TECHNICAL SPECIFICATION

ISO/TS 19700

First edition
2007-03-15

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

Reference number
ISO/TS 19700:2007(E)

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Published in Switzerland

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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:

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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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.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 µL/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 $\mu LL^{-1}min$ 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 ± 1) l·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 ± 5 mm.

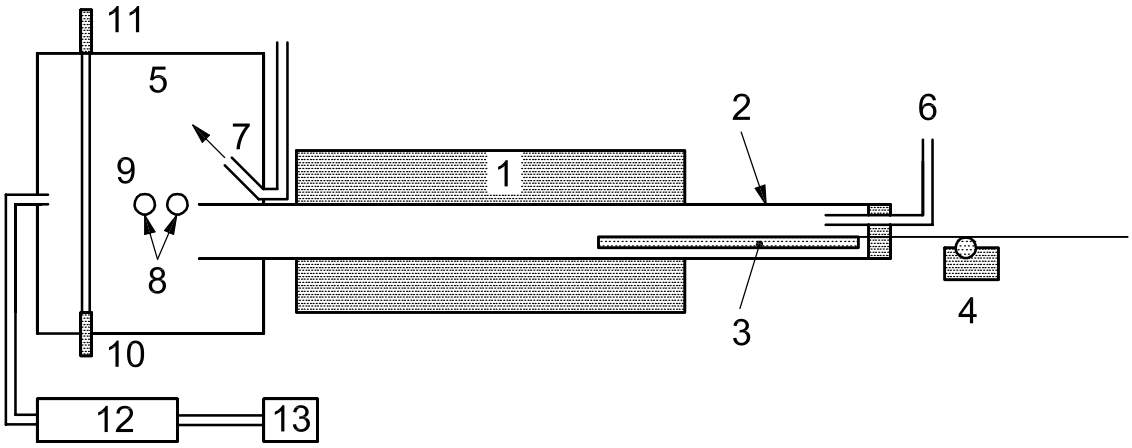
5.2 Tube furnace

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 ± 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 ± 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.

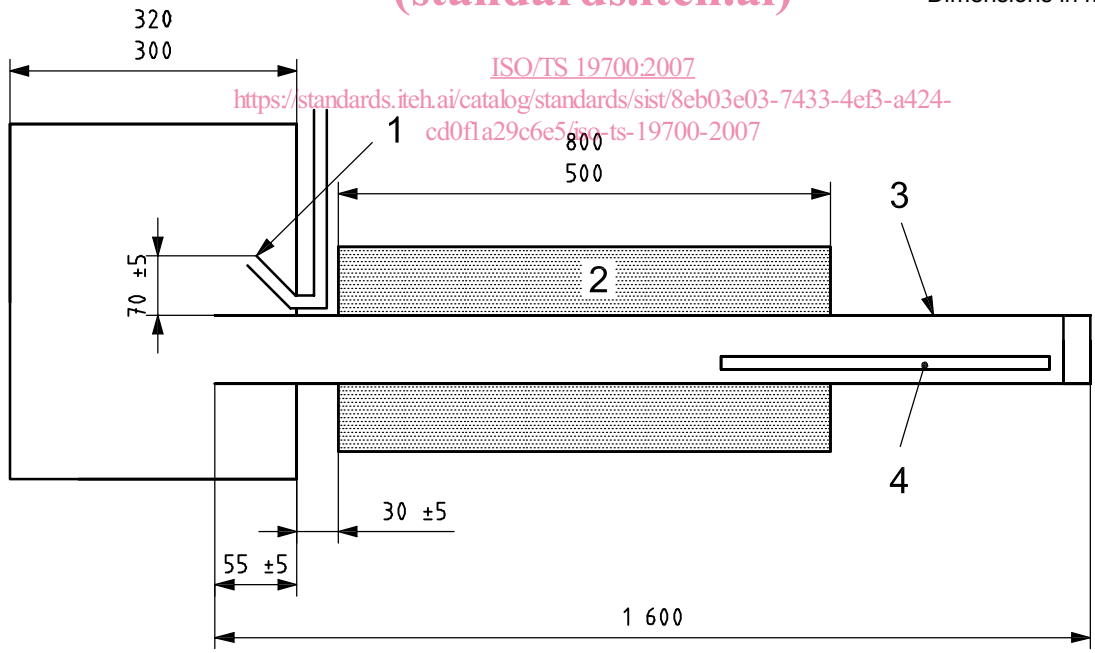


Key

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}.