TECHNICAL REPORT

ISO/TR 16312-2

Second edition
2021-01

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
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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

This second edition cancels and replaces the first edition (ISO/TR 16312-2:2007) which has been technically revised.

The main changes compared to the previous edition are as follows:

— fire models have been updated following the publication of certain other standards, including ISO/TS 19021 and ISO/TS 5660-5;

— deprecated methods have been moved to Annex A.

A list of all parts in the ISO 16312 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 on a small scale. The accuracy of the physical fire model, then, depends on two features:

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

b) the degree to which the yields of the important combustion products obtained from the 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. Since the publication of the first edition of this document, in which a methodology for effecting this comparison was cited in Reference [1], ISO 29903-1 has been developed.

This document 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 document covers 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 10 physical fire models discussed in this document, plus 4 depreciated methods in Annex A. Additional apparatus can be added as they are developed or adapted with the intent of generating information regarding the toxic potency of smoke.

For all models in this document, several 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.

Reference documents useful for discussions of analytical methods, bioassay procedures, and prediction of the toxic effects of fire effluents are listed in the Bibliography at the end of this document.

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 document 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. This is achieved by 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 ISO 19703 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
vitiation-controlled
type of conditions under which the volume concentration of oxygen is intentionally controlled or reduced in the combustion environment

Note 1 to entry: Vitiation controlled conditions represent an oxygen depleted fire environment.

[SOURCE: ISO/TS 5660-5:2020, 3.3, modified.]

3.2
ventilation-controlled
type of conditions un which the supply rate of (ambient or vitiated) air to the combustion environment is intentionally controlled or limited

Note 1 to entry: Ventilation-controlled conditions represent a fire environment with limited fresh air supply.

[SOURCE: ISO/TS 5660-5:2020, 3.4, modified.]

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.

ISO 12828-1, ISO 12828-2 and ISO/TS 12828-3 are guidance documents for model validity. This includes limits of detection and quantification, range of application, trueness and fidelity in terms of repeatability and reproducibility.

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.

The conditions of pyrolysis and combustion may differ locally and globally in a physical fire model, leading to difficulties in scale with real-fire conditions in reduced experiments.

The experimental conditions may be vitiation-controlled and/or ventilation-controlled, or may be unknown and vary during the test.

It is essential that the physical fire model enable accurate determinations of chemical effluent composition. Validation of the method according to ISO 12828-2 is a suitable way to validate the chemical analysis.

4.5 Effluent characterization

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, a base set of combustion products has been identified for routine analysis. Novel materials may evolve previously unidentified toxic products. Thus, a more detailed chemical analysis may 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 contains a fuller discussion of the utility of integrating assays. It is desirable that the physical fire model accommodate a bioassay method. However, due to bioethics practices, such use and comparisons are limited. The use of laboratory animals as test subjects or living tissues are means of insuring inclusion of the impact of all combustion gases. However, it is recognized that the adoption and use of such protocols may be prohibited in some jurisdictions and tend to disappear. An animal-free protocol can capture the effects of known combustion gases, but can miss the impact of any unexpected or uncommon and highly toxic species, the smoke components of which are most in need of identification.

5 Significance and use

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.

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.

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.

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

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

The methods that do not include animal exposure and are not amenable to such an adaptation might not allow identification of extreme and/or unusual toxicity.

Note that four depreciated methods are detailed in Annex A.

6 Physical fire models

6.1 Smoke chambers - Closed cabinet toxicity tests (international)

6.1.1 NBS smoke chamber

6.1.1.1 Application

This physical fire model is described in ASTM E662, with a vertically-orientated sample and heat flux limited to 25 kW/m^2. It was first designed to generate smoke optical density data. The physical fire model has also been implemented by the European Union in EN 2824, EN 2825, and EN 2826 for determination of smoke density and gas components in smoke. It is also used in ABD-0031 (Airbus) and BSS 7239 (Boeing) for smoke in passenger aircraft.

6.1.1.2 Principle

A vertically mounted specimen, 76 mm square and up to 25 mm thick, is exposed to a radiant heater for a minimum of 10 min. Tests are conducted at 25 kW/m^2 with and without pilot flame. The gases are sampled through probes positioned at various positions in the smoke box depending on the standard applied.

6.1.1.3 Fire stage(s)

The fire stage(s) according to ISO 19706 are not clearly defined and may change during the test.

6.1.1.4 Types of data

The standard procedure includes measurement of smoke obscuration and specific effluent gas concentrations (CO_2, CO, HCN, HCl, HF, HBr, NO_x, SO_2) with a large number of analytical techniques. Depending on the standard applied, gas data can be provided continuously during test or 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.

6.1.1.5 Presentation of results

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

6.1.1.6 Apparatus assessment

6.1.1.6.1 Advantages

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

6.1.1.6.2 Disadvantages

The combustion conditions are not well characterized as they are linked to oxygen consumption inside the chamber. At the beginning of the test, they are well-ventilated if it is flaming but their evolution depends on sample behaviour. Vitiation can occur and affects the yields of combustion products.

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.

6.1.1.6.3 Repeatability and reproducibility

No data reported.

6.1.1.7 Toxicological results

6.1.1.7.1 Advantages

The initial conditions are few and well prescribed.

6.1.1.7.2 Disadvantages

Possible vitiation could lead to time dependent generation of toxicants, which are only sampled at a specified time in some applications of the standard.

Condensation could occur on the wall of the chamber leading to removal of some gases from the sampled environment. The prescribed set of gases to be measured may be insufficient for estimating lethal toxic potency.

6.1.1.8 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.

6.1.1.9 Validation

No data reported.

6.1.1.10 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 extreme or unusual toxic potency of smoke is not identified.

6.1.2 ISO smoke chamber

6.1.2.1 Application

This physical fire model is described in ISO 5659-2 and ASTM E 1995, with a horizontally-orientated sample and heat fluxes up to 50 kW/m^2. It was first 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. The physical fire model has also been implemented by the European Union in EN 45545-2 for determination of burning behaviour, smoke density and gas components in smoke on materials used for the railway sector.

ISO/TS 19021 has been developed to propose suitable specifications to operate this fire model with a FTIR gas analyser according to ISO 19702 (see Reference [33]).

6.1.2.2 Principle

A schematic of this closed cabinet test is shown in Figure 1. A horizontally mounted specimen, 75 mm^2 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 and at 50 kW/m^2, with and without pilot flame. The gases are sampled through probes positioned at various positions of the smoke box depending on the standard applied. ISO/TS 19021 proposes a small multi-hole probe close to the ceiling of the chamber.

6.1.2.3 Fire stage(s)

The fire stage(s) according to ISO 19706 are not clearly defined and may change during the test.

6.1.2.4 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). ISO/TS 19021 uses measurement of concentrations with FTIR following ISO 19702. Depending on the standard applied, gas data could be provided continuously during the test or at the time when the maximum smoke concentration is reached.

6.1.2.5 Presentation of results

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

6.1.2.6 Apparatus assessment

6.1.2.6.1 Advantages

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

6.1.2.6.2 Disadvantages

The combustion conditions are not well characterized as they are linked to oxygen consumption inside the chamber. At the beginning of the test, they are well-ventilated if it is flaming but their evolution depends on sample behaviour. Vitiation can occur and affects the yields of combustion products.

The gases are mixed by natural convection and possible stratification can lead to non-representative sampling of the combustion gases.

6.1.2.6.3 Repeatability and reproducibility

Inter-laboratory evaluations have been performed for the smoke density test and gave satisfactory results for a range of materials. Inter-laboratory evaluation of toxic gas production has been reported in ISO/TS 19021.

6.1.2.7 Toxicological results

6.1.2.7.1 Advantages

The initial conditions are few and well prescribed.

6.1.2.7.2 Disadvantages

Possible vitiation could lead to time dependent generation of toxicants, which are only sampled at a specified time in some applications of the standard. ISO/TS 19021 proposes a continuous monitoring, but this sampling has to be reduced in order to avoid modifying the behaviour of the material tested.

Condensation could occur on the wall of the chamber leading to removal of some gases from the sampled environment. The prescribed set of gases to be measured may be insufficient for estimating lethal toxic potency.

6.1.2.8 Miscellaneous

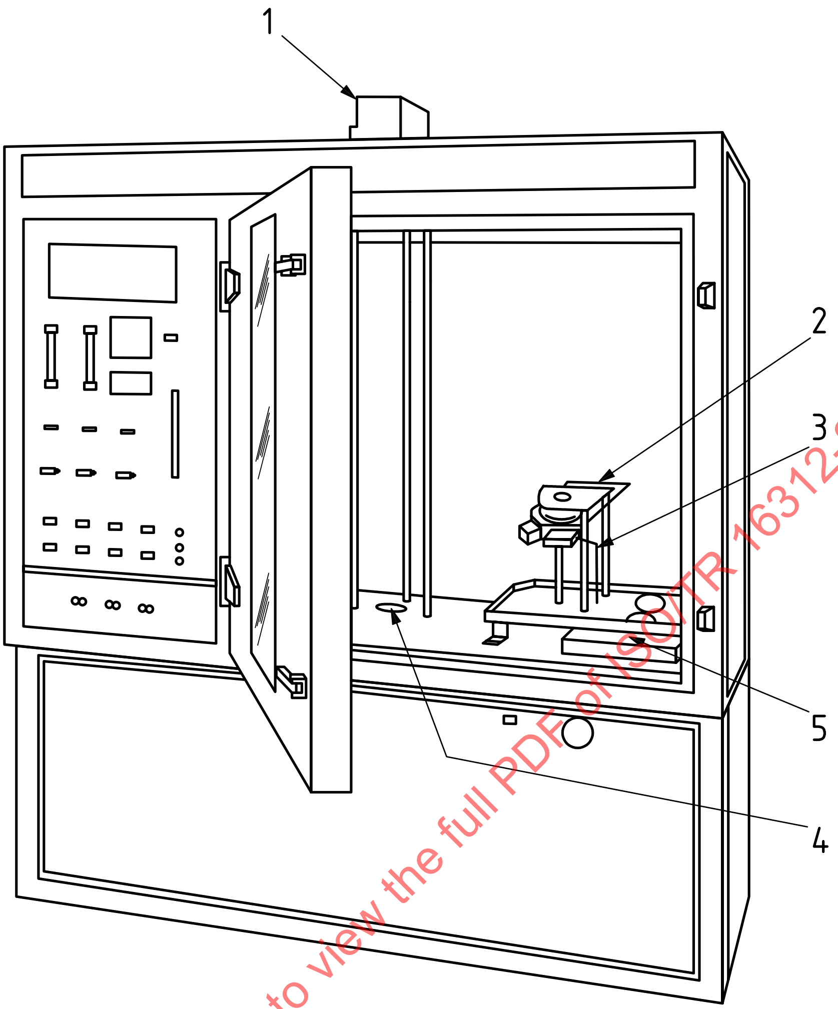
No animals are exposed in the test, nor is the apparatus compatible with such an addition. The use of the chemical data is typically limited to a comparison with critical concentrations of listed toxic gases.

6.1.2.9 Validation

There are several reported comparisons of toxic gas generation with data from real-scale fire tests in ISO/TS 19021. They highlight the difficulty in relating this fire scenario to any real-scale application.

6.1.2.10 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 extreme or unusual toxic potency of smoke is not identified.



Key

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

Figure 1 — Schematic of the closed cabinet toxicity test apparatus

6.2 NES 713 (United Kingdom)

6.2.1 Application

This apparatus was designed to provide values of a toxicity index for use in short-listing materials and end products for warship marine use[8]. It is also known as UK Ministry of Defence Standard DEFSTAN 02-713.

6.2.2 Principle

A photograph of this closed cabinet test is shown in Figure 2. A specimen of size chosen to provide optimal analytical precision (typically a few grams) 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.

6.2.3 Fire stage(s)

The fire stage from ISO 19706 is as follows:

— 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.

6.2.4 Types of data

The standard procedure includes measurement of CO, CO_2, formaldehyde, NO_x, HCN, acrylonitrile, phosgene, SO_2, H_2S, HCI, 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.

6.2.5 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.

6.2.6 Apparatus assessment

6.2.6.1 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.

6.2.6.2 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 are needed for fire safety engineering calculations. Colorimetric tubes are not a reliable measurement technique for combustion products due to possible interferences.

6.2.6.3 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.

6.2.7 Toxicological results

6.2.7.1 Advantages

The initial conditions are few and well prescribed.

6.2.7.2 Disadvantages

The gases selected are those considered by the originators of the standard to be a hazard in warship fires. The levels specified were 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.

6.2.8 Miscellaneous

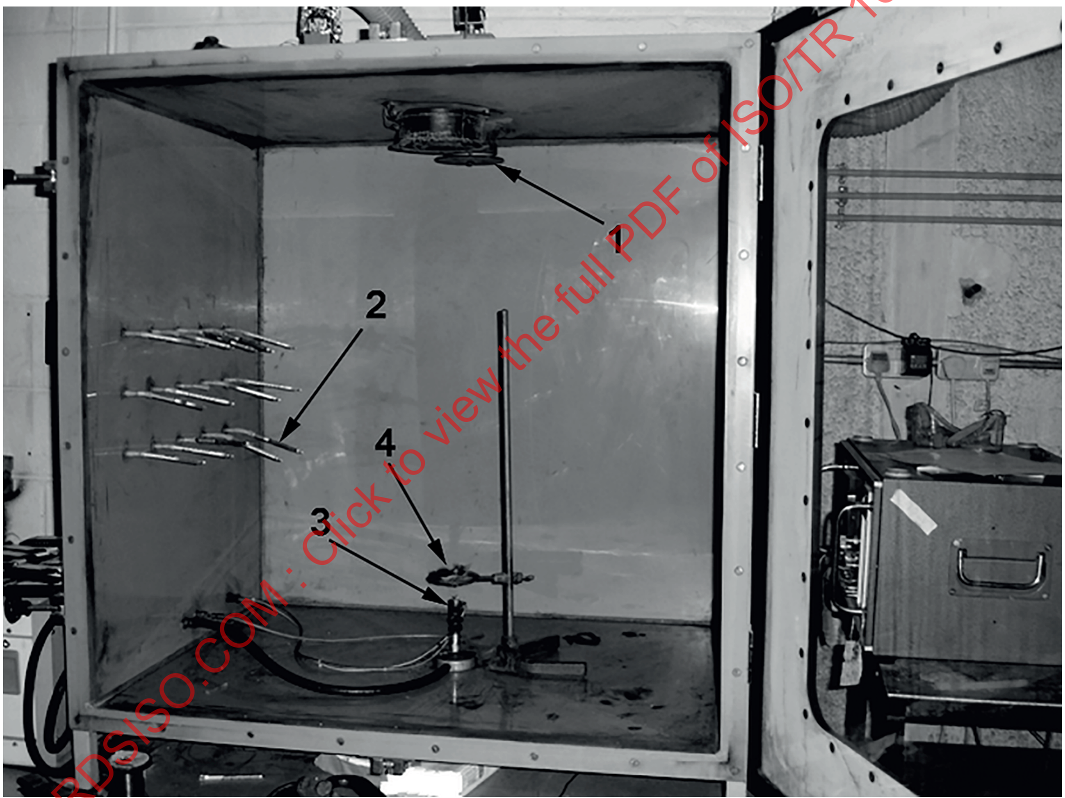
No animals are exposed in the test, nor is the apparatus compatible with such an addition.

6.2.9 Validation

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

6.2.10 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 and its weak analytical method. 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 extreme or unusual toxic potency of smoke will not be identified.



Key

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

Figure 2 — Photograph of the NES 713 apparatus

6.3 Rotative cages smoke toxicity tests

6.3.1 Japanese and Korean methods

6.3.1.1 Application

This model was designed to obtain toxic potency data for building and furnishing materials[9],[10]. It is the basis for the method prescribed in the fireproof performance test and evaluation procedure which is used by performance evaluation bodies designated by the Ministry of Land, Infrastructure and Transport of Japan, and also used in the Republic of Korea as KSF2271.

6.3.1.2 Principle

This is a two-chamber apparatus (see Figure 3). The sample is placed in a gas furnace and effluents are released to the first chamber. 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.

The criteria are based on time to incapacitation, normally expressed as relative to that obtained with a “Red Lauan” wood reference, giving a time to incapacitation of 6,8 min in Japan. Korea uses a criterion of 9 min.

6.3.1.3 Fire stage(s)

The fire stage from ISO 19706 is as follows:

— 3.a, small, localized fire, generally in a poorly ventilated compartment.

6.3.1.4 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.

6.3.1.5 Presentation of results

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

6.3.1.6 Apparatus assessment

6.3.1.6.1 Advantages

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

6.3.1.6.2 Disadvantages

The test is limited to a single fire stage.

6.3.1.6.3 Repeatability and reproducibility

In a 4-laboratory examination of the method for six materials[9], the interlaboratory standard deviation of the times to incapacitation of the mice was under 15 %. The agreement of duplicate tests within each laboratory was within 5 %.

6.3.1.7 Toxicological results

6.3.1.7.1 Advantages

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

6.3.1.7.2 Disadvantages

The test requires specialized equipment for animal exposure.

6.3.1.8 Miscellaneous

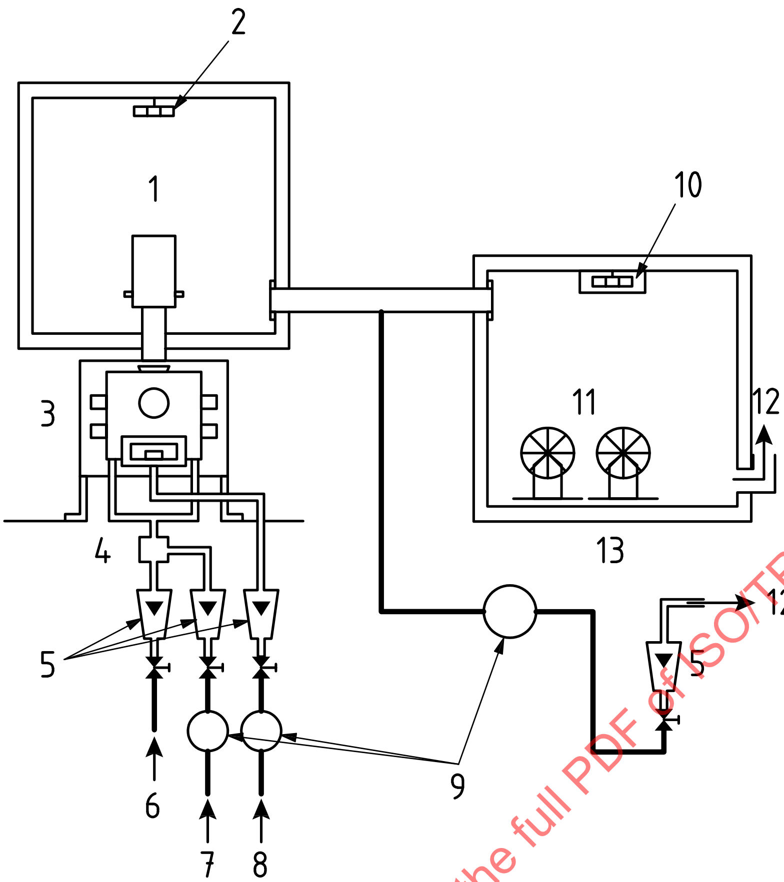
This is primarily an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation can be added with little interference in the standard method.

6.3.1.9 Validation

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

6.3.1.10 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, multiple tests would need to be performed.



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 3 — Schematic of the rotative cages smoke-toxicity apparatus

6.3.2 Chinese method

6.3.2.1 Application

The device (GB 20285) is used to test the risk of smoke toxicity of combustible materials and has been applied to GB 8624 (Classification for burning of building materials and products) as additional classification criteria for the combustion performance of materials.

6.3.2.2 Principle

The schematic of the apparatus is shown in Figure 4. The homogeneous strip material is heated by a tubular furnace with constant velocity primary air and stable heating. The stable pyrolysis and combustion of the material can be realized, and the smoke with stable composition concentration can be obtained.

For different materials, animal tests are carried out under sufficient pyrolysis and flameless conditions. The smoke concentration that indicates the end of the animal study is used as the basis for smoke toxicity classification of materials.

6.3.2.3 Fire stage(s)

According to ISO 19706 the fire stage(s) is as follows:

— 1.b, oxidative pyrolysis from externally applied radiation.

6.3.2.4 Types of data

The smoke concentration, smoke yield, and smoke toxicity degree of the tested materials is obtained.

6.3.2.5 Presentation of results

Smoke toxicity of materials.

6.3.2.6 Apparatus assessment

6.3.2.6.1 Advantages

Steady fire effluents can be quantitatively produced, and the concentration and animal exposure results can be obtained, which is conducive to the study of tolerance toxicity mechanism. The smoke toxicity of different building materials can be classified into safety, quasi-safety and dangerous levels according to the smoke concentration, which is helpful to the promotion of low-smoke and low-toxicity building materials.

6.3.2.6.2 Disadvantages

It represents only one fire stage. It is adapted to simple materials.

6.3.2.7 Repeatability and reproducibility

This method has good repeatability and reproducibility. It is adopted in GB 8624 (Classification for burning behaviour of building materials and products) and GB 20286 (Requirements and mark on burning behaviour of fire retarded products and subassemblies in public place).

6.3.2.8 Toxicological results

6.3.2.8.1 Advantages

The symptoms of the animals after exposure can be observed directly, and the general anatomy and histopathological examination can be carried out to accurately describe the toxic effect and degree of the smoke.

6.3.2.8.2 Disadvantages

It is difficult to control the respiratory rate and degree of the exposure animal when they are awake, therefore, the inhaled dose or concentration is difficult to obtain.

6.3.2.9 Miscellaneous

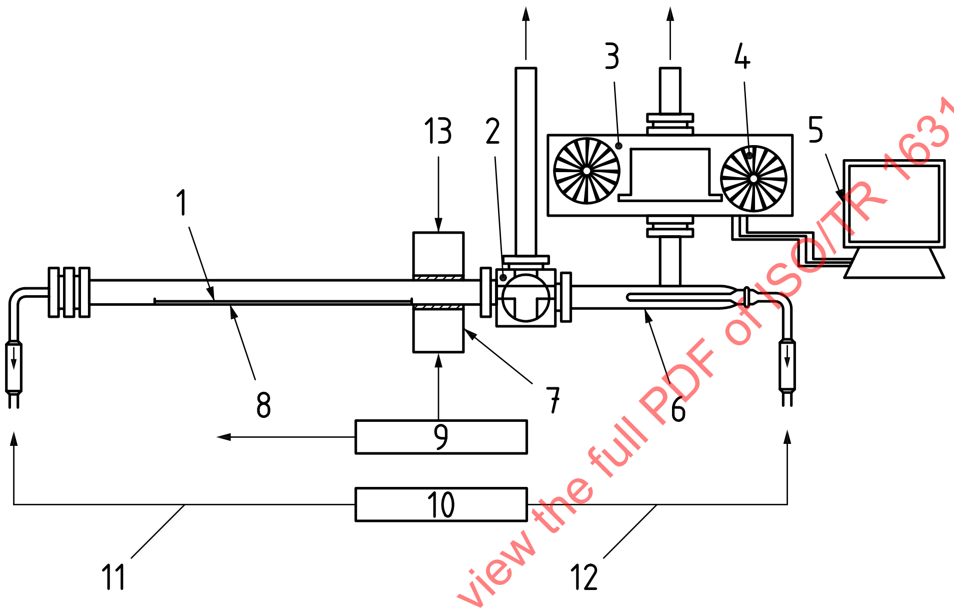
In this process, the smoke composition and concentration of the material can be used for smoke toxicity evaluation and can be compared with the experimental results presented by this method.

6.3.2.10 Validation

Sichuan fire laboratory stated that the same toxicity of fire smoke of the same material was obtained using ISO/TS 19700 and this method.

6.3.2.11 Conclusion

This method can classify the smoke toxicity of building materials according to the concentration, which is conducive to the promotion of low-smoke and low-toxicity materials. Steady-state smoke, smoke concentration and animal exposure effect could be obtained, which is conducive to toxicological research. This method can only represent one fire stage.



Key

1	test specimen boat	8	quartz tube
2	three-way cock	9	furnace movement system
3	exposure box	10	air supply system
4	rotor cage	11	primary air
5	computer	12	secondary air
6	air mixing tube	13	temperature control system
7	tubular furnace		

Figure 4 — Schematic of the apparatus

6.4 Cone calorimeter (international)

6.4.1 Application

The physical fire model in this apparatus is the one used in ISO 5660-1, ASTM E 1354[11], NFPA 271[12] and NFPA 272[13]. The apparatus is designed to generate measurement of the rate of heat release (RHR) and smoke from samples of materials and finished products. An alternative fire model to allow vitiated conditions has been recently developed as ISO/TS 5660-5. Coupling with ISO 19702 FTIR for gas analysis is under development in ISO/TC 92/SC 1.

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.

6.4.2 Principle

A schematic of the apparatus is shown in Figure 5. 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 in the range 0-100 kW/m^2. In addition, ISO/TS 5660-5 allows the exploration of oxygen conditions from 21 % down to 15 %. 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. Additional coupling with FTIR allows measurement of CO_2, CO, HCN, HCl, HF, HBr, NO_x, SO_2.

6.4.3 Fire stage(s)

The fire stage(s) from ISO 19706, are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 2, well-ventilated flaming.

When a ISO/TS 5660-5 test chamber is used, additional conditions can also be explored, such as:

— 1.c, anaerobic pyrolysis from externally applied radiation;

— 3.a, small, localized fire, generally in a poorly ventilated compartment;

— 3.b, post-flashover fire.

6.4.4 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.

6.4.5 Presentation of results

There are calculation procedures for rate of heat release, effective heat of combustion, smoke generation and gas yields. ISO/TS 21397:—[1)] focusses on calculation procedures to obtain gas emission vectors and gas yields.

6.4.6 Apparatus assessment

6.4.6.1 Advantages

The representation and exposure of finished products is accurate. There are numerous apparatus worldwide for "classical cone" and few for vitiated cone. Obtaining toxic-potency data linked to the heat-release rate enables linking of 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.

6.4.6.2 Disadvantages

Under ISO 5660-1 conditions, the flames are highly over-ventilated, so the gas yields are not clearly linked to real-scale results, especially for post-flashover fires[1].

Under ISO/TS 5660-5 conditions, the conditions between pyrolysis and combustion can be different, and post-combustion can occur, modifying the results. This is reduced by the presence of a chimney.

The fraction of the air flow passing through the combustion zone is unknown, making values of the global equivalence ratio somewhat uncertain.

1) To be published.

The effluents are flowed across to the radiant heater, leading to possible modifications.

6.4.6.3 Repeatability and reproducibility

Multiple inter-laboratory evaluations for rate of heat release have been performed successfully.

For gas yield, ISO 19702 presents in appendix several results from SAFIR research programme where such data has been produced. ISO/TS 12828-3 also summarizes some values.

6.4.7 Toxicological results

6.4.7.1 Advantages

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

6.4.7.2 Disadvantages

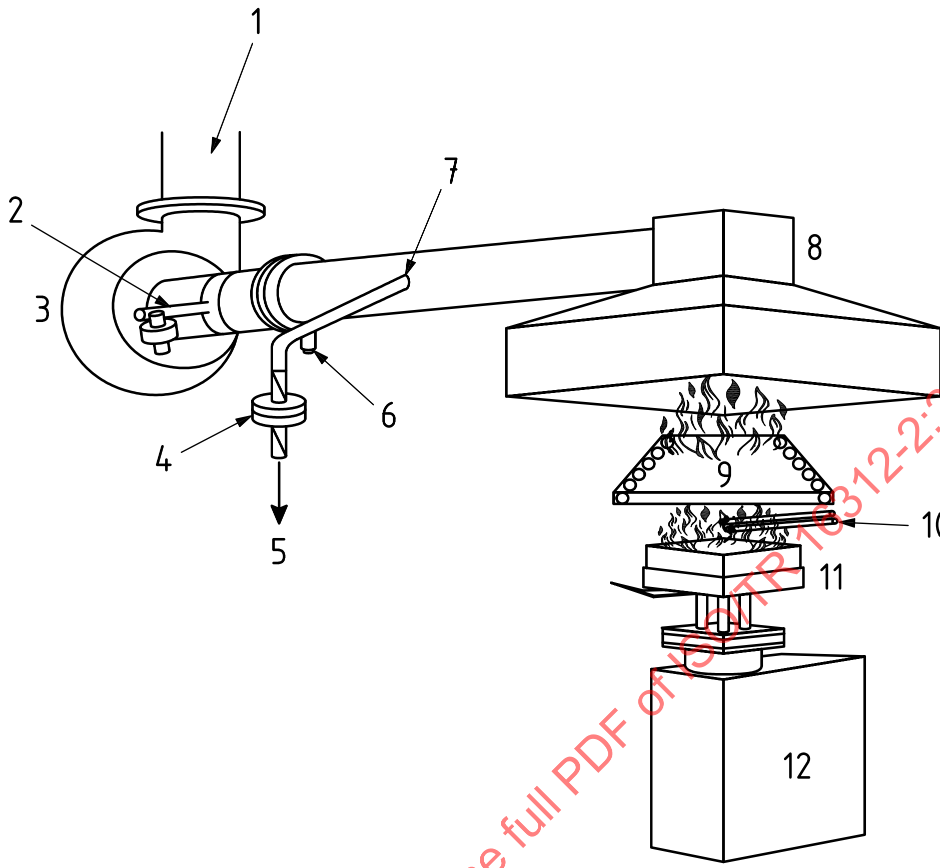
The product gases are highly diluted in the exhaust stream, making quantitative assessment of some toxic gases difficult and highlighting the importance of LoQ determination according to ISO 12828-1. The gases pass through the conical heater, which can cause some chemical change. The prescribed set of gases measured might be insufficient to estimate lethal toxic potency.

6.4.8 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.

6.4.9 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.



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 5 — Schematic of the cone calorimeter

6.5 Flame propagation apparatus (International)

6.5.1 Application

The physical fire model in this apparatus is the one described in ISO 12136, and also in NFPA 287 and ASTM E 2058. 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.

6.5.2 Principle

A schematic of the apparatus is shown in Figure 6. 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.

6.5.3 Fire stage(s)

The fire stage(s) from ISO 19706, are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 1.c, anaerobic pyrolysis from externally applied radiation;

— 2, well-ventilated flaming;

— 3.a, small, localized fire, generally in a poorly ventilated compartment;

— 3.b, post-flashover fire.

6.5.4 Types of data

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

6.5.5 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.

6.5.6 Apparatus assessment

6.5.6.1 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.

6.5.6.2 Disadvantages

While the yield of CO has been related to real-scale results[16], this comparison has not been made for other components of fire effluent. There are few apparatus worldwide. In vitiated conditions, problems of post-combustion can occur.

6.5.6.3 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[16].

6.5.7 Toxicological results

6.5.7.1 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.

6.5.7.2 Disadvantages

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

6.5.8 Miscellaneous

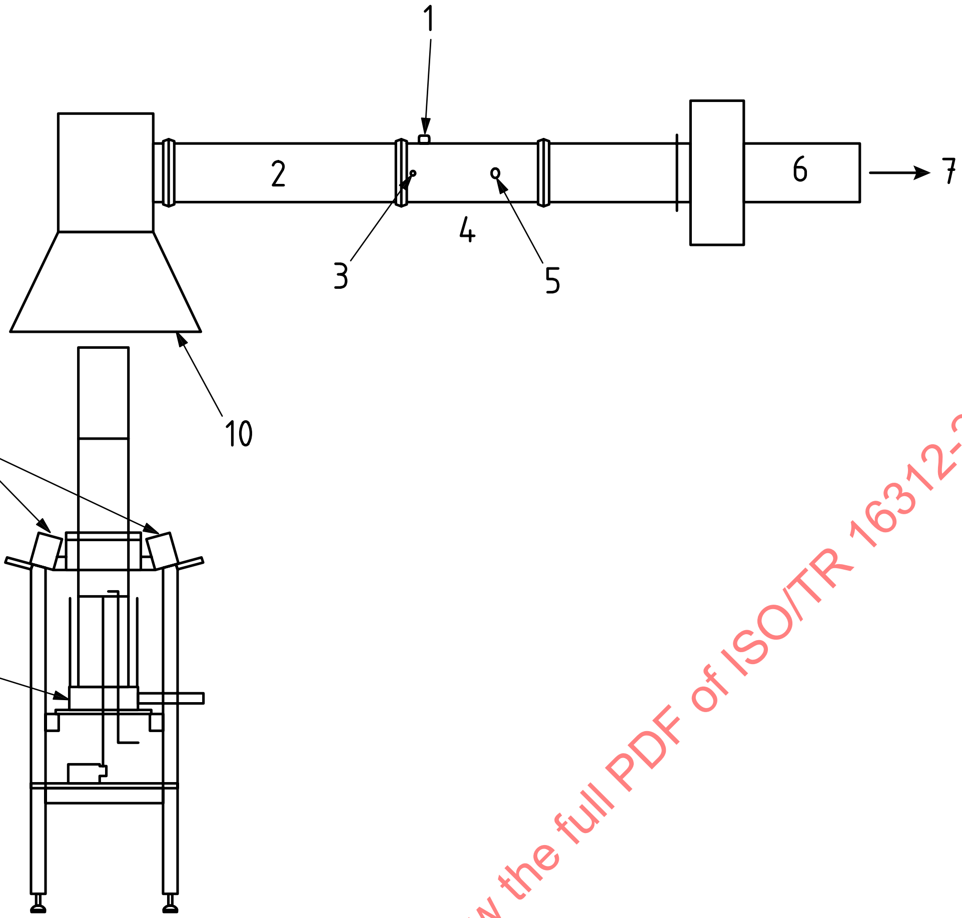
The prescribed method does not include test animals. Thus, smoke of extreme and/or unusually toxicity is not identified.

6.5.9 Validation

CO yields have been related to real-scale results.

6.5.10 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.



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 6 — Schematic of the flame propagation apparatus

6.6 Tube furnace methods

6.6.1 Static tube furnace (International)

6.6.1.1 Application

This apparatus initially came from France, but is used nowadays in railways at European level (EN 45545-2) and at international level (UITP E6, NATO AFAP-3). It was initially described in NFX 70-100[27],[28] (see Figure 7) and 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.

6.6.1.2 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.

6.6.1.3 Fire stage(s)

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

Appropriate fire stage(s) from ISO 19706 are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 1.c, anaerobic pyrolysis from externally applied radiation;

— 2, well-ventilated flaming;

— 3.a, small, localized fire, generally in a poorly ventilated compartment;

— 3.b, post-flashover fire.

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.

6.6.1.4 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, NOx (NO and NO_2), formaldehyde and acrolein using ILC, HPLC and classical analytical methods according to ISO 19701. FTIR according to ISO 19702 can also be used.

6.6.1.5 Presentation of results

The data are presented as gas yields.

6.6.1.6 Apparatus assessment

6.6.1.6.1 Advantages

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

6.6.1.6.2 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 may limit gas detection. Several runs are needed to measure the full range of toxic products. The lack of an igniter may lead to unrepeatable flaming. Flaming will be 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 may 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 are needed for fire safety engineering calculations.

6.6.1.6.3 Repeatability and reproducibility

Inter-laboratory evaluations have been performed in which the gases have been introduced into the furnace[27], with good results. Data on materials are available in ISO 19701. Data on the temperature profile of furnaces are available and these are fairly uniform across the laboratories.

6.6.1.7 Toxicological results

6.6.1.7.1 Advantages

The method does not generate direct toxicological results. However, the test enables the yields of toxic gases to be determined under controlled conditions.

6.6.1.7.2 Disadvantages

The toxic potency of a material in its end-use condition cannot be evaluated.

6.6.1.8 Miscellaneous

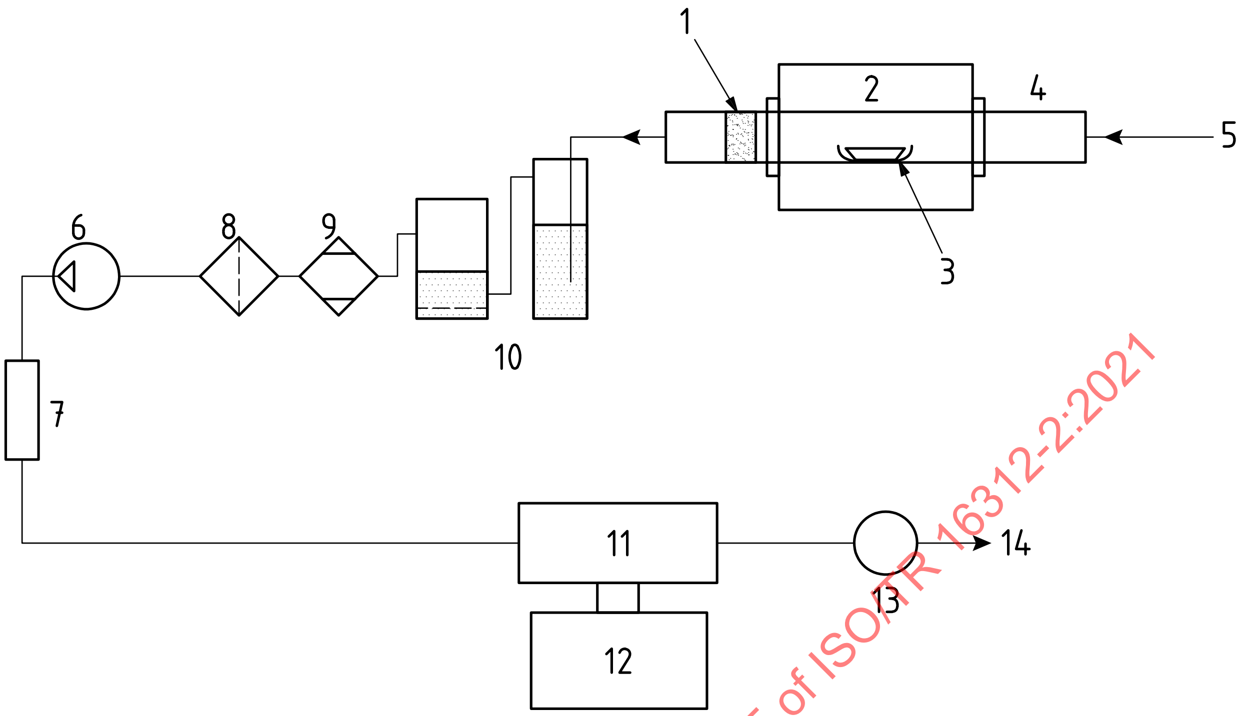
No animals are used with this method.

6.6.1.9 Validation

Results have been tested against gas yields in real-scale tests of materials used on trains[29]. Reasonable correlations were found for toxicity of structural materials between (a) the toxicity index found with the NFX 70-100 method (at 400 °C and 600 °C) when combined with mass loss, determined using ISO 5660-1 and (b) the fractional effective dose (FED).

6.6.1.10 Conclusion

This method can be used to screen materials for their yields of known toxicants from the pyrolysis, under-ventilated flaming and well-ventilated flaming of homogeneous materials. However, if the combustion conditions vary for different materials, comparison among those materials can be difficult. The small sample size limits the use for evaluation of finished products. The absence of animal-exposure data means that extreme or unusual toxic potency of smoke cannot be identified.



Key

1	silica wool	8	particle filter
2	furnace	9	drying filter
3	boat and sample	10	impingers
4	tube	11	gas analysers
5	dry-air inlet	12	data collection
6	pump	13	gas meter
7	flowmeter	14	exhaust

Figure 7 — Schematic of the NFX 70-100 tube furnace

6.6.2 Tube furnace (Germany)

6.6.2.1 Application

This apparatus (see Reference [22] and DIN 53436-1), was designed to thermally degrade solid or fluid substances under predefined temperature conditions in an adjustable air flow. Originally, it was coupled to an apparatus to expose rodents in order to determine the toxic effects of the gaseous emissions, but this part of DIN 53436-3 has been withdrawn, as well as the calculation methods stated in DIN 53436-5. The furnace can also be linked to suitable analytical methods, i.e. Fourier-transformed infrared spectroscopy (FTIR), to quantify gaseous species. The original purpose was to generate toxic potency data for liquid/solid test articles, building and furnishing materials and end products.

6.6.2.2 Principle

A schematic of the apparatus is shown in Figure 8. This is a flow-through system. The sample, approximately, is cut from the end product and heated by radiation, convection and heat conduction in a tube furnace. The furnace moves continuously across the specimen in the tube, countercurrent to the air-flow direction, to facilitate a constant combustion rate over the duration of the 30 min test. Due to the possibility of using different temperature exposures and to vary the air flow, substances can be tested under smouldering or under flaming conditions. The airflow carrying the combustion effluents can be analysed with suitable continuous methods (i.e. FTIR) or accumulating methods (i.e. bubblers or filters which subsequently undergo analysis).

6.6.2.3 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, are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 1.c, anaerobic pyrolysis from externally applied radiation;

— 3.a, small, localized fire, generally in a poorly ventilated compartment;

— 3.b, post-flashover fire.

6.6.2.4 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[22],[26].

6.6.2.5 Presentation of results

Depending on the utilized analysis method, concentrations of the gaseous emissions can be determined for different furnace temperatures. When used with animal exposure, 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.

6.6.2.6 Apparatus assessment

6.6.2.6.1 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. If applicable, 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.

6.6.2.6.2 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.

6.6.2.7 Repeatability and reproducibility

Three-laboratory evaluation of this method using reference materials has been performed[22],[26] and has shown sufficient repeatability and reproducibility.

6.6.2.8 Toxicological results

6.6.2.8.1 Advantages

When used with animal exposure, 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.

When used with analytical methods the method produces results for different stages of a fire (temperature and ventilation conditions can be changed). The test enables the yields of toxic gases to be determined under controlled conditions.

6.6.2.8.2 Disadvantages

Flaming is quenched on the upper surface of the tube, resulting in distorted concentrations of some combustion products reaching the test animals.

6.6.2.9 Miscellaneous

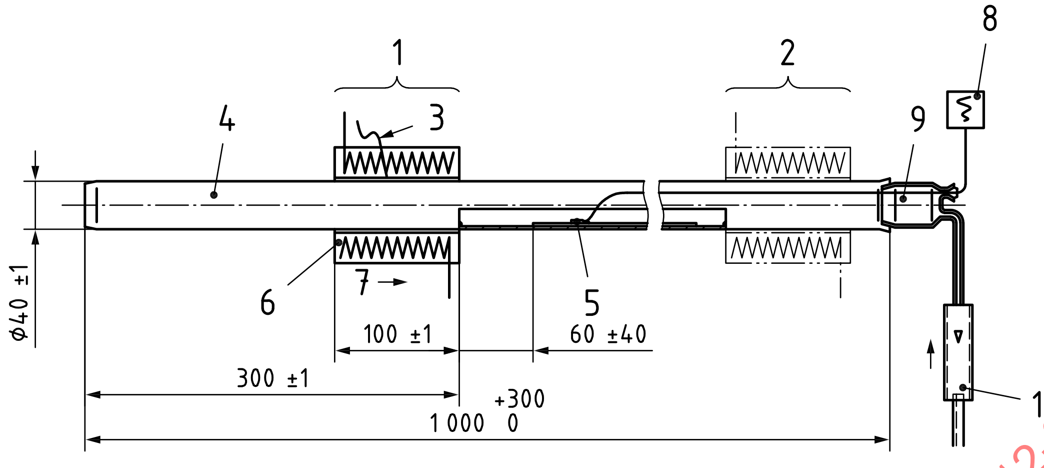
This was designed as an animal-exposure test with limited chemical instrumentation. However, additional analytical instrumentation and analysis methods have been developed since then and can be added with little interference with the standard method.

6.6.2.10 Validation

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

6.6.2.11 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.



Key

1 position 1
2 position 2
3 thermocouple
4 quartz tube
5 cuvette with reference specimen
6 tube furnace
7 feeding direction
8 recorder
9 connector
10 flow meter

Figure 8 — Schematic of the DIN 53436-1:2015 tube furnace

6.6.3 ISO/TS 19700 Tube furnace (International)

6.6.3.1 Application

This apparatus is currently described in ISO/TS 19700 and was previously described in BS 7990. It was designed to obtain toxic gas yields for decomposing and burning materials under various fire conditions.

6.6.3.2 Principle

A schematic of the apparatus is shown in Figure 9. This is a flow-through system. The sample is fed into the tube furnace at a fixed rate (typically 1 g/min^{-1}) alongside a fixed air flow, which may be above, at, or below the stoichiometric (chemical) air requirement. As the sample moves into the furnace, so it experiences increasing radiant flux intensity (and some conductive and convective heating), until it ignites, then the flame spreads to a slightly cooler part of the furnace. At low oxygen concentrations, where ignition is more difficult, the sample reaches a hotter part of the furnace before igniting, and again, the flame will stabilize itself as it spreads a little way back up the tube. The fixed fuel feed rate and fixed air flow allows the equivalence ratio to be pre-determined. The fire effluent leaving the tube furnace is diluted to a total flow of 50 l/min^{-1}, providing a constant concentration base, and a large excess of gas for the full range of analyses.

6.6.3.3 Fire stage(s)

The fire stage(s) from ISO 19706, are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 1.c, anaerobic pyrolysis from externally applied radiation;

— 2, well-ventilated flaming;

— 3.a, small, localized fire, generally in a poorly ventilated compartment;

— 3.b, post-flashover fire.

6.6.3.4 Types of data

Oxygen concentrations are measured to confirm the fire stage and as input for estimation of hypoxia. The concentrations of CO_2, CO, HF, HCl, HBr, NO, NO_2, HCN, SO_2, H_3PO_4, acrolein, formaldehyde and a range of other organic species may be measured as gas concentrations in the diluted fire effluent or collected for a fixed period through bubblers. Smoke generation is determined using a light/photocell system and expressed as optical density and smoke yield. Although this method is intended primarily for chemical analysis, animal exposure can be carried out for irritancy, acute lethality and other toxicological investigations.

6.6.3.5 Presentation of results

For a given equivalence ratio and temperature, the test produces a concentration and yield of each toxicant and the extinction coefficient and specific extinction area of smoke. The data can be used to calculate an estimated fractional effective dose (FED) of the effluent. With animal exposure, LC_{50} values can be determined.

6.6.3.6 Apparatus assessment

6.6.3.6.1 Advantages

The apparatus allows small-scale replication, under steady state conditions, of three fire stages and is well suited for the highly toxic, vitiated stages (3a and 3b). This has been shown for solid thermoplastic products only – for a number of other products this has not yet been achieved.

6.6.3.6.2 Disadvantages

Pre-testing can be necessary for determining the desired operating conditions for test specimens of unknown composition. Samples of non-homogeneous products, accommodated by the furnace, might not be indicative of end-use configuration. The lack of an igniter can lead to unrepeatable flaming. 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.

6.6.3.6.3 Repeatability and reproducibility

ISO/TS 19700 presents interlaboratory reproducibility data for selected homogenous thermoplastic materials.

6.6.3.7 Toxicological results

6.6.3.7.1 Advantages

The test enables the yields of toxic gases to be determined under controlled conditions and the estimation of an FED. With the addition of animal exposure, LC_{50} data can be obtained.

6.6.3.7.2 Disadvantages

The standard method does not generate direct toxicological results.

6.6.3.8 Miscellaneous

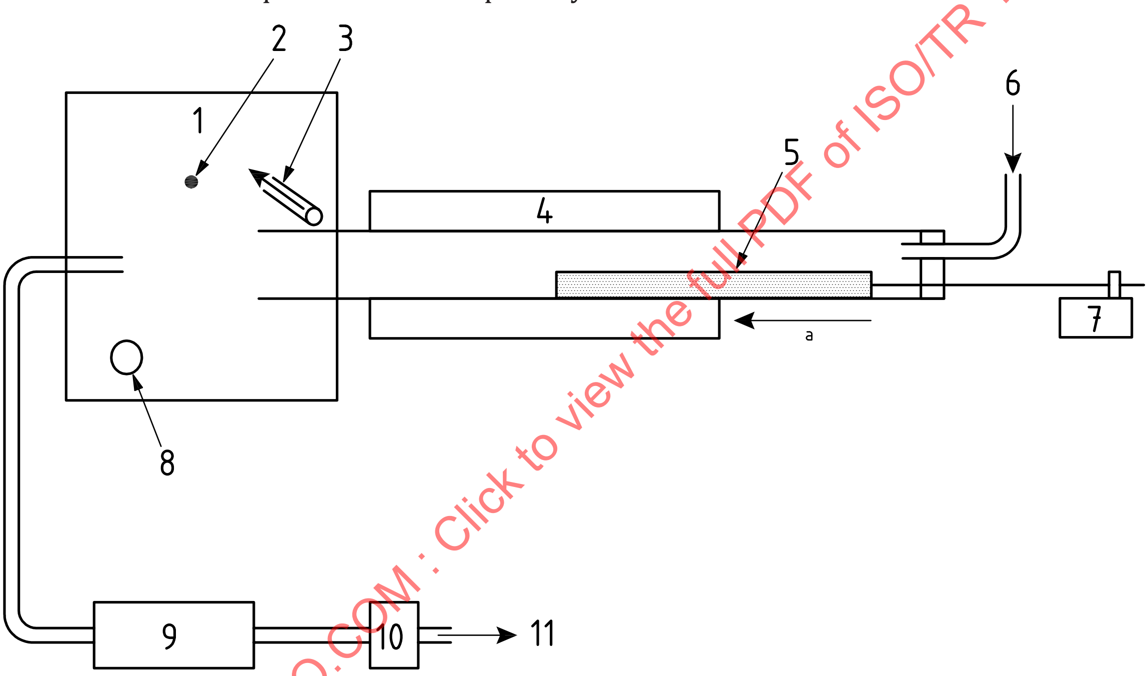
There can be advantages in supplementing the lower air flows used for vitiated combustion with a balance of nitrogen. On one occasion, this variation gave significantly different toxic-product yields for a particular material.

6.6.3.9 Validation

Published work shows a correlation between CO yields in real-scale fires and those found in the tube furnace[31],[32].

6.6.3.10 Conclusion

This method generates combustion product yield data for a range of equivalence ratios and a range of fire stages. With validation, this can be a useful test for obtaining estimates of the toxic potency of smoke from materials and some end products for input to fire hazard models. The addition of animal-exposure data can lead to quantitative toxic potency information.



Key

1 mixing chamber
2 gas sampling
3 secondary air
4 furnace
5 sample boat
6 primary air
7 sample-drive mechanism
8 smoke meter
9 aspirated bubbler chain
10 metering pump
11 exhaust
a Direction of sample movement.

Figure 9 — Schematic of the ISO/TS 19700 tube furnace

7 Summary of test methods

Apparatus (section)	Fire model	Intended fire stage	Apparatus assessment		Toxicological results	
			advantages	disadvantages	advantages	disadvantages
NBS smoke chamber (6.1.1)	Vertically-orientated 76 mm x 76 mm sample Heat flux 25 kW/m^2. With and without pilot flame.	1.b, oxidative pyrolysis from externally applied radiation; 2, well-ventilated flaming.	Simple to use and widely available. Test specimen can be a reasonable representation of a finished product.	Combustion conditions not well characterized. Not adapted to melting and dripping materials. Gases mixed by natural convection.	Initial conditions are few and well prescribed.	Possible vitiation. Possible condensation.
ISO smoke chamber (6.1.2)	Horizontally-oriented 75 mm x 75 mm sample Heat flux 25 and 50 kW/m^2. With and without pilot flame.	1.b, oxidative pyrolysis from externally applied radiation; 2, Well-ventilated flaming.	Simple to use and widely available. Test specimen can be a reasonable representation of a finished product.	Combustion conditions not well characterized. Gases mixed by natural convection.	Initial conditions are few and well prescribed.	Possible vitiation. Possible condensation.
NES 713 (6.2)	Specimen exposed to a premixed Bunsen burner flame. Atmosphere mixed with a fan then sampled for gas measurement.	2, well-ventilated flaming. However, this might not relate to a real fire.	Simple to use. Test specimen can be a reasonable representation of a finished product.	The specimen combustion might not be representative of the real fire conditions. Colorimetric tubes are not a reliable measurement technique for combustion products due to possible interferences.	Initial conditions are few and well prescribed.	The gases selected are representing hazard in warships. The coefficients for the toxicity index calculation are not current. The basis for the index equation is unclear.
Japanese and Korean rotative cages (6.3.1)	Two-chamber apparatus, animal model exposure and optionally gas analysis.	3.a, small, localized fire, generally in a poorly ventilated compartment.	Single combustion test condition well defined. The method could be adapted to calculate yields of toxicants.	The test is limited to a single fire stage.	A direct measure of the incapacitation capability of the smoke. It can identify instances of extreme and unusual fire effluents toxic potency.	Requires specialized equipment for animal exposure.

Apparatus (section)	Fire model	Intended fire stage	Apparatus assessment		Toxicological results	
			advantages	disadvantages	advantages	disadvantages
Chinese method (6.3.2)	Homogeneous strip material is heated by a tubular furnace with constant velocity primary air and stable heating.	1.b, oxidative pyrolysis from externally applied radiation.	Steady fire effluents can be quantitatively produced, and the concentration and animal exposure results can be obtained.	The test is limited to a single fire stage.	The symptoms of the animals after exposure can be observed directly, and the general anatomy and histopathological examination can be carried out to accurately describe the toxic effect and degree of the smoke.	It is difficult to control the respiratory rate and degree of exposure of the animal when they are awake. Therefore, the inhaled dose or concentration is difficult to obtain.
Cone calorimeter (6.4)	Sample up to 100 mm x 100 mm in area. Thermal radiation in the range 0-100 kW/m^2. In addition, ISO/TS 5660-5 allows exploring oxygen conditions from 21 % down to 15 %.	In ISO 5660-1: 1.b, oxidative pyrolysis ; from externally applied radiation; 2, well-ventilated flaming. In addition in ISO/TS 5660-5: 1.c, anaerobic pyrolysis from externally applied radiation; 3.a, small, localized fire, generally in a poorly ventilated compartment; 3.b, post-flashover fire.	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 of 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.	In ISO 5660-1 conditions, the flames are highly over-ventilated, so the gas yields are not clearly linked to real-scale results, especially post-flashover fires. In ISO/TS 5660-5 conditions, the conditions between pyrolysis and combustion may be different, and post-combustion may occur, modifying the results. The fraction of the air flow passing through the combustion zone is unknown, making values of the global equivalence ratio somewhat uncertain. The effluents are flowed across to the radiant heater, leading to possible modifications.	Since the heat-release rate and smoke density correlate to real-scale data in pre-flashover flaming fires, it is possible that toxic gas yields also correlate, but this has not been confirmed.	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.

Apparatus (section)	Fire model	Intended fire stage	Apparatus assessment		Toxicological results	
			advantages	disadvantages	advantages	disadvantages
Flame propagation apparatus (6.5)	Samples up to 102 mm x 102 mm in area (horizontal sample) or 102 mm wide x 300 mm high (vertical sample). Pilot flame. Control of the equivalence ratio.	1.b, oxidative pyrolysis from externally applied radiation; 1.c, anaerobic pyrolysis from externally applied radiation; 2, well-ventilated flaming; 3.a, small, localized fire, generally in a poorly ventilated compartment; 3.b, post-flashover fire.	The representation and exposure of finished products is accurate. 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.	There are few apparatus worldwide. In vitiated conditions, problems of post-combustion can occur.	The CO yield correlates with real-scale, flaming fires. The method can provide a good indication of the yields of major toxic products for comparable full-scale fire conditions.	There have been no reported attempts to obtain toxic potency data. Thus, there is no list of prescribed gas measurements.
Static tube furnace (6.6.1)	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.	The method does not specify any particular fire stage.	The apparatus is easy to use. The operating conditions can be easily modified.	The small specimen size limits the apparatus to the 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. The lack of an igniter may lead to unrepeatable flaming.	The test enables the yields of toxic gases to be determined under controlled conditions.	The toxic potency of a material in its end-use condition cannot be evaluated.

Apparatus (section)	Fire model	Intended fire stage	Apparatus assessment		Toxicological results	
			advantages	disadvantages	advantages	disadvantages
Tube furnace (Germany) (6.6.2)	This apparatus was designed to thermally degrade solid or fluid substances under predefined temperature conditions in an adjustable air flow.	The method does not specify any particular fire stage.	The test conditions are well defined. The flow-through system provides for a constant atmosphere composition with a low residence time. The test conditions are defined in terms of sample mass, furnace temperature and air flow.	The relation between the sample exposure and that in real-scale is questionable. There can be significant condensation on surfaces. The lack of an igniter can lead to unrepeatable flaming. The combustion conditions depend upon the behaviour of each individual specimen and can change during the course of a test run.	When used with animal exposure, the method produces both a qualitative (mode of action) and a quantitative measure of smoke lethality. When used with analytical methods, the method produces results for different stages of a fire. The test enables the yields of toxic gases to be determined under controlled conditions.	Flaming is quenched on the upper surface of the tube, resulting in distorted concentrations of some combustion products reaching the test animals.
ISO/TS 19700 tube furnace (6.6.3)	This is a flow-through system. The sample is fed into the tube furnace at a fixed rate alongside a fixed air flow, which may be above, at, or below the stoichiometric (chemical) air requirement.	1.b, oxidative pyrolysis from externally applied radiation; 1.c, anaerobic pyrolysis from externally applied radiation; 2, well-ventilated flaming; 3.a, small, localized fire, generally in a poorly ventilated compartment; 3.b, post-flashover fire.	The apparatus allows small-scale replication, under steady state conditions, of three fire stages and is well suited for the highly toxic, vitiated stages	Pre-testing can be needed to determine the desired operating conditions for test specimens of unknown composition. Samples of non-homogeneous products, might not be indicative of end-use configuration. The lack of an igniter can lead to unrepeatable flaming. Additional data input on burning rates at different fire stages is required for fire safety engineering calculations.	The test enables the yields of toxic gases to be determined under controlled conditions and the estimation of an FED. With the addition of animal exposure, LC_{50} data can be obtained.	The standard method does not generate direct toxicological results.

Annex A
(informative)

Deprecated methods

A.1 Cup-furnace smoke-toxicity test method

A.1.1 Application

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

A.1.2 Principle

A schematic of the apparatus is shown in Figure A.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.

A.1.3 Fire stage(s)

The fire stage(s) from ISO 19706, are as follows:

— 1.b, oxidative pyrolysis from externally applied radiation;

— 2, well-ventilated flaming.

A.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.

A.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.

A.1.6 Apparatus assessment

A.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.

A.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 necessary for fire safety engineering calculations.

A.1.6.3 Repeatability and reproducibility

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

A.1.7 Toxicological results

A.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.

A.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.

A.1.8 Miscellaneous

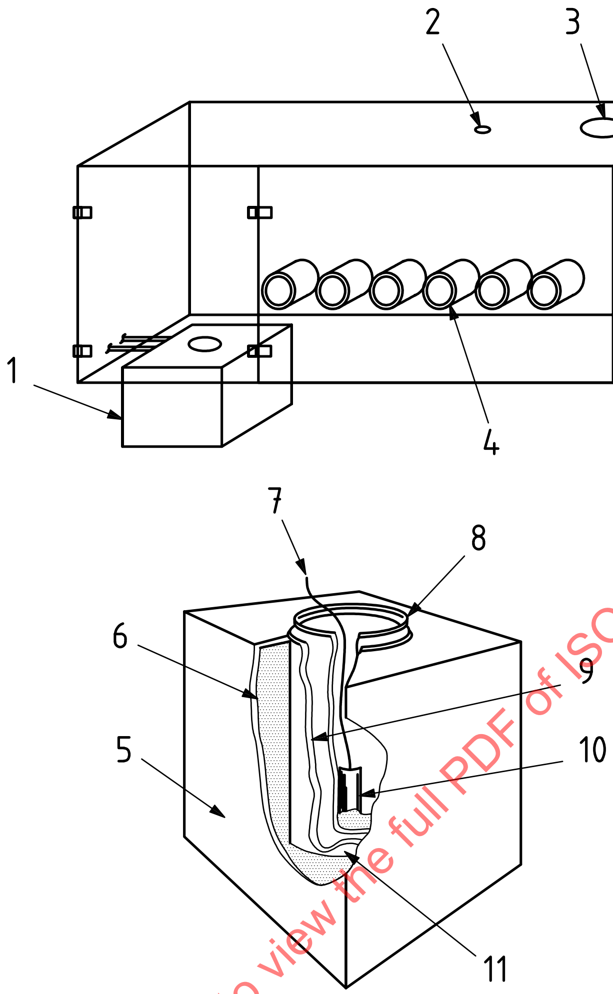
This is primarily an animal-exposure test with chemical instrumentation to quantify the expected major toxicants. Additional analytical instrumentation can be added with little interference with the standard method. The apparatus can be used without test animals, but it then loses the ability to identify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

A.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.

A.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.



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 A.1 — Schematic of the cup-furnace smoke-toxicity apparatus

A.2 Radiant furnace toxicity test method (United States)

A.2.1 Application

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

A.2.2 Principle

A photograph of the apparatus is shown in Figure A.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.