



Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)¹

This standard is issued under the fixed designation D 2863; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 This test method covers a fire-test-response procedure. This test method describes a procedure for measuring the minimum concentration of oxygen that will just support flaming combustion in a flowing mixture of oxygen and nitrogen.

1.2 Methods are provided for testing materials that are structurally 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 15 kg/m³. The methods may also be applicable to some cellular materials having an apparent density of less than 15 kg/m³. A method is provided for testing flexible sheet or film materials while supported vertically.

NOTE 1—Although this test method has been found applicable for testing other materials, the precision of the test method has not been determined for these materials, or for specimen geometry's and test conditions outside those recommended herein.

1.3 This test method may be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statement are given in Note 2.

NOTE 2—**Warning:** During the course of combustion, gases or vapors, or both, are evolved which may be hazardous to personnel.

NOTE 3—This test method and ISO 4589-2 are technically equivalent

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30).

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when using the Type A gas measurement and control device accuracy as described in 6.4.

2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning, Plastics and Electrical Insulating Materials for Testing²

D 1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples³

D 1622 Test Method for Apparent Density of Rigid Cellular Plastics²

D 2444 Test Method for Impact Resistance of Thermoplastic Pipe and Fittings by Means of a Tup (Falling Weight)⁴

D 4802 Specification for Poly(Methyl Methacrylate) Acrylic Plastic Sheet⁵

E 176 Terminology of Fire Standards⁶

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁷

2.2 ISO Standards:

4589-2 Plastics—Determination of Flammability by Oxygen Index—Part 2, Ambient Temperatures⁸

7823-1 Poly(Methylmethacrylate) Sheets—Types, Dimensions and Characteristics—Part 1—Cast Sheets⁸

3. Terminology

3.1 Definitions:

3.1.1 *ignition*—for the purpose of this standard shall imply the initiation of flaming combustion.

3.1.2 *oxygen index (OI)*—the minimum concentration of oxygen determined by the method in 9.1, expressed as volume percent, in a mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at $23 \pm 2^\circ\text{C}$ under the conditions of this test method.

3.1.3 Definitions of terms relating to fire are in accordance with Terminology E 176.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 05.05.

⁴ Annual Book of ASTM Standards, Vol 08.04.

⁵ Annual Book of ASTM Standards, Vol 08.03.

⁶ Annual Book of ASTM Standards, Vol 04.07.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

*A Summary of Changes section appears at the end of this standard.

3.2 Symbols Specific To This Test Method:

- 3.2.1 C_o —oxygen concentration in percent volume.
- 3.2.2 C_F —final value of oxygen concentration in percent volume.
- 3.2.3 C_i —each of the oxygen concentration percentages used during measurement of the last six responses in the N_T series.
- 3.2.4 O —neither the period or extent of burning exceeds the relevant limit specified in Table 1.
- 3.2.5 X —the period or extent of burning exceeds the relevant limit specified in Table 1.
- 3.2.6 N_L —series of “X” or “O” results.
- 3.2.7 N_T —series of “X” or “O” results plus five ($N_T = N_L + 5$).
- 3.2.8 σ^* —standard deviation of the oxygen concentration.
- 3.2.9 d —internal between oxygen concentration levels in percent volume.
- 3.2.10 k —a factor to be determined from Table 2.
- 3.2.11 n —number of measurements of oxygen concentration.

4. Summary of Test Method

4.1 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 behavior of the specimen is observed to compare the period for which burning continues, or the length of specimen burnt, with specified limits for each burning. By testing a series of specimens in different oxygen concentrations, the minimum oxygen concentration is determined.

5. Significance and Use

5.1 This test method provides for the measuring of the minimum concentration of oxygen in a flowing mixture of oxygen and nitrogen that will just support flaming combustion of plastics. Correlation with burning characteristics under actual use conditions is not implied.

5.2 In this procedure, the specimens are subjected to one or more specific sets of laboratory test conditions. If different test conditions are substituted or the end-use conditions are changed, it may not be possible by or from this test to predict

changes in the fire-test-response characteristics measured. Therefore, the results are valid only for the fire-test-exposure conditions described in this procedure.

6. Apparatus

6.1 *Test Chimney*, consisting of a heat-resistant glass tube of 75 to 100 mm inside diameter and 450 to 500 mm height. The bottom of the chimney or the base to which the tube is attached shall contain noncombustible material to mix and distribute evenly the gas mixture entering at this base. Glass beads 3 to 5 mm in diameter in a bed 80 to 100 mm deep have been found suitable. The chimney shall be mounted securely on the base to prevent air leaks. See Fig. 1.

NOTE 4—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 for restricting the column opening.

NOTE 5—It is helpful to place a wire screen above the noncombustible material to catch falling fragments and aid in keeping the base of the column clean.

6.2 *Specimen Holder*—Any small holding device that will support the specimen at its base and hold it vertically in the center of the chimney is acceptable. For physically self-supporting specimens, a typical arrangement (see Fig. 1) consists of a laboratory thermometer clamp inserted into the end of a glass tube held in place by glass beads or otherwise firmly supported. For supported film or sheet test specimens, the specimen shall be supported by both vertical edges in a frame equivalent to that illustrated by Fig. 2, with reference marks at 20 and 100 mm below the top of the frame. The profile of the holder and its support shall be smooth to minimize induction of turbulence in the rising flow of gas.

6.3 *Gas Supplies*, comprising pressurized sources of oxygen or nitrogen, or both, not less than 98 % pure or clean dry air, or both, (containing 20.9 % oxygen), as appropriate.

6.3.1 The gas mixture entering the chimney shall have a moisture content of < 0.1 %, 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.

NOTE 6—It should not be assumed that bottled oxygen or nitrogen will always contain < 0.1 % of water; moisture contents of 0.003 to 0.01 % are typical for commercial supplies as filled bottles > 98 % 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 %.

6.4 *Gas Measurement and Control Devices*, suitable for establishing the following accuracies when the gas velocity through the chimney is 40 ± 2 mm/s at $23 \pm 2^\circ\text{C}$;

6.4.1 *Type A*—Maintaining the volumetric concentration of oxygen in the gas mixture entering the chimney with an accuracy of ± 0.5 % of the mixture and for adjusting the concentration with a precision of ± 0.1 % of the mixture;

6.4.2 *Type B*—Maintaining the volumetric concentration of oxygen in the gas mixture entering the chimney with an accuracy of ± 1.0 % of the mixture and for adjusting the concentration with a precision of ± 0.5 % of the mixture.

TABLE 1 Criteria for Oxygen Index Measurements^A

Test Specimen Type (See Table 3)	Ignition Procedure	Alternative Criteria	
		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	propagating ignition	180	80 mm below the upper reference mark (on the frame)

^A These criteria do not necessarily produce equivalent oxygen index 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 burning drips descending the vertical faces, passes the level indicated in the column.

TABLE 2 Determination of k

Responses for the Last Five Measurements	Values of k for which the first N_L determinations are:				Responses for the Last Five Measurements ^A
	(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	OXXOX
XOXXO	1.17	1.24	1.25	1.25	OXOOX
XOXXX	0.61	0.73	0.76	0.76	OXXXX
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

^A Values of k for which the first N_L determinations are (b) X, XX, XXX, and XXXX are as given in Table 2 opposite the appropriate response in Column 6, but with the sign of k reversed, that is: $OI = C_F - kd$ (see 9.1).

NOTE 7—Systems of measurement and control that have proved satisfactory include the following:

- (a) For Type A—Needle valves on individual and mixed gas supply lines, a paramagnetic oxygen analyzer that continuously samples the mixed gas, and a flowmeter to indicate when the gas flow through the chimney is within the required limits;
- (b) For Type A or B—Calibrated orifices, gas pressure regulators and pressure gages on the individual gas supply lines; or
- (c) For Type B—Needle valves and calibrated flowmeters on the individual gas supply lines.

6.4.3 These systems require calibration after assembly to ensure that the compounded errors of the component parts do not exceed the requirements of 6.4.

6.4.3.1 Means shall be provided for checking or ensuring that the temperature of the gas mixture entering the chimney is $23 \pm 2^\circ\text{C}$. If this involves an internal probe, its position and profile shall be designed to minimize induction of turbulence within the chimney.

6.5 *Flame Igniter*, comprising a tube, with an inside diameter of 2 ± 1 mm, that can be inserted into the chimney to apply the test flame.

6.5.1 The flame fuel shall be methane or natural gas of at least 97 % purity, without premixed air. The fuel supply shall be adjusted so that the flame projects 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.

6.6 *Timing Device*, capable of measuring periods up to 5 min with an accuracy of ± 0.5 s.

6.7 *Fume Extraction System*, having 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 8—If soot-generating materials are being tested, the glass chimney may require cleaning to maintain good visibility, and the gas inlets, or inlet screen may also require cleaning to function properly.

6.8 *Thin Film Rolling Tool*—A 2 ± 0.1 mm stainless steel wire with a 0.3 ± 0.05 mm slit at one end, equivalent to that illustrated in Fig. 3.

7. Test Specimens

7.1 Cut or mold at least 15 specimens. Use Table 3 to determine specimen dimensions.

NOTE 9—It is likely that, for materials where the oxygen index is known to within $\pm 2\%$ by volume 15 test specimens will be sufficient. However, for materials of unknown oxygen index, or which exhibit erratic burning characteristics, between 15 and 30 test specimens are likely to be required.

NOTE 10—If non-standard size specimens are used, a difference in oxygen index may result.

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

7.1.2 The edges of the specimens shall be smooth and free from fuzz or burrs of material left from machining or molding.

7.1.3 Record position and orientation of test specimens with respect to any asymmetry in the sample material (see Note 12).

NOTE 11—Oxygen index results are likely to be significantly affected by differences in burning behavior, due to material inhomogeneity (for example, different levels of shrinkage when heated for specimens cut in different directions from asymmetrically-oriented thermoplastics film).

7.2 For preparation of Type VI specimens, use the rolling tool described in 6.8.

7.2.1 The rolled film is obtained by first inserting one corner of the film into the slit of the thin film rolling tool (see 6.8) and then winding the film around the wire in a spiral of 45° , as shown in Fig. 3. 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. After the winding is finished, tape the last end of the roll while the material is still on the stainless steel wire to prevent loosening. Then pull the wire out of the rolled film.

7.2.2 Cut off the rolled film at a distance of 20 mm from the top end. See Fig. 4.

7.3 For monitoring the distance over which burning occurs, mark the specimen with transverse lines at one or more levels which are dependent upon the specimen form and the ignition

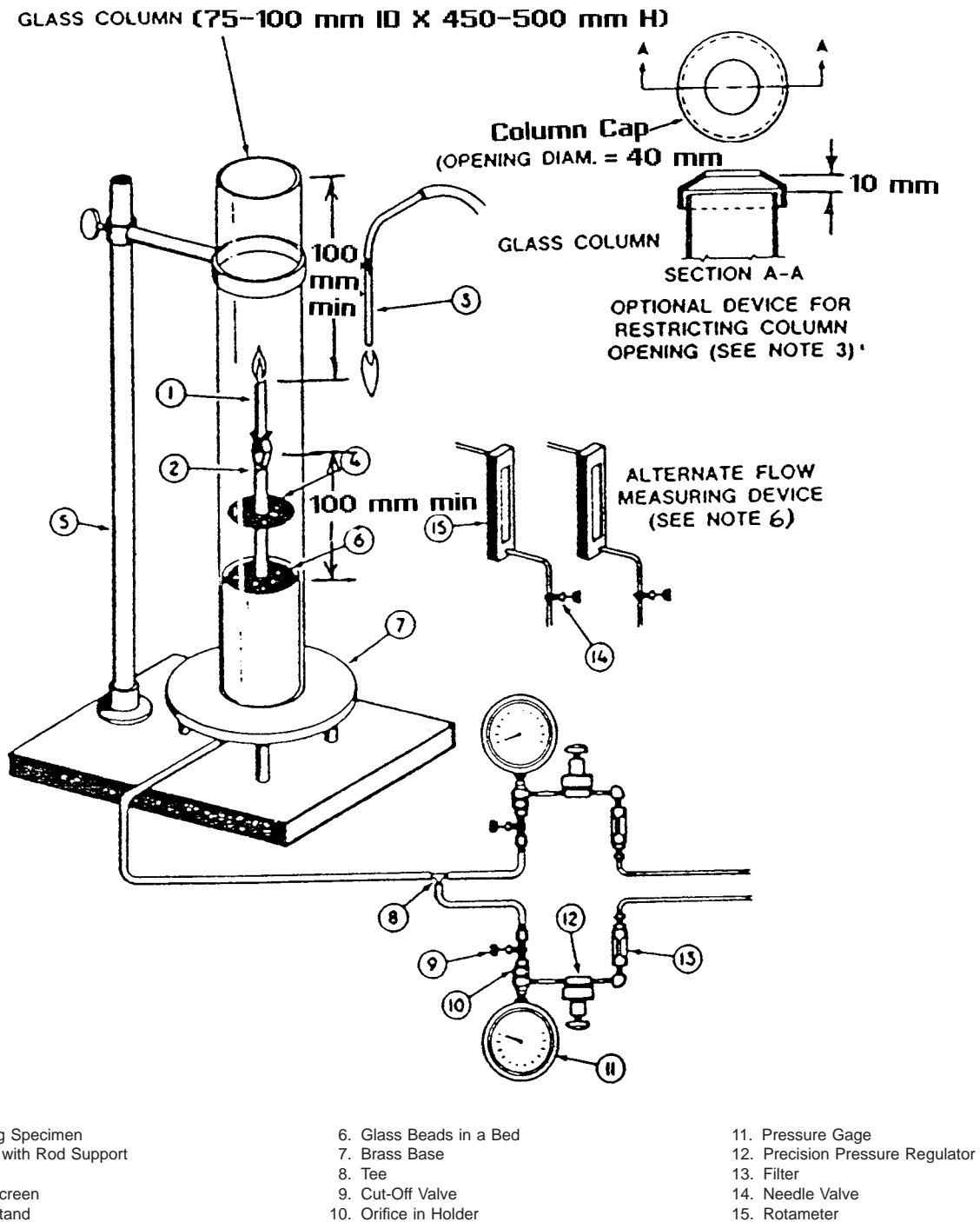


FIG. 1 Typical Equipment Layout

procedure to be used. Structurally 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 Test specimens of Type I, II, III, IV or VI are to be tested in accordance with Procedure A (see 8.8), and shall be marked 50 mm from the end to be ignited.

7.3.2 The reference marks for testing specimens of Type V are carried by the supporting frame (see Fig. 2), but it is

acceptable to mark thermally stable materials at 20 mm and at 100 mm from the end to be ignited, for convenience.

7.3.3 If specimens of Type I, II, III, IV and VI are to be tested in accordance with Procedure B (see 8.8 and 8.10), they shall be marked at 10 mm and at 60 mm from the end to be ignited.

7.4 Unless otherwise specified, each test specimen shall be conditioned for at least 88 h at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ relative humidity (RH) immediately prior to use.

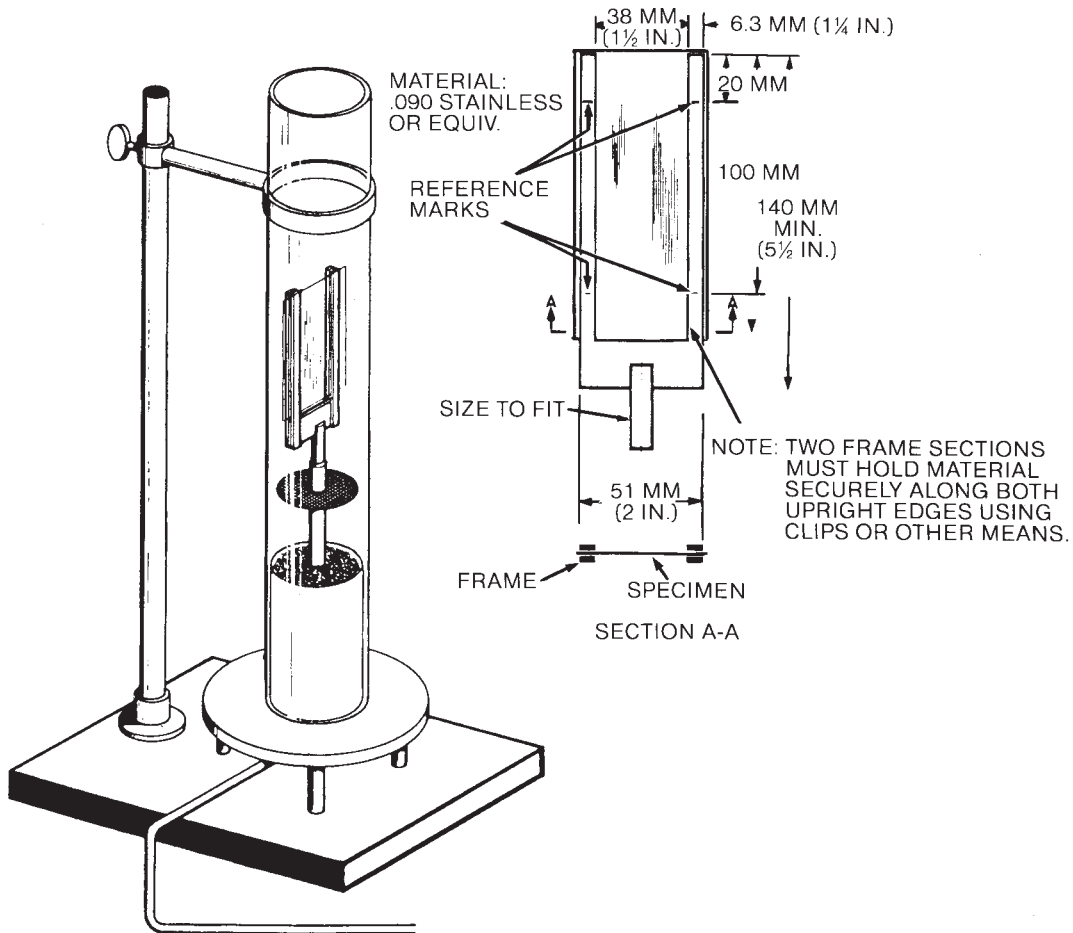


FIG. 2 Frame Design

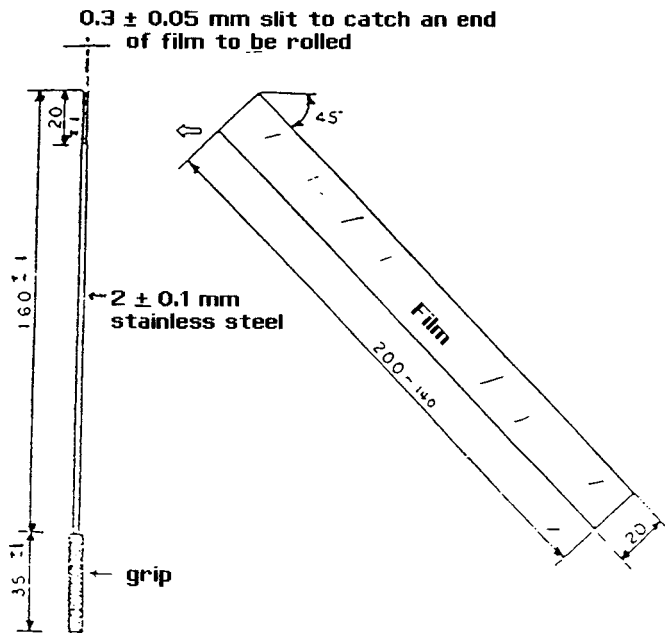


FIG. 3 Wire With a Slit

7.5 For cellular materials, the density shall be determined in accordance with Test Method D 1622.

NOTE 12—It is possible that the oxygen index samples of cellular

TABLE 3 Test Specimen Dimensions

Test Specimen Type ^A	Dimensions			Material Form
	Length, mm	Width, mm	Thickness, mm	
I	80 to 150	10 ± 0.5	4 ± 0.25	for molding 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
IV ^C	70 to 150	6.5 ± 0.5	3 ± 0.25	alternative size for self-supporting molding or sheet materials
V ^B	140 ± 5	52 ± 0.5	≤ 10.5	for flexible film or sheet
VI ^{B,D}	140 to 200	20	0.02 to 0.10	for thin film; limited to film that can be rolled by the wire specified in 6.8

^A Test specimens of Types I, II, III, and IV are suitable for materials that are self-supporting at these dimensions. Test specimens of Form V and VI are suitable for materials that require support during testing.

^B Results obtained using Type III, V, and VI 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.

^C The Type IV (ASTM) specimen will eventually be discontinued in favor of the Type 1 (ISO) specimen.

^D The test specimen of Type VI is suitable for thin film that is self-supporting when it is rolled. Dimensions in the table are of the specimen size from which the rolled form is made. If the film is very thin, it is possible that proper results will only be obtained if two or more layers are combined in the preparation of the roll to obtain proper results.

materials that contain volatile flammable blowing agents that diffuse from the sample will change with time.

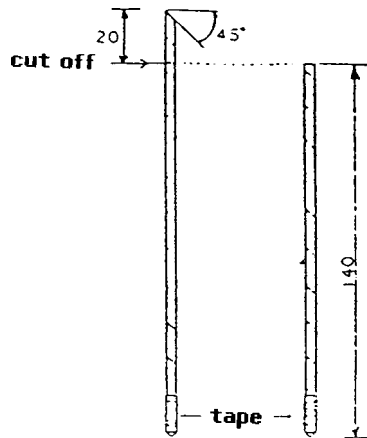


FIG. 4 Rolled Film

8. Procedure A

8.1 Calibrate the flow-measuring system using a water-sealed rotating drum meter (wet test meter) in accordance with Test Method D 1071 or by equivalent calibration devices. The maximum interval between recalibration shall be six months. A cast PMMA specimen shall be used as a verification material at least once a month. See Annex A1 for calibration method.

8.2 Maintain the ambient temperature for the test apparatus at $23 \pm 2^\circ\text{C}$. If necessary, keep the test specimens in an enclosure at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ RH and take the test specimens out of the enclosure just before testing.

8.3 Recalibrate equipment components, more frequently if necessary (see 8.1 and Annex A1).

8.4 Select an initial concentration of oxygen to be used. Whenever possible, base the initial concentration on experience of results for similar materials. Alternatively, try to ignite a test specimen in air, and note the burning behavior. If the specimen burns rapidly, select an initial concentration of about 18 % 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 extinguishing in air.

8.5 Ensure that the test chimney is vertical (see Fig. 1). Mount a specimen vertically in the center 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 Fig. 1 or Fig. 2 as appropriate).

8.6 Set the gas mixing and flow controls so that an oxygen/nitrogen mixture at $23 \pm 2^\circ\text{C}$, containing the desired concentration of oxygen, is flowing through the chimney at a rate 40 ± 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.

8.7 Verify the temperature at the lower end of the chimney to be $23 \pm 2^\circ\text{C}$ and record the oxygen concentration used as the volume percent calculated according to the equations given in Annex A2.

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

8.8.1 For specimen Types I, II, III, IV and VI use Method A, top surface ignition, as described in 8.9.

8.8.2 For specimen Type V, use Method B, propagating ignition, as described in 8.10.

NOTE 13—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 structurally 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 Test Method A. Test Method B may then be used for specimens of Type I, II, III, IV or VI.

NOTE 14—Some materials 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.

8.9 Test Method A—Top Surface Ignition:

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

8.9.2 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, just briefly, to observe whether or not the entire top surface of the specimen is burning.

8.9.3 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.

8.10 Test Method B—Propagating Ignition:

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

8.10.2 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 Type I, II, III, IV or VI on the specimen.

8.10.3 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 15—The burning portion includes any burning drips that run down the surface of the specimen.

8.11 Assessing the Burning Behavior of Individual Test Specimens:

8.11.1 Commence measurement of the period of burning as soon as the specimen has been ignited in accordance with 8.9 or 8.10 as applicable, and observe its burning behavior. If burning ceases but spontaneous re-ignition occurs within 1 s, continue the observation and measurements.

8.11.2 If neither the period or extent of burning exceeds the relevant limit specified in Table 1 for the applicable specimen, note the duration and extent of burning. This is recorded as an “O” response. (See Appendix X1.)

8.11.3 Alternatively, if either the period or extent of burning exceeds the relevant limit specified in Table 1 note the burning behavior accordingly, and extinguish the flame. This is recorded as “X” response. (See Appendix X1.)

8.11.4 Note also the burning characteristics of the material, for example, dripping, charring, erratic burning, glowing combustion or after-glow.

8.11.5 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 $23 \pm 2^\circ\text{C}$, or replace it with another so conditioned. Install the next specimen.

NOTE 16—For screening purposes, it is acceptable to invert sufficiently long specimens or trim them to remove the burnt end, and re-use them. 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.

8.12 *Selecting Successive Oxygen Concentrations:*

8.12.1 The procedure described in 8.13 and 8.14 is based upon the “up-and-down method for small samples,” using the specific case where $N_T - N_L = 5$ (see 8.14.2 and 8.14.4), with an arbitrary step size for certain changes to be made in the oxygen concentration used.

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

8.12.2.1 Decrease the oxygen concentration if the burning behavior of the preceding specimen gave an “X” response, otherwise

8.12.2.2 Increase the oxygen concentration if the preceding specimen gave an “O” response.

8.12.3 Choose the size of the change in oxygen concentration in accordance with 8.13 and 8.14, as appropriate.

8.13 *Determining the Preliminary Oxygen Concentration:*

8.13.1 Repeat the procedures specified in 8.5 to 8.6 inclusive, using oxygen concentration changes of any convenient step size, until the oxygen concentrations, in percent volume, have been found that differ by $\leq 1.0\%$ 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 8.14.

NOTE 17—The two results, at oxygen concentrations $\leq 1.0\%$ apart, which give opposite responses, do not have to be from successive specimens.

NOTE 18—At times, the concentration that gave the “O” response will not be lower than that which gave the “X” response. Such apparent inconsistencies, that are likely to be caused by the variability of the test, the equipment, or the material, are not uncommon.

NOTE 19—A format convenient for recording the information required by this and subsequent clauses is illustrated in Appendix X1.

8.14 *Oxygen Concentration Changes:*

8.14.1 Using, again, the preliminary oxygen concentration 8.13, test one specimen by repeating 8.5 to 8.11 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.

8.14.2 Change the oxygen concentration, in accordance with 8.12 using concentration changes (d) of 0.2% (see Note 20) of the total gas mixture to test further specimens in accordance with 8.4-8.12 inclusive, noting the values of C_o and corresponding responses, until a different response to that obtained in 8.14.1 is recorded.

8.14.3 The result from 8.14.1 plus those of like response from 8.14.2 constitute the N_L series of results. (See example in Appendix X1, Part 2).

NOTE 20—Where experience has shown that the requirements of 8.14.6 are usually satisfied by a value of d other than 0.2% , that value may be selected as the initial value of d .

8.14.4 Test four more specimens, in accordance with 8.4-8.12 inclusive, maintaining $d = 0.2\%$, and note the C_o used for, and response of, each specimen. Designate the oxygen concentration used for the last specimen as C_F .

8.14.5 These four results together with the last result from 8.14.2 (that is, that which differed in response from that of 8.14.1) constitute the remainder of the N_T series, so that:

$$N_T = N_L + 5$$

See example in Appendix X1, Part 2.

8.14.6 Calculate the estimated standard deviation, σ^* , 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 is satisfied, calculate the oxygen index in accordance with 9.1, otherwise:

$$\frac{2\sigma^*}{3} < d < 1.5\sigma^*$$

8.14.6.1 If $d < 2\sigma^*/3$, repeat 8.14.2-8.14.6 using increased values for d , until the condition is satisfied, or

8.14.6.2 If $d > 1.5\sigma^*$, repeat 8.14.2-8.14.6 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. Calculation

9.1 *Oxygen Index:*

9.1.1 Calculate the oxygen index (OI), expressed as a percentage by volume, from the following relationship:

$$OI = C_F + kd$$

where:

C_F = the final value of oxygen concentration, in percent volume to one decimal place, used in the series of N_T measurements performed in accordance with 8.14 and noted in accordance with 8.14.4,

d = the interval, in percent volume to at least one decimal place, between oxygen concentration levels used and controlled in accordance with 8.14, and

k = a factor to be obtained from Table 2 as described in 9.2.

9.1.2 For the purpose of calculation of σ^* , as required by 8.14.6 and 9.3, the OI shall be calculated to two decimal places.

9.1.3 For the purpose of reporting *OI* results, express *OI* values to the nearest $\pm 0.1\%$, with exact intermediate results being rounded downwards.

9.2 Determination of *k*:

9.2.1 The value and sign of *k* are dependent upon the pattern of the responses of specimens tested in accordance with 8.14. Determine them from Table 2 as follows:

9.2.1.1 If the response of the specimen tested according to 8.14.1 was “O”, so that the first contrary response (see 8.14.2) was an “X”, refer to Column 1 of Table 2 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.14.4. 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 8.14.1 and 8.14.2, or

9.2.1.2 If the responses of the specimen tested according to 8.14.1 was “X”, so that the first contrary response was an “O”, refer to the sixth column of Table 2 to select the row for which the last four response symbols correspond to those found when testing in accordance with 8.14.4. 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 8.14.1 and 8.14.2 but the sign of *k* must be reversed, so that negative values shown in Table 2 for *k* become positive, and vice versa.

NOTE 21—An example of the determination of the calculation of an *OI* is given in Annex A2.

9.3 Standard Deviation of Oxygen Concentration Measurements:

9.3.1 For the purposes of 8.14.6 calculate the estimated standard deviation, σ^* , of oxygen concentration measurements from the relationship:

$$\sigma^* = \left[\frac{\sum(C_i - OI)^2}{n - 1} \right]^{\frac{1}{2}}$$

where:

- C_i = in turn, each of the percent oxygen concentrations used during measurement of the last six responses in the N_T series of measurements;
- OI* = the oxygen index value, calculated in accordance with 9.1; and
- n* = the number of measurements of oxygen concentration contributing to $\sum(C_i - OI)^2$.

NOTE 22—For this test method, $n = 6$, in accordance with 8.14.6. For $n < 6$, the test method loses precision. For $n > 6$, alternative statistical criteria would apply.

Table 2 values of *k* for calculating oxygen index concentration from determinations made by Dixon’s “Up-and-Down” method.

10. Procedure B—Comparison With a Specified Minimum Value for Oxygen Index (Short Procedure)

10.1 If the actual oxygen index of a material is needed or in case of a dispute, Procedure A shall be used.

10.2 Set up the apparatus and test specimen in accordance with 8.1-8.7 except that the specified minimum concentration of oxygen shall be selected for the purposes of 8.4.

10.3 Ignite the test piece in accordance with 8.8.

10.4 Using up to three specimens, assess the burning behavior of each specimen in accordance with 8.11.

10.4.1 If for at least two out of the three specimens thus tested the flame is extinguished before the relevant criteria from Table 1 are exceeded, that is, 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 Section 8, as appropriate.

11. Report

11.1 Report the following information:

11.1.1 A reference to this test method;

11.1.2 Date of testing;

11.1.3 A statement that test results relate only to the behavior of the test specimens under the conditions of this test method and that these results must not be used to infer the fire hazards of the material in other forms or under other fire conditions;

11.1.4 Identification of the material tested, including, where relevant, the type of material, density, previous history, the specimen orientation with respect to any anisotropy in the material or sample, and the date of manufacture with lot number;

11.1.5 The oxygen index (*OI*) as determined in 9.1;

11.1.6 The test specimen type or dimensions;

11.1.7 The gas measurement and control device accuracy (Type A or B);

11.1.8 The ignition procedure used (Test Method A or B);

11.1.9 When Procedure B is used, indicate the relevant specified minimum oxygen index (*OI*) of the material and report if the material tested had a lower or higher oxygen index (*OI*);

11.1.10 If applicable, the estimated standard deviation and the oxygen concentration increment used, if other than 0.5 %;

11.1.11 A description of any relevant characteristics or behavior, such as charring, dripping, severe shrinkage, erratic burning, after-glow; and

11.1.12 Any variations from the requirements of this test method.

12. Precision and Bias

12.1 Table 4 is based on a round robin⁹ conducted in 1999 in accordance with Practice E 691, involving 8 materials tested by 12 laboratories. For each material, the samples were prepared by the supplier of the material and conditioned at the laboratories that tested them. Each laboratory obtained 2 test results for each material. All laboratories utilized gas measurement and control devices in accordance with 6.4.1 (Type A) for accuracy and precision.

12.2 Table 4 does not include three laboratories that participated in the round robin and utilized measurement and control devices in accordance with 6.4.2 (Type B) for accuracy and precision. The results from these laboratories could not be

⁹ Supporting data are available from ASTM Headquarters. Request RR: D20-1031.

TABLE 4 Oxygen Index (OI), %

Material	Specimen Type	Procedure	Average	S _r ^A	S _R ^B	r ^C	R ^D
PMMA-1	III	A	17.7	0.10	0.23	0.28	0.65
PMMA-2	III	A	17.8	0.13	0.25	0.37	0.70
PVC, plasticized	I	A	38.4	0.60	2.03	1.67	5.68
ABS, FR	I	A	26.8	0.58	1.09	1.61	3.07
PF, thermoset	I	A	49.7	0.36	1.74	1.01	4.87
PS, foam	II	A	20.9	0.44	0.97	1.22	2.71
PC, sheet	V	B	26.1	0.31	1.42	0.88	3.97
PET, film	VI	A	21.9	0.64	1.48	1.79	4.15

^AS_r is the within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories:

$$S_r = [[(S_1)^2 + (S_n)^2]/n]^{1/2}$$

^BS_R is the between-laboratories reproducibility, expressed as standard deviation:

$$S_R = [S_L^2 + S_L^2]^{1/2}$$

where S_L is the standard deviation of laboratory means.

^Cr is the within-laboratory critical interval between two test results = 2.8 × S_r.

^DR is the between-laboratories critical interval between two test results = 2.8 × S_R.

incorporated into this precision statement, due to the limited number of participants to comply with Practice E 691 guidelines. Therefore, the resulting precision is provided in Annex A3.

NOTE 23—Two statistically designed interlaboratory round robins for precision evaluation were conducted earlier, one with 18 laboratories and 5 materials (supporting data are available from ASTM Headquarters, request RR: D20-0102) and one with 29 laboratories and 12 materials.⁹ The first study indicated a higher standard deviation for specimens with higher oxygen index, while the second study indicated a dependence of precision with the type of specimen used.

NOTE 24—**Caution:** The explanation of “r” and “R” (12.3-12.3.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The test results and precision in Table 4

should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 12.3-12.3.3 would then be valid for such data.

12.3 *Concept of “r” and “R” in Table 4*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing 2 specimens for each test result, then:

12.3.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “r” value for that material. The concept of “r” is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment in the same laboratory.

12.3.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the “R” value for that material. The concept of “R” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.3.3 Any judgement in accordance with 12.3.1 or 12.3.2 would have an approximate 95 % (0.95) probability of being correct.

12.4 *Bias*—There are no recognized standards on which to base an estimate of bias for this test method.

13. Keywords

13.1 candle-like combustion; minimum oxygen concentration; oxygen; oxygen concentration; oxygen index

ANNEXES

(Mandatory Information)

A1. CALIBRATION OF EQUIPMENT

A1.1 *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.

A1.2 Gas-Flow Rates:

A1.2.1 The system for indicating the gas-flow rate through the chimney, to satisfy 6.4 and 8.6 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.

A1.2.2 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, for example, by using the following equation:

$$F = 1.27 \times 10^6 \frac{q_v}{D^2}$$

where:

F = flow rate through the chimney, mm/s,

q_v = total gas-flow at 23 ± 2°C through the chimney, L/s,

D = diameter of the bore of the chimney, mm.

A1.3 Oxygen Concentration:

A1.3.1 The concentration of oxygen in the mixture of gases flowing into the chimney shall be checked to an accuracy of 0.1 % of mixture, either by sampling the chimney atmosphere for analysis or by using an independently calibrated analyzer in situ. If an oxygen analyzer is incorporated in the equipment, this shall be calibrated using the following gases, each of which shall conform with 6.3 for purity and moisture content:

A1.3.1.1 Any two gases selected from the following: nitrogen; oxygen; or clean air; and

A1.3.1.2 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.

A1.4 *Verification of Complete Equipment*—For monthly verification, in accordance with 8.1, use Type III specimens of the PMMA. The PMMA shall be a non-modified transparent cast sheet based on a homopolymer of methylmethacrylate in

accordance with Specifications D 4802, Category A-1 (ISO7823-1 for Cast Sheets). The PMMA shall have an oxygen index (OI) between 17.0 and 18.5.

NOTE A1.1—Other PMMA sheets such as cast sheets based on copolymer of methylmethacrylate, extruded or melt calendared PMMA sheets may give a different burning behavior depending on the comonomer used, its contents or molecular weight which effects melt behavior when being burned.

A2. CALCULATION OF OXYGEN CONCENTRATION

A2.1 Oxygen concentrations required for the purposes of Section 8 shall be calculated according to the equation:

$$C_o = \frac{100 V_o}{V_o + V_N} \quad (\text{A2.1})$$

where:

C_o = oxygen concentration, in percent by volume,
 V_o = volume of oxygen per volume of mixture, at 23°C,
 and
 V_N = volume of nitrogen per volume of mixture, at 23°C.

NOTE A2.1—If an oxygen analyzer is used, the oxygen concentration should be determined using the readout from the particular instrument used.

NOTE A2.2—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 % purity or with nitrogen containing 0.5 % of oxygen, the oxygen concentration, in percent by volume, should be calculated using the relationship:

$$C_o = \frac{98.5 V'_o + 20.9 V'_A + 0.5 V'_N}{V'_o + V'_A + V'_N} \quad (\text{A2.2})$$

where:

V'_o = volume of oxygen stream used, per volume of mixture,
 V'_A = volume of air stream used, per volume of mixture, and
 V'_N = 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.

A3. PRECISION AND BIAS USING TYPE B CONTROL DEVICE ACCURACY AND PRECISION

A3.1 Table A3.1 is based on a limited round robin⁹ conducted in 1999, along with the round robin in Section 14. This limited round robin does not comply with Practice E 691 because only 3 laboratories participated on a limited number of materials using the Type B control devices for accuracy and precision. For each material, the samples were prepared by the supplier of the material and conditioned at the laboratories that tested them. Each laboratory obtained either 1 or 2 test results for each material. All laboratories utilized gas measurement and control devices in accordance with 6.4.2 (Type B) for accuracy and precision.

A3.2 **Caution**—The explanations of “r” and “R” (A3.3-

A3.3.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The test results and precision in Table A3.1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles in Practice E 691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of A3.3-A3.3.3 would then be valid for such data.

A3.3 *Concept of “r” and “R” in Table 4*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing 2 specimens for each test result, then:

TABLE A3.1 Oxygen Index (OI), %^A

Material	Specimen Type	Procedure	Average	S_r^B	S_R^C	r^D	R^E
PMMA-1	III	A	17.1	0.20	0.43	0.57	1.21
PMMA-2	III	A	17.4	0.00	0.61	0.00	1.72
PVC, plasticized	I	A	48.0	0.27	7.84	0.77	22.0
ABS, FR	I	A	26.5	0.27	11.1	0.95	31.1
PF, thermoset	I	A	52.2	0.15	0.82	0.42	2.30
PS, foam	II	A	23.3	0.00	3.04	0.00	8.50

^ABased on data from only two laboratories.

^B S_r is the within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories:

$$S_r = [[(S_1)^2 + (S_n)^2]/n]^{1/2}$$

^C S_R is the between-laboratories reproducibility, expressed as standard deviation:

$$S_r = [S_1^2 + S_L^2]^{1/2}$$

where S_L is the standard deviation of laboratory means.

^D r is the within-laboratory critical interval between two test results = $2.8 \times S_r$.

^E R is the between-laboratories critical interval between two test results = $2.8 \times S_R$.

A3.3.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “r” value for that material. The concept of “r” is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment in the same laboratory.

A3.3.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the “R” value for that material. The concept of “R” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

A3.3.3 Any judgement in accordance with A3.3.1 or A3.3.2 would have an approximate 95 % (0.95) probability of being correct.

APPENDIX

(Nonmandatory Information)

X1. TYPICAL TEST RESULTS SHEET

Material: Phenolic laminate
 Specimen Type: I (4 mm thick)
 Ignition procedure: A
 Conditioning procedure: 23/50
 Oxygen concentration increment (d): 0.2

Oxygen Index (concentration, %): 29.5 (rounded to 0.1 %)
 σ^* : 0.152
 Date of test: 13/10/80
 Laboratory No. 19 Test No. 1

Determination of oxygen concentration for one pair of "X" and "O" response at $\leq 1\%$ O₂ concentration interval (in accordance with 8.13)

Oxygen concentration, %
 Burning period, s
 Length burnt, mm
 Response, "X" or "O"

25.0	35.0	30.0	32.0	31.0				
10	>180	140	>180	>180				
O	X	O	X	X				

Oxygen concentration of the "O" response for the pair = 30.0 (being the concentration to be used again for the first measurement in Part 2)

Determination of oxygen index value (in accordance with 8.4)

Step size to be used for successive changes in oxygen concentration of $d\% = 0.2\%$ (initially to be 0.2 %, unless otherwise instructed):

Oxygen concentration, %
 Burning period, s
 Length burnt, mm
 Response, "X" or "O"

N _T series measurements										
N _T series measurements (8.6.1 + 8.6.2)						(8.6.3)			C _F	
30.0	29.8	29.6	29.4			29.4	29.6	29.4	29.6	29.8
>180	>180	>180	150			150	>180	110	185	>180
X	X	X	-----	-----	-----	O	X	O	O	X
Column (2, 3, 4 or 5): 4						Row (1 to 16): 7				
k value from table 4: 1.25						Hence k = - 1.25				

$OI = C_F + kd = 29.8 + (-1.25 \times 0.2)$
 = 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 Part 3)

Part 3: Verification of Step size d % oxygen concentration (in accordance with 8.14.6 and 9.3)–

Last six result	Oxygen Concentration, %			
	C _F	OI	C _i - OI	(C _i - OI) ²
1	29.8	29.55	0.25	0.0625
2	29.6	29.55	0.05	0.0025
3	29.4	29.55	-0.15	0.0225
4	29.6	29.55	0.05	0.0025
5	29.4	29.55	-0.15	0.0225
n	29.6	29.55	0.05	0.0025
	Total $\Sigma(C_i - OI)^2$			0.115
	Column C _i contains the oxygen concentrations used for the measurements of C _F and for each of the 5 preceding measurements, for n = 6			

Estimation of Standard deviation:

$$\sigma^* = \left[\frac{\Sigma(C_i - OI)^2}{n - 1} \right]^{1/2} = \left[\frac{0.115}{5} \right]^{1/2} = 0.152$$

$$\frac{2\sigma^*}{3} = 0.101 \quad d = 0.2 \quad \frac{3\sigma^*}{2} = 0.227$$

If $2 \frac{\sigma^*}{3} < d < \frac{3\sigma^*}{2}$ or if $0.2 = d > \frac{3\sigma^*}{2}$; OI is valid

Otherwise

if $2 \frac{\sigma^*}{3} > d$, repeat Part 2 using a larger value for d; or

if $\frac{3\sigma^*}{2} < d$, repeat Part 2 using a smaller value for d.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 2863 – 00:

- (1) A round robin was conducted in 1999 and Section 12 (Precision and Bias) was replaced with the precision results for the Type A measurement and control device.
- (2) The 1999 round robin also included limited data using the precision results for the Type B measurement and control

device, which was added as Annex A3.

- (3) Added requirements for the PMMA verification material in A1.4.
- (4) Deleted Appendix X2, which cited a previous round robin, and added Note 23 to indicate that this old round robin data is available from ASTM Headquarters.

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