SLOVENSKI STANDARD
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Polimerni materiali - Ugotavljanje vnetljivosti s kisikovim indeksom

Plastics -- Determination of flammability by oxygen index

Plastiques -- Essais de réaction au feu -- Détermination de l'indice d'oxygène

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International Standard 4589

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Plastics — Determination of flammability by oxygen index

Plastiques — Essais de réaction au feu — Détermination de l'indice d'oxygène

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Plastics — Determination of flammability by oxygen index

0 Introduction

This International Standard has been prepared to rationalize procedures developed independently for similar methods of test by various national standards bodies.

1 Scope and field of application

This International Standard 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.

Oxygen index results obtained using the methods described in this International Standard 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. Such requirements should be referred to in other standards. 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 International Standard 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 for testing horizontal burning characteristics.

2 References

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

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

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

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

ISO 845, *Cellular plastics and rubbers — Determination of density.*

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

ISO 2859, *Applications of statistical methods — Sampling procedures and tables of inspection by attributes.*

ISO 3167, *Plastics — Preparation and use of multi-purpose test specimens.*

ISO 3582, *Cellular plastic and cellular rubber materials — Laboratory assessment of horizontal burning characteristics of small specimens subjected to a small flame.*

ISO 4893, *Plastics — Preparation of test specimens from thermosetting materials by injection moulding.*[1)]

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility.*

ISO 6400, *Plastics — Test specimens from semi-crystalline thermoplastic moulding materials by compression moulding — Preparation of reference test specimens with a reproducible state.*[1)]

1) At present at the stage of draft.

3 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 at which the burning behaviour of 50 % of specimens representing the material under test would exceed at least one of the specified limits for burning.

4 Definition

For the purposes of this International Standard, the following definition applies.

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

5 Apparatus

The following apparatus shall be arranged as indicated by the diagrams in figures 1 and 2 as appropriate:

5.1 Test chimney: a heat-resistant glass tube supported vertically on a base through which oxygen-containing gas mixtures can be introduced.

The preferred dimensions of the chimney are 450 mm minimum height and 75 mm minimum diameter cylindrical bore. 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 a flow rate within the chimney of 30 mm/s (see the note). 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 means for distributing evenly the gas mixture entering the chimney. The preferred means comprises solid glass beads of between 3 and 5 mm diameter, in a layer between 80 and 100 mm deep. Other means, 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.

NOTE — For tubes of 75 to 100 mm diameter, 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.

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 be smooth to minimize induction of turbulence in the rising flow of gas.

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 % 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 — It should not be assumed that bottled oxygen or nitrogen will always contain $<0,1$ % (m/m) of water; moisture contents of 0,003 % to 0,01 % (m/m) are typical for commercial supplies as filled bottles >98 % (m/m) pure, but as such bottled gases are depressured 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 establishing 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 ± 10 mm/s at 23 ± 2 °C.

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

NOTE — 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; or

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 compounded 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 ± 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 ± 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 ±0,2 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 — 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.

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.

Table 1 — Equipment calibration frequencies

Item	Maximum period
Gas-flow rate controls	6 months
Oxygen concentration controls	6 months
Gas system joints (as required by clause A.2 in annex A)	
a) for joints disturbed during use or cleaning of the apparatus	24 h
b) for undisturbed joints	6 months

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.

NOTE — 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.

7.2 Test specimen dimensions and their 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.

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

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

NOTES

1 Some material specifications may require choice and identification of the "state of the test specimen" used; for example, in a "defined state" or a "basic state" for a styrene-based polymer or copolymer.

2 In the absence of a relevant material specification, one or more procedures from ISO 293, ISO 294, ISO 295, ISO 2818, ISO 3167, ISO 4893 or ISO 6400 may be used.

3 Oxygen index results may be significantly affected by differences in ease of ignition or of burning behaviour, due to material inhomogeneity (for example, different levels of shrinkage when heated for specimens cut in different directions from asymmetrically-oriented thermoplastic film).

Table 2 — Test specimen dimensions

Test specimen form [1]	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

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.

7.3 Marking of test specimens

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.

7.3.1 Marks for testing by top surface ignition

Test specimens of form I, II, III or IV, to be tested in accordance with procedure A (see 8.2.1), shall be marked 50 mm from the end to be ignited.

7.3.2 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 form I, II, III and IV are to be tested in accordance with procedure B (see 8.2.1 and 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 ± 2 °C and (50 ± 5) % RH immediately prior to use.

NOTE — Samples of cellular materials that may contain volatile flammable material should be purged of such volatile material prior to conditioning at 23 °C and 50 % RH. 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. Facilities for cutting specimens from cellular material that may contain volatile flammable material must be suitable for the hazards involved.

8 Procedure

8.1 Setting up the apparatus and test specimen

8.1.1 Maintain the ambient temperature for the test apparatus at 23 ± 2 °C. If necessary, keep the test specimens in an enclosure at 23 ± 2 °C and (50 ± 5) % RH 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, this may be based 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 %; if the specimen does not continue to burn in air, select an initial concentration of at least 25 %, depending upon the difficulty of ignition or the period of burning before extinguishment 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 figure 2 as appropriate).

8.1.5 Set the gas mixing and flow controls so that an oxygen/nitrogen mixture at 23 ± 2 °C, containing the desired concentration of oxygen, is flowing through the chimney at a rate of 40 ± 10 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 according to the equations given in annex C.

8.2 Igniting the test specimen

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

a) for specimen forms I, II, III and IV (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 International Standard, the initiation of flaming combustion.

NOTES

1 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 or IV.

2 Some materials may exhibit a non-flaming type of combustion (for example, 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 surface 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, with 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 on the support frame or, if used for specimens of form I, II, III or IV, 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 visibly 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.

8.3 Assessing burning behaviour

For the purposes of 8.4 to 8.6 inclusive, observe and terminate the burning of individual test specimens as follows.

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 in < 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, for example 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 ± 2 °C, or replace it with another so conditioned.

NOTE — 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.

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,

Table 3 — Criteria for oxygen index measurements [1)]

Test specimen form (see table 2)	Ignition procedure	Alternative criteria	
		Period of burning after ignition, s	Extent of burning [2)]
I, II, III and IV	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 table 3.

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