INTERNATIONAL STANDARD

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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

Annexes

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Foreword

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

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.

International Standard ISO 4589-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 4, *Burning behaviour*.

Together with parts 1 and 3 (see below), this part of ISO 4589 cancels and replaces ISO 4589:1984.

This revision has been prepared to introduce the following changes relative to the 1984 edition:

a) to amplify the requirements for equipment calibration (see clause 6 and annex A);

b) to reduce the permissible deviations for the gas flow rate through the chimney at 40 mm/s from ± 10 mm/s to ± 2 mm/s;

c) to introduce a relatively short procedure, as procedure C, intended for use for comparison purposes, to determine whether or not the oxygen index of a material lies above a specified minimum value;

d) to introduce a new specimen (form VI) and a corresponding procedure for testing of thin films. Precision data for the new procedure are given in an informative annex.

ISO 4589 consists of the following parts, under the general title *Plastics — Determination of burning behaviour by oxygen index*:

- *Part 1: Guidance*
- *Part 2: Ambient-temperature test*
- *Part 3: Elevated-temperature test*

Annexes A and B form an integral part of this part of ISO 4589. Annexes C and D are for information only.

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Plastics — Determination of burning behaviour by oxygen index —

Part 2:
Ambient-temperature test

1 Scope

This part of ISO 4589 specifies methods for determining the minimum concentration 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 values.

Methods are provided for testing materials that are self-supporting in the form of vertical bars or sheet up to 10,5 mm thick. These methods are suitable for solid, laminated or cellular materials characterized by an apparent density greater than 100 kg/m^3. The methods may 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 sheet or film materials while supported vertically.

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

Oxygen index results obtained using the methods described in this part of ISO 4589 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 part of ISO 4589-2 must not be used 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.

NOTES

1 It may 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 For assessing the flame propagation properties of cellular materials of density < 100 kg/m^3, attention is drawn to the method of ISO 3582:1978, *Cellular plastic and cellular rubber materials — Laboratory assessment of horizontal burning characteristics of small specimens subjected to a small flame*, for testing horizontal burning characteristics.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4589. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4589 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:1995, *Plastics — Injection moulding of test specimens of thermoplastic materials.*

ISO 295:1991, *Plastics — Compression moulding of test specimens of thermosetting materials.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 2859-1:1989, *Sampling procedures for inspection by attributes — Part 1: Sampling plans indexed by acceptable quality level (AQL) for lot-by-lot inspection.*

ISO 2859-2:1985, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

3 Definition

For the purposes of this part of ISO 4589, the following definition applies.

3.1 oxygen index: The minimum concentration of oxygen, by volume percentage, in a mixture of oxygen and nitrogen introduced at 23 °C ± 2 °C that will just support combustion of a material under specified test conditions.

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 burnt, with specified limits for such burning. By testing a series of specimens in different oxygen concentrations, the minimum oxygen concentration is estimated (see 8.6).

Alternatively, for comparison with a specified minimum oxygen index value, three test specimens are tested using the relevant oxygen concentration, at least two of which are required to extinguish before any relevant burning criterion is exceeded.

5 Apparatus

5.1 Test chimney, consisting of a heat-resistant glass tube supported vertically on a base through which oxygen-containing gas mixtures can be introduced (see figures 1 and 2).

The preferred dimensions of the chimney are 450 mm minimum height and 95 mm minimum 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.

NOTE 3 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, may be used, 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 distributing evenly the gas mixture entering the chimney. The preferred device comprises a suitable diffuser and a mixing chamber with metal foil. Other devices, such as radial manifolds, may be used, if shown to give equivalent results. A porous screen may be mounted below the level of the specimen holder, to prevent falling combustion debris from fouling the gas entry and distribution paths.

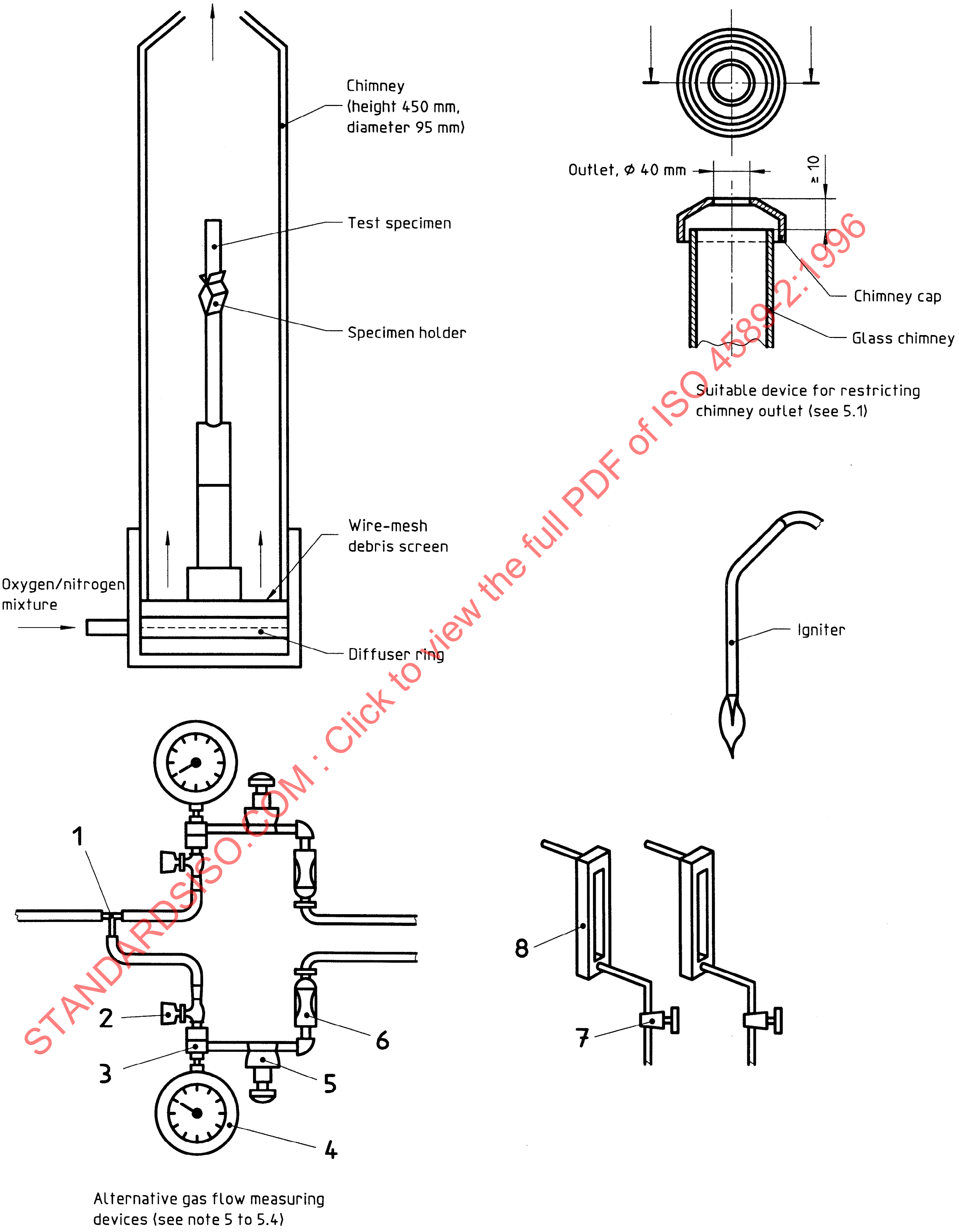
The chimney support may incorporate a levelling device and indicator, to facilitate vertical alignment of the chimney and a test specimen supported therein. A dark background may be provided to facilitate observation of flames within the chimney.

5.2 Test specimen holder, 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 the specimen may 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 2, with reference marks at 20 mm and 100 mm below the top of the frame.

The profile of the holder and its support should preferably be smooth to minimize induction of turbulence in the rising flow gas.

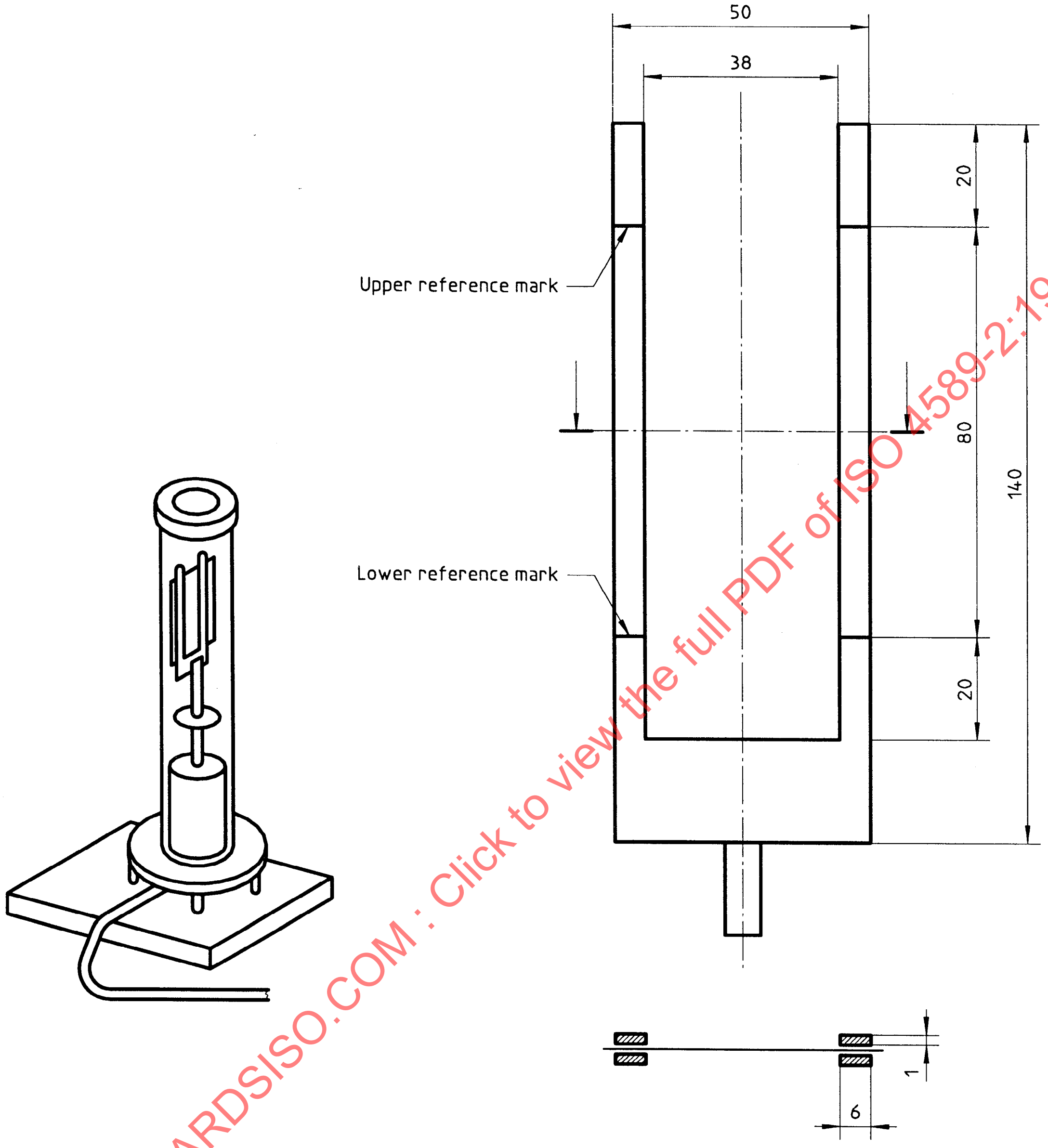
Dimensions in millimetres



1 Gas premixing point
2 Cut-off valve
3 Orifice in holder
4 Pressure gauge
5 Precision pressure regulator
6 Filter
7 Needle valve
8 Gas flow meter

Figure 1 — Diagram of typical apparatus for determination of oxygen index

Dimensions in millimetres with tolerances of ± 0,25 mm



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

Figure 2 — Support frame for non-self-supporting test specimens

5.3 Gas supplies, comprising pressurized sources of oxygen and/or nitrogen not less than 98 % (*m/m*) pure and/or clean air [containing 20,9 % (*V/V*) oxygen], as appropriate.

The moisture content of the gas mixture entering the chimney shall be < 0,1 % (*m/m*), 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 oxygen concentration in the gas mixture rising in the chimney, below the level of the test specimen, is < 0,2 % (*V/V*).

NOTE 4 It should not be assumed that bottled oxygen or nitrogen will always contain $< 0{,}1$ % (*m/m*) of water; moisture contents of 0,003 % (*m/m*) to 0,01 % (*m/m*) are typical for commercial supplies as filled bottles of purity $\geqslant 98$ % (*m/m*), 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 % (*m/m*).

5.4 Gas measurement and control devices, suitable for measuring the concentration of oxygen in the gas mixture entering the chimney with an accuracy of ± 0,5 % (*V/V*) of the mixture and for adjusting the concentration with a precision of ± 0,1 % (*V/V*) of the mixture when the gas velocity through the chimney is 40 mm/s ± 2 mm/s at 23 °C ± 2 °C.

Means shall be provided for checking or ensuring that the temperature of the gas mixture entering the chimney is 23 °C ± 2 °C. If this involves an internal probe, its position and profile shall be designed to minimize induction of turbulence within the chimney.

NOTE 5 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 flowmeter to indicate when the gas flow through the chimney is within the required limits;

b) calibrated orifices, gas pressure regulators and pressure gauges on the individual gas supply lines;

c) needle valves and calibrated flowmeters on the individual gas supply lines.

Systems b) and c) may require calibration after assembly to ensure that the cumulative errors of the component parts do not exceed the requirements of 5.4.

5.5 Flame igniter, comprising 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 fuel supply shall be adjusted so that the flame will project 16 mm ± 4 mm vertically downwards from the outlet when the tube is vertical within the chimney and the flame is burning within the chimney atmosphere.

5.6 Timing device, capable of measuring periods up to 5 min with an accuracy of ± 0,5 s.

5.7 Fume extraction system, 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.

NOTE 6 If soot-generating materials are being tested, the glass chimney may require cleaning to maintain good visibility, and the gas inlets, or inlet screen, and temperature sensor (if fitted) may also require cleaning to function properly. Suitable precautions should be taken to protect personnel from noxious materials or burns during testing or cleaning operations.

5.8 Tool for preparing rolled film, consisting of a stainless-steel rod of 2 mm diameter, with a slit in one end (see figure 3).

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 complies with the periods stated in table 1.

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.

NOTE 7 For a material for which the oxygen index is known to within ± 2, 15 test specimens may be sufficient. For materials of unknown oxygen index, or which exhibit erratic burning characteristics, between 15 and 30 test specimens may be required.

Table 1 — Equipment calibration frequencies

Item	Maximum period
Gas system joints (as required by clause A.1 in annex A)	
a) for joints disturbed during use or cleaning of the apparatus	Immediately
b) for undisturbed equipment	6 months
Cast PMMA sample	1 month
Gas flow rate controls	6 months
Oxygen concentration controls	6 months

Dimensions in millimetres

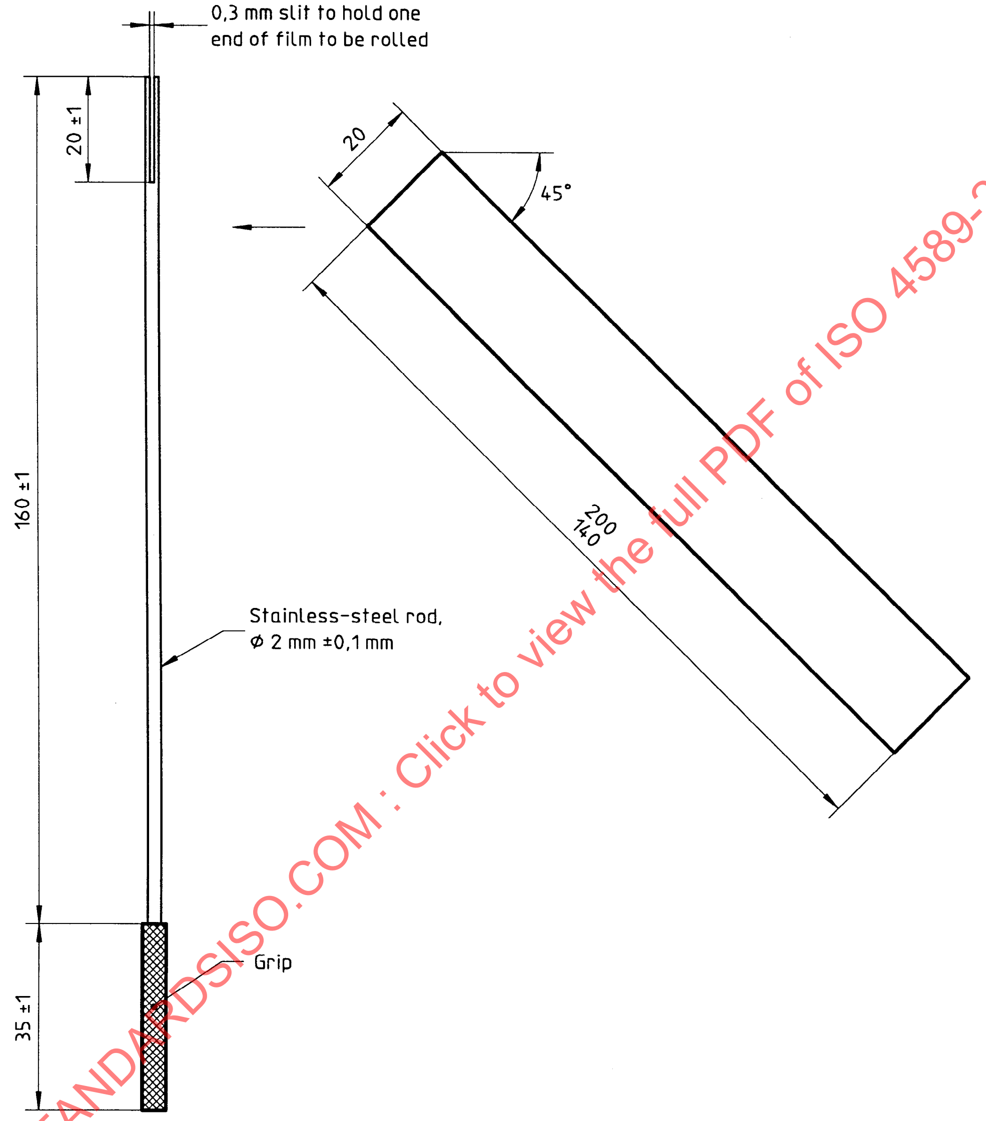


Figure 3 — Tool for preparing rolled-film specimens

7.2 Test specimen dimensions and preparation

Using, if applicable, procedures that comply with the appropriate material specification (see note 8) or ISO methods (see note 9) 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 5.8. 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 3. 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 4).

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 (see note 10).

NOTES

8 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.

9 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.

10 Oxygen index 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).

11 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 form1)	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 2)	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 2)	$140^{\ 0}_{-5}$	52 ± 0,5	⩽ 10,5	For flexible film or sheet
VI 3)	140 to 200	20	0,02 to 0,10 4)	For thin film "as received"; limited to the film that can be rolled by the specified rod 4)

1) 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.

2) Results obtained using form III or form V test specimens may 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.

3) 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.

4) The film is limited to thicknesses that can be rolled by the specified rod (see figure 3). If the film is very thin, it may 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

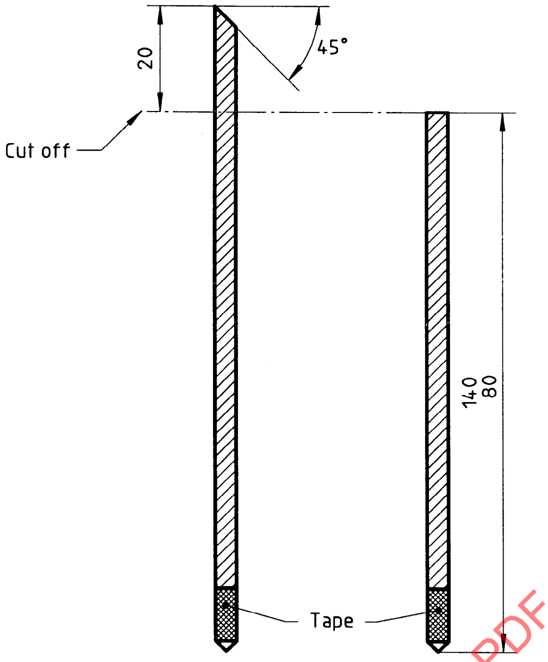


Figure 4 — Rolled specimen

7.3 Marking of test specimens

7.3.1 General

For monitoring the distance over which a specimen burns, it may be marked 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 8.2.2) 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 2), but such specimens may be marked at 20 mm and at 100 mm from the end to be ignited, for 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 8.2.3), 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 at least 88 h at 23 °C ± 2 °C and (50 ± 5) % relative humidity immediately prior to use.

NOTE 12 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 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

NOTE 13 For a shortened procedure to determine compliance relative to a specified minimum value of the oxygen index, without determining the actual oxygen index of the material under test, see clause 10.

8.1 Setting up the apparatus and test specimen

8.1.1 Maintain the ambient temperature for the test ap.paratus 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 may be taken when required.

8.1.2 Recalibrate equipment components, if necessary (see clause 6 and annex A).

8.1.3 Select an initial concentration 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 concentration of about 18 % (*V/V*) of oxygen; if the test specimen burns gently or unsteadily, select an initial oxygen concentration of about 21 % (*V/V*); if the specimen does not continue to burn in air, select an initial concentration of at least 25 % (*V/V*), depending upon the difficulty of ignition or the period of burning before extinguishing in air.

8.1.4 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 2 as appropriate).

8.1.5 Set the gas mixing and flow controls so that an oxygen/nitrogen mixture at 23 °C ± 2 °C, containing the desired concentration of oxygen, is flowing through 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.

Record the oxygen concentration used as the volume per cent calculated in accordance with the equations given in annex B.

8.2 Igniting the test specimen

8.2.1 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 8.2.2;

b) for specimen form V, use procedure B (propagating ignition) as described in 8.2.3.

Ignition shall imply, for the purposes of this part of ISO 4589, the initiation of flaming combustion.

NOTES

14 For tests on materials that exhibit steady burning and spread of combustion in oxygen concentrations at, or close to, their oxygen index value, or for self-supporting specimens of ⩽ 3 mm thickness, procedure B (with specimens marked in accordance with 7.3.2) may 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.

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

8.2.2 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 top 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 top end of the specimen.

8.2.3 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 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, III, 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 16 The burning portion includes any burning drips that may run down the surface of the specimen.

8.3 Assessing the burning behaviour of individual test specimens

8.3.1 Commence measurement of the period of burning as soon as the specimen has been ignited in accordance with 8.2.2 or 8.2.3, as applicable, and observe its burning behaviour. If burning ceases but spontaneous re-ignition occurs within 1 s, continue the observation and measurements.

8.3.2 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.

8.3.3 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 so conditioned.

NOTES

17 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.

18 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 oxygen concentration required for combustion, but cannot be included among those used for estimation of the oxygen index, 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[1)]	
		Period of burning after ignition s	Extent of burning[2)]
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)

1) These criteria do not necessarily produce equivalent oxygen index results for specimens of differing shape or tested using different ignition conditions or procedures.

2) The extent of burning is exceeded when any part of the visibly burning portion of a specimen, including burning drips descending the vertical faces, passes the level defined in the fourth column of the table.

8.4 Selecting successive oxygen concentrations

The procedure described in 8.5 and 8.6 is based upon the "up-and-down method for small samples"[1], using the specific case where $N_T - N_L = 5$ (see 8.6.2 and 8.6.3), with an arbitrary step size for certain changes to be made in the oxygen concentration used.

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

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

otherwise

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

Choose the size of the change in oxygen concentration in accordance with 8.5 or 8.6, as appropriate.

8.5 Determining the preliminary oxygen concentration

Repeat the procedures specified in 8.1.4 to 8.4 inclusive, , using oxygen concentration changes of any convenient step size, until the oxygen concentrations, in volume per cent, have been found that differ by $\leqslant$ 1,0 % (*V/V*) and of which one gave an "O" response and the other an "X" response. From this pair of oxygen concentrations, note that which gave the "O" response as the preliminary oxygen concentration level and then proceed in accordance with 8.6.

NOTES

19 The two results, at oxygen concentrations $\leqslant$ 1,0 % (*V/V*) apart, which give opposite responses do not have to be from successive specimens.

20 That concentration which gave the "O" response does not have to be lower than that which gave the "X" response.

21 A format convenient for recording the information required by this and subsequent clauses is illustrated in annex C.

8.6 Oxygen concentration changes

8.6.1 Using, again, the preliminary oxygen concentration (see 8.5), test one specimen by repeating 8.1.4 to 8.3 inclusive. Record both the oxygen concentration (c_o) used and the response, "X" or "O", as the first of the N_L and of the N_T series of results.

8.6.2 Change the oxygen concentration, in accordance with 8.4, using concentration changes (d) of 0,2 % (*V/V*) (see note 22) of the total gas mixture to test further specimens in accordance with 8.1.4 to 8.4 inclusive, noting the values of c_o and the corresponding responses until a different response to that obtained in 8.6.1 is recorded.

The result from 8.6.1, plus those of like response from 8.6.2, constitute the N_L series of results. (See example in annex C, section 2.)

NOTE 22 Where experience has shown that the requirements of 8.6.4 are usually satisfied by a value of d other than 0,2 % (*V/V*), that value may be selected as the initial value of d.

8.6.3 Test four more specimens, in accordance with 8.1.4 to 8.4 inclusive, maintaining $d = 0{,}2\ \%\ (V/V)$; and note the c_o used for, and response of, each specimen. Designate the oxygen concentration used for the last specimen as c_f.

These four results, together with the last result from 8.6.2 (i.e. that which differed in response from that of 8.6.1), constitute the remainder of the N_T series, so that

$$N_T = N_L + 5$$

(See example in annex C, section 2.)

8.6.4 Calculate the estimated standard deviation, $\hat{\sigma}$, of the oxygen concentration 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 oxygen index in accordance with 9.1; otherwise

a) if $d < 2\hat{\sigma}/3$, repeat steps 8.6.2 to 8.6.4, using increased values for d, until the condition is satisfied, or

b) if $d > 1{,}5\hat{\sigma}$, repeat steps 8.6.2 to 8.6.4, using decreased values for d, until the condition is satis-

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[1] DIXON, W.J., *American Statistical Association Journal*, pp. 967-970 (1965).

fied, 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 oxygen index OI, expressed as a percentage by volume, from the relationship

$$\text{OI} = c_f + kd$$

where

c_f is the final value of the oxygen concentration, in volume per cent to one decimal place, used in the series of N_T measurements performed in accordance with 8.6, and noted in accordance with 8.6.3;

d is the interval, in volume per cent to at least one decimal place, between oxygen concentration levels used and controlled in accordance with 8.6;

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 8.6.4 and 9.3, the OI shall be calculated to two decimal places.

For the purposes of reporting OI results, express OI values to the nearest 0,1, 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 determinations 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
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. $\text{OI} = c_f - kd$ (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 8.6, and may be determined from table 4 as follows:

a) If the response of the specimen tested in accordance with 8.6.1 was "O", so that the first contrary response (see 8.6.2) 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 8.6.3. 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 8.6.1 and 8.6.2.

or

b) If the response of the specimen tested in accordance with 8.6.1 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 8.6.3. 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 8.6.1 and 8.6.2, but the sign of k is reversed, so that negative values shown in table 4 for k become positive, and *vice versa*.

NOTE 23 An example of the determination of k and the calculation of an OI is given in annex C.

9.3 Standard deviation of oxygen concentration measurements

For the purposes of 8.6.4, calculate the estimated standard deviation $\hat{\sigma}$ of the oxygen concentration measurements from the relationship

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

where

c_i represents, in turn, each of the per cent oxygen concentrations 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 concentration contributing to $\Sigma(c_i - \text{OI})^2$.

NOTE 24 For this method, $n = 6$, in accordance with 8.6.4. For $n < 6$, the method loses precision. For $n > 6$, alternative statistical criteria would apply.

9.4 Precision of results

This method may be expected to be capable of the limits given in table 5 for materials that ignite without difficulty and burn steadily.

Table 5 — Estimated precision limits

Approximate values for 95 % confidence	Within laboratories	Between laboratories
Standard deviation	0,2	0,5
Repeatability (r)	0,5	—
Reproducibility (R)	—	1,4

NOTE — The precision data were determined from an international interlaboratory trial in 1978/1980 involving 16 laboratories and 12 samples.

NOTE 25 Materials that exhibit erratic combustion behaviour may increase the limits in table 5 by a factor of up to 5. On the other hand, it may be found that, for materials that exhibit very consistent burning behaviour, $d > 1{,}5\,\hat{\sigma}$ even if d is reduced to 0,1 % (V/V), indicating that greater precision is possible. For practical purposes, the accuracy and precision requirements specified for apparatus by this part of ISO 4589 are inadequate for significant discrimination if using $d < 0{,}1$ % (V/V), and results obtained using this method have not been found to be significantly different for $d \leqslant 0{,}2$ % (V/V). More precise determination of the minimum oxygen concentration to just support combustion would require different apparatus and the use of different statistical relationships and factors to determine the value from a longer series of measurements.

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

NOTE 26 In case of dispute, or if the actual oxygen index of a material is needed, the procedure given in clause 8 is applicable.

10.1 Set up the apparatus and test specimen in accordance with 8.1 except that the specified minimum concentration of oxygen shall be selected for the purposes of 8.1.3.

10.2 Ignite the test specimen in accordance with 8.2.

10.3 Using up to three specimens, assess the burning behaviour of each specimen in accordance with 8.3.1, 8.3.2 and 8.3.3.

If for at least two out of the three specimens thus tested the flame is extinguished before the relevant criteria from table 3 are exceeded, i.e. an "O" response is recorded, then record that the oxygen index of the material is not less than the specified value. Otherwise, record that the oxygen index of the material is less than the specified value or determine the oxygen index in accordance with clause 8, as appropriate.

11 Test report

The test report shall include the following information:

a) a reference to this part of ISO 4589;

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);

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

g) if applicable, the estimated standard deviation and the oxygen concentration increment used, if other than 0,2 % (*V/V*);

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 part of ISO 4589.

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 oxygen concentration levels in the chimney from the concentration 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 8.1.5, shall be checked using a calibrated flow meter, or an equivalent device, with an accuracy equivalent to ± 0,2 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 the following equation:

$$F = 1{,}27 \times 10^6 \frac{q_V}{D^2}$$

where

F is the flow rate through the chimney, in millimetres per second;

q_V is the total gas flow at 23 °C ± 2 °C through the chimney, in litres per second;

D is the diameter of the bore of the chimney, in millimetres.

A.3 Oxygen concentration

The concentration of oxygen in the mixture of gases flowing into the chimney shall be checked to an accuracy of 0,1 % (V/V) of mixture, either by sampling the chimney atmosphere for analysis or by using an independently calibrated oxygen analyser *in situ*. If an oxygen analyser is incorporated in the equipment, this shall be calibrated using the following gases, each of which shall conform with 5.3 for purity and moisture content:

a) any two gases selected from the following:

— nitrogen;

— oxygen;

— clean air;

and

b) a mixture of any two of the preceding gases having an oxygen concentration within the range of concentrations to be used for most test specimens.

A.4 Calibration of complete equipment

The performance of the equipment may be checked, for a specific test procedure, by testing a calibrated material and comparing the measured results with the expected result for the calibrated material. For the selection, availability and use of calibrated materials, see table A.1.

Table A.1 — Reference-material oxygen index values

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. While stocks of surplus materials from the 1978/1980 interlaboratory trial last, samples with the oxygen index levels given in this table will be available from the Rubber and Plastics Research Association, Shawbury, Shrewsbury, Shropshire, United Kingdom (sole supplier).

Material	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[1]	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

1) 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.

Annex B
(normative)

Calculation of oxygen concentration

B.1 Oxygen concentrations required for the purposes of clause 8 shall be calculated using the equation

$$c_O = \frac{100\ V_O}{V_O + V_N} \qquad \ldots (B.1)$$

where

c_O is the oxygen concentration, in per cent by volume;

V_O is the volume of oxygen per volume of mixture, at 23 °C;

V_N is the volume of nitrogen per volume of mixture, at 23 °C.

If an oxygen analyser is used, determine the oxygen concentration using the readout from the particular instrument used.

If the result is calculated from flow or pressure data for individual gas streams contributing to the mixture, it is necessary to allow for the proportion of oxygen present in streams other than a pure oxygen supply. For example, for mixtures made using air mixed with oxygen of 98,5 % (*V/V*) purity or with nitrogen containing 0,5 % (*V/V*) of oxygen, calculate the oxygen concentration, in per cent by volume, using the relationship

$$c_O = \frac{98{,}5V'_O + 20{,}9V'_A + 0{,}5V'_N}{V'_O + V'_A + V'_N} \qquad \ldots (B.2)$$

where

V'_O is the volume of oxygen stream used, per volume of mixture;

V'_A is the volume of air stream used, per volume of mixture;

V'_N is the volume of nitrogen stream used, per volume of mixture;

assuming that the streams are at the same pressure at 23 °C.

For mixtures based on two gas streams, V'_O, V'_A or V'_N becomes zero, as appropriate.

Annex C
(informative)

Typical test results sheet

Test results sheet for oxygen index determined in accordance with ISO 4589-2

Material: *Phenolic laminate*
Specimen form: *III (4 mm thick)*
Ignition procedure: [✓ A] B
Conditioning procedure: 23 [✓ 23/50]
Oxygen concentration increment (d): *0,2* % (V/V)

Oxygen index [concentration, % (V/V)]: *29,5*
[rounded to 0,1 % (V/V)]
$\hat{\sigma}$: *0,152*
Date of test: *1995-05-26*
Laboratory No.: *19* Test No.: *1*

Section 1: Determination of oxygen concentration for one pair of "X" and "O" responses at ⩽ 1 % (V/V) O_2 concentration interval (in accordance with 8.5)

Oxygen concentration, % (V/V)	*25,0*	*35,0*	*30,0*	*32,0*	*31,0*				
Burning period, s	*10*	*>180*	*140*	*>180*	*>180*				
Length burnt, mm									
Response ("X" or "O")	*O*	*X*	*O*	*X*	*X*				

Oxygen concentration of the "O" response for the pair = *30,0* % (V/V)
(this is the concentration to be used again for the first measurement in section 2)

Section 2: Determination of oxygen index (in accordance with 8.6)

Step size to be used for successive changes d in oxygen concentration = *0,2* % (V/V)
[initially to be 0,2 % (V/V), unless otherwise instructed]

<table>
<tr><td></td><td colspan="12">N_T series measurements</td></tr>
<tr><td></td><td colspan="7">N_L series measurements (8.6.1 and 8.6.2)</td><td colspan="4">(8.6.3)</td><td>c_f</td></tr>
<tr><td>Oxygen concentration, % (V/V)</td><td>30,0</td><td>29,8</td><td>29,6</td><td>29,4</td><td></td><td></td><td></td><td>29,4</td><td>29,6</td><td>29,4</td><td>29,6</td><td>29,8</td></tr>
<tr><td>Burning period, s</td><td>>180</td><td>>180</td><td>>180</td><td>150</td><td></td><td></td><td></td><td>150</td><td>>180</td><td>110</td><td>165</td><td>>180</td></tr>
<tr><td>Length burnt, mm</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Response ("X" or "O")</td><td>X</td><td>X</td><td>X</td><td></td><td></td><td></td><td></td><td>O</td><td>X</td><td>O</td><td>O</td><td>X</td></tr>
<tr><td></td><td colspan="7">Column (2, 3, 4 or 5): 4</td><td colspan="5">Row (1 to 16): 7</td></tr>
<tr><td></td><td colspan="12">k value from table 4: 1,25</td></tr>
<tr><td></td><td colspan="7"></td><td colspan="5">Hence k = – 1,25</td></tr>
</table>

OI = $c_f + kd$ = *29,8 + (– 1,25 × 0,2)*
= *29,5* % (to one decimal place, for reporting OI)
= *29,55* % (to two decimal places, for calculation of and verification of d as required in section 3)