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Plastics — Determination of burning behaviour by oxygen index —

Part 1: General requirements

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

Partie 1: Exigences générales

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Foreword

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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 4589-1:1996), which has been technically revised.

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

Introduction

The oxygen index (OI) test at ambient temperature was first described by Fenimore and Martin[3] in 1966. The first use of the method in standards was ASTM D2863:1970[2] and it has since been published in a wide range of national and international standards. It was published as ISO 4589 in 1984 and has now been revised as ISO 4589-2. The OI test at elevated temperatures is described in ISO 4589-3.

In the period since ASTM D2863 became a standard, a considerable number of papers have been published about this test. An example is the review in Reference [6] relating to the relevance of the test to real fire situations. Other papers have suggested empirical formulae relating OI to the amounts of added fire retardant, or describe practical investigations on the equipment performance (see Reference [7]). A clear consensus on the value of the two variants of the test has emerged, however, and it is the purpose of this document to discuss the use of the equipment and the applicability of both test methods.

Plastics — Determination of burning behaviour by oxygen index —

Part 1: General requirements

1 Scope

This document specifies the general requirements for the oxygen index (OI) test which are further described in ISO 4589-2 and ISO 4589-3 as follows:

— ISO 4589-2 describes a method for determining the minimum volume fraction of oxygen 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;

— ISO 4589-3 describes methods of carrying out the same determination over a range of temperatures typically between 25 °C and 150 °C (although temperatures up to 400 °C can be used).

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 4589-2:2017, *Plastics — Determination of burning behaviour by oxygen index — Part 2: Ambient-temperature test*

ISO 4589-3:2017, *Plastics — Determination of burning behaviour by oxygen index — Part 3: Elevated-temperature test*

ISO 13943, *Fire safety — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943 and the following 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

3.1
oxygen index
OI
limiting oxygen index
LOI
minimum volume fraction of oxygen in a mixture of oxygen and nitrogen, at 23 °C ± 2 °C, that just supports flaming combustion of a material under specified test conditions

Note 1 to entry: It is expressed as a percentage, e.g. OI = 34,6 %.

[SOURCE: ISO 13943:2008, 4.248, modified — Note 1 to entry has been revised.]

4 Principles of the test

In ISO 4589-2, the material can be tested either as a rigid specimen or as a flexible specimen in a specified holder which is mounted in a transparent chimney in which a mixture of oxygen and nitrogen flows upwards in a laminar flow pattern. After specimen conditioning, the test is carried out at room temperature. This constitutes the test in its simplest form. In the top-surface ignition procedure, the applied flame is allowed to impinge on the top surface of the specimen for a maximum time of 30 s coupled with periodic removal of the flame every 5 s to check if the specimen is burning. This also ensures that the specimen temperature does not rise excessively, since this would normally lower the OI value. In the propagating-ignition procedure, the flame is allowed to impinge to a depth of approximately 6 mm down the vertical sides of the specimen. In the thin-film procedure, the film is wrapped in a 45° spiral around a rod and taped, after which the rod is withdrawn and the top end of the specimen is cut off at a distance of 20 mm from the top.

In ISO 4589-3, the material is tested in an identical manner, except that the test takes place in a heated column in which both the incoming gas and the gas passing up the column are heated. At the start of the test, the specimen and specimen holder are preheated in the gas flow for 240 s ± 10 s to allow them to reach temperature equilibrium prior to testing. The flame is applied for the same length of time as in ISO 4589-2.

5 Applicability of the test

The test is used for the quality control of materials, particularly to check the incorporation of flame retardants in the material under test, and for research and development. This test is often used for inclusion in material data sheets. This test, in isolation, is insufficient to evaluate the burning behaviour and should not be used for regulations relating to safety control and consumer protection. The test provides a sensitive measure of burning materials under controlled laboratory conditions. The results are dependent upon the specimen size, shape and orientation. Aside from these restrictions, the OI test is widely used in the polymer industry as well as by cable-manufacturing companies and by those manufacturing flame retardants.

The elevated-temperature test (ISO 4589-3) provides information on the effect of a range of temperatures on the OI value. As a result, the value of the test is different from that of the single-point measurement at room temperature in that it gives an understanding of the behaviour of materials over a temperature range. This is of particular value in detecting, for example, the potential loss of effectiveness of some added flame retardant or of some technology which has been shown to be significant in certain cases. It is also useful in monitoring any chemical changes taking place at the higher temperatures which can serve to enhance or reduce the tendency to burn.

The flammability temperature test (ISO 4589-3:2017, Annex A) provides a means for assessing the way in which materials behave in a normal atmosphere by determining the temperature at which the OI of a specimen is 20,9 %.

ISO 4589-2 and ISO 4589-3 can be used to compare the particular burning characteristics of a series of plastic materials. The burning characteristics of a material are complex, and one test alone is insufficient to evaluate the material behaviour. It should be stressed that a number of tests are required to describe all the burning characteristics of a material.

It is essential that these small-scale laboratory tests be regarded as material tests only. They are primarily for assistance in development, monitoring consistency and/or pre-selection of materials, and are not for use as the sole means of assessing the potential fire hazard of a material in use.

The specific requirements of different industries have resulted in the issue of a number of similar standards, but they are not completely identical, often using different burners and conditions of ignition. These different burners and conditions may give different test results and care should be exercised in comparing results from these tests when conducted in accordance with different standards.

6 Specimen preparation

Specimen preparation should always be carried out carefully. It is important to ensure that the surfaces are clean and free from any flaws, as failure to observe these precautions can profoundly affect the burning behaviour. No shortcuts should be taken in conditioning of specimens.

7 Apparatus

7.1 General

Several types of apparatus exist that meet the specifications of ISO 4589-2 and ISO 4589-3. Some models have flow meters, valves or oxygen analysers. Some models are modular and allow upgrading to a heated model at a later date. A full description of apparatus can be found in ISO 4589-2:2017, Clause 5 and ISO 4589-3.

7.2 Measuring device

ISO 4589-2 indicates that measurement may be made either by the use of flow meters or by the use of an oxygen analyser. It is essential to use the proper calibration information for flow meters and to calibrate oxygen analysers using a standard gas. It is also necessary to check the overall equipment from time to time at the intervals specified in ISO 4589-2 to ensure that there are no leaks in the system. This is critical if for any reason the equipment is dismantled and reassembled.

7.3 Column design

The preferred inside diameter of the column for the ambient test (ISO 4589-2) is 75 mm to 100 mm. The reason for this was clearly identified in Reference [8] which showed that there was entrainment of air from outside the column. In the case of ISO 4589-3, it is recommended that a 75 mm minimum diameter column be used, again with a restricted aperture since entrainment of air was more of a problem in this equipment. Without the aperture, there would be errors introduced into the oxygen index values for particular materials. The preferred shape and size of the opening to eliminate this effect is given in ISO 4589-2 and ISO 4589-3.

7.4 Sample holder

There are two types of holder, depending on the specimen: one for rigid specimens, the other for flexible specimens. Care should be taken to ensure that the specimen holder cools to ambient temperature in ISO 4589-2 testing. Alternatively, a second holder can be used.

The elevated-temperature test (ISO 4589-3) encounters a number of problems, one of which relates to the sample holder for flexible thermoplastic materials. The recommended wire cage support (see ISO 4589-3:2017, Figure 5) is not optimum for some products. Another practice is to support the test piece between two capillary glass tubes, the assembly being lightly bound together with a single tie of fine nichrome or stainless-steel wire (nominally 200 µm gauge) and held in a small standard clamp. This non-standard practice should be used carefully and recorded in the test report.

8 Operating conditions

8.1 Calibration

The importance of following the calibration procedures as specified in both ISO 4589-2 and ISO 4589-3 cannot be stressed too highly. It is recommended that a regular check be carried out by testing a specified material, such as poly(methyl methacrylate) (PMMA), and that records of all values obtained should be kept. If there is any significant change in these values then the complete calibration procedure should be carried out to determine the cause of the change.

The PMMA should be a non-modified transparent cast sheet based on a homopolymer of methyl methacrylate in accordance with ISO 7823-1.[1] Other PMMA sheets, such as cast sheets based on a copolymer of methyl methacrylate and extruded or melt-calendered PMMA sheets can give a different burning behaviour depending on the comonomer used, the composition and the molecular mass since these characteristics affect melt behaviour during burning.

8.2 Flame application time

The conditions under which the flame is applied to the specimen should be carefully controlled. The reason for this caution is that the longer the flame is applied the higher the specimen temperature becomes. This will normally result in a fall in the OI value since it is generally the case that the higher the temperature, the lower the OI value obtained for most materials.

8.3 Gas flow

While early work indicated that the upward laminar flow in the column could be varied by ± 25 % (i.e. the linear velocity is 40 mm/s ± 10 mm/s) for the test conducted at room temperature, it has been clearly shown that such a wide variation could not be tolerated for the elevated-temperature test since the test requires a constant concentration of oxygen, which depends on flow and temperature. In ISO 4589-3, both flow and temperature are therefore closely defined, flow being controlled to within ± 2 % (i.e. the linear velocity is 40 mm/s ± 0,8 mm/s). Tighter flow control is now also applicable for ISO 4589-2 at ± 5 % (i.e. 40 mm/s ± 2 mm/s). A description on how to estimate the flow rate can be found in ISO 4589-2:2017, A.2.

8.4 Elevated-temperature routine

It is important to follow precisely the same routine from test to test when testing in accordance with ISO 4589-3, to ensure that the equilibration is identical. It has been found that the specimen holder should be in position while establishing the correct temperature and when checking that the temperature is within specification, prior to carrying out a test.

8.5 Pass/fail criteria

The flammability temperature test (ISO 4589-3:2017, Annex A) identifies a pass/fail criterion at a specified temperature and is widely used for demonstrating satisfactory behaviour at a limiting temperature. This method is only suitable for testing well characterized grades of materials. However, great caution should be exercised when testing unknown compounds in which apparently satisfactory behaviour is observed at temperatures above the flammability temperature. An example is shown in Table 1, determined using ISO 4589-3:2017, Annex A, on a compound already established in the marketplace with a flammability temperature claimed to be above 300 °C.

Table 1 — An example of Pass/Fail at test temperature

Temperature (°C)	**Pass/Fail**
262	P
272	P
274	F
277	P
277 (second test)	F
277 (third test)	P
284	P
304	P
305	P

Passes above 280 °C were associated with visible degradation of the compound, such that the flammable volatiles were swept from the column during the conditioning period prior to ignition. Therefore, careful observation and testing are very important prior to adopting ISO 4589-3:2017, Annex A in any testing routine.

9 Conclusions

ISO 4589-2 and ISO 4589-3 are of particular value in monitoring quality control and in the pre-selection of materials used in many applications, as well as for studying the effect of flame retardants added to synthetic polymers. However, Clause 5 has already described the limitations which should always be kept in mind when using these tests.