects immediate or delayed in onset. Blood samples are taken immediately after exposure for subsequent analysis.



Key

1 secondary air stream
2 dilution unit
3 quartz tube
4 test specimen
5 thermocouple
6 flowmeter
7 inlet air
8 exposure chamber
9 additional gas-sampling port
10 exhaust
11 filter unit
12 cooler
13 condensate
14 CO, CO_2 and O_2 monitors

a Movement of furnace.

Figure 10 — Schematic of the DIN 53436 tube furnace

6.10.5 Presentation of results

Sufficient tests are performed to determine LC_{50} values and confidence limits for within exposure and within-plus-post-exposure periods, including threshold levels for critical sublethal effects. Also included are the yields of gaseous and particulate effluent components, identification of the critical mode of toxicological action and identification of specimens exhibiting unexpected toxicity.

6.10.6 Apparatus assessment

6.10.6.1 Advantages

The test conditions are well defined. The flow-through system provides for a constant atmosphere composition with a low residence time. The basic apparatus is versatile, providing control over both fuel and air ratios and temperatures. It is theoretically possible to cover a range of fire stages under defined equivalence ratios by modifying the operating protocol. The effluent is generated in a steady state so that multiple analytical procedures can be used sequentially rather than concurrently. There is direct access to the test animals for specific determination during the course of exposure or immediately thereafter. The apparatus can be connected to commonly used animal-exposure systems.

6.10.6.2 Disadvantages

The tube is of small diameter, limiting the sample size. Thus the relation between the sample exposure in the test and that in real-scale is questionable, especially for non-homogeneous products. For condensable effluent components, there can be significant condensation on surfaces, resulting in lower measured yields. The lack of an igniter can lead to unrepeatable flaming. The test conditions are defined in terms of sample mass, furnace temperature and air flow. The combustion conditions depend upon the behaviour of each individual specimen and can change during the course of a test run (i.e., intermittent flaming/non-flaming). It is, therefore, difficult to compare the test conditions to those in full-scale fires. In common with many physical fire models, no indication is given about the rate of burning, so highly fire-retarded materials can be forced to burn at the same rate as materials without any fire retardants. Therefore, additional data input on burning rates at different fire stages is required for fire safety engineering calculations.

6.10.6.3 Repeatability and reproducibility

Three-laboratory evaluation of this method using reference materials has been performed [24], [25] and has shown sufficient repeatability and reproducibility.

6.10.7 Toxicological results

6.10.7.1 Advantages

The method produces both qualitative (mode of action) and quantitative measure of smoke lethality by utilizing a series of independent endpoints (lethality, clinical observations, organ damage, blood analysis, functional changes), including the onset, duration, recovery and intensity of effects. This test can identify instances of extreme and unusual smoke toxic potency and identify the acute health risks of highest concern. It can also identify cases where unusual toxicity occurs as a result of constituents not identified by the analytical procedures applied or through post-combustion physical interactions of airborne constituents. The method can be adapted to measure incapacitation (hind-leg flexion or immobilization) and respiratory tract irritation.

6.10.7.2 Disadvantages

Flaming is quenched on the upper surface of the tube, resulting in distorted concentrations of some combustion products reaching the test animals. The method requires expensive and specialized equipment and it is necessary to be licensed for animal experimentation.

6.10.8 Miscellaneous

This is primarily an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation can be added with little interference with the standard method. The apparatus can be used without test animals, but it then loses the ability to identify the principal cases of real interest.

6.10.9 Validation

No comparison of the toxic potency and gas yield data against real-scale test data have been published.

6.10.10 Conclusion

This method is useful for obtaining toxicological data and gas yields from pyrolysis of homogeneous materials. The small sample size limits the use for evaluation of finished products. It can be used to determine whether the chemical measurements are sufficient to explain the observed toxicology.

6.11 Tube furnace (France)

6.11.1 Application

This apparatus described in NFX 70-100 [26], [27] (see Figure 11) is designed to generate concentrations of gases in fire effluents produced by combustion in a tubular furnace. The data collected are used for the evaluation of a toxicity index.

6.11.2 Principle

This is a flow-through system designed for use in choosing materials, not finished products. The 1 g sample is thermally degraded in a tube furnace at 350 °C, 400 °C, 600 °C and/or 800 °C. Auto-ignition to flaming occurs episodically.

6.11.3 Fire stage(s)

The method does not specify any particular fire stage or stages.

Appropriate fire stage(s) from ISO 19706:2007[35], Table 1, are as follows:

— 1.b, oxidative pyrolysis (low- and well-ventilated), if sample does not auto-ignite;

— 2, well-ventilated flaming;

— 3, under-ventilated flaming.

The different stages are reproduced by the choice of furnace temperature and the resulting behaviour of the test specimen. A stage is difficult to determine because the local amount of oxygen available for combustion (and so the fire stage) depends on the combustion rate of the sample.

6.11.4 Types of data

The standard procedure includes measurement of total mass lost, concentrations and yields of CO, CO_2, HCl, HBr, HF, HCN, SO_2, NO_x (NO and NO_2), formaldehyde and acrolein using ILC, HPLC and classical analytical methods. FTIR can also be used.

6.11.5 Presentation of results

The data are presented as gas yields.

6.11.6 Apparatus assessment

6.11.6.1 Advantages

The apparatus is easy to use. The operating conditions (temperature, air flow, mass of sample) can be easily modified.

6.11.6.2 Disadvantages

The small specimen size limits the apparatus to testing of homogenous materials. The thermal exposure is unrealistic for non-homogenous finished products. The combustion conditions can vary during a test and, thus, cannot be readily identified with any particular fire stage. Samples of low-density materials have a low sample mass, which can limit gas detection. It is necessary to make several runs to measure the full range of toxic products. The lack of an igniter can lead to unrepeatable flaming. Flaming is quenched on the upper surface of the tube, resulting in distorted concentrations of some combustion products. In common with many physical fire models, no indication is given about the rate of burning, so highly fire-retarded materials can be forced to burn at the same rate as materials without any fire retardants. Therefore, additional data input on burning rates at different fire stages is required for fire safety engineering calculations.

