TECHNICAL SPECIFICATION

ISO/TS 19700

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Controlled equivalence ratio method for the determination of hazardous components of fire effluents

Méthode du rapport d'équivalence contrôlée pour la détermination des substances dangereuses des effluents du feu

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Foreword

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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 other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 19700 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

Introduction

The framework for the long-term standardization of fire safety in support of performance-based design (ISO/TC 92 SC 4) requires general engineering methods for specific performance aspects of fire safety, but is applicable to all types of structural systems, products and processes. These are referred to in the document as Level 2, Group B standards. One such aspect of fire safety is the yields of toxic products evolved in fires. This Technical Specification has been developed to measure toxic product yields from materials and products over a range of decomposition conditions in fires. The decomposition conditions are defined in terms of fuel/air equivalence ratio, temperature and flaming behaviour.

The toxic potency of a fire effluent represents the combination of a number of factors, including the concentrations of toxic products, gases, and smoke particles. The concentrations of toxic products in turn depend upon a number of factors, one of which is the yield of each toxic product from the burning fuel. In order to make a performance-based assessment of the toxic hazard in a fire, one required input is the yield of toxic products under specified fire conditions.

For any specific material or product, the effluent yields in fires depend upon the thermal decomposition conditions. The most important variables are whether the decomposition is non-flaming or flaming, and for flaming decomposition the fuel/oxygen ratio. Based upon these variables, it is possible to classify fires into a number of types, as detailed in ISO/TS 19706:2004, Table 1.

The use of this Technical Specification provides data on the range of toxic product yields likely to occur in different types and stages of full-scale fires. More comprehensive data on the relationships between decomposition conditions and product yields can be obtained by using a wider range of apparatus settings. Guidance on the choice of additional decomposition conditions, the application of test data to ISO 13344 and ISO 13571, to health and safety and environmental situations and the use of the tube-furnace method for bioassay purposes is provided in the annexes.

This Technical Specification makes use of the same apparatus and a similar basic methodology as specified in IEC 60695-7-50. The test method has been developed to fulfil the requirements of ISO 16312-1 and ISO/TS 19706, for data on the yields of toxic products in fire effluents evolved under different fire conditions as part of the data required for input to the toxic-hazard-assessment calculation methods described in ISO 13571. The data may also be used as input for the toxic-potency calculation methods described in ISO 13344 and ISO 13571.

Controlled equivalence ratio method for the determination of hazardous components of fire effluents

1 Scope

This Technical Specification describes a tube-furnace method for the generation of fire effluent for the identification and measurement of its constituent combustion products, in particular, the yields of toxic products under a range of fire decomposition conditions.

It uses a moving test specimen and a tube furnace at different temperatures and air flow rates as the fire model. The use of this apparatus is generally applicable to individual materials, to products that are layered such that the layering will not result in a significant change in product yields with time in real fires, i.e. to products where the upper surface does not provide major protection to the sub-layers.

This method has been designed as a TC 92 Level Group B performance-based engineering method to provide data for input to hazard assessments and fire-safety engineering design calculations. The method can be used to model a wide range of fire conditions by using different combinations of temperature, non-flaming and flaming decomposition conditions and different fuel/oxygen ratios in the tube furnace. These include the following types of fires, as detailed in ISO/TS 19706:2004, Table 1:

— Stage 1: Non-flaming:

  — Stage 1b) Oxidative pyrolysis from externally applied radiation;

— Stage 2: Well-ventilated flaming (representing a flaming developing fire) (see Note 1);

— Stage 3: Less well-ventilated flaming (see Note 2):

  — Stage 3a) Small vitiated fires in closed or poorly ventilated compartments;

  — Stage 3b) Post-flashover fires in large or open compartments.

NOTE 1 Where the fire size is small in relation to the size of the compartment, the flames are below the base of the hot layer and the fire size is fuel-controlled.

NOTE 2 Where the fire size may be large in relation to the size of the compartment, the flames are partly above the base of the hot layer and the fire size is ventilation-controlled.

For each flaming fire type, the minimum conditions of test are specified in terms of the equivalence ratio ϕ as follows:

Stage 2: $\phi < 0{,}75$;

Stages 3a) and 3b) $\phi = 2 \pm 0{,}2$.

Guidance on the choice of additional decomposition conditions is given in Annex A.

The data on toxic product concentrations and yields obtained using this Technical Specification may be used as part of the assessment of toxic potencies, in conjunction with toxic potency calculation methods in ISO 13344, and as an input to the toxic hazard assessment from fires in conjunction with fire growth and effluent dispersal modelling, and fractional effective dose (FED) calculation methods in ISO 13571.

Application of data from the tube-furnace test to the calculation of lethal toxic potency according to ISO 13344, and to the assessment of toxic hazards in fires according to ISO 13571 is considered in Annex B and Annex C, respectively.

Guidance on application of data from the tube-furnace test to health and safety assessments of combustion products, and to the assessment of environmental hazards of combustion products from fires is given in Annex D and Annex E, respectively. Guidance on the use of the tube-furnace method for bioassay purposes is given in Annex F.

The test method described in this Technical Specification can be used solely to measure and describe the properties of materials, products or systems in response to heat or flame under controlled laboratory conditions. It is not suitable to be used by itself for describing or appraising the fire hazard of materials, products or systems under actual fire conditions, or as the sole source on which regulations pertaining to toxicity can be based.

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 291:2005, *Plastics — Standard atmospheres for conditioning and testing*

ISO 554:1976, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 5660-2:2002, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 2: Smoke production rate (dynamic measurement)*

ISO 13344:2004, *Estimation of the lethal toxic potency of fire effluents*

ISO 13571, *Life-threatening components of fire — Guidelines for the estimation of time available for escape using fire data*

ISO/IEC 13943, *Fire safety — Vocabulary*

ISO 19701:2005, *Methods for sampling and analysis of fire effluents*

ISO 19702:2006, *Toxicity testing of fire effluents — Guidance for analysis of gases and vapours in fire effluents using FTIR gas analysis*

ISO/TS 19706:2004, *Guidelines for assessing the fire threat to people*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13344, ISO 13571, ISO 13943, and the following apply.

3.1
combustible load
mass of the components of a test specimen capable of combustion in the furnace

NOTE This usually includes all components of a specimen excluding inert fillers and other non-combustible components, such as metal frames.

3.2
equivalence ratio
ϕ
fuel mass to oxygen mass ratio in the test divided by the stoichiometric fuel mass to oxygen mass ratio

NOTE For the tube-furnace method, this is the mass loss rate of combustible effluent from the test specimen, in milligrams per minute ($mg \cdot min^{-1}$), divided by the mass flow rate of oxygen in the primary air introduced into the furnace, in milligrams per minute ($mg \cdot min^{-1}$), divided by the stoichiometric fuel mass to oxygen mass ratio for the material under test.

3.3
exposure dose
Ct
product of a gaseous toxicant or of a fire effluent which is available for inhalation, i.e. the integrated area under the concentration(C)-time(t) curve

3.4
extinction coefficient
natural logarithm of the ratio of incident light intensity to transmitted light intensity, per unit light path length

3.5
fractional effective concentration
FEC
ratio of the concentration of an irritant to that expected to produce a given effect on an exposed subject of average susceptibility

NOTE 1 As a concept, FEC may refer to any effect, including incapacitation, lethality or even other endpoints. Within the context of this Technical Specification, FEC refers only to incapacitation.

NOTE 2 When not used with reference to a specific irritant, the term FEC represents the summation of FECs for all irritants in a combustion atmosphere.

NOTE 3 When FEC = 1, the defined effect (incapacitation or death) is predicted to occur.

3.6
fractional effective dose
FED
ratio of the Ct product for an asphyxiant toxicant to that Ct product of the asphyxiant expected to produce a given effect on an exposed subject of average susceptibility

NOTE 1 As a concept, FED may refer to any effect, including incapacitation, lethality or even other endpoints. Within the context of this Technical Specification, FED refers only to incapacitation.

NOTE 2 When FED = 1, the defined effect (incapacitation or death) is predicted to occur.

3.7
LC_{50}
concentration of a toxic gas or fire effluent statistically calculated from concentration-response data to produce lethality in 50 % of test animals within a specified exposure and post-exposure time

NOTE The typical units are $\mu L/L$ for a gaseous toxicant and gm^{-3} for fire effluent.

3.8
LCt_{50}
product of LC_{50} and the exposure duration over which it was determined

NOTE The typical units are $\mu L L^{-1} min$ for a gaseous toxicant and $gm^{-3} min$ for fire effluent. This constitutes a measure of lethal toxic potency.

3.9
mass-charge concentration
concentration of fire effluents from a material defined in terms of the mass of material exposed to burning conditions (mass charge) and the volume into which the effluent is dispersed, expressed in $g \cdot m^{-3}$

3.10
mass loss concentration
concentration of fire effluents from a material defined in terms of the mass of material decomposed (mass loss) and the volume into which the effluent is dispersed, expressed in $g \cdot m^{-3}$

3.11
mass loss exposure dose
mass loss concentration multiplied by the exposure time, expressed in $g \cdot m^{-3}$min

[BS 7899-2:1999, definition 2.22]

3.12
smoke extinction area
SEA
ratio of the smoke extinction coefficient, in reciprocal metres (m^{-1}) to the mass loss concentration of the test specimen, expressed as grams per cubic metre ($g \cdot m^{-3}$) having units of metres squared per gram ($m^2 \cdot g^{-1}$)

3.13
smoke obscuration
reduction, usually expressed as a percentage, in the intensity of light due to its passage through smoke

3.14
smoke production
integral of the smoke production rate over the steady-state burn period being considered

3.15
volume yield
volume of an effluent component at 20 °C and 101,325 kPa divided by the mass loss of the test specimen associated with the production of that volume of the effluent component

3.16
yield
mass of an effluent component divided by the mass loss of the test specimen associated with the production of that mass of the effluent component

4 Principle

Since the yields of products in fires depend upon the decomposition conditions (references [1] to [5]), it is possible to examine the relationships between product yield and a range of variables affecting the decomposition conditions using this apparatus and the methodology described. The specified test conditions represent a minimum set designed to obtain data for oxidative pyrolysis under non-flaming conditions, for well-ventilated flaming conditions at an equivalence ratio of less than 0,75, and for vitiated flaming conditions at an equivalence ratio of more than 2. The test is designed to replicate real fire conditions, and it is essential that proper observations are made to ensure that those conditions are being met.

Samples of a material or product are combusted under steady-state conditions in one or more of four environments whose temperature and equivalence ratio are representative of a particular stage of a fire. The four types of fire to be represented are: oxidative pyrolysis, well-ventilated flaming developing fires, small flaming vitiated fires, and post-flashover vitiated fires, as defined in ISO/TS 19706.

A test specimen in granular or rod form, or a product, is placed in a quartz boat, and introduced at a constant rate into a furnace tube through the hot zone of a fixed tubular furnace. A stream of primary air is passed through the furnace tube and over the test specimen at constant flow rate, to support combustion. The fire

effluent is expelled from the quartz furnace tube into a mixing and measuring chamber, where it is diluted with secondary air to a nominal total air flow rate of $(50 \pm 1)\ l \cdot min^{-1}$ through the chamber and then exhausted to waste.

In the oxidative pyrolysis mode, the furnace temperature is set below the auto-ignition temperature. The three flaming modes are accomplished by using vapour temperatures above typical auto-ignition temperatures. For flaming decomposition conditions, different fuel-to-oxygen ratios, and hence different equivalence ratios, are obtained when different, constant primary air flows are used in relation to the constant rate of introduction of the fuel. To achieve the required gasification rates, materials may be combusted under different conditions from each other.

The secondary, dilution air is added to generate a greater sample flow and cooler effluent which permits a large number of gas and smoke sampling procedures to be used without the need for a large number of replicate tests.

The requirement in each test run is to obtain stable, steady-state decomposition conditions for at least 5 min during which the concentrations of effluent gases and particles can be measured. The time taken for steady-state conditions to be established varies, depending upon the nature of the test specimen and the test conditions.

The concentrations of carbon dioxide and oxygen are recorded to establish the steady-state period and samples of the effluent mixture are taken from the chamber during the steady-state period for analysis. Smoke obscuration and smoke yield are calculated from measurement of the attenuation of a light beam by the combustion effluent stream in the mixing chamber. A sample of smoke is drawn through a filter, and the mass of particles is determined.

5 Apparatus

5.1 General apparatus

The apparatus consists of a tube furnace and a quartz tube which passes through the furnace and into a mixing and measurement chamber. A drive mechanism pushes the specimen boat into the furnace tube at a preset, controlled rate. A constant, predetermined stream of primary air is provided at the furnace-tube entry and a preset, secondary supply into the mixing and measurement chamber. Gas samples are taken from the mixing and measurement chamber. A light/photo cell system is used to determine smoke density across the mixing and measurement chamber.

The arrangement of the apparatus is shown in Figure 1. Unless otherwise stated, all tolerances are $\pm$ 5 mm.

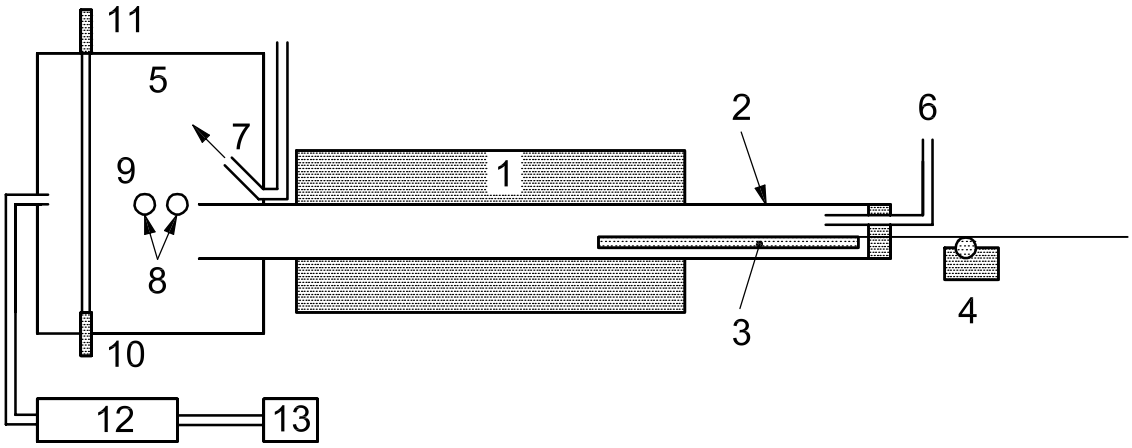
5.2 Tube furnace

The tube furnace shall have a heating zone length of 500 mm to 800 mm and an inside diameter of 50 mm to 65 mm. The furnace shall be equipped with an adjustable electric heating system capable of maintaining the furnace temperature to within $\pm$ 2 % of the set temperature. The heating element should preferably be rated at 1 300 °C (see Note 1).

With the peak furnace temperature set at (650 ± 10) °C, the temperature shall not decrease by more than 100 °C over a length of at least $\pm$ 125 mm from the point of peak temperature measurement. The method used to determine this temperature profile is given in 7.2 (see Note 2).

NOTE 1 The furnace is similar to that used in IEC 60754-2.

NOTE 2 This will also reduce the likelihood of a hot spot in the furnace, to which the pyrolysis rate will be sensitive.

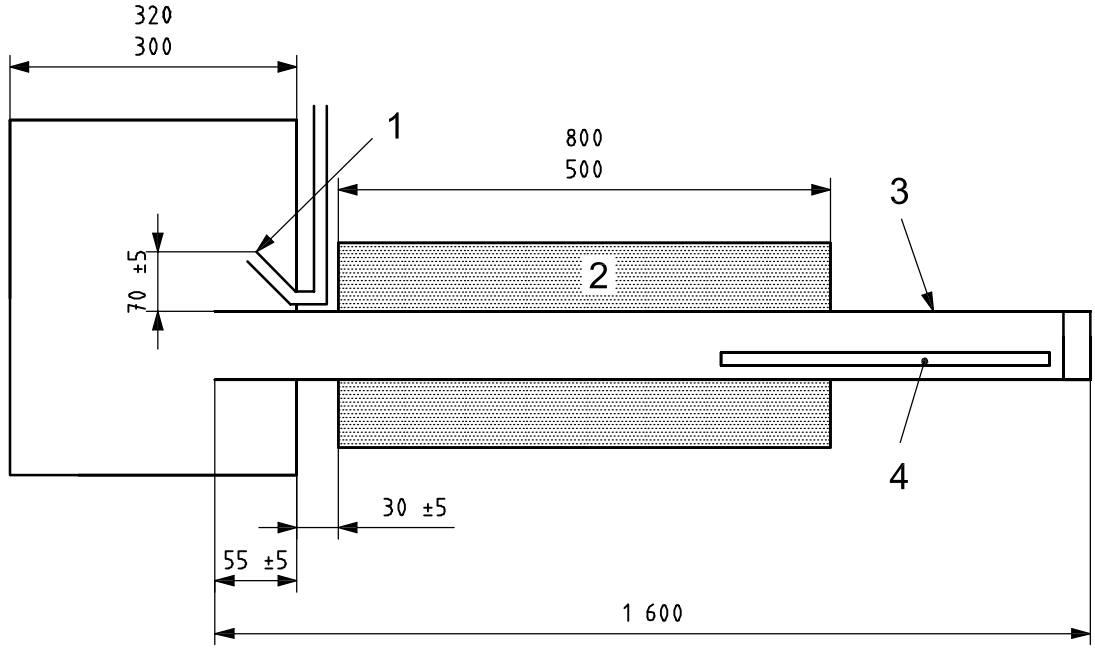


Key

1 tube furnace
2 quartz furnace tube
3 test-specimen boat
4 test-specimen boat drive mechanism
5 mixing and measurement chamber
6 primary air inlet
7 secondary air inlet
8 ports for sampling lines
9 smoke-particle filter
10 tube containing light source
11 tube containing photodetector
12 gas bubblers
13 pump with flow meter

a) General arrangement of apparatus

Dimensions in millimetres



Key

1 secondary air inlet 45° to vertical
2 tube furnace
3 furnace tube
4 sample boat 800

b) Critical dimensions of assembly

Figure 1 — Tube-furnace decomposition and sampling apparatus

5.3 Calibrated thermocouples

Calibrated stainless-steel sheathed thermocouples, 1,5 ± 0,1 mm in diameter, shall be used for measuring the temperature in the furnace tube, the temperature in the mixing and measurement chamber and for calibrating the furnace. Three thermocouples are required.

5.4 Quartz furnace tube

The quartz furnace tube, as shown in Figure 2, is made of clear heat-resistant quartz, resistant to the effects of fire effluent. The tube is 1 600 mm long, and has an external, approximately concentric diameter of (47,5 ± 1) mm and a wall thickness of (2 ± 0,5) mm. The outside diameter shall permit a smooth fit within the tube furnace (5.2) and allow expansion at operating temperatures.

The input end of the furnace tube shall have a closure with openings in it to allow the primary air inlet and the specimen boat drive to pass through (see Note 1).

The downstream end of the furnace tube shall pass through a heat-resisting sealed gland and shall protrude 55 ± 5 mm into the mixing and measurement chamber (5.7). (see Note 2).

The distance between the mixing and measurement chamber and the exit of the furnace shall be 30 ± 5 mm.

NOTE 1 A PTFE gland seal has been found to be suitable.

NOTE 2 A gland made from glass wool inside a brass collar has been found to be suitable.

5.5 Test-specimen boat

The test-specimen boat, as shown in Figure 2, is made from quartz glass (see Note 1), of diameter (41 ± 2) mm, with a length of 800 mm and a wall thickness of (2 ± 0,5) mm (see Notes 2 and 3). The boat should be cleaned after each test (see Note 4).

NOTE 1 A convenient method for making a suitable test-specimen boat for a 47,5 mm diameter furnace tube is to use quartz tubing with a nominal diameter less than that of the furnace tube (nominal 41 mm). This can then be sliced in half to provide a semi-circular cross-section, nominally of 41 mm width, 18 mm depth and 800 mm length.

NOTE 2 A test-specimen-boat diameter (41 mm) of just less than the furnace-tube internal diameter (47 mm) provides the maximum sample capacity.

NOTE 3 A boat length of 800 mm has been found suitable for testing most materials. Where materials take a long time to reach steady-state burning, or where a steady-state period of longer that 5 min is required, longer boats may be used.

NOTE 4 A convenient method of cleaning both the boat and tube is to remove obvious residues mechanically, then fire at 1 000 °C, followed by washing in water to remove any inorganic residues.

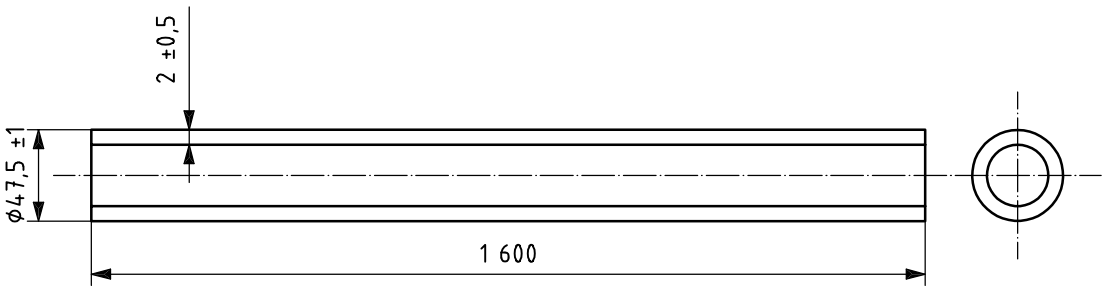
5.6 Test-specimen-boat drive mechanism

The test-specimen boat is connected to a hooked drive bar, which passes through a gland seal (see 5.4) at the upstream end of the furnace tube, and connects to a drive mechanism. The drive mechanism advances the sample boat at a typical rate of (40 ± 1) mm·min^{-1}. The drive mechanism shall allow different speeds to be used, because the actual rate is dependent upon the flame spread characteristics of the sample (see Note).

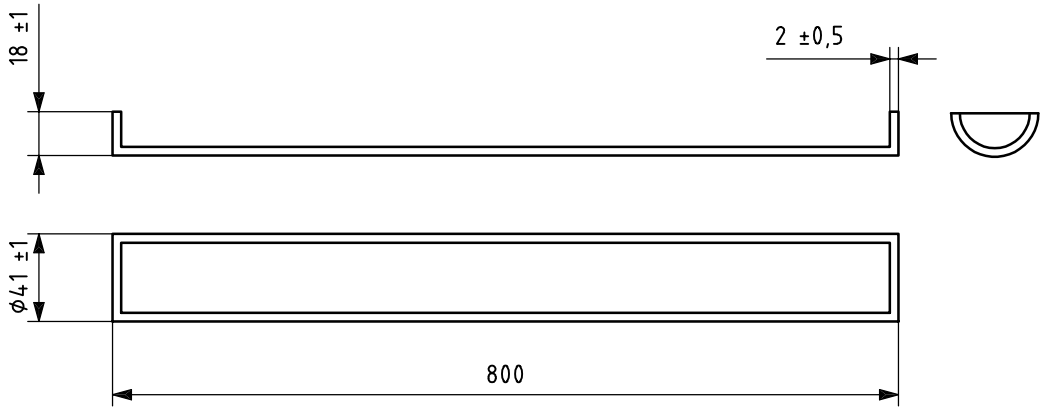
The mechanism shall enable the specimen boat to be rapidly retracted into the upstream, external part of the furnace tube at the end of the test burn. This may be achieved manually after detaching the push rod from the drive mechanism.

NOTE A drive advance rate of 40 mm·min^{-1} has been found suitable for most materials under most decomposition conditions. For some fast-burning or low-density materials, it has been found necessary to use advance rates of up to 60 mm·min^{-1}.

Dimensions in millimetres



a) Quartz furnace tube



b) Test-specimen boat

Figure 2 — Dimensions of a suitable quartz furnace tube and test-specimen boat

5.7 Mixing and measurement chamber

This shall consist of an approximately cubic box with a side length of 30 to 32 cm (see Figure 3) although the exact dimensions are not critical. The box should accommodate the necessary sampling and measurement points (gas sampling probes to bubblers, etc., particulate filters and smoke meter) (see Note 1). The front of the chamber has a door providing a seal when shut. The walls of the box are made of PMMA, polycarbonate, polyethylene or other suitable material, except for the back wall of the chamber and the rear portion of the roof which are made of stainless steel, so as to be resistant to heat and any flames emanating from the end of the furnace tube (see Notes 2 and 3).

The roof of the chamber is fitted with a safety blow-out panel 75 mm in diameter, made of aluminium foil approximately 0,04 mm thick (see Note 3).

Sampling ports and probes are provided in the mixing chamber for taking samples of the test atmosphere. The open end of the sampling probe shall be (30 ± 5) mm from the wall of the mixing and measurement chamber (see Note 4).

A port approximately 35 mm in diameter is provided at the base of the rear face of the chamber for the test atmosphere to be exhausted to waste.

Ports are provided in the mixing and measurement chamber for the insertion of a light source and detector for the measurement of smoke density (see Note 5). Measurement points are located away from the rising plume and the chamber walls; these may be sited in any convenient location. Suitable methods for the prevention of the deposition of particles on the surfaces of both the light source and detector shall be used (see Note 6).

A thermocouple (5.3), extending approximately 50 mm into the mixing and measurement chamber, is located as shown in Figure 3, for monitoring of the temperature in the chamber during the tests.

NOTE 1 The volume of the mixing and measurement chamber needs to be large enough to accommodate the sampling points but smaller than the total volume of air flowing through the box in 1 min.

NOTE 2 A suitable chamber can be made from a commercially available desiccator cabinet with nominal dimensions of 310 mm × 310 mm × 340 mm (see Figure 3). This would have an internal volume of 33 l compared to the air flow volume of 50 l in 1 min. The furnace-tube entry wall of the chamber cabinet should be covered by a stainless-steel plate fitted to the inner surface; the top of the plate extending 140 mm across the chamber roof to provide heat protection for the plastic surfaces.

NOTE 3 This is important for safety reasons.

NOTE 4 The sampling points are positioned away from the furnace-tube exit plume and chamber walls but can be sited in any convenient location. Suitable locations are shown in Figure 3.

NOTE 5 A suitable smoke-measurement path length has been found to be approximately 300 mm.

NOTE 6 A suitable method has been to mount the photodectector and lamp vertically as in Figure 3 and pass part of the chamber diluent air into tubes containing the light source and detector at a rate of 500 ml·min^{-1}. A further modification to reduce particle deposition is to mount the photodetector and lamp horizontally.

5.8 Analysis of gases

This Technical Specification requires the determination of certain combustion gases. The means of gas sampling and analysis shall be those given in ISO 19701 and ISO 19702.

Carbon dioxide and oxygen concentrations shall be sampled and determined continuously throughout the test. These data are used to identify and monitor the steady-state burn period and also to quantify the gas yields. The concentration of carbon monoxide shall also be determined continuously to quantify the gas yield.

The oxygen meter shall be capable of an accuracy of 0,01 %. Details of a suitable oxygen meter and sampling system are given in ISO 5660-1.

The following other gases shall be determined during the steady-state burn period (see Note):

— organic irritants including formaldehyde and acrolein;

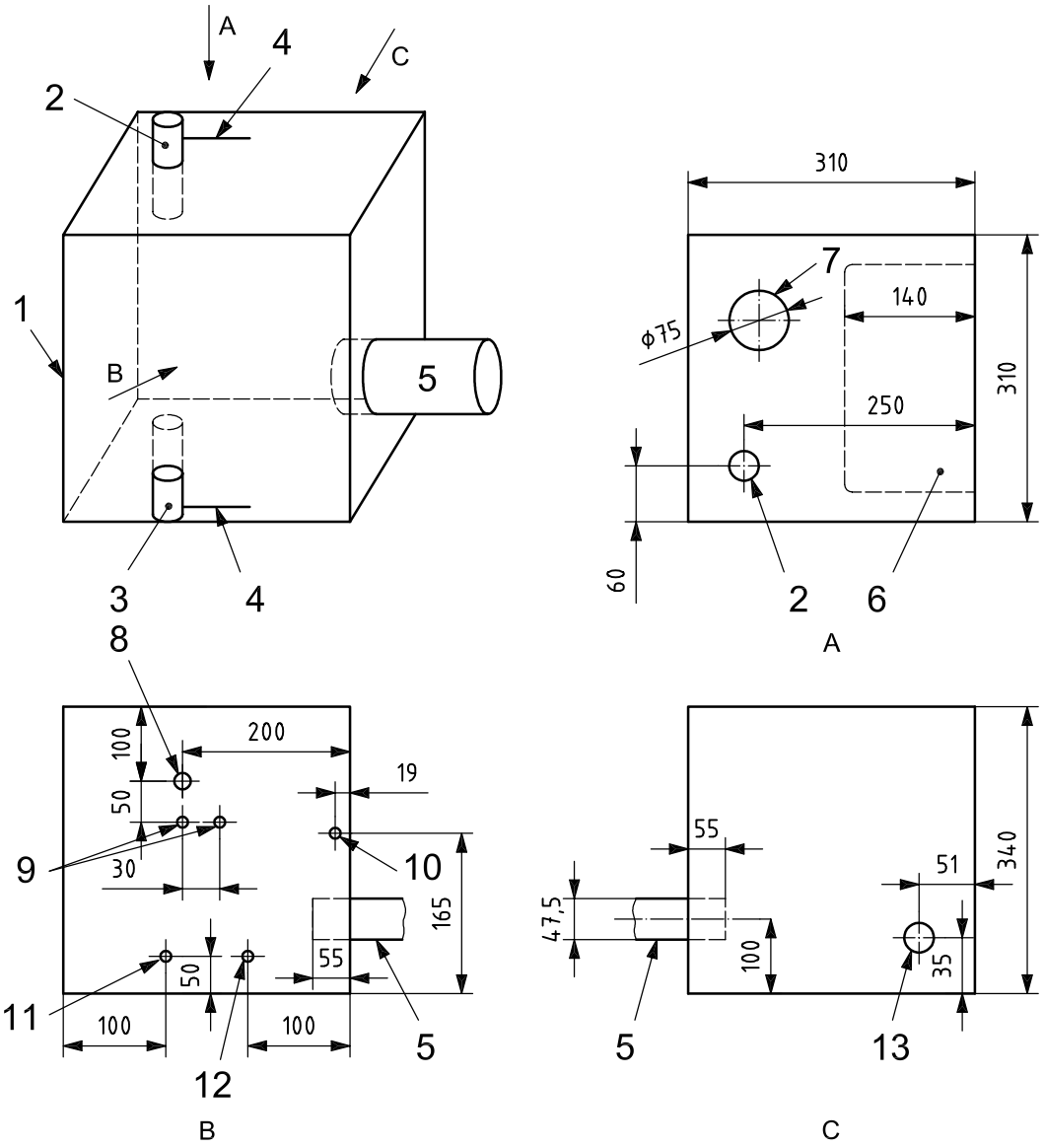
— total organic fraction;

— acid gases, including hydrogen cyanide, hydrogen chloride, hydrogen bromide, hydrogen fluoride, nitrogen oxides and sulfur dioxide if the presence of the relevant elements is suspected.

Other gases may need to be determined, if suspected from knowledge of the compound (see Note).

NOTE The above list is not to be considered inclusive. A compound may be excluded from the effluent analysis if one or more of the elements in that compound is determined not to be present in the test specimen, or if citable evidence shows the compound is not likely to be present in a toxicologically significant quantity. For more sophisticated analyses, other individual organic irritants (e.g. other organo-aldehydes, isocyanates, organo-nitriles, etc.) may be measured directly. The selection of organics to be monitored should be justified in the report and based on citable scientific literature on combustion-product analyses or elemental composition of the material tested.

Dimensions in millimetres



Key

1	door	8	smoke particle filter
2	tube containing photodetector	9	ports for sampling lines
3	tube containing light source	10	secondary air inlet
4	purge tubes for photodetector and light source	11	port for thermocouple
5	quartz furnace tube	12	port for tube to sample atmosphere in furnace tube for measurement of oxygen concentration
6	stainless-steel plate	13	exhaust port
7	safety blow-out panel		

A top
B front
C back

Figure 3 — Dimensions of mixing and measurement chamber

5.9 Determination of smoke

5.9.1 Aerosols and particulates

These are continuously sampled in the mixing and measurement chamber through a particle filter at an appropriate flow.

NOTE A glass microfibre filter 0,26 mm thick with a 1,6 µm particle retention characteristic and a diameter of 37 mm has been found to be suitable.

5.9.2 Optical density of smoke

The smoke optical density is calculated from measurement of the attenuation of a laser light beam by the combustion-product stream in the mixing chamber. Smoke obscuration is recorded continuously for the steady-state burn period of the test.

A suitable smoke-determining system is given in ISO 5660-2 (see Note).

Two glass neutral-density dispersion filters, accurately calibrated at the laser wavelength of 632,8 nm, are required to calibrate the smoke-determining system. The filters used shall not be of the coated type, because these filters can give rise to interference effects with laser light and can deteriorate with time. The filters shall have nominal optical densities (D) of 0,3 and 0,8. Corresponding values of extinction coefficient, k, are obtained from the formula:

$$k = (2{,}303D)L^{-1}$$

where L is the distance between the entrances of the light emitter/detector system.

NOTE Experimental work has been performed with ISO 5660-2 with systems using a white light source with collimating optics. Such systems have been shown to yield generally similar results, but not under all conditions. Theoretical predictions have been verified experimentally. White light systems may be used if they are shown to have an equivalent accuracy.

6 Establishment of air supplies

6.1 The primary and secondary air supplies to the apparatus shall be clean and free from excessive moisture that could interfere with burning characteristics or combustion-product analysis. The water content and/or the relative humidity of the air shall be reported (see Note 1).

6.2 Both the primary and secondary air flows are delivered at a constant, predetermined rate, positive pressure and monitored using in-line flow meters. Air flow rates must be calibrated at the point of entry to the furnace tube and chamber (see Note 2).

6.3 The primary air shall be introduced through the closure at the input end of the furnace tube.

6.4 The secondary air shall be introduced into the mixing box using piping of internal diameter 3 mm to 4 mm, passing through the wall of the mixing and measurement chamber and ending (70 ± 5) mm above and in line with the end of the furnace tube and pointing upwards at an angle of approximately 45°. The secondary air supply intercepts the rising plume to facilitate the efficient mixing of the test atmosphere (see Note 3).

NOTE 1 Oil free compressed air passed through a carbon trap and silica gel, or bottled air, has been found to be suitable.

NOTE 2 The flow meters shall be calibrated using a bubble meter. A correction for back pressure at the in-line flow meters may be necessary.

NOTE 3 This system will give good mixing of the furnace effluent and the secondary air, and removes the need for a mechanical stirring device.

7 Establishment of furnace temperature and setting of furnace temperature

7.1 General

There are two stages included in the temperature standardization. The first stage is to establish that the temperature profile (change of temperature with distance through the furnace tube) of the particular furnace to be used is suitable; the second stage is to determine the temperature setting needed for the particular experimental-run condition to be carried out.

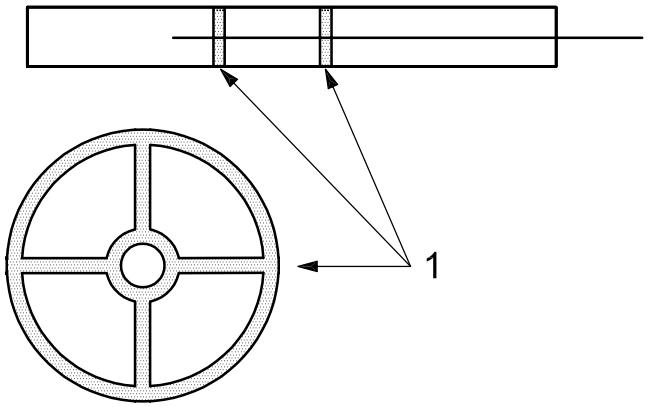
7.2 Establishing furnace temperature profile to determine furnace suitability

Set up the furnace, with an empty quartz furnace tube in place, under static conditions (i.e. with no air flow through the furnace tube). Close the furnace tube at one end with a bung to prevent air flow through the furnace and set the furnace temperature controller at 680 °C. Introduce the calibrated thermocouple (5.3) into the quartz furnace tube, with the tip of the thermocouple in air within a 10 mm radius of the centre of the quartz furnace tube (see Note).

Allow the furnace to reach equilibrium. Then measure the temperature profile along the furnace tube by taking measurements at intervals of no greater than 25 mm to find the point of maximum temperature. This should be near the centre of the furnace and the maximum temperature should be (650 ± 10) °C. If the maximum temperature is outside this range, adjust the furnace temperature controller to bring the maximum temperature into this range.

From the results obtained, determine the location of the point of maximum temperature and record the temperature at that point. Make further measurements also at intervals of no greater than 25 mm on each side of the location of the point of maximum temperature, until points are reached at which the temperature decrease relative to the maximum temperature exceeds 100 °C. For the furnace to be acceptable, these points should lie between 125 mm and 250 mm from the location of the point of maximum temperature.

NOTE A suitable support for the thermocouple is shown in Figure 4.



Key

1 position of thermocouple supports inside furnace tube to position thermocouple in centre of tube

Figure 4 — Wire thermocouple support rings allowing thermocouple to move along in required position

7.3 Setting the temperature for an individual experimental-run condition

Once it has been determined that the furnace temperature profile is suitable, the only temperature setting necessary for an experimental run is the maximum temperature under air flow conditions (T_{run}). In order to set the maximum temperature for an experimental run, set up the furnace with the quartz furnace tube in place and set up the appropriate primary air flow through the furnace tube. Introduce the calibrated thermocouple (5.3) into the quartz furnace tube as described in 8.2 and allow it to reach equilibrium. Once a primary air flow has been established, the point of maximum temperature moves downstream relative to its location under static conditions. In order to compensate for this shift, position the thermocouple tip (75 ± 10) mm downstream of the point of maximum temperature established under static conditions (see 8.2). This position introduces a minimum error for flow rates of up to 20 l·min^{-1}. Then adjust the furnace temperature controller until a temperature within ± 5 °C of the desired value for T_{run} is obtained.

NOTE It is necessary to specify the furnace conditions for the test. The conditions given above are based on experimental work using typical commercially available furnaces 500 to 600 mm long. If the furnace hot zone is too short then the test specimen and decomposition products might not be heated for a sufficient time. Hot zones longer than 600 mm are less likely to present problems, but it is possible that the longer period for which the test specimen and decomposition products are heated could result in small differences in the combustion-product yields. The above has been found to be satisfactory in use.

8 Test specimen preparation

8.1 The test specimen preferably should be in the form of a rod of uniform cross-sectional area. Details of the test specimen and its form shall be included in the test report (as described in 12.2) (see Note).

NOTE Test specimens may be in various forms depending upon the nature of the material. It has been shown that the form of the sample itself can have an effect on the results. The use of this apparatus is generally limited to homogeneous materials, and layered materials where the outer layers do not prevent involvement of the inner layers during the course of a fire.

8.2 The test specimen shall be uniformly distributed along the length of the sample boat, so that a constant flow of decomposition products is produced as the test specimen passes through the furnace. The specimen combustible loading should be approximately 25 mg·mm^{-1} (20 g spread over 800 mm). To give this loading, a specimen rod with a density of 1 g·ml^{-1} would have a cross-sectional area of 25 mm^2 (see next paragraph).

Specimen preparation may also have a minor effect on test results. This may occur with, for example, non-melting materials, cables, layered materials, etc., and it is important that details of the test specimen shall be reported. The results for non-melting, granular materials may be affected by the size of the granules.

In the case of materials that are subject to rapid flame spread or that might distort or shrink on introduction into the furnace, the test specimens may be divided into short lengths. These specimen forms are acceptable, providing the material is uniformly distributed along the length of the boat such that the specimen loading per unit length is known and so that the rate of decomposition can be determined.

Materials having densities below approximately 0,05 g·ml^{-1} can be so large that they interfere with the air flow through the furnace tube at a specimen loading of 25 mg·mm^{-1}. To overcome this problem, it is acceptable to reduce the specimen loading and increase the test-specimen-boat advance rate to compensate (see 5.6).

For materials which contain an inert matrix or fillers which do not form part of the combustible mass, the mass loading of material in the test specimen should be increased to compensate.

8.3 Before the test, specimens shall be conditioned to constant mass at a temperature of (23 ± 2) °C, and a relative humidity of (50 ± 5) %, in accordance with ISO 554.

Constant mass is considered to be reached when two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0,1 % of the mass of the test piece or 0,1 g, whichever is the greater (see next paragraph).

Materials such as polyamides, which require more than 1 week in a conditioning atmosphere to reach equilibrium, may be tested after conditioning in accordance with ISO 291. This period shall be not less than 1 week, and shall be described in the test report.

9 Selection of test decomposition conditions

9.1 Selection of decomposition conditions for fire hazard analysis or fire-safety engineering

Test specimens shall be decomposed or combusted under one or all of the conditions set forth below.

ISO/TS 19706:2004, Table 1 defines the various fire stages, 1b, 2, 3a and 3b. For stage 2, the procedure provides an equivalence ratio ϕ of $< 0{,}75$. For stages 3a and 3b, the procedure provides a ϕ of $> 2{,}0 \pm 0{,}2$.

Preliminary test runs, according to the procedures of Clause 10, will need to be carried out to determine the test conditions for materials of unknown decomposition behaviour. It is essential to measure carbon dioxide and oxygen concentrations, mass loss and possibly smoke, to establish the conditions for steady-state decomposition according to the defined fire types. Guidance on the selection of additional decomposition conditions is presented in Annex A.

A test run is only valid if the selected steady-state conditions (see 10.1) are maintained for a period of at least 5 min during the test. If ignition occurs during a non-flaming run, or fails to occur during a flaming run, then the furnace temperature shall be raised or lowered in 25 °C steps until the required behaviour is obtained. A new test run shall then be carried out with a fresh test specimen. For flaming behaviour, it is also necessary to ensure that the primary air flow rates are correct, as specified in 9.3, 9.4 and 9.5.

9.2 Stage 1b: oxidative pyrolysis from externally applied radiation

9.2.1 Place a test-pecimen combustible loading of 25 mg·mm^{-1} in the specimen boat.

9.2.2 Set the furnace temperature to obtain a T_{run} of 350 °C.

9.2.3 Set the primary air flow to 2 l·min^{-1}.

9.2.4 If flaming decomposition occurs during the run, repeat at temperatures progressively 25 °C lower until continuous, non-flaming decomposition is obtained throughout the steady-state period.

9.3 Stage 2: well-ventilated flaming

9.3.1 Place a test-specimen combustible loading of 25 mg·mm^{-1} in the specimen boat.

9.3.2 Set the furnace temperature to obtain a T_{run} of 650 °C. Set the primary air flow rate to 10 l·min^{-1} and the secondary air flow rate to 40 l·min^{-1}.

9.3.3 Complete a test run as described in the procedure in 10.1.

9.3.4 From the average percent oxygen concentration in the mixing and measurement chamber (M_{O2}) calculated to 2 decimal places; calculate the oxygen depletion (D_{O2}) as follows:

$$D_{O2} = 20{,}95 - M_{O2}$$

If $D_{O2} < 3{,}14$ % and $> 1{,}8$ % then $\phi < 0{,}75$ and the run meets the criteria of this Technical Specification for well-ventilated flaming.

If $D_{O2} > 3{,}14$ % then $\phi > 0{,}75$ and the run is unacceptable. Repeat with a primary air flow rate of 15 l·min^{-1}. Under these conditions, $\phi < 0{,}75$ and the run meets the criteria of this Technical Specification for well-ventilated flaming.

If $D_{O2} > 1{,}5$ % and $< 1{,}8$ % then $\phi < 0{,}75$ but the combustible fuel content is too low to obtain reliable data. Repeat the run with a specimen mass loading of $\times$ 1,5.

9.3.5 If flaming decomposition does not occur or is intermittent during the run, repeat at temperatures progressively 25 °C higher, until continuous flaming decomposition is obtained throughout the steady-state period. If continuous flaming cannot be obtained, this shall be reported.

If $D_{O2} < 1{,}5$ % then $\phi < 0{,}75$ but the combustible fuel content is too low to obtain reliable data. Repeat the run with a specimen mass loading of $\times$ 2.

9.4 Stage 3a: small vitiated fires in closed or poorly ventilated compartments

9.4.1 Place a test-specimen combustible loading of 25 mg·mm^{-1} in the sample boat.

9.4.2 Set the furnace temperature to obtain a T_{run} of 650 °C.

9.4.3 Complete the test as defined in Clause 10.

9.4.4 From D_{O2} data (obtained from 9.3) calculate the primary air flow rate (P) as follows:

$$P = D_{O2} \times 1{,}193\ 3$$

This will provide a ϕ or 2,0 $\pm$ 0,2.

9.4.5 If flaming decomposition does not occur or is intermittent during the run, repeat at temperatures progressively 25 °C higher, until continuous flaming decomposition is obtained throughout the steady-state period. If continuous flaming cannot be obtained, this shall be reported.

9.5 Stage 3b: post-flashover fires in open compartments

As in 9.4, except that the furnace temperature is set at a T_{run} of 825 °C.

10 Procedure

10.1 Decomposition of the test sample

10.1.1 The furnace tube and boat shall be clean before each test (see Note). The mixing box shall be free of any loose material before each test. A blank test run carried out before each series of tests will determine the cleanliness of the apparatus.

NOTE A convenient method of cleaning both the boat and tube is to remove obvious residues mechanically, then fire at 1 000 °C, followed by washing in water to remove any inorganic residues.

10.1.2 Bring the tube furnace to the required temperature at the required primary air-flow rate in accordance with 9.2 – 9.5, as applicable.

10.1.3 Set the secondary air flow to provide a total air flow through the mixing chamber of 50 l·min^{-1} ($\pm$1 l·min^{-1}).

10.1.4 Calibrate the sampling and measurement equipment.

10.1.5 Introduce the sample boat containing a test specimen of known mass, prepared in accordance with Clause 8, into the furnace tube with the front end of the sample boat just outside the air inlet end of the tube-furnace entrance.

10.1.6 Start the experimental run by switching on the specimen-boat drive mechanism to introduce the sample boat containing the test specimen into the furnace at a rate of 40 $mm \cdot min^{-1}$ (see Note).

NOTE For some fast-burning and low-density materials, it has been found necessary to use advance rates of up to 60 $mm \cdot min^{-1}$. In this case, it may be necessary to change the specimen mass and air flow rates to maintain a constant fuel-mass/air-flow ratio. Depending upon the nature of the specimen, or where high primary air flow rates present other difficulties, it is permissible to use different primary air flow rates, provided the fuel-mass/air-flow ratio is maintained constant (e.g. 25 $mg \cdot mm^{-1}$ specimen loading at an air flow rate of 10 $l \cdot min^{-1}$ corresponds to 12,5 $mg \cdot mm^{-1}$ specimen loading at an air flow rate of 5 $l \cdot min^{-1}$).

10.1.7 Look inside the furnace tube to determine the presence or absence of flaming, to determine when ignition occurs and that flaming is continuous during flaming decomposition experiments, also to confirm that flaming does not occur during non-flaming decomposition experiments, see Note. The fire condition can also be verified from the gas analysis where flaming will be indicated by relatively high CO_2 values.

NOTE A convex mirror at the primary air inlet end of the furnace tube has been found useful for this.

10.1.8 Observe the recordings from the gas analysers and the smoke density monitor during the early stages of the run. When these have reached approximately constant levels, then dynamic steady-state conditions have been achieved. Record the time and begin chamber sampling at this point.

10.1.9 Continue to observe the test specimen and recordings from the gas monitors. The decomposition conditions need to remain approximately steady for a minimum of 5 min to enable the specimen decomposition behaviour and toxic product yields to be characterized because data for concentrations of all measured parameters are averaged over this steady-state period.

10.1.10 When the up-stream end of the sample boat enters the furnace tube, the run is completed. Switch off the sample boat drive and gas sampling systems, pumps, bubblers, etc. Immediately withdraw the sample boat to its starting location in the furnace tube and extinguish any flame by temporarily interrupting the primary air flow.

10.1.11 When the test specimen and boat have cooled, remove from the furnace tube and weigh according to 10.2.3. Store in a desiccator and reweigh when the boat and contents have reached constant mass.

10.1.12 The steady-state period shall be used as the basis for calculating results. If flaming combustion cannot be obtained, or where only intermittent flaming occurs, these should be reported. The results of the tests are not valid in terms of the specified fire types 1, 2, 3a or 3b, if steady-state conditions are not obtained for a minimum of 5 min (see last paragraph).

During dynamic steady-state conditions, the concentrations of carbon dioxide and oxygen in the mixing and measurement chamber should remain approximately constant, without any long-term trend (greater than $\pm$ 10 %) or short-term fluctuations (greater than $\pm$ 20 %). Carbon monoxide (CO) and smoke concentrations may be somewhat more variable in some cases. If larger fluctuations occur then this should be reported. If steady-state conditions are not maintained for at least 5 min then it may be necessary to use a longer furnace boat, or reduce the rate of introduction of the furnace boat to increase the duration of the run. A 20 min run has been found to provide a steady-state period of at least 10 min duration for most materials. The oxygen depletion shall remain approximately constant, that is within 3 % O_2 during the steady-state period.

Where difficulties are encountered in obtaining steady flaming conditions, these can often be overcome by varying the fuel load or decomposition temperature. If ignition occurs during a non-flaming run, or fails to occur during a flaming run, then the furnace temperature should be raised or lowered in 25 °C steps until the required behaviour is obtained. A new test run should then be carried out with a fresh test specimen. For flaming behaviour, it is also necessary to ensure that the primary air flow rates are correct for one of the three states: well-ventilated flaming, small vitiated or post-flashover vitiated. The important consideration is that there is excess oxygen during well-ventilated flaming, and a fuel-rich system for vitiated flaming. This is achieved by following the procedure in this Clause.

10.2 Sampling and analysis of fire effluent and measurement of smoke density

10.2.1 General

The concentrations of all effluent components (gases and respirable aerosols) that contribute significantly to the computation of an LC_{50} or IC_{50} shall be measured throughout the steady-state test period. The following shall be measured:

— carbon monoxide;

— carbon dioxide;

— oxygen (consumed);

— hydrogen cyanide (if the presence of nitrogen is suspected);

— hydrogen chloride (if the presence of chlorine is suspected);

— hydrogen bromide (if the presence of bromine is suspected);

— hydrogen fluoride (if the presence of fluorine is suspected);

— smoke optical density;

— sulfur dioxide (if the presence of sulfur is suspected);

— oxides of nitrogen (if the presence of nitrogen is suspected);

— formaldehyde;

— acrolein;

— total organic content shall also be reported (expressed as total hydrocarbons or mass of effluent carbon in organic form);

— total particulates;

— smoke optical density.

NOTE 1 This list is not to be considered inclusive. A compound may be excluded from the effluent analysis if one or more of the elements in that compound is determined not to be present in the test specimen or if documented experience shows that the compound is not likely to be present in a toxicologically significant quantity. For more sophisticated analyses, other individual organic irritants (e.g. other organo-aldehydes, isocyanates, organonitriles, inorganic phosphates, etc.) may be measured directly. The selection of organics to be monitored should be justified in the report and based on citable scientific literature on combustion-product analyses or elemental composition of the material tested.

NOTE 2 Clause 7 of ISO/TS 19706:2004 provides guidance regarding the potential import of unmeasured components.

NOTE 3 ISO 19701 and ISO 19702 describe analytical chemical procedures for measurement of effluent gases and aerosols.

10.2.2 Sampling of fire effluent

The fire effluent shall be sampled continuously or at intervals of no more than 30 s from the mixing and measurement chamber. The total extracted flow shall not exceed approximately 5 $l \cdot min^{-1}$ but, if additional samples are required, they shall be taken from the fire effluent exit nearest to the mixing chamber. ISO 19701 provides information on the sampling of fire effluent.

10.2.2.1 Sampling and analysis of permanent gases

A continuous flow of approximately 2 l·min^{-1} for the analysis of O_2, CO, and CO_2 shall pass through a suitable drying agent and a smoke filtration system *en route* to the appropriate analysers. The concentrations of these gases shall be determined continuously.

NOTE Details of a suitable filtration and drying system and analysers for these are given in ISO 5660-1.

10.2.2.2 Sampling and analysis of acid gases

The following acid gases shall be determined, unless eliminated by elemental analysis or citable knowledge:

HCN, HCl, HBr, HF, NO_x, SO_2.

These gases may be determined by the following methods or by Fourier transform infra-red analysis (FTIR), see ISO 19702.

Pass a continuous sample of the fire effluent at an appropriate flow rate (see Note 1) through two gas bubblers placed in series (Note 2), each containing an appropriate volume of the appropriate absorbent (see Note 3). The sampling shall be carried out at a constant rate throughout the period during which dynamic steady-state conditions are maintained (Note 3).

NOTE 1 Calibrate the flow rate using a bubble meter (see ISO 19701).

NOTE 2 A flow rate of 1 l·min^{-1} passing through two 250 ml Dreschel bottles fitted with sintered glass of porosity zero, each containing 150 ml of absorbing solution.

NOTE 3 0,1 M aqueous sodium hydroxide solution has been found suitable for the acid gases listed above, except for SO_2 which should be passed through 3 % aqueous hydrogen peroxide solution and NO_x which needs to be determined by a dedicated method. Details are given in ISO 19701.

10.2.2.3 Sampling and analysis of organic gases

10.2.2.3.1 Specific organic gases

A large number of organic irritant gases can be produced. Of these, formaldehyde and acrolein are strong irritants and should be determined (see ISO 19701 and Note 1 of 10.2.1).

10.2.2.3.2 Total organic content

Since the organic fraction of the effluent contains many irritant species, it is important to obtain some indication of the organic fraction of the effluent and, if appropriate, the concentrations and yields of individual organic compounds (see Note 1).

NOTE 1 A total hydrocarbon analysis does not give rise to an accurate measure of the potency of the organics because the organic fraction of the smoke contains many irritant species. The acute toxic potency of non-oxidized organics is relatively low compared to that of the partially oxidized compounds, but the sum of the latter concentrations are generally much smaller than the sum of the former concentrations. This determination does not indicate organo-irritancy but critically does indicate the fraction of gases not determined and allows carbon balances to be established.

The minimum requirement is for an estimate of the total organic content of the effluent (see 5.8 and 10.2.1).

One of the following methods shall be used to measure the organic content of the effluent.

a) A total hydrocarbon analyser shall be used to obtain an approximate estimate of the total hydrocarbon content of the effluent.

NOTE 2 A limitation of this method is that, since the identities of the organic compounds in the effluent are unknown, the appropriate response factors are also unknown.

b) The fraction of carbon in the effluent in the form of organic carbon shall be determined by oxidizing a sample of the effluent from the mixing and measurement chamber and measuring the CO_2 concentration. The organic fraction shall then be determined by comparing the CO_2 concentration in the oxidized sample with the concentrations of CO_2, CO and soot particulates in the mixing and measurement chamber, in accordance with 11.5.

c) For test specimens with a known composition, the organic fraction shall be estimated approximately from the CO_2, CO and soot particulates in the mixing and measurement chamber, and the estimated carbon-mass-loss concentration from the test specimen.

d) Direct measurements shall be made of a range of individual organic species. This shall include irritants (e.g. acrolein and formaldehyde), and may also include other toxic organic species (e.g. benzene) or environmental pollutants (e.g. dibenzodioxins or dibenzofurans).

10.2.2.4 Sampling of aerosols and particulates

Sample the fire effluent in the mixing and measurement chamber continuously through particulate filters at appropriate flow rates using the same procedure as in 10.2.3. Weigh the filter before sampling and within 10 min after sampling, and also after conditioning to constant mass (to minimize the effects of condensed moisture), to enable calculation of the mass of particulate deposited.

10.2.2.5 Measurement of smoke density

The reduction, expressed as a percentage, in the intensity of light due to its passage through smoke, as determined by the output from the photo-detector, shall be recorded continuously.

10.2.3 Determination of the mass of the specimen residue

At the end of the run and when the sample boat has cooled sufficiently, remove it from the entrance of the furnace tube and store in a desiccator to achieve constant mass.

Inspect the residue and measure the length of the uniformly decomposed region from the up-stream end of the boat. Remove the uniformly decomposed portion of the residue, weigh it and calculate the residue mass per millimetre.

NOTE This represents the part of the test specimen that has been decomposed, and has evolved the products measured in 10.2.

Remove the uniformly decomposed portion of the residue, weigh it and calculate the mass of residue per millimetre.

10.3 Validity of test run

A test run is only valid if the selected steady-state conditions (see Clause 9 and 10.1) are maintained for a period of at least 5 min during the test. If ignition occurs during a non-flaming run, or fails to occur during a flaming run, then the furnace temperature should be raised or lowered in 25 °C steps until the required behaviour is obtained. A new test run should then be carried out with a fresh test specimen. For flaming behaviour it is also necessary to ensure that the primary air flow rates are correct as specified in 9.3, 9.4, and 9.5.

11 Calculations

11.1 General

The calculated values are averages over the duration of the steady-state part of the test. This period is defined in terms of the concentrations of carbon dioxide, carbon monoxide and oxygen (see 9.1). Steady consumption of O_2 and production of CO_2 is taken to be indicative of steady production of all volatile products.

The results shall be calculated to 2 significant figures.

11.2 Mass-charge concentration and mass-loss concentration

11.2.1 Mass-charge concentration

Calculate the mass-charge concentration, $C_{\text{m.charge}}$, in grams per cubic metre ($g \cdot m^{-3}$), from the following Equation:

$$C_{\text{m.charge}} = \frac{\dot{m}}{\dot{a}}$$

where

$\dot{m}$ is the rate of introduction of the test specimen mass into the furnace, in milligrams per minute ($mg \cdot min^{-1}$);

$\dot{a}$ is the total air flow rate through the mixing and measurement chamber, in litres per minute ($l \cdot min^{-1}$) (i.e. 50 $l \cdot min^{-1}$).

11.2.2 Mass-loss concentration

a) Calculate the mass-loss concentration as follows.

Calculate the mass loss per unit length, m_{loss}, in milligrams per millimetre ($mg \cdot mm^{-1}$), from the following Equation:

$$m_{\text{loss}} = m_{\text{load}} - m_{\text{res}}$$

where

m_{load} is the test-specimen mass loading, in milligrams per millimetre ($mg \cdot mm^{-1}$);

m_{res} is the test-specimen residue mass, in milligrams per millimetre ($mg \cdot mm^{-1}$).

b) Calculate the mass-loss rate, $\dot{m}_{\text{loss}}$, in milligrams per minute ($mg \cdot min^{-1}$) from the following Equation:

$$\dot{m}_{\text{loss}} = m_{\text{loss}} \times \dot{b}$$

where $\dot{b}$ is the test-specimen-boat advance rate, in millimetres per minute ($mm \cdot min^{-1}$).

c) Calculate the mass-loss concentration, $C_{\text{m.loss}}$, in grams per cubic metre ($g \cdot m^{-3}$), from the following Equation:

$$C_{\text{m.loss}} = \frac{\dot{m}_{\text{loss}}}{\dot{a}}$$

where $\dot{a}$ is as given in 11.2.1.

11.3 Smoke density

The smoke density is reported as the smoke extinction coefficient, k, and the smoke specific extinction area, σ_f. which are calculated as follows.

Calculate the smoke extinction coefficient, k, in reciprocal metres (m^{-1}) from the following Equation:

$$k = \frac{1}{L} \ln\left(\frac{I_o}{I}\right)$$

where

I_o is the intensity of a beam of parallel light rays, in candelas (cd) measured in a smoke-free environment with a photodetector having the same spectral sensitivity as the human eye;

I is the intensity of the same beam of parallel light rays, in candelas (cd), measured after traversing the environment containing the smoke;

L is the length of the beam of light that has traversed the environment containing the smoke, in metres (m).

Calculate the smoke specific extinction area, σ_f, in metres squared per gram ($m^2 \cdot g^{-1}$), from the following Equation:

$$\sigma_f = \frac{k}{C_{m.loss}}$$

where

k is the smoke extinction coefficient, in reciprocal metres (m^{-1});

$C_{m.loss}$ is the mass-loss concentration of the test specimen calculated in accordance with 11.2.2, in grams per cubic metre ($g \cdot m^{-3}$).

11.4 Yield

Calculate the yield of each effluent component, Y, (dimensionless) from the following Equation:

$$Y = (M/V_m) \times (F_v/C_{m.loss}) \times 10$$

where

M is the molar mass of the component, in grams per mole ($g \cdot mol^{-1}$);

V_m is the molar volume of the component at 20 °C temperature and 101,325 kPa atmospheric pressure, assuming that it behaves as an ideal gas, in litres per mole ($l \cdot mol^{-1}$), i.e. 24,055 $l \cdot mol^{-1}$;

F_v is the measured volume fraction of the component in the mixing and measurement chamber, as a percentage;

$C_{m.loss}$ is the mass-loss concentration, calculated in accordance with 11.2.2, in grams per cubic metre ($g \cdot m^{-3}$).

To aid calculations, values of the molar mass factor (M/V_m) are given in Table 1 for a number of effluent components.

Table 1 — Values of (M/V_m)

Component	Molar mass $g \cdot mol^{-1}$	(M/V_m) $g \cdot l^{-1}$
CO_2	44,01	1,830
CO	28,01	1,164
HCN	27,03	1,124
NO_2	46,01	1,913
NO	30,01	1,248
HCl	36,46	1,516
HBr	80,91	3,364
HF	20,01	0,832
SO_2	64,06	2,663
Acrolein (C_3H_4O)	56,06	2,331
Formaldehyde (CH_2O)	30,03	1,248

NOTE Another yield concept that may be used is that of the "volume yield". The volume yield of an effluent is the volume of the component (at 20 °C and 101,325 kPa pressure) divided by the mass-loss concentration of the test specimen associated with the production of that volume of the component. The volume yield, X, in litres per gram ($l \cdot g^{-1}$) can be calculated from the following Equation:

$$X = F_v \times 10^{-3} / C_{m,loss}$$

11.5 Organic fraction

Where the fraction of carbon in the effluent in the form of organic carbon, $F_{C.org}$, is to be determined [see 10.2.2.3.2b)], this shall be calculated using the following Equation:

$$F_{C.org} = \frac{[CO_2]_{oxidized} - \left([CO_2]_{chamber} + [CO]_{chamber} + [soot]_{chamber} \right)}{[CO_2]_{oxidized}}$$

where

$F_{C.org}$ is the fraction of the effluent carbon in the form of organic carbon;

$[CO_2]_{oxidized}$ is the volume fraction of CO_2, as a percentage, in the oxidized sample;

$[CO_2]_{chamber}$ is the volume fraction of CO_2, as a percentage, in the mixing and measurement chamber;

$[CO]_{chamber}$ is the volume fraction of CO, as a percentage, in the mixing and measurement chamber;

$[soot]_{chamber}$ is the soot concentration [measured in grams per cubic metre ($g \cdot m^{-3}$)] expressed as an equivalent volume fraction of CO_2, as a percentage, in the mixing and measurement chamber (assuming that soot is 100 % carbon).

NOTE Where the carbon content of the mass loss can be estimated from the known composition of the test specimen, it is possible to make an approximation of the fraction of effluent carbon in the form of organic carbon by calculating the theoretical CO_2 concentration that would be formed in the mixing and measurement chamber, if all the specimen mass loss was oxidized to CO_2. This can then be substituted for $[CO_2]_{oxidized}$ in the above equation.

12 Test report

The test report shall be as comprehensive as possible and shall include any observations made during the test and comments on any difficulties experienced during testing. The units for all measurements shall be clearly stated in the test report.

The test report shall contain the information given in 12.1 to 12.3.

12.1 Name and address of the testing laboratory, names of responsible persons at the testing laboratory, test identification and date; laboratory ambient conditions (temperature and humidity).

12.2 Description of the test specimen, including details of its preparation and (if relevant) its configuration and condition relative to the end use of the product being examined. This shall include details concerning its homogeneity, layering (e.g. laminations), whether solid, cellular or granular, and other structural aspects.

12.3 The test report shall include at least the following information for data obtained during the steady-state decomposition period:

a) the decomposition conditions [fire stage 1, 2, 3a) or 3b)];

b) the run temperature (T_{run}), in degrees Celsius (°C);

c) the primary air flow rate, in litres per minute ($l \cdot min^{-1}$);

d) the burning behaviour (flaming/non-flaming) and stability (see 10.1);

e) the mass-charge concentration and mass-loss concentration of the test specimen;

f) the mean concentration of each component of the effluent (as listed in 10.2.1);

g) the mean yield of each component of the effluent, calculated in accordance with 11.4;

h) the mean smoke extinction coefficient and the smoke-specific extinction area of the smoke in the mixing and measurement chamber;

i) the methods used to determine the effluent yields;

j) the report shall contain the following statement.

> “The following test results relate only to the toxic gases and smoke generated from the test specimen under the particular conditions of the test stated: they are intended for use as an input for calculations of toxic potency using, for example, ISO 13344 and as an input to fire hazard assessment of toxic potency using, for example ISO 13571. They are not intended as a means of assessing the full potential fire hazards of the material or product in their end use, and the interpretation and application of these test data require care.”

13 Repeatability and reproducibility

13.1 Repeatability

Standard deviations and 95 % limits for PMMA tests for fire types 3b and 2 are shown in the following table for tests within one laboratory.

Material **PMMA**
Fire type **Developed vitiated, type 3b**
Test parameters
Temperature **825 °C**
Air flow **2,7 l/min^{-1}**
Equivalence ratio **3,3**

Test results	CO_2 yield g/g	CO yield g/g	Smoke yield m^2g^{-1}	Combustion mass concentration g dm^{-3}
No. of tests	5	5	5	5
Mean	0,34	0,13	0,047	0,38
Standard deviation	0,016	0,003 2	0,007 1	0,020
Relative standard deviation, %	4,7	2,6	15,2	5,2
± 95 % limits	0,032	0,006 4	0,014	0,039

Material **PMMA**
Fire type **Well-ventilated type 2**
Test parameters
Temperature **650 °C**
Air flow **22,6 l/min^{-1}**
Equivalence ratio **0,4**

Test results	CO_2 yield g/g	CO yield g/g	Smoke yield m^2g^{-1}	Combustion mass concentration g dm^{-3}
No of tests	5	5	5	5
Mean	1,36	0,0082	0,061	0,058
Standard deviation	0,055	0,000 9	0,004 9	0,003 0
RSD %	4,0	11,3	8,0	5,1
± 95 % limits	0,11	0,001 8	0,01	0,005 9

13.2 Reproducibility

The reproducibility of test data has not yet been fully quantified. A statement is in preparation and will be incorporated into this Technical Specification after the compilation of suitable test data.

13.3 Accuracy

Accuracy is the relationship between tube furnace and real-scale fire tests.

The tube furnace has been shown to be capable of obtaining both stable non-flaming and flaming decomposition conditions. For flaming decomposition conditions, it has been shown to perform over a wide range of fuel/air ratios typical of different types of fires (references [1] to [3]). A more specific form of validation is to compare the yields of key toxic products, such as CO, obtained in the tube furnace with those obtained in large-scale and full-scale compartment fire tests. Comparison of the yields of CO and other products has shown a good agreement with those obtained in large-scale compartment fire tests (reference [1]).

Annex A
(informative)

Guidance on choice of additional decomposition conditions

Since the yields of products in fires depend upon the decomposition conditions (references [1] to [5]), it is possible to examine the relationships between product yield and a range of variables affecting the decomposition conditions using this apparatus and the methodology described (references [6] to [8]). The specified test conditions represent a minimum set designed to obtain data for oxidative pyrolysis under non-flaming conditions, for well-ventilated flaming conditions at an equivalence ratio of less than 0,75 and for vitiated flaming conditions at an equivalence ratio of 2.

For a more comprehensive analysis of yields under non-flaming conditions, it is possible to vary the furnace temperature in separate runs over a range from low temperatures at which little or no thermal decomposition occurs, to a temperature at which flaming ignition occurs. By this means, product yields can be measured throughout the temperature range over which non-flaming oxidative decomposition is possible for a particular material.

For flaming decomposition conditions, the product yields for any specific material are affected mainly by the fuel/oxygen ratio under which the test is carried out (from which the equivalence ratio is calculated, see below). For any material, it is therefore possible to map the relationship between equivalence ratio and product yields. This then enables comparison of the behaviour of materials over the entire range of equivalence ratios over which flaming decomposition can be obtained.

In full-scale fires, the equivalence ratios, and hence the product yields and concentrations in the fire compartment, change as the fire develops. Data on the effects of equivalence ratio on product yields for materials can therefore be useful in calculations of time-concentration curves for products from those materials involved in full-scale fires.

For any tube-furnace test run, the equivalence ratio ϕ is given by the mass-loss rate of combustible effluent from the material under test, in milligrams per minute ($mg \cdot min^{-1}$), divided by the mass flow rate of oxygen in the primary air introduced into the furnace, in milligrams per minute ($mg \cdot min^{-1}$), relative to the stoichiometric fuel mass to oxygen mass ratio for the material under test, i.e.:

$$\phi = \frac{\dot{m}_{\text{loss}} \times \Psi_{\text{O}}}{O}$$

where

$\dot{m}_{\text{loss}}$ is the mass-loss rate of the test specimen, in milligrams per minute ($mg \cdot min^{-1}$) calculated in accordance with 11.2.2b);

Ψ_{O} is the stoichiometric oxygen mass to fuel mass ratio;

O is the oxygen supply rate, in milligrams per minute ($mg \cdot min^{-1}$), given by the following Equation;

$$O = P \times 0{,}209\,5 \times 1\,330$$

where

P is the primary air flow rate, in litres per min ($l \cdot min^{-1}$);

1 330 is a factor to convert the volume of oxygen to mass of oxygen at 20 °C.

The stoichiometric oxygen mass to fuel mass ratio, Ψ_O (also known as the stoichiometric oxygen demand of the material) can be obtained in one of three ways, depending upon the information available on the test material, as follows:

a) from the elemental composition or empirical formula for the test material;

b) from the net heat of complete combustion for the test material (ΔH_T).

It has been empirically determined that when a material burns, for every gram of oxygen consumed, the heat released is approximately 13,1 kJ. Thus, if the net heat of complete combustion for the test material (ΔH_T) is known (e.g. as measured by bomb calorimetry), the stoichiometric oxygen mass to fuel mass ratio, Ψ_O, for the particular test material can be calculated from the following Equation:

$$\Psi_O = (\Delta H_T)/13{,}1$$

c) from the oxygen depletion in the mixing and measurement chamber and the mass-loss concentration of the test specimen during a well-ventilated tube-furnace test run, the stoichiometric oxygen mass to fuel mass ratio, Ψ_O, can be calculated from the following Equation:

$$\Psi_O = \frac{D_{O2} \times 1\,330}{C_{m.loss}}$$

where

D_{O2} is the oxygen depletion in the mixing and measurement chamber, as a volume fraction (see 9.3);

$C_{m.loss}$ is the mass-loss concentration of the test specimen, in grams per cubic metre ($g \cdot m^{-3}$), calculated in accordance with 11.2.2c).

Annex B
(informative)

Calculation of lethal toxic potency for combustion products according to ISO 13344 using tube-furnace data

The following procedure can be used in conjunction with ISO 13344 to calculate estimated LC_{50} and LCt_{50} concentrations for a material tested in the tube furnace under a specified thermal-decomposition condition:

a) Measure toxic gas concentrations and material mass-loss concentrations during a furnace run as described in this Technical Specification.

b) Calculate the fractional effective dose (FED) according to ISO 13344. This then represents the FED for the mass-loss concentration of the material specimen tested.

c) If the calculated FED is less than (or greater than) 1, calculate toxic gas concentrations for a greater (or smaller) material mass-loss concentration (for example, the gas concentrations for a mass-loss concentration double that used for the test would be twice the original).

d) Recalculate the FED for the new toxic gas mixture.

e) When the calculated $FED = 1$ then the mass-loss concentration represents the estimated LC_{50} concentration for the material under test. The estimated LCt_{50} concentration is given by the following Equation:

$$LCt_{50} = LC_{50} \times 30$$

Annex C
(informative)

Application of data from the tube-furnace test to assessment of toxic hazard in fires according to ISO 13571

The data from the tube-furnace test may be used as part of the information needed for toxic hazard assessment in a fire. Toxic hazard assessments include the following considerations:

a) the point in time during a fire by which a person will have been exposed to a concentration of smoke and irritants capable of impairing the efficiency of escape or causing incapacitation;

b) the point in time during a fire by which a person will have been exposed to a dose of asphyxiants capable of causing incapacitation and/or significant post-exposure health effects;

c) the point in time during a fire by which a person will have been exposed to a dose of lung irritants capable of causing post-exposure lung inflammation capable of causing long-term health effects or death.

There are a number of ways in which such assessments can be made as described in ISO 13571. Part of the data required for these assessments include either:

a) the lethal toxic potencies [mass loss LCt_{50} values (see Annex B)] for materials involved in the fire; or

b) the yields of individual toxic gases from materials involved in the fire.

For the first method, mass loss LCt_{50} data obtained from the tube-furnace test can be combined with mass-loss time-concentration curves for the fire, to calculate the time at which an exposed subject is predicted to have received a lethal exposure dose.

For the second method, the yield of toxic gases measured using the tube-furnace test can be combined with mass-loss time-concentration curves for the fire, to provide estimates of the time-concentration curves for individual toxic gases in the fire. These data can then be used with the fractional effective concentration (FEC), fractional effective dose (FED) and fractional lethal dose (FLD) methods described in ISO 13571, to estimate times to different incapacitation and lethal endpoints for exposed persons.

Annex D
(informative)

Guidance on application of data from the tube-furnace test to health and safety assessments of combustion-products

The data from the tube-furnace may be used in health and safety assessments for exposures to combustion products. For this application, it is necessary first to identify the material being decomposed and the decomposition conditions or fire type. The material is then decomposed using the tube-furnace method and the yields of toxic products are measured. Health and safety assessments usually involve situations for which long-term exposures or repeated short-term exposures occur. The range of combustion-products for which measurements are required will be considerably wider than those required for acute toxic hazards involved in fire survival.

From measurements of the yields of individual toxic combustion products from the material (or materials) and data on the expected mass-loss concentration in the workplace, it is possible to estimate potential workplace concentrations. Since combustion-product atmospheres always consist of mixtures of large numbers of toxic products, it is necessary to consider the overall toxicity of the mixed combustion products.

Guidance on occupational exposure limits for individual toxic substances is provided in document EH40 (HSE, UK)[9]. EH40 Clauses 38 to 49 provide guidance on the assessment of the overall toxic effects of mixtures.

Annex E
(informative)

Guidance on application of data from the tube-furnace tests to assessment of environmental hazards of combustion products from fires

Fires produce a range of pollutants capable of contaminating the indoor and outdoor environment, including both air contamination during the fire and contamination of water and solid surfaces by deposition. These include many of the substances of concern with regard to direct exposure during a fire, but also a further range of substances which may present a continued health hazard from effects on indoor air quality or from oral ingestion. The latter include substances such as polyaromatic hydrocabons, polyhalogenated biphenyls and halogenated dibenzodioxins and dibenzofurans. See PRI/26 PD[10]. As with other substances produced during fires, the yields of these substances can vary considerably with the combustion conditions (see reference [5]).

The tube-furnace method can therefore be used to identify and measure the yields of these substances for application to estimates of the production of environmental contaminants from fires. However, the analytical methods used to determine the fire effluent will need to be changed because the list of compounds affecting general assessment of environmental hazards of combustion products from fires, their relative importance, and the length of exposure are all likely to be different from the corresponding input to the acute exposure hazards considered in the main text of this Technical Specification.

Annex F
(informative)

Use of the tube-furnace method for bioassay purposes

The tube-furnace method was originally used for bioassay work, to examine the mechanisms whereby combustion products cause incapacitation during fires. Work with combustion-product atmospheres generated from a range of materials and with individual gases (CO, HCN, low O_2 and CO_2) established that incapacitation (asphyxiation) was caused by these gases and models were developed to enable these effects to be predicted from the chemical composition of the effluent atmospheres (see references [11] and [13]). A further important effect of exposure to fire effluent was found to be sensory irritation, and work was done to measure sensory irritancy for a range of effluent atmospheres (see references [14] and [15]).

It is possible that bioassay methods may still be required for the evaluation of toxic product evolution for certain special cases. The procedure described in this Technical Specification can be used for this purpose but minor modifications of the apparatus are required. Rodents can be exposed nose-only by placing them in rodent restrain tubes which can be inserted in ports in the mixing and measurement chamber. In order to obtain a 30 min exposure period, the sample advance rate can be slowed or a longer boat used. It is advantageous to obtain a “square wave” exposure profile for bioassay exposure, particularly when evaluating sensory irritancy. Two methods have been used to achieve this previously for the tube furnace. For one method, a small chamber was fitted on the side of a rectangular chamber similar to that described. Nose-only exposure tubes were fitted to the side chamber. The side chamber was flushed with air during a pre-exposure monitoring period. In order to start an exposure, a flap between the main and side chamber was lifted so that exposure to the steady-state atmosphere commenced.

Another chamber used for most biosassays consisted of a horizontally placed, low volume, cylindrical chamber with a door in the middle. The steady-state atmosphere was established in one-half of the chamber (the mixing chamber part). The bioassy pre-exposure conditions were set up in the other end. Nose-only exposure tubes were attached to the chamber which was flushed with air. To begin the exposure, the extract line was moved from the mixing chamber end to the bioassy end, so that the steady-state atmosphere was established in the bioassy end within a few seconds. The method is described in reference [14].

The concept of lethal toxic potency was derived originally from experiments on rats, on the assumption that an exposure dose of effluent lethal to rats is likely to be equally lethal to humans. The lethal toxic potency is expressed in terms of the exposure dose of effluent lethal to 50 % of exposed rats (LCt_{50}), which depends upon the concentration of toxic fire effluent to which the animals are exposed, multiplied by the exposure time (including a 14 day post-exposure period during which any additional deaths are scored). The concentration of toxic fire effluent can be expressed in a number of ways, but the most commonly used parameters are related to the material under test in terms of the mass-charge or mass-loss concentrations and the yields of individual toxic products.

Although estimates of toxic potency have been made primarily using animal exposures, it is now considered that, for most test specimens, reasonable estimates of toxic potency can be made, based upon chemical analytical measurements of the composition of fire effluent atmospheres. Therefore, the method described in the Technical Specification is intended principally for use with chemical analysis measurements.

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