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Plastics — Determination of burning behaviour by oxygen index —

Part 2:
Ambient-temperature test

Plastiques — Détermination du comportement au feu au moyen de l'indice d'oxygène —

Partie 2: Essai à la température ambiante

Reference number
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Contents

Foreword

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

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

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

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 4, *Burning behaviour*.

This second edition cancels and replaces the first edition (ISO 4589-2:1996), which has been technically revised. It also incorporates the Amendment ISO 4589-2:1996/Amd.1:2005.

A list of all parts in the ISO 4589 series can be found on the ISO website.

Introduction

Oxygen index (OI) results obtained using the methods described in this document can provide a sensitive measure of the burning characteristics of materials under certain controlled laboratory conditions, and hence may be useful for quality control purposes. The results obtained are dependent upon the shape, orientation and isolation of the test specimen and the conditions of ignition. For particular materials or applications, it may be necessary or appropriate to specify different test conditions. Results obtained from test specimens of differing thickness or by using different ignition procedures may not be comparable and no correlation with flammability behaviour under other fire conditions is implied.

Results obtained in accordance with this document are not applicable to describe or appraise the fire hazard presented by a particular material or shape under actual fire conditions, unless used as one element of a fire risk assessment that takes into account all of the factors pertinent to the assessment of the fire hazard of a particular application for the material.

For assessing the flame propagation properties of cellular materials of density < 100 kg/m^3, attention is drawn to the method described in ISO 3582.

Plastics — Determination of burning behaviour by oxygen index —

Part 2:
Ambient-temperature test

1 Scope

This document specifies methods for determining the minimum volume fraction of oxygen, in admixture with nitrogen, that will support combustion of small vertical test specimens under specified test conditions. The results are defined as oxygen index (OI) values.

Methods are provided for testing materials that are self-supporting in the form of vertical bars or sheets up to 10,5 mm thick. These methods are suitable for solid, laminated or cellular materials characterized by an apparent density 100 kg/m^3 or greater. The methods might also be applicable to some cellular materials having an apparent density of less than 100 kg/m^3. A method is provided for testing flexible sheets or film materials while supported vertically.

For comparative purposes, a procedure is provided for determining whether or not the OI of a material lies above some specified minimum value.

NOTE It might not be possible to apply these methods satisfactorily to materials that exhibit high levels of shrinkage when heated, e.g. highly oriented thin film.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291:2008, *Plastics — Standard atmospheres for conditioning and testing*

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 2859-2, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*

ISO 4589-1, *Plastics — Determination of burning behaviour by oxygen index — Part 1: General requirements*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 7823-1, *Plastics — Poly(methyl methacrylate) sheets — Types, dimensions and characteristics — Part 1: Cast sheets*

ISO 13943, *Fire safety –Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4589-1 and ISO 13943 apply.

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

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

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

4 Principle

A small test specimen is supported vertically in a mixture of oxygen and nitrogen flowing upwards through a transparent chimney. The upper end of the specimen is ignited and the subsequent burning behaviour of the specimen is observed to compare the period for which burning continues, or the length of specimen burned, with specified limits for such burning. By testing a series of specimens in different volume fractions of oxygen, the OI is estimated (see 8.7).

Alternatively, for comparison with a specified OI, three test specimens are tested using the relevant volume fraction of oxygen, at least two of which are required to given an "O" response (see 8.4) in order to meet the specified OI.

5 Apparatus

5.1 Test chimney

The test chimney shall consist of a heat-resistant glass tube supported vertically on a base through which oxygen-containing gas mixtures can be introduced (see Figure 1).

The recommended dimensions of the chimney are 450 mm to 500 mm height and 75 mm to 100 mm inside diameter.

The upper outlet shall be restricted as necessary by an overhead cap having an outlet small enough to produce an exhaust velocity of at least 90 mm/s from that outlet. The flow rate shall be calculated by using the formula found in A.2.

NOTE 1 Measurement of flow rate or flow speed at the specimen position helps for checking gas leaks.

NOTE 2 A cap converging to an outlet of 40 mm diameter at a level at least 10 mm above the top of the cylindrical chimney has been found satisfactory.

Chimneys of other dimensions, with or without restricted outlets, are suitable for use, if shown to give equivalent results. The bottom of the chimney, or the base upon which the chimney is supported, shall incorporate a device for evenly distributing the gas mixture entering the chimney. The preferred device comprises a diffuser and a mixing chamber with metal foil (honeycomb) or glass beads. Other devices, such as radial manifolds are suitable for use, if shown to give equivalent results. The mounting of a porous screen below the level of the specimen holder is helpful to prevent falling combustion debris from fouling the gas entry and distribution paths. One option is to construct the chimney in such a way that it can be divided in half, so as to make the setting of samples and cleaning easier.

The use of a levelling device and indicator incorporated within the chimney support will aid vertical alignment of the chimney and of a test specimen supported therein. If a dark background is provided, this will aid the observation of flames within the chimney.

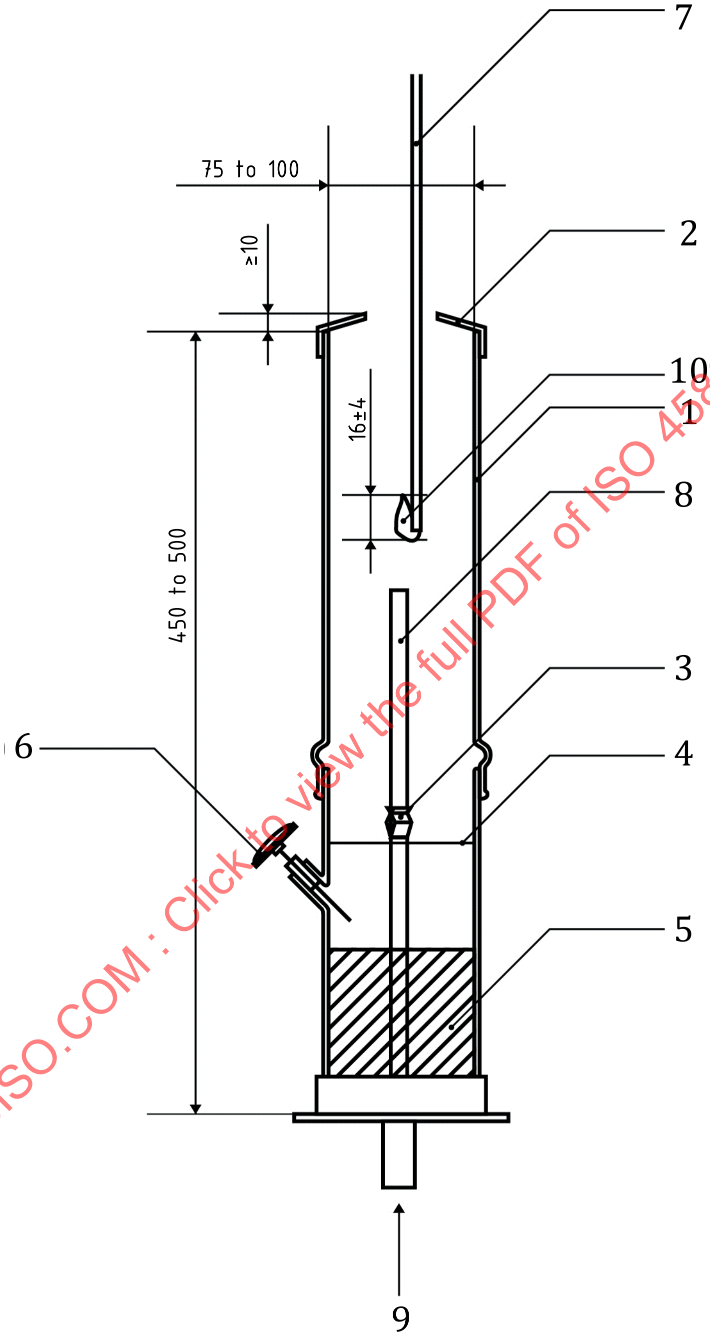
5.2 Test specimen holder

The specimen holder shall be suitable for supporting a specimen vertically in the centre of the chimney.

For self-supporting materials, the specimen shall be held by a small clamp, which is at least 15 mm away from the nearest point at which it is possible that the specimen will burn before the extent-of-burning criterion is exceeded. For supported film or sheet test specimens, the specimen shall be supported by both vertical edges in a frame equivalent to that illustrated by Figure 6, with reference marks at 20 mm and 100 mm below the top of the frame.

It is recommended that the profile of the holder and its support is smooth to minimize induction of turbulence in the rising flow gas.

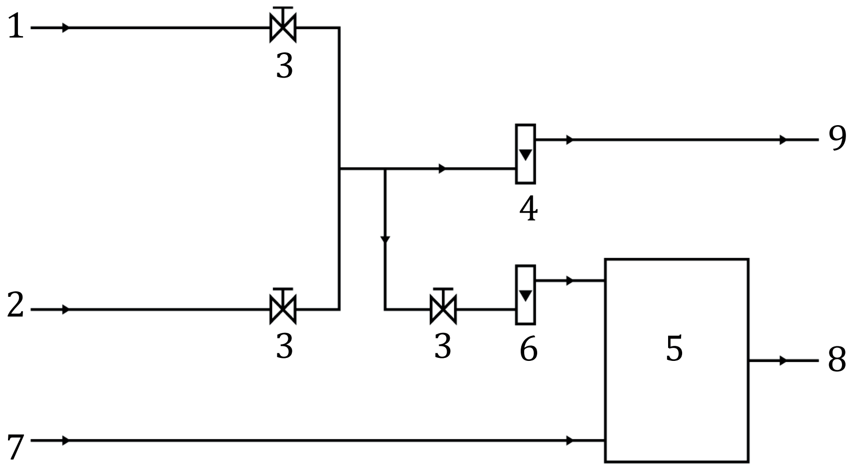
Dimensions in millimetres



Key

1	chimney	6	optional temperature measurement device
2	chimney cap	7	tube
3	specimen holder	8	test specimen
4	wire-mesh debris screen	9	oxygen/nitrogen mixture
5	diffuser and a mixing chamber	10	flame igniter

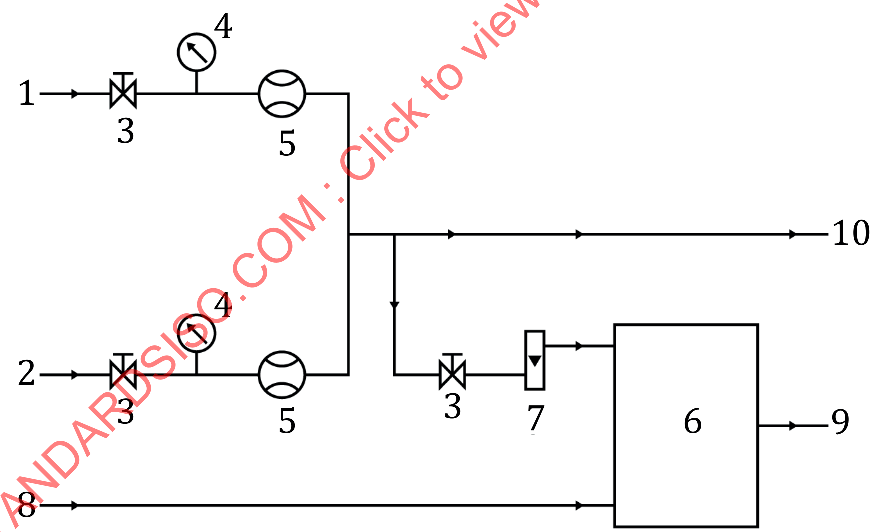
Figure 1 — Typical apparatus for determination of oxygen index



Key

1	oxygen	6	flow meter
2	nitrogen	7	reference gas
3	needle valve	8	to exhaust
4	calibrated flow meter	9	to chimney
5	oxygen analyser		

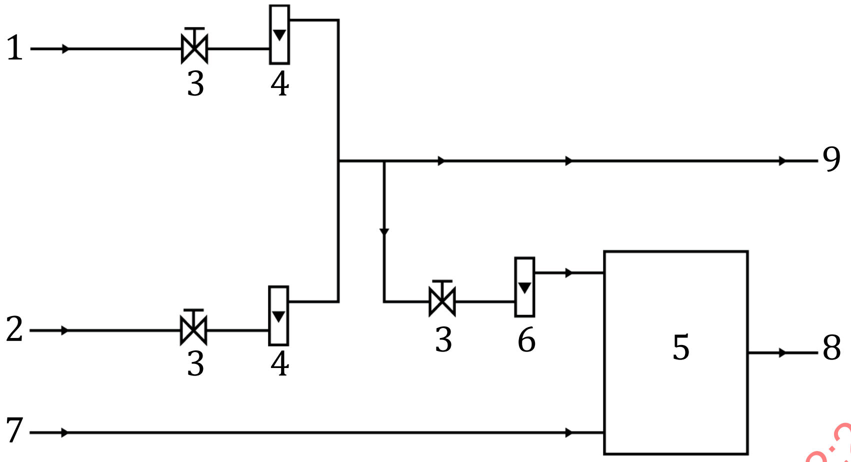
Figure 2 — Typical flow system incorporating the elements described in 5.4, a)



Key

1	oxygen	6	oxygen analyser
2	nitrogen	7	flow meter
3	needle valve	8	reference gas
4	pressure gauge	9	to exhaust
5	calibrated orifice	10	to chimney

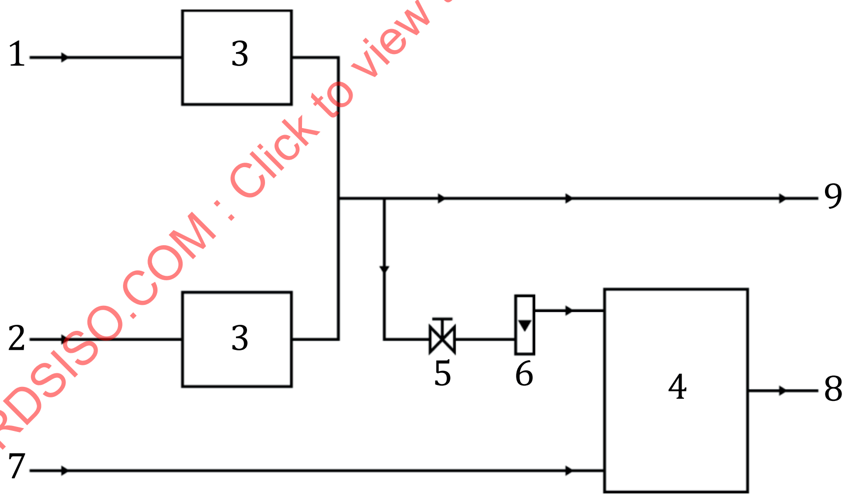
Figure 3 — Typical flow system incorporating the elements described in 5.4, b)



Key

1 oxygen
2 nitrogen
3 needle valve
4 calibrated flow meter
5 oxygen analyser
6 flow meter
7 reference gas
8 to exhaust
9 to chimney

Figure 4 — Diagram of typical flow system incorporating the elements described in 5.4, c)

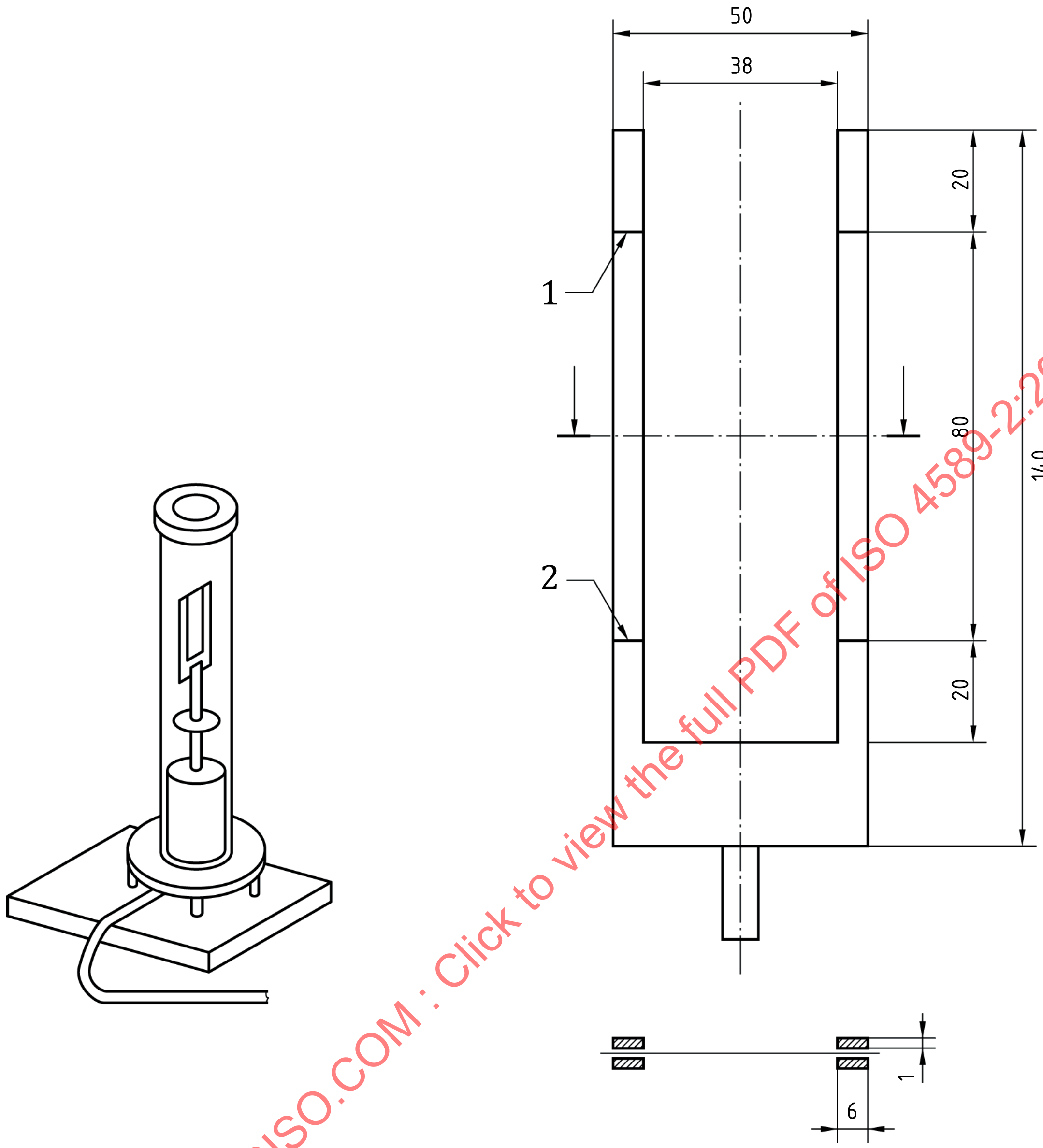


Key

1 oxygen
2 nitrogen
3 calibrated mass flow controller
4 oxygen analyser
5 needle valve
6 flow meter
7 reference gas
8 to exhaust
9 to chimney

Figure 5 — Typical flow system incorporating the elements described in 5.4, d)

Dimensions in millimetres with tolerances ± 0,25 mm



Key

1 upper reference mark

2 lower reference mark

NOTE The test specimen is held securely along both upright edges between forks made of stainless steel.

Figure 6 — Support frame for non-self-supporting test specimens

5.3 Gas supplies

The gas supplies shall comprise pressurized sources of oxygen and/or nitrogen not less than 98 % (mass fraction) pure and/or clean air [containing 20,9 % (volume fraction) oxygen], as appropriate.

The moisture content of the gas mixture entering the chimney shall be < 0,1 % (mass fraction), unless the results have been shown to be insensitive to higher moisture levels in the gas mixture. The gas supply system shall incorporate a drying device, or provision for monitoring or sampling the gas supply for moisture content, unless the moisture content of the gas supplies is known to be acceptable.

The constituent gas supply lines shall be linked in a manner which thoroughly mixes the gases, before they enter the gas distribution device at the base of the chimney, so that the variation in volume fraction of oxygen in the gas mixture rising in the chimney, below the level of the test specimen, is < 0,2 %.

NOTE Bottled oxygen or nitrogen does not always contain < 0,1 % (mass fraction) of water; moisture contents of 0,003 % (mass fraction) to 0,01 % (mass fraction) are typical for commercial supplies as filled bottles of purity ≥ 98 % (mass fraction), but as such bottled gases are depressurized to below about 1 MPa, the moisture content of the gas drawn off may rise above 0,1 % (mass fraction).

5.4 Gas control devices

The gas control devices shall be suitable for setting the volume fraction of oxygen in the gas mixture entering the chimney with a resolution of 0,1 % of the mixture and for adjusting the volume fraction with an accuracy of ± 0,2 % of the mixture when the gas velocity within the chimney is 40 mm/s ± 2 mm/s at 23 °C ± 2 °C. The flow rate shall be calculated by using the formula found in A.2.

NOTE 1 It is found suitable to measure the temperature of gas mixture inside the column by an optional temperature measurement device shown in Figure 1.

NOTE 2 Systems of measurement and control that have proved satisfactory include the following:

a) needle valves on individual and mixed gas supply lines, a paramagnetic oxygen analyser that continuously samples the mixed gas, and a calibrated flow meter to indicate when the gas flow through the chimney is within the required limits (see Figure 2);

b) calibrated orifices, gas pressure regulators and pressure gauges on the individual gas supply lines, and an oxygen analyser that continuously samples the mixed gas (see Figure 3);

c) needle valves and calibrated flow meters on the individual gas supply lines, and an oxygen analyser that continuously samples the mixed gas (see Figure 4);

d) calibrated mass flow controllers on the individual gas supply lines, and an oxygen analyser that continuously samples the mixed gas (see Figure 5).

NOTE 3 Any system incorporating appropriate needle valves, and/or calibrated orifices, and/or gas pressure regulators, and/or calibrated flow meters in conjunction with a suitable means to measure the volume fraction of oxygen to the requirements of 5.3 and 5.4 has been found suitable for the performance of this test procedure.

5.5 Oxygen analyser

The oxygen analyser shall be suitable for measuring the volume fraction of oxygen in the gas mixture entering the chimney with a resolution of 0,1 % and an accuracy of ± 0,1 % of the mixture.

NOTE 1 It has been found that paramagnetic oxygen analysers meet the accuracy requirements.

NOTE 2 In case the volume fraction of oxygen around the position of the specimen differs from that controlled at the air supply line, troubleshooting will be necessary.

5.6 Flame igniter

The flame igniter shall comprise a tube that can be inserted into the chimney to apply to the test specimen a flame issuing from an outlet of 2 mm ± 1 mm diameter at the end of the tube.

The flame fuel shall be propane without premixed air. The purity of the propane gas shall be not less than 98 %. The fuel supply shall be adjusted so that, for the set volume fraction of oxygen, the total flame height is 16 mm ± 4 mm when the tube is vertical within the chimney.

The use of a flame height gauge is recommended.

5.7 Timing device

The timing device shall be capable of measuring periods up to 5 min with an accuracy of ± 0,5 s.

5.8 Fume extraction system

The fume extraction system shall be capable of providing sufficient ventilation or exhaust to remove fumes or soot expelled from the chimney without disrupting the gas flow rate or temperatures in the chimney.

WARNING — Take suitable precautions to protect personnel from noxious materials or burns during testing or cleaning operations.

5.9 Tool for preparing rolled film

The tool for preparing rolled film shall consist of a stainless-steel rod of 2 mm diameter, with a slit in one end (see Figure 7).

6 Calibration of equipment

For compliance with this method, calibrate the equipment periodically in accordance with the instructions given in Annex A so that the maximum interval between recalibration and use conforms to the periods stated in Table 1.

Table 1 — Equipment calibration frequencies

Item	Maximum period
Leak tests for gas system joints (as required by A.1)	
a) for joints disturbed during use or cleaning of the apparatus	Immediately
b) for undisturbed equipment	6 months
Gas flow rates (as required by A.2)	6 months
Oxygen analyser (as required by A.3)	1 week
Performance check of complete equipment (as required by A.5)	1 month

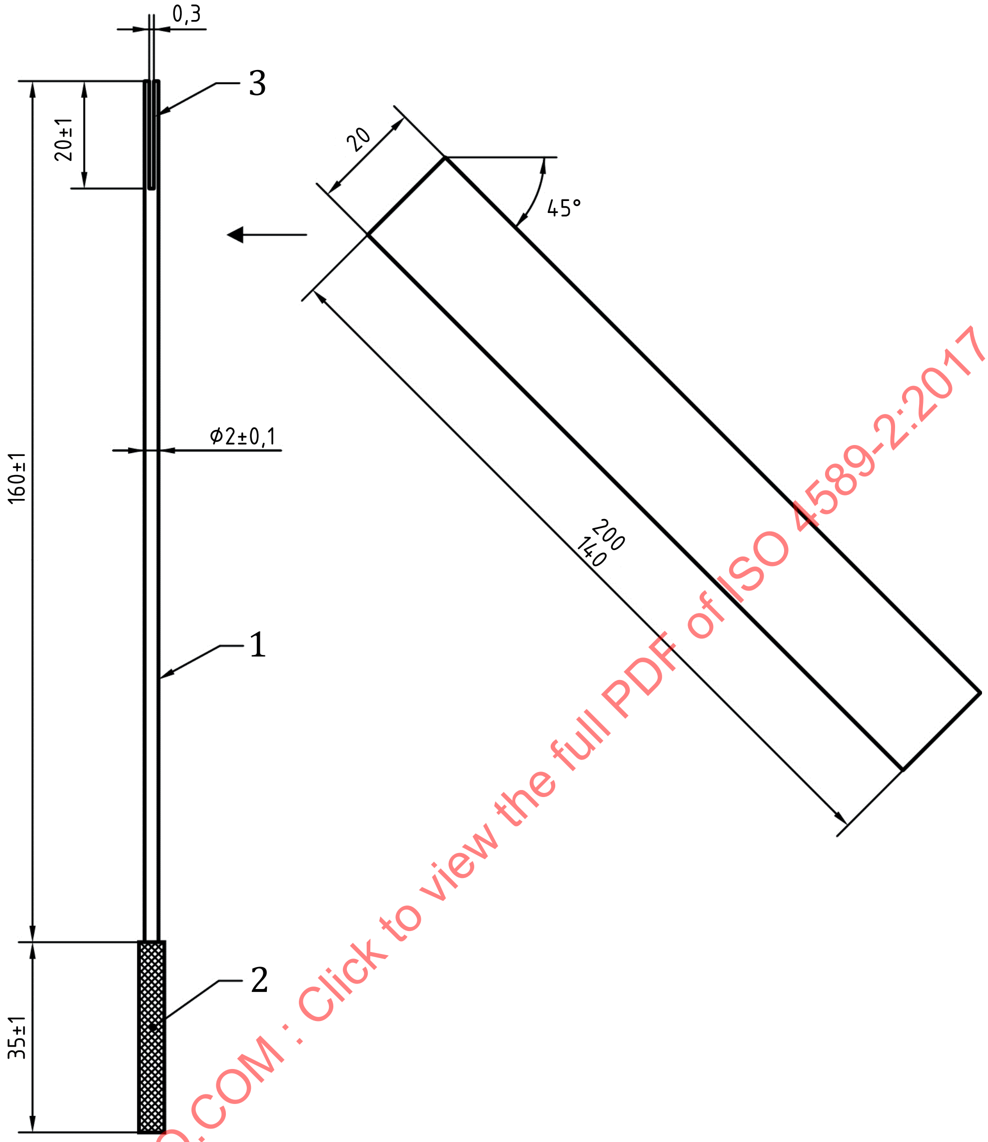
7 Preparation of test specimens

7.1 Sampling

Obtain a sample sufficient for preparation of at least 15 test specimens. The sample shall be taken, if relevant, in accordance with the material specification, otherwise in accordance with ISO 2859-1 or ISO 2859-2, as applicable.

For a material for which the OI is known to be within ± 2, 15 test specimens can be sufficient. For materials of unknown OI, or which exhibit erratic burning characteristics, between 15 and 30 test specimens may be required.

Dimensions in millimetres



Key

1	stainless-steel rod
2	grip
3	slit to hold one end of film to be rolled

Figure 7 — Tool for preparing rolled-film specimens

7.2 Test specimen dimensions and preparation

Using, if applicable, procedures that comply with the appropriate material specification or ISO methods for specimen preparation, mould or cut test specimens that satisfy the dimensions specified for the most appropriate specimen form given in Table 2.

To prepare a rolled specimen from a thin film, use the tool described in 5.9. Insert one corner of the film into the slit and then wind the film round the rod in a spiral of 45°. Ensure that the 45° angle is maintained during the winding process so that the film reaches exactly to the end of the tool, to produce a test piece of the correct length, as shown in Figure 7. After the winding is finished, tape the last end of the roll while the material is still on the stainless steel rod to prevent loosening. Then pull the rod out of the rolled film. Cut off the rolled film at a distance of 20 mm from the top end (see Figure 8).

Ensure that the surfaces of the specimens are clean and free from flaws that could affect burning behaviour, e.g. peripheral moulding flash or burrs from machining.

Note the position and orientation of test specimens with respect to any asymmetry in the sample material.

Some material specifications may require choice and identification of the “state of the test specimen” used; e.g. in a “defined state” or a “basic state” for a styrene-based polymer or copolymer.

In the absence of a relevant specification, one or more procedures from ISO 293, ISO 294, ISO 295, ISO 2818 or ISO 3167 may be used.

OI results may be significantly affected by differences in ease of ignition or burning behaviour, due to material inhomogeneity (e.g. different levels of shrinkage when heated for specimens cut in different directions from asymmetrically oriented thermoplastics film).

If a thin film burns in such a manner that erratic combustion behaviour including heat shrinkage and fluctuation of data results, the specimen form VI, i.e. a rolled film, should preferably be used. It gives reproducible results, similar to those given by specimen form I. Precision data obtained by interlaboratory trials on specimen form VI are given in Annex D.

Table 2 — Test specimen dimensions

Test specimen form [a]	**Dimensions**			**Typical use**
	Length mm	**Width** mm	**Thickness** mm	
I	80 to 150	10 ± 0,5	4 ± 0,25	For moulding materials
II	80 to 150	10 ± 0,5	10 ± 0,5	For cellular materials
III [b]	80 to 150	10 ± 0,5	≤ 10,5	For sheet materials “as received”
IV	70 to 150	6,5 ± 0,5	3 ± 0,25	Alternative size for self-supporting moulding or sheet materials, for electrical purposes
V [b]	$140\,^{\;0}_{-5}$	52 ± 0,5	≤ 10,5	For flexible film or sheet
VI [c]	140 to 200	20	0,02 to 0,10 [d]	For thin film “as received”; limited to the film that can be rolled by the specified rod [d]

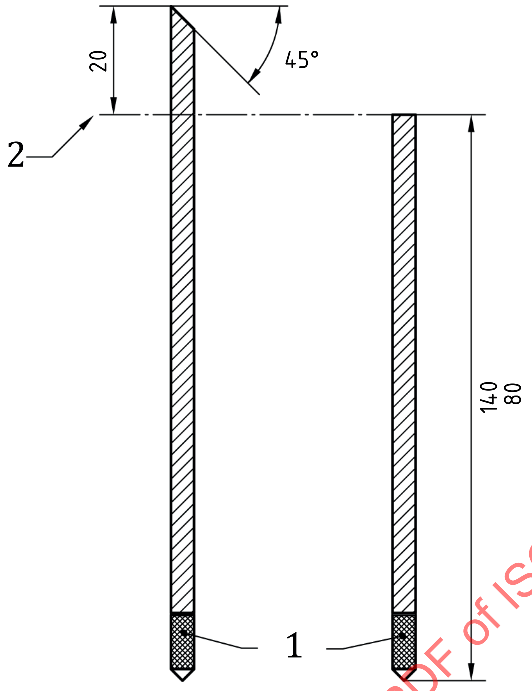
[a] Test specimens of forms I, II, III and IV are suitable for materials that are self-supporting at these dimensions. Test specimens of form V are suitable for materials that require support during testing.

[b] Results obtained using form III or form V test specimens are likely to only be comparable for specimens of the same form and thickness. It is assumed that the amount of variation in thickness for such materials will be controlled by other standards.

[c] The test specimen of form VI is suitable for a thin film that is self-supporting when it is rolled. Dimensions in the table are of an original film from which the rolled form is made. See 7.2 for the preparation of rolled film.

[d] The film is limited to thicknesses that can be rolled by the specified rod (see Figure 7). If the film is very thin, it will potentially be necessary to combine two or more films together in the preparation of the rolled film so as to obtain results similar to those normally obtained with specimen form VI.

Dimensions in millimetres



Key

1 tape

2 cut off

Figure 8 — Rolled specimen

7.3 Marking of test specimens

7.3.1 General

For monitoring the distance over which a specimen burns, it is convenient to mark the specimen with transverse lines at one or more levels which are dependent upon the specimen form and the ignition procedure to be used. Self-supporting specimens are preferably marked on at least two adjacent faces. If wet inks are used, the marks shall be dry before the specimen is ignited.

7.3.2 Marks for testing by top surface ignition

Test specimens of form I, II, III, IV or VI to be tested in accordance with procedure A (see 8.3.2) shall be marked 50 mm from the end to be ignited.

7.3.3 Marks for testing by propagating ignition

The reference marks for testing specimens of form V are carried by the supporting frame (see Figure 6), but marking such specimens at 20 mm and at 100 mm from the end to be ignited will provide convenience when testing heat-stable materials.

If specimens of forms I, II, III, IV and VI are to be tested in accordance with procedure B (see 8.3.3), they shall be marked at 10 mm and at 60 mm from the end to be ignited.

7.4 Conditioning

Unless otherwise specified in other established standards, each test specimen shall be conditioned for 24 h at 23 °C ± 2 °C and (50 ± 5) % relative humidity. After 24 h conditioning, if the stability of the mass of the specimen reaches to 0,1 % or less changes, then allow to conduct the test. Otherwise, follow ISO 291:2008, 8.1 to keep the specimen in conditioning chamber for at least 88 h.

Specimens of cellular materials that may contain volatile flammable material should preferably be purged of such volatile material prior to conditioning at 23 °C and 50 % relative humidity. Test specimens may be purged satisfactorily by pre-conditioning at 60 °C in suitable ventilated ovens for 168 h. Larger blocks of such materials may require longer pre-treatment. It is important that facilities for cutting specimens from cellular material that may contain volatile flammable material are suitable for the hazards involved.

8 Procedure for determination of oxygen index

8.1 General

For a shortened procedure to determine compliance relative to a specified minimum value of the OI, without determining the actual OI of the material under test, see Clause 10.

8.2 Setting up the apparatus and test specimen

8.2.1 Maintain the ambient temperature for the test apparatus at 23 °C ± 2 °C. If necessary, keep the test specimens in an enclosure at 23 °C ± 2 °C and (50 ± 5) % relative humidity from which each test specimen shall be taken when required.

8.2.2 Recalibrate equipment components, if necessary (see Clause 6 and Annex A). Before conducting a test, clean the glass chimney to maintain good visibility. If necessary, clean also the gas inlets, or inlet screen, and the temperature sensor (if fitted).

8.2.3 Select an initial volume fraction of oxygen to be used. When possible, base this on experience of results for similar materials. Alternatively, try to ignite a test specimen in air, and note the burning behaviour. If the specimen burns rapidly, select an initial volume fraction of about 18 % of oxygen; if the test specimen burns gently or unsteadily, select an initial oxygen volume fraction of about 21 %; if the specimen does not continue to burn in air, select an initial volume fraction of at least 25 %, depending upon the difficulty of ignition or the period of burning before extinguishing in air.

8.2.4 Ensure that the test chimney is vertical (see Figure 1). Mount a specimen vertically in the centre of the chimney so that the top of the specimen is at least 100 mm below the open top of the chimney and the lowest exposed part of the specimen is at least 100 mm above the top of the gas distribution device at the base of the chimney (see Figure 1 or 6 as appropriate).

8.2.5 Set the gas mixing and flow controls so that an oxygen/nitrogen mixture at 23 °C ± 2 °C, containing the desired volume fraction of oxygen, is flowing within the chimney at 40 mm/s ± 2 mm/s. Let the gas flow purge the chimney for at least 30 s prior to ignition of each specimen, and maintain the flow without change during ignition and combustion of each specimen. The flow rate shall be calculated by using the formula found in A.2.

Record the volume fraction of oxygen used, which is measured by an oxygen analyser or calculated in accordance with the formulae given in Annex B.

8.3 Igniting the test specimen

8.3.1 General

Select one of two alternative ignition procedures which are dependent upon the specimen form as follows:

a) For specimen forms I, II, III, IV and VI (see Table 2), use procedure A (top surface ignition) as described in 8.3.2.

b) For specimen form V, use procedure B (propagating ignition) as described in 8.3.3.

Ignition shall imply, for the purposes of this document, the initiation of flaming combustion.

For tests on materials that exhibit steady burning and spread of combustion in volume fractions of oxygen at, or close to, their OI value, or for self-supporting specimens of ≤ 3 mm thickness, procedure B (with specimens marked in accordance with 7.3.3) can be found to give more consistent results than procedure A. Procedure B may then be used for specimens of form I, II, III, IV or VI.

Some materials may exhibit a non-flaming type of combustion (e.g. glowing combustion) instead of, or at a lower volume fraction of oxygen than that required for, flaming combustion. When testing such materials, it is necessary to identify the type of combustion for which the OI is required or measured.

8.3.2 Procedure A — Top surface ignition

For top surface ignition, the igniter is used to initiate burning only on the top surface of the upper end of the specimen.

Apply the lowest visible part of the flame to the upper end of the specimen using a sweeping motion, if necessary, to cover the whole surface, but taking care not to maintain the flame against the vertical faces or edges of the specimen. Apply the flame for up to 30 s, removing it every 5 s for just sufficient time to observe whether or not the entire top surface of the specimen is burning.

Consider the specimen to be ignited, and commence measurement of the period and distance of burning, as soon as removal of the igniter, after a contact period increment of 5 s, reveals burning supported by the whole of the upper end of the specimen.

8.3.3 Procedure B — Propagating ignition

For propagating ignition, the igniter is used to produce burning across the top and partially down the vertical faces of the specimen.

Lower and move the igniter sufficiently to apply the visible flame to the upper end face of the specimen and also, to a depth of approximately 6 mm, to its vertical faces. Continue to apply the igniter for up to 30 s, including interruptions for inspection of the specimen every 5 s, until its vertical faces are burning steadily or until the visibly burning portion first reaches the level of the upper reference mark either on the support frame or, if used for specimens of form I, II, Ill, IV or VI, on the specimen.

Consider the specimen to be ignited, for the purpose of measuring the period and extent of burning, as soon as any part of the visible burning portion reaches the level of the upper reference mark.

NOTE The burning portion includes any burning drips that may run down the surface of the specimen.

8.4 Assessing the burning behaviour of individual test specimens

8.4.1 Commence measurement of the period of burning as soon as the specimen has been ignited in accordance with 8.3.2 or 8.3.3, as applicable, and observe its burning behaviour. If burning ceases but spontaneous re-ignition occurs within 1 s, continue the observation and measurements.

8.4.2 If neither the period nor the extent of burning exceeds the relevant limit specified in Table 3 for the applicable specimen, note the duration and extent of burning. This is recorded as an "O" response.

Alternatively, if either the period or extent of burning exceeds the relevant limit specified in Table 3, note the burning behaviour accordingly, and extinguish the flame. This is recorded as an "X" response.

Note also the burning characteristics of the material, e.g. dripping, charring, erratic burning, glowing combustion or after-glow.

8.4.3 Remove the specimen and clean, as necessary, any surfaces within the chimney or on the igniter that have become contaminated with soot, etc. Allow the chimney to regain a temperature of 23 °C ± 2 °C, or replace it with another conditioned one.

NOTE If many tests are being carried out, it is appropriate to use two chimneys and two specimen holders, thus allowing one chimney and holder to cool down while the second set is being used for the next test.

If sufficiently long, the specimen may be inverted, or trimmed to remove the burnt end, and re-used. Results from such specimens can save material when establishing an approximate value for the minimum volume fraction of oxygen required for combustion, but cannot be included among those used for estimation of the OI, unless the specimen is reconditioned at the temperature and humidity appropriate for the material involved.

Table 3 — Criteria for oxygen index measurements

Test specimen form (see Table 2)	Ignition procedure	Alternative criteria [a]	
		Period of burning after ignition s	Extent of burning [b]
I, II, III, IV and VI	A Top surface ignition	180	50 mm below the top of the specimen
	B Propagating ignition	180	50 mm below the upper reference mark
V	B Propagating ignition	180	80 mm below the upper reference mark (on the frame)

[a] These criteria do not necessarily produce equivalent OI results for specimens of differing shape or tested using different ignition conditions or procedures.

[b] The extent of burning is exceeded when any part of the visibly burning portion of a specimen, including drips of flaming specimen and/or a portion of thermal deformation with flaming, passes the level defined in the fourth column of the table.

8.5 Selecting successive volume fractions of oxygen

The procedure described in 8.6 and 8.7 is based upon Reference [9], using the specific case where $N_T - N_L = 5$ (see 8.7.2 and 8.7.3), with an arbitrary step size for certain changes to be made in the oxygen volume fraction used.

During the testing, select the oxygen volume fraction to be used for testing the next test specimen as follows:

a) decrease the oxygen volume fraction if the burning behaviour of the preceding specimen gave an "X" response;

otherwise

b) increase the oxygen volume fraction if the preceding specimen gave an "O" response.

Choose the size of the change in oxygen volume fraction in accordance with 8.6 or 8.7, as appropriate.

8.6 Determining the preliminary volume fraction of oxygen

Repeat the procedures specified in 8.2.4 to 8.5 inclusive, using volume fraction of oxygen changes of any convenient step size, until the volume fractions of oxygen have been found that differ by ≤ 1,0 % and of which one gave an "O" response and the other an "X" response. From this pair of volume fractions of oxygen, note that which gave the "O" response as the preliminary volume fraction of oxygen level and then proceed in accordance with 8.7.

NOTE 1 The two results, at volume fractions of oxygen ≤ 1,0 % apart, which give opposite responses do not have to be from successive specimens.

NOTE 2 That volume fraction which gave the "O" response does not have to be lower than that which gave the "X" response.

NOTE 3 A format convenient for recording the information required by this and subsequent clauses is illustrated in Annex C.

8.7 Volume fraction of oxygen changes

8.7.1 Using, again, the preliminary volume fraction of oxygen (see 8.6), test one specimen by repeating 8.2.4 to 8.4 inclusive. Record both the oxygen volume fraction (c_O) used and the response, "X" or "O", as the first of the N_L and of the N_T series of results.

8.7.2 Change the volume fraction of oxygen, in accordance with 8.5, using volume fraction changes (d) of 0,2 % (see last paragraph) of the total gas mixture to test further specimens in accordance with 8.2.4 to 8.5 inclusive, noting the values of c_O and the corresponding responses until a different response to that obtained in 8.7.1 is recorded.

The result from 8.7.1, plus those of like response from 8.7.2, constitute the N_L series of results (see example in Annex C, Section 2).

Where experience has shown that the requirements of 8.7.4 are usually satisfied by a value of d other than 0,2 %, that value may be selected as the initial value of d.

8.7.3 Test four more specimens, in accordance with 8.2.4 to 8.5 inclusive, maintaining d = 0,2 %; and note the volume fraction of oxygen c_O used, and response of, each specimen. Designate the volume fraction of oxygen used for the last specimen as c_f.

These four results, together with the last result from 8.7.2 (i.e. that which differed in response from that of 8.7.1), constitute the remainder of the N_T series, so that:

$$N_T = N_L + 5$$

where

N_L is the series of "X" or "O" results;

N_T is the series of "X" or "O" results plus five.

(See example in Annex C, Section 2.)

8.7.4 Calculate the estimated standard deviation, $\hat{\sigma}$, of the oxygen volume fraction measurements from the last six responses in the N_T series (including c_f), in accordance with 9.3. If the condition

$$\frac{2\hat{\sigma}}{3} < d < 1{,}5\hat{\sigma}$$

is satisfied, calculate the OI in accordance with 9.1; otherwise

a) if $d < 2\hat{\sigma}/3$, repeat steps 8.7.2 to 8.7.4, using increased values for d, until the condition is satisfied, or

b) if $d > 1{,}5\hat{\sigma}$, repeat steps 8.7.2 to 8.7.4, using decreased values for d, until the condition is satisfied, except that d shall not be reduced below 0,2 unless so required by the relevant material specification.

9 Calculations and expression of results

9.1 Oxygen index

Calculate the OI from the relationship:

$$\mathrm{OI} = c_f + (k \times d)$$

where

c_f is the final value of the volume fraction of oxygen, reported to three decimal places, used in the series of N_T measurements in accordance with 8.7, and noted in accordance with 8.7.3;

d is the interval, to at least three decimal places, between oxygen volume fraction levels used and controlled in accordance with 8.7;

k is a factor to be obtained from Table 4, as described in 9.2.

For the purposes of calculation of $\hat{\sigma}$ as required by 8.7.4 and 9.3, the OI shall be calculated to four decimal places (i.e. two decimal places when OI is expressed as a percentage).

For the purposes of reporting OI results, express OI values to the nearest 0,001 (i.e. to the nearest 0,1 when OI is expressed as a percentage), with exactly intermediate results being rounded downwards.

Table 4 — Values of *k* for calculating the oxygen index concentration from determinations made by Dixon's "up-and-down" method

1	2	3	4	5	6
Responses for the last five measurements	**Values of *k* for which the first N_L determination are**				
	a) O	OO	OOO	OOOO	
XOOOO	-0,55	-0,55	-0,55	-0,55	OXXXX
XOOOX	-1,25	-1,25	-1,25	-1,25	OXXXO
XOOXO	0,37	0,38	0,38	0,38	OXXOX
XOOXX	-0,17	-0,14	-0,14	-0,14	OXXOO
XOXOO	0,02	0,04	0,04	0,04	OXOXX
XOXOX	-0,50	-0,46	-0,45	-0,45	OXOXO
XOXXO	1,17	1,24	1,25	1,25	OXOOX
XOXXX	0,61	0,73	0,76	0,76	OXOOO
XXOOO	-0,30	-0,27	-0,26	-0,26	OOXXX
XXOOX	-0,83	-0,76	-0,75	-0,75	OOXXO

Table 4 *(continued)*

1	2	3	4	5	6
Responses for the last five measurements	**Values of k for which the first N_L determination are**				
	a) O	OO	OOO	OOOO	
XXOXO	0,83	0,94	0,95	0,95	OOXOX
XXOXX	0,30	0,46	0,50	0,50	OOXOO
XXXOO	0,50	0,65	0,68	0,68	OOOXX
XXXOX	-0,04	0,19	0,24	0,25	OOOXO
XXXXO	1,60	1,92	2,00	2,01	OOOOX
XXXXX	0,89	1,33	1,47	1,50	OOOOO
	Values of k for which the first N_L determinations are				Responses for the last five measurements
	b) X	XX	XXX	XXXX	
	are as given in the above table opposite the appropriate response in column 6, but with the sign of k reversed, i.e. OI = $c_f - kd$ (see 9.1).				

9.2 Determination of *k*

The value and sign of *k* are dependent upon the pattern of the responses of specimens tested in accordance with 8.7, and shall be determined from Table 4 as follows:

a) If the response of the specimen tested in accordance with 8.7.1 was “O”, so that the first contrary response (see 8.7.2) was an “X”, refer to column 1 of Table 4 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.7.3. The value and sign of *k* will be that shown in column 2, 3, 4 or 5 for which the number of “O”s shown in row a) of the table corresponds to the number of “O” responses found for the N_L series, in accordance with 8.7.1 and 8.7.2.

or

b) If the response of the specimen tested in accordance with 8.7.1 was “X”, so that the first contrary response was an “O”, refer to the sixth column of Table 4 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.7.3. The value of *k* will be that shown in column 2, 3, 4 or 5 for which the number of “X”s shown in row b) of the table corresponds to the number of “X” responses found for the N_L series, in accordance with 8.7.1 and 8.7.2, but the sign of *k* is reversed, so that negative values shown in Table 4 for *k* become positive, and vice versa.

NOTE An example of the determination of *k* and the calculation of an OI is given in Annex C.

9.3 Standard deviation of oxygen volume fraction measurements

For the purposes of 8.7.4, calculate the estimated standard deviation $\hat{\sigma}$ of the oxygen volume fraction measurements from the relationship

$$\hat{\sigma} = \left[\frac{\sum_{i=1}^{n} \left(c_i - \mathrm{OI} \right)^2}{n - 1} \right]^{1/2}$$

where

c_i represents, in turn, each of the oxygen volume fractions used during measurement of the last six responses in the N_T series of measurements;

OI is the oxygen index value, calculated in accordance with 9.1;

n is the number of measurements of the oxygen volume fraction contributing to $\sum\left(c_i - \mathrm{OI}\right)^2$.

NOTE For this method, $n = 6$, in accordance with 8.7.4. For $n < 6$, the method loses precision. For $n > 6$, different statistical criteria would apply.

9.4 Precision of results

9.4.1 An interlaboratory study was conducted in 1999 between ISO and ASTM using ISO 4589-2:1996 and ASTM D2863-95 as protocols for the test criteria. The precision data were determined from interlaboratory tests involving 12 laboratories, on eight polymeric materials, with two replicates of each material. The resulting data were analysed in accordance with ISO 5725-2, and are summarized in Table 5. Precision data obtained from an interlaboratory trial carried out in 1978-80 is shown in Annex E.

NOTE 1 The use of an oxygen analyser was not mandatory in 1999.

NOTE 2 Reference [11] includes recent data on the ISO 4589-2:1996 method, which are obtained by annual French interlaboratory tests made within the frame work of railway fire test by certified laboratories.

Table 5 — Precision data

Material	Specimen type	Procedure	Oxygen index (OI), %		
			Average	**Repeatability**	**Reproducibility**
a-1	III	A	17,7	0,09	0,14
a-2	III	A	17,8	0,35	0,35
b	III	A	38,4	4,44	6,16
c	I	A	26,8	3,33	3,33
d	I	A	49,7	5,45	5,66
e	II	A	20,9	0,91	1,30
f	V	B	26,1	2,37	3,11
g	VI	A	21,9	1,74	2,87

9.4.2 The following uncertainty calculation is based upon Reference [11]. The uncertainty on OI is maximized depending on the value of d:

For $d = 0,2$: $\mathrm{OI}_{\max} = 0,54$

For $d = 0,4$: $\mathrm{OI}_{\max} = 0,66$

These values can be accepted as uncertainty limits for the oxygen index results. They depend only on the increment volume fraction condition and on the application of the standard by use of appropriate metrological actions on the truth and accuracy of the oxygen volume fraction.

10 Comparison with a specified minimum value of the oxygen index (short procedure)

10.1 General

In case of dispute, or if the actual OI of a material is needed, the procedure given in Clause 8 is applicable.

10.2 Setting up the apparatus and test specimen

Set up the apparatus and test specimen in accordance with 8.2 except that the specified minimum volume fraction of oxygen shall be selected.

10.3 Igniting the test specimen

Ignite the test specimen in accordance with 8.3.

10.4 Assessing the burning behaviour of the test specimens

Using up to three specimens, assess the burning behaviour of each specimen in accordance with 8.4.1, 8.4.2 and 8.4.3.

10.5 Expressing of results

If for at least two out of the three specimens tested, the flame is extinguished before the relevant criteria from Table 3 is exceeded, i.e. an “O” response is recorded, then record that the OI of the material is not less than the specified value. Otherwise, record that the OI of the material is less than the specified value or determine the OI in accordance with Clause 8, as appropriate.

11 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 4589-2;

b) a statement that the test results relate only to the behaviour of the test specimens under the conditions of this test and that these results shall not be used to infer the fire hazards of the materials in other forms or under other fire conditions;

c) all details necessary for identification of the material tested, including, where relevant, the type of material, its density, its previous history and the specimen orientation with respect to any anisotropy in the material or sample;

d) the test specimen form (I to VI) and dimensions;

e) the ignition procedure used (A or B), and the igniter used, if other than the standard propane flame;

f) the oxygen index or a reference to short procedure together with the relevant specified minimum OI and a report as to whether or not the material tested had a higher OI;

e.g. OI – 34,6 %

g) the estimated standard deviation and the oxygen volume fraction increment used, if other than 0,2 %;

h) a description of any relevant ancillary characteristics or behaviour, such as charring, dripping, severe shrinkage, erratic burning or after-glow;

i) any deviations from the requirements of this document.

Annex A
(normative)

Calibration of equipment

A.1 Leak tests

Leak tests shall be carried out thoroughly on all joints where leaks could change the volume fraction of oxygen levels in the chimney from the volume fraction levels set or indicated.

A.2 Gas flow rates

The system for indicating the gas flow rate through the chimney, to satisfy 5.4 and 8.2.5, shall be checked using a calibrated flow meter, or an equivalent device, with an accuracy equivalent to ± 2 mm/s flow rate through the chimney. The flow rate shall be estimated by dividing the total gas flow rate through the chimney by the cross-sectional area of the bore of the chimney, e.g. by using Formula (A.1):

$$F = \left(\frac{4}{\pi}\right) \times 10^6 \left(\frac{q_V}{D^2}\right) = 1{,}27 \times 10^6 \left(\frac{q_V}{D^2}\right) \qquad \text{(A.1)}$$

where

F is the flow rate within the chimney, expressed in millimetres per second (mm/s);

q_V is the total gas flow at 23 °C ± 2 °C within the chimney, expressed in litres per second (l/s);

D is the diameter of the bore of the chimney, expressed in millimetres (mm).

A.3 Oxygen analyser

For zeroing, feed the analyser with oxygen-free nitrogen gas (purity 99,99 % or better), with the same flow rate and pressure as for the sample gases. Adjust the analyser response to (0,0 ± 0,2) %. Calibration shall be similarly achieved using the gas satisfying following conditions and adjusting the analyser response to the volume fraction of the oxygen source.

a) Oxygen gas with purity of 99,99 % or more.

b) Volume fraction of oxygen of gas for span adjustment should have volume fraction of oxygen higher than that of what is going to be measured, and the accuracy of volume fraction of that gas should be higher than ± 0,2 %.

A.4 Mass flow controller

The mass flow controllers shall be calibrated by the manufacturer or by a qualified calibration laboratory.

A.5 Performance check of complete equipment

The performance of the equipment may be checked, for a specific test procedure, by testing a reference material and comparing the measured results with the expected result for the reference material.

In order to use a reference material, an analysis of results should be conducted on previous tests to establish the expected results (OI and tolerance).

Some typical materials that can be used as reference samples are given in Table A.1. Note that the actual OI and tolerance should be determined by either the supplier or the laboratory.

For the purpose of performance check, PMMA of non-modified transparent cast sheet based on a homopolymer of methyl methacrylate in accordance with ISO 7823-1 can be used.

Table A.1 — Reference-material oxygen index values

Material	**OI value, %**	
	Procedure A **Top surface ignition**	**Procedure B** **Propagating ignition**
Melamine-formaldehyde (MF)	41,0 to 43,6	39,6 to 42,5
PMMA, 3 mm thick [a]	17,3 to 18,1	17,2 to 18,0
PMMA, 10 mm thick	17,9 to 19,0	17,5 to 18,5
Phenolic foam, 10,5 mm thick	39,1 to 40,7	39,6 to 40,9
PVC film, 0,02 mm thick	Not applicable	22,4 to 23,6

[a] These results relate to the particular material used for, and available following, the interlaboratory trial referred to in the introductory paragraph above. For monthly calibration, in accordance with Table 1, using 3 mm-thick test pieces (form IV) of any cast PMMA free from additives, the average of three results for such material should lie, with 95 % confidence, in the range 17,3 ± 0,2.

The results obtained for particular samples of certain materials tested using the procedures of this method in an interlaboratory trial involving 16 laboratories from seven different countries have been expressed in this table as the range within which a single test result should be found, with 95 % confidence, for each particular material/test procedure combination.