



# Standard Test Method for Measuring Liquid and Solid Material Fire Limits in Gaseous Oxidants<sup>1</sup>

This standard is issued under the fixed designation G125; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a procedure for measuring the threshold-limit conditions to allow equilibrium of combustion of materials in various oxidant gases under specific test conditions of pressure, temperature, flow condition, fire-propagation directions, and various other geometrical features of common systems.

1.2 This test method is patterned after Test Method D2863-95 and incorporates its procedure for measuring the limit as a function of oxidant concentration for the most commonly used test conditions. Sections 8, 9, 10, 11, 13, and for the basic oxidant limit (oxygen index) procedure are quoted directly from Test Method D2863-95. Oxygen index data reported in accordance with Test Method D2863-95 are acceptable substitutes for data collected with this standard under similar conditions.

1.3 This test method has been found applicable to testing and ranking various forms of materials. It has also found limited usefulness for surmising the prospect that materials will prove “oxygen compatible” in actual systems. However, its results do not necessarily apply to any condition that does not faithfully reproduce the conditions during test. The fire limit is a measurement of a behavioral property and not a physical property. Uses of these data are addressed in Guides G63 and G94.

NOTE 1—Although this test method has been found applicable for testing a range of materials in a range of oxidants with a range of diluents, the accuracy has not been determined for many of these combinations and conditions of specimen geometry, outside those of the basic procedure as applied to plastics.

NOTE 2—Test Method D2863-95 has been revised and the revised Test

<sup>1</sup>This test method is under the jurisdiction of ASTM Committee G04 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods. Portions have been adopted from Test Method D2863-95, which is under the jurisdiction of ASTM Committee D20 on Plastics.

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Method has been issued as D2863-97. The major changes involve sample dimensions, burning criteria and the method for determining the oxygen index. The aim of the revisions was to align Test Method D2863 with ISO 4589-2. Six laboratories conducted comparison round robin testing on self-supporting plastics and cellular materials using D2863-95 and D2863-97. The results indicate that there is no difference between the means provided by the two methods at the 95 % confidence level. No comparison tests were conducted on thin films. The majority of ASTM Committee G4 favors maintaining the D2863-95 as the backbone of G125 until comprehensive comparison data become available.

1.4 One very specific set of test conditions for measuring the fire limits of metals in oxygen has been codified in Test Method G124. Test Method G124 measures the minimum pressure limit in oxygen for its own set of test conditions. Its details are not reproduced in this standard. A substantial database is available for this procedure, although it is much smaller than the database for Test Method D2863-95. (**Warning**—During the course of combustion, gases, vapors, aerosols, fumes or any combination of these are evolved which may be hazardous.) (**Warning**—Adequate precautions should be taken to protect the operator.)

1.5 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.6 *This basic standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to directly 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. The standard has more applicability in this regard at predicting the fire behavior of materials and components that are close in size to the test condition, than for systems that are much different (for example: comparing a test rod to a valve seat rather than comparing a test rod to a house or a particle).*

1.7 *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.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D618 Practice for Conditioning Plastics for Testing
- D1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples
- D2444 Test Method for Determination of the Impact Resistance of Thermoplastic Pipe and Fittings by Means of a Tup (Falling Weight)
- D2863 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)
- D2863-95 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)
- D2863-97 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)
- G63 Guide for Evaluating Nonmetallic Materials for Oxygen Service
- G94 Guide for Evaluating Metals for Oxygen Service
- G124 Test Method for Determining the Combustion Behavior of Metallic Materials in Oxygen-Enriched Atmospheres
- G128 Guide for Control of Hazards and Risks in Oxygen Enriched Systems

### 2.2 Other Standards:

- ISO 4589-2 Plastics—Determination of burning behavior by oxygen index—Part 2: Ambient temperature test<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *oxygen compatibility, n*—the ability of a substance to coexist with both oxygen and a potential source(s) of ignition within the acceptable risk parameter of the user (at an expected pressure and temperature). (See Guide G128.)

3.1.2 *oxygen index, n*—the minimum concentration of oxygen, expressed as a volume percent, in a mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at room temperature under the conditions of Test Method D2863. (See Test Method D2863.)

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *fire limit, n*—the threshold limit conditions that will just support sustained combustion of a material under a combination of specified conditions and at least one variable parameter (typically oxidant concentration, diluent nature, pressure, temperature, geometry, flow or flame parameters, etc.).

3.2.2 *oxidant compatibility, n*—the ability of a substance to coexist with both an oxidant and a potential source(s) of ignition within the acceptable risk parameter of the user (at an expected pressure and temperature).

3.2.3 *oxidant index, n*—the minimum concentration of an oxidant such as oxygen, nitrous oxide, fluorine, etc., expressed as a volume percent, in a mixture of the oxidant with a diluent such as nitrogen, helium, carbon dioxide, etc., that will just support sustained combustion of a material initially at given conditions of temperature, pressure, flow conditions, propagation direction, etc. (See also, *oxygen index*.)

3.2.3.1 *Discussion*—The oxidant index may be more specifically identified by naming the oxidant: oxygen limit (or index), nitrous oxide limit (or index), fluorine limit (or index), etc. Unless specified otherwise, the typical oxidant is taken to be oxygen, the typical diluent is taken to be nitrogen, and the typical temperature is taken as room temperature.

3.2.4 *pressure limit*—the minimum pressure of an oxidant (or mixture) that will just support sustained combustion of a material initially at given conditions of oxidant concentration, temperature, flow condition, propagation direction, etc.

3.2.4.1 *Discussion*—The pressure limit may be more specifically identified by naming the oxidant: oxygen pressure limit, nitrous oxide pressure limit, fluorine pressure limit, etc.

3.2.5 *temperature limit*—the minimum temperature of an oxidant (or mixture) that will just support sustained combustion of a material initially at given conditions of oxidant concentration, temperature, flow condition, propagation direction, etc.

3.2.5.1 *Discussion*—The temperature limit may be more specifically identified by naming the oxidant: oxygen temperature limit, nitrous oxide temperature limit, fluorine temperature limit, etc.

## 4. Summary of Test Method

4.1 The threshold limit condition (minimum oxidant concentration, minimum pressure, minimum temperature, etc.) that will just support sustained combustion under equilibrium conditions is measured in a test apparatus. The equilibrium is established by the relation between the heat generated from the combustion of the specimen (that may be augmented by the heat of decomposition of some oxidants) and the heat lost to the surroundings as measured by one or the other of two arbitrary criteria, namely, a time of burning or a length of specimen burned. This point is approached from both sides of the critical threshold condition in order to establish the fire limit.

## 5. Significance and Use

5.1 This test method provides for measuring of the minimum conditions of a range of parameters (concentration of oxidant in a flowing mixture of oxidant and diluent, pressure, temperature) that will just support sustained propagation of combustion. For materials that exhibit flaming combustion, this is a flammability limit similar to the lower flammability limit, upper flammability limit, and minimum oxidant for

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> ISO 4589-2 First edition 1996-07-15, International Organization for Standardization, Geneva, Switzerland, 1996.

combustion of gases (1).<sup>4</sup> However, unlike flammability limits for gases, in two-phase systems, the concept of upper and lower flame limits is not meaningful. However, limits can typically be determined for variations in other parameters such as the minimum oxidant for combustion (the oxidant index), the pressure limit, the temperature limit, and others. Measurement and use of these data are analogous to the measurement and use of the corresponding data for gaseous systems. That is, the limits apply to systems likely to experience complete propagations (equilibrium combustion). Successful ignition and combustion below the measured limits at other conditions or of a transient nature are not precluded below the threshold. Flammability limits measured at one set of conditions are not necessarily the lowest thresholds at which combustion can occur. Therefore direct correlation of these data with the burning characteristics under actual use conditions is not implied.

## 6. Abstract

6.1 A well-established procedure for measuring an oxidant limit, the oxygen index, of plastics (See Test Method D2863) is reviewed, then variations commonly used to collect data for oxidant compatibility purposes are described. In the test, a series of specimens is placed in a preadjusted oxidant mixture and deliberately ignited. Specimens that do not “burn” are retested in higher concentrations. Specimens that do burn are retested in lower concentrations. When the operator is confident that the threshold has been determined by a suitable number and spread of negative tests below the threshold, the lowest positive is reported as the oxidant index.

6.2 Similar test methods apply when the oxidant concentration is held constant and the temperature, pressure or other key factor is varied. In some cases, apparatus modification or replacement is necessary, such as a pressurized vessel is required to complete some tests (see Test Method G124). Relatively little work (1-18) has been done using oxidants other than oxygen, diluents other than nitrogen, pressure, temperature, or other properties as the variable parameter.

## 7. Variations

7.1 A number of variations of the procedure have been used. The principle variables have been oxidant, diluent, pressure, temperature, flow condition and flow direction. Relatively little work has been done for most of these variables (1-18). There is some qualitative and even quantitative understanding of the manner in which these variables affect the fire limits of materials, but the understanding is largely incomplete. Finally, the database for most combinations of variables is small (only Test Method D2863-95 and Test Method G124 have significant databases) and so the ability to draw strong conclusions is limited. Nonetheless, where data is obtained for two or more materials, these data are useful to the evaluation of those materials. Care is necessary in comparing materials that have not been tested in similar procedures.

7.2 *Oxidants*—Changing the oxidant may cause the greatest changes in results for other constant conditions (1, 2, 3).

Oxidants behave dramatically different, because their basic chemistry with differing materials is different. For example, even though nitrous oxide is a combination of nitrogen and oxygen, it behaves much differently than a similar oxygen/nitrogen mixture. During combustion, nitrous oxide decomposes to release heat that renders it more able to support combustion than a simple mixture. Fluorine is very reactive and produces more gaseous product species which changes its behavior in higher purity oxidant. There are data available in varying amounts for the oxidants: oxygen, nitrous oxide, fluorine, nitrogen trifluoride, and nitrogen (nitrogen is an oxidant in some cases, a diluent in others).

7.3 *Diluents*—Varying diluents can have a significant effect although much less impressive than oxidant, pressure or even flow direction (1-8). Diluent’s thermal conductivity and heat capacity appear to be the most significant properties. Reactivity is a second issue. For example, nitrogen does not participate in most polymer combustions but can react with some metals and exhibit widely different diluent natures. Among the diluents used to date are nitrogen, helium, argon, carbon dioxide, neon, and xenon.

7.4 *Pressures*—Pressure has a dramatic effect on the fire limit (1, 4, 5, 8, 9, 10, 11). The role of pressure is complex, yet it is one of the most important variables because oxygen systems employ a range of pressures to 82 MPa (12000 psig).

7.5 *Temperatures*—The role of temperature appears to be among the more straightforward higher temperatures appear to imply lower fire limits. The effect can be gradual or abrupt. For example PTFE will not burn in the oxygen index test at room temperature, but burns nicely at just a few degrees above room temperature (9, 12).

### 7.6 *Flow and Propagation Schemes:*

7.6.1 Variations in the flow scheme and the direction of propagation have dramatic effect on the fire limit. The earliest work on oxygen index (8) demonstrated that for polymers, a much lower index resulted if the flow carried the hot combustion products over the unburned portions of a specimen. Later work confirms the observation (9, 13, 14) (Therefore in most polymer testing, lower limits were measured if the specimens were bottom ignited with upward flow or top ignited with downward flow than with the standard top ignition with upward flow. The effect is similar but less dramatic with metals combustion. Indeed, the standard top-ignition upward-flow conditions of Test Method D2863 and bottom-ignition conditions of Test Method G124 were chosen to facilitate the measurement and its precision rather than to obtain the lowest-possible limit measurement. Similarly, in stagnant systems, a concentration of inert combustion products, diluents, and even impurities in the oxidant gases can yield higher limits than otherwise. Limited work has been done with most of the combinations of vertical (upward or downward) flow and vertical directions of propagation.

7.6.2 Variations in the flow scheme have been used (3, 6, 7) in which a fire was established in the bore (intraluminal flame) of a flowing horizontal tube. These demonstrated that the effect of diluents can be inverted at high flow rates and that there can be an optimum velocity that yields a minimum fire limit.

### 7.7 Geometries:

7.7.1 The influence of geometry is not well understood, but work shows that specimen size (8) is not a particularly significant variant in polymer tests performed as in Sections 8, 9, 10, 11, 13, and 14, but that the change from rod to tubing can have a dramatic effect on the fire limit of stainless steel but may have a much smaller effect on carbon steel (5).

7.7.2 Powders and liquids have been tested (15, 16) with slight modification of Test Method D2863-95. Typically, powders have had lower fire limits than their bulk counterparts. Few materials can be tested as both liquid and solid. However, data suggest that if materials could be tested as solids or gases, the gases would exhibit a lower fire limit (1).

## 8. Apparatus

8.1 *Test Column*, consisting of a heat-resistant glass tube of 75 mm minimum inside diameter and 450 mm minimum height. The bottom of the column 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 (an example is shown in Fig. 1).

NOTE 3—A column with a 95-mm inside diameter and 210 mm high with a restricted upper opening (diameter = 50 mm) has been found to give equivalent results.

NOTE 4—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.

8.2 *Specimen Holder*—Any small holding device that will support the specimen at its base and hold it vertically in the center of the column 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 other forms, such as film and thin sheet, the frame shown in Fig. 2 shall be used and held in place by the above tube. The test specimen must be held securely along both upright edges by the frame, using clips or other means.

8.3 *Gas Supply*—Commercial grade (or better) oxygen and nitrogen shall be used. If an air supply is used with oxygen or nitrogen, it must be clean and dry.

8.4 *Flow Measurements and Control Devices*—Suitable flow measurement and control devices shall be available in each line that will allow monitoring the volumetric flow of each gas into the column with 1 % in the range being used. After the flow is measured in each line, the lines should be joined to allow the gases to mix before being fed into the column.

NOTE 5—One satisfactory flow control consists of calibrated jeweled orifices<sup>5</sup> pressure regulating devices, and gas gages. An equally satisfactory system consists of needle valves and rotameters meeting the requirements of 8.4.

8.5 *Ignition Source*—The igniter shall be a tube with a small orifice (1 to 3 mm in diameter) having a hydrogen, propane, or

other gas flame at the end that can be inserted into the open end of the column to ignite the test specimen. A suitable flame may be from 6 to 25 mm long.

8.6 *Timer*—A suitable timer capable of indicating at least 10 min and accurate at 5 s shall be used.

8.7 *Soot, Fumes, and Heat Removal*—To ensure the removal of toxic fumes, soot, heat, and other possible noxious products, the column shall be installed in a hood or other facilities providing adequate exhaust.

NOTE 6—If soot-generating specimens are being tested, the glass column becomes coated on the inside with soot and should be cleaned as often as necessary for good visibility.

## 9. Test Specimens

9.1 Cut a sufficient number of specimens (normally 5 to 10) from the material to be tested. Use Table 1 to determine specimen dimensions.

9.1.1 Test the specimens in the as-received condition unless otherwise agreed upon.

9.1.2 Moisture content of some materials has been shown to affect the oxygen index. Where a material is suspected to be affected by retained moisture, condition the specimens in accordance with Procedure A of Test Methods D618.

NOTE 7—If non-standard size specimens are used, a difference in oxygen index may result.

9.1.3 For Type C specimens, make comparisons only between materials of similar densities.

NOTE 8—For certain types of cellular plastics, the direction of anisotropy may have an effect and should be evaluated unless a particular direction has previously been agreed upon.

9.1.4 Test Type D materials in the as-received thickness, but make comparisons only between material of the same thickness.

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

## 10. Procedure

10.1 Calibrate the flow-measuring system using a water-sealed rotating drum meter (wet test meter) in accordance with Test Method D1071 or by equivalent calibration devices. It is recommended that this calibration be repeated at least every six months.

NOTE 9—One step in the calibration should be to check carefully for leaks at all joints.

10.2 The test shall be conducted at room temperature conditions in accordance with Practice D618.

10.3 Clamp the specimen vertically in the approximate center of the column with the top of the specimen at least 100 mm below the top of the open column.

NOTE 10—If a restricted opening column is used (see Note 4), the top of the specimen should be at least 40 mm below the opening.

10.4 Select the desired initial concentration of oxygen based on experience with similar materials. If there is no experience with similar material, light a specimen in the air and note the burning. If the specimen burns rapidly, start at a concentration

<sup>5</sup> Andersen, J.W., and Friedman, R., "An Accurate Gas Metering System for Laminar Flow," RSINA, Vol 20, 1949.

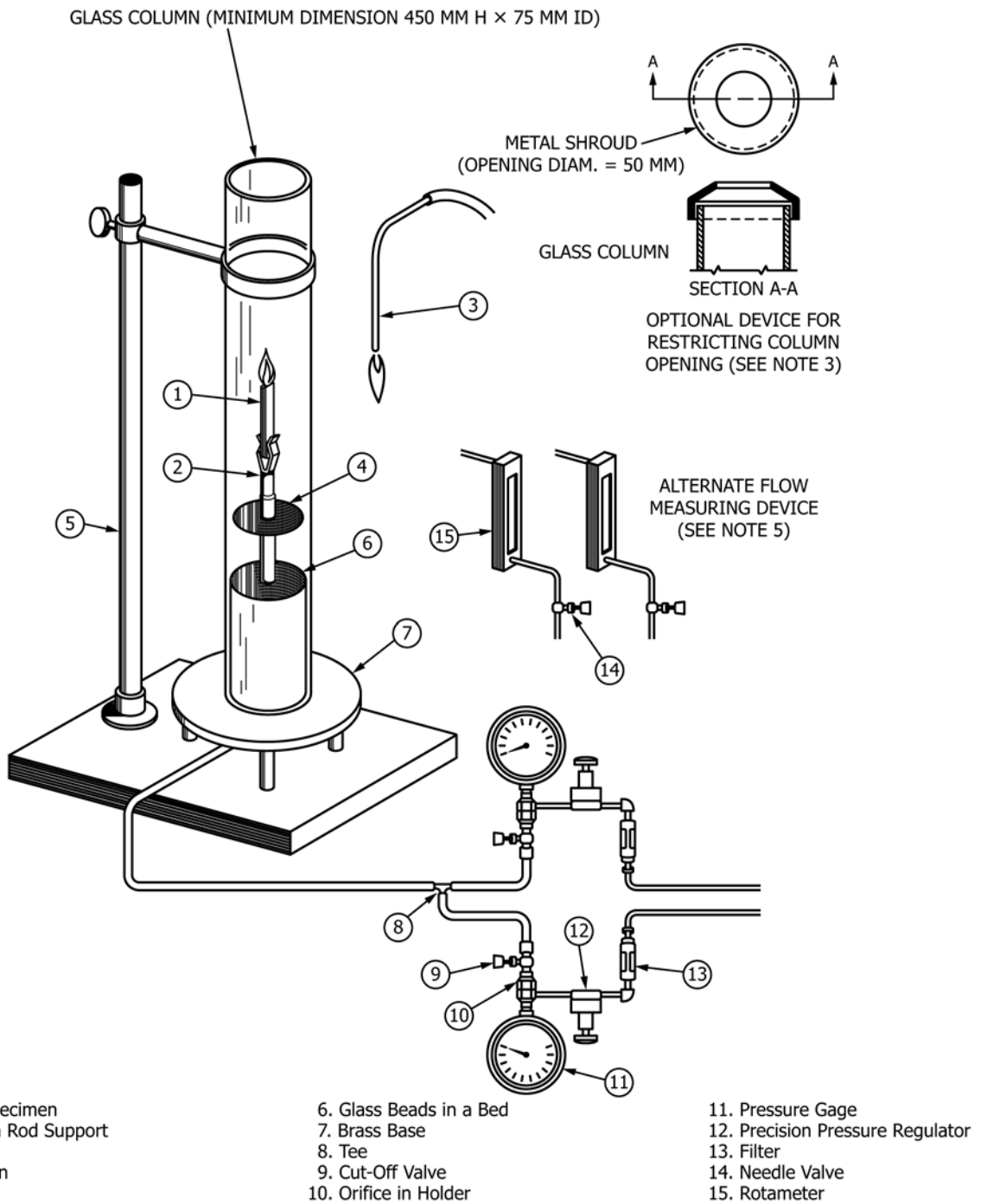


FIG. 1 Typical Equipment Layout

of about 18 %, but if the specimen goes out, select a concentration of about 25 % or higher depending on the difficulty of ignition and time of burning.

10.5 Set the flow valves so that the desired initial concentration of oxygen is flowing through the column. The gas flow rate in the column shall be  $4 \pm 1$  cm/s as calculated at standard temperature ( $0^{\circ}\text{C}$ ) and pressure (101.3 kPa) from the total flow of gas in  $\text{mm}^3/\text{s}$ , divided by the area of the column in  $\text{mm}^2$ .

10.6 Allow the gas to flow for 30 s to purge the system.

10.7 Ignite the entire top of the specimen with the ignition flame so that the specimen is well lighted. Remove the ignition flame and start the timer.

10.7.1 Type A, B, and C specimens are well lighted when the entire top is burning.

10.7.2 Type D specimens are well lighted if ignition occurs before any portion of the flame front passes the 20-mm reference mark on the frame. This test method is not applicable to materials that shrink below the 20-mm mark before ignition.

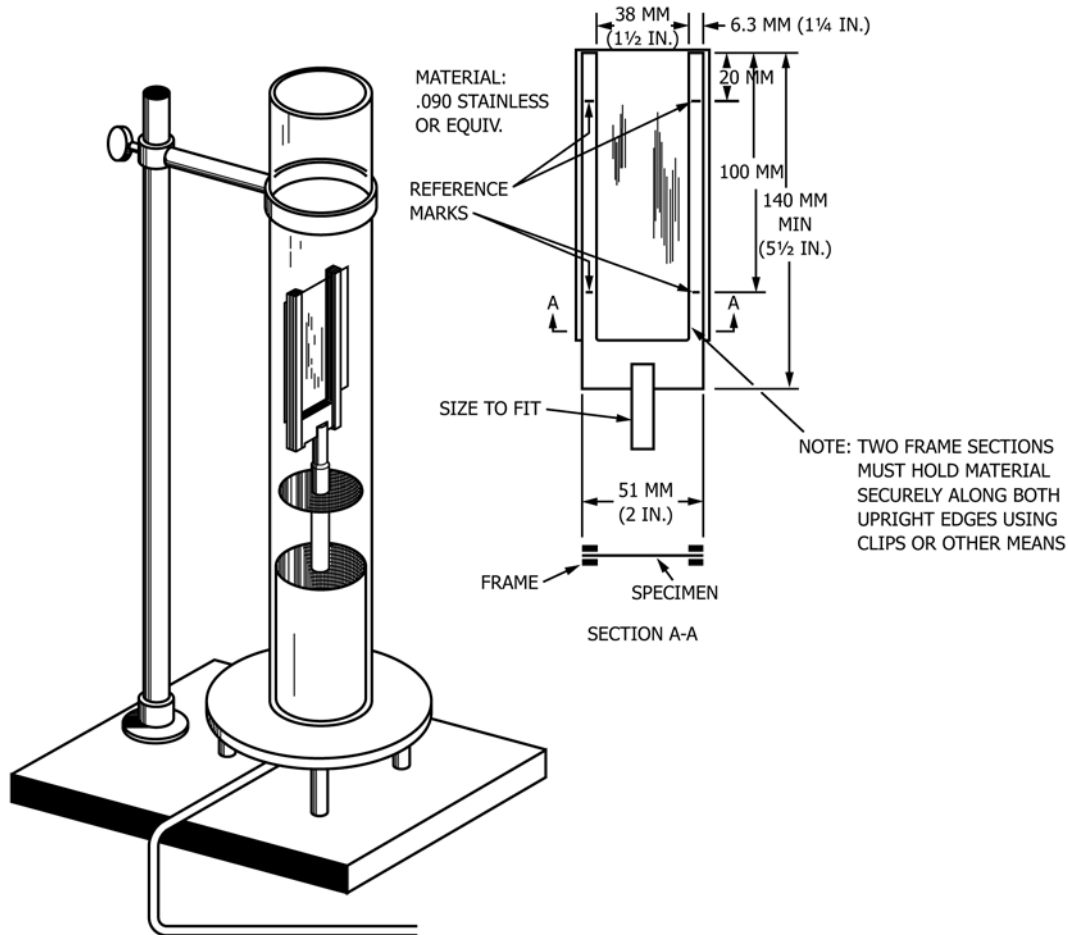


FIG. 2 Frame Design

TABLE 1 Specimen Dimensions, mm

| Type | Plastic Form                                    | Width      | Thickness   | Length     |
|------|---|------------|-------------|------------|
| A    | Physically self-supporting                      | 6.5 ± 0.5  | 3.0 ± 0.5   | 70 to 150  |
| B    | Alternate for self-supporting flexible plastics | 6.5 ± 0.5  | 2.0 ± 0.25  | 70 to 150  |
| C    | Cellular plastic                                | 12.5 ± 0.5 | 12.5 ± 0.5  | 125 to 150 |
| D    | Film or thin sheet                              | 52 ± 0.5   | as received | 140 ± 5    |

NOTE 11—Certain Type D materials have been found to shrink excessively at oxygen concentrations below the critical value but burn at values above the critical value. Care should be taken in testing such materials.

10.8 The concentration of oxygen is too high and must be reduced if the specimen burns in accordance with one of the following criteria:

| Type                 | A and B                 | C                       | D                              |
|----------------------|-------------------------|-------------------------|--------------------------------|
| Criteria for burning | at least 3 min or 50 mm | at least 3 min or 75 mm | past the 100-mm reference mark |

10.8.1 Do not adjust the oxygen concentration after igniting the specimen.

10.9 The concentration of oxygen must be raised if the flaming of the specimen extinguishes before meeting the criterion in 10.8.

NOTE 12—When testing Type D specimens, the support frame may come within 12 mm of the glass chimney. It has been found that the chimney may become quite hot and cause a decrease in oxygen index. Where this is found, it is suggested that the glass chimney be allowed to come back to room temperature before running the next test specimen. Certain laboratories accomplish this by alternating two chimneys.

10.10 Adjust the oxygen concentration, insert a new specimen, or if the previous specimen is long enough, turn it end for end or cut off the burned end, then purge and re-ignite.

10.11 Continue repeating 10.6 – 10.10 until the critical concentration of oxygen is determined. This is the lowest oxygen concentration that will meet the criterion of 10.8. At the next lower oxygen concentration that will give a difference in oxygen index of 0.2 % or less, the specimen should not meet the criterion of 10.8.

NOTE 13—The critical oxygen concentration has been found to be dependent on the temperature of the specimen at ignition and the temperature of the gas mixture.

NOTE 14—For a material having consistent burning characteristics, the difference in oxygen concentration between burning as defined in 10.8 and extinguishing as defined in 10.9, will be reproducible within 0.1 to 0.3 % depending on the sensitivity of the flow measuring equipment and upon the particular oxygen concentration involved. Some materials, however, exhibit erratic burning characteristics because of inhomogeneity, char formation, dripping, bending, etc., which cause less reproducible results.

In such cases, the critical concentration may be determined by a statistical testing method.<sup>6</sup>

10.12 Perform the test at least three times by starting at a slightly different flow rate still within the 30 to 50-mm/s limits and again performing 10.5 – 10.11.

10.13 *Routine Inspection Tests*—For routine inspection or specification purposes, a specified number of specimens may be tested at a specified oxygen concentration, all other conditions being controlled as in 10.110.1 – 10.7. The specification should be written in terms of the maximum number of specimens burning according to 10.8.

NOTE 15—Such a specification might read, for example “Not more than \_\_\_\_\_ of \_\_\_\_\_ specimens shall burn at least 3 min or 50 mm at an oxygen level of \_\_\_\_\_ %.”

NOTE 16—As indicated in 7.6, a much lower index results if the test is initiated with sample bottom ignition and subsequent upward flame propagation concurrent with the oxidant mixture flow. This alternate method should be considered when the purpose of the test goes beyond materials ranking, that is, when estimating suitability of materials in oxidant environments from an ignition and fire propagation safety point of view. Although the bottom-ignition yields more conservative results than the standard method and may simulate closer real-life ignition events, the caveat of 1.6 should be considered. For bottom ignition tests, use samples at the shorter length indicated in Table 1. With the exception of sample ignition location, all other procedures are similar with the standard method.

## 11. Calculation

11.1 Calculate the oxygen index,  $n$ , or the material for each replicate in 10.12, as follows:

$$n, \% = (100 \times O_2) / (O_2 + N_2) \quad (1)$$

where:

$O_2$  = volumetric flow of oxygen, mm<sup>3</sup>/s, at the concentration determined in 10.11, and

$N_2$  = corresponding volumetric flow rate of nitrogen, mm<sup>3</sup>/s.

11.1.1 If air is used and either oxygen or nitrogen is added as required, calculate  $n$  assuming that air contains 20.9 % oxygen as follows:

$$n, \% = (100 \times O_2) + (20.9 \times A) / (O_2 + N_2 + A) \quad (2)$$

where:

$A$  = volumetric flow rate of air, mm<sup>3</sup>/s,

$O_2$  = volumetric flow rate of oxygen, and

$N_2$  = volumetric flow rate of nitrogen.

## 12. Interpretation of Results

12.1 *Spirit of the Test*—The fire limit is taken as the boundary conditions that enables sustained propagation of combustion in a specified system. It is a condition in which combustion does occur and would typically yield complete propagation. However, one does not need to burn an infinite length specimen over an infinite period to report that sustained

equilibrium combustion occurs. Also, some materials do not combust under many test conditions or behave erratically. It is useful to be able to report results for several categories of these materials. For example, to report the oxygen index as 100 indicates the material will burn at that concentration. To report a material that does not burn as having an oxygen index >100 is a physically meaningless description. This section describes rationale for assigning interpretations to observed specimen tests.

12.2 *Equilibrium Combustion*—The user seeks an end point condition in which propagation of combustion is sustained. For polymers, a fire is taken as sustained if it propagates a distance of 50 mm along the surface of a specimen or if it burns for a period of 3 min. Both of these criteria suggest that the propagation has continued beyond the point at which the igniter may have produced an upset condition conducive to combustion. At either of these points, the test may be terminated and the result assigned as a positive result. However, the combustion along this distance and through this time must exhibit equilibria and create the impression that it would proceed on for an indefinite period were the sample longer or were more time to be allowed. The fire need not be calm and uniform to qualify as “equilibrium;” however, any variations in intensity or erratic nature should not be systematically decaying.

12.2.1 *Example*—A 100-mm long specimen is ignited. It burns a distance of 50 mm beyond the ignition zone and then extinguishes. If it had been 50-mm long, it would have achieved the burn distance criterion to report as a positive in the oxygen index test. To interpret this result, one would weigh several factors. If the nature of the combustion was progressively decaying after propagation outside the ignition zone, then this combustion was not equilibrium, regardless of the burn length, and it is a negative. If the combustion had been “equilibrium”) meaning that it was uniform of that its nature oscillated in a repetitive fashion that might suggest nonuniformity of the specimen, then it is more like a positive result, since any specimen might burn to completion. This argument is stronger if the specimen exhibited swings in intensity and if the extinguishment occurred during a low intensity swing. However, if a large number of tests were run to lend statistical confidence that the apparent nonuniformity is a reliable combustion-thwarting mechanism, then the results collectively might validate assignment of a negative.

12.2.2 *Example*—A fluid specimen is ignited. It burns for a period of time greater than the typical 3-min positive result criterion then extinguishes leaving a large amount of liquid remaining. During the combustion there may be a gradual decay in its intensity. The substantial amount of fluid that is still present can be reignited at the same conditions. To interpret this result, one would weigh several factors. If the fluid was likely to contain several fractions of differing combustibility, and if one of those fractions were completely combusted under equilibrium conditions, then one might expect to see the combustion intensity decay as the fraction of the constituent in the fluid decayed, and this result would be a positive, because the combustion affected the entire (fraction) of the specimen. However, if the fluid was merely warmed

<sup>6</sup> Such a statistical method as the Bruceton Staircase Method at the  $F_{50}$  mean failure value may be used. See the equations in Test Method D2444. Also see Dixon, W. J. and Massey, F. J., Jr., *Introduction of Statistical Analysis*, (2d Ed.), McGraw-Hill Book Co., Inc., New York, NY, 1957, Chapter 19, or Natrella, Mary, “Experimental Statistics.” Section 10–4. *National Institute of Standards and Technology Handbook 91*, 1963. Other procedures, such as using ten specimens at each oxygen concentration tried, have also proved successful.

during the ignition process, and then cooled during the combustion, so that the flame grew progressively less intense, then it is not equilibrium combustion, and the result is a negative. Complex combinations of these results can occur. If upon repeated tests, one were to observe that the combustion period was related to the initial volume of fluid, then this supports an inference that the test is a positive (more initial fraction yields more combustion time). Similarly, if repeated ignition of the specimen results in combustion of approximately the same time (for similar initial volumes), this argues that preheat in causing a transient combustion and that the result is a negative.

**12.3 Materials That Ignite but do not Propagate**—Many materials can be ignited but the fire is not sustaining. Failure to sustain is a common result in polymer tests in the oxygen index test when the oxygen concentration is below the index, and they are merely reported as negative test results. However, there are a number of polymers and composite materials (polymers with inert matrices) that can be ignited in pure oxygen but which exhibit progressively decaying combustion and extinguish if watched for a sufficient period of time. These results can be reported as an oxygen index of “Did Not Propagate” or DNP. For example one could report an oxygen index as DNP.

**12.4 Materials That do not Ignite**—Some material such as ceramics do not appear to ignite. They can be bathed in flame for extended periods of time and do not appear to participate in any reaction during the attempted ignition. These materials can be reported as “Did Not Ignite,” or DNI. For example, one could report an oxygen index as DNI.

**12.5 Multiple Limits**—Some materials can exhibit “erratic extinguishment” above the index. Wharton (17) reports on a nylon material that yielded reproducible negative results in one oxygen concentration range in the oxygen index test (suggesting the condition was below the fire point), but that also yielded reproducible positive burns at lower oxygen concentrations and negative results at still lower concentrations (an apparent multiple limit). The higher-concentration negatives were attributed to combustion that was so intense that it melted the specimen allowing the melt to drip away and carry the combustion with it, yielding a reproducible negative. Since the fire limit is the minimum condition, the lower of the two measurements is the value that should be reported. This is the reason the Summary (Section 4) calls for the limit to be “approached from both sides of the critical threshold.” The user should keep in mind that a single valid positive result is positive proof that the test condition is above the limit, but that assorted mechanisms may yield false negatives above the actual threshold. Therefore, there is always an element of uncertainty that the threshold may be lower than any set of tests suggests, and to reduce the uncertainty that the threshold may be lower than any set of tests suggests, and to reduce the probability of this, one may need to build a statistical base of

valid negative tests both near the threshold at throughout the range of conditions that are below the apparent limit.

### 13. Report

13.1 Report the following information:

13.1.1 Description of the material tested including the type, density, and general direction of anisotropy (for Type C specimens), source, manufacturers code number, form, previous history, and conditioning (if any),

13.1.2 Test specimen dimensions,

13.1.3 Special test conditions if any (that is, bottom ignition).

13.1.4 Individual oxygen index values found for each of the tests, and average oxygen index value.

13.1.5 Description of any unusual behavior such as charring, dripping, bending, and the like, and,

13.1.6 The precautionary caveat herein shall be incorporated in its entirety in the test report issued.

### 14. Precision and Bias

14.1 From a statistically designed round-robin testing program<sup>7,8</sup> on Type A specimens in which 18 laboratories checked five materials, the following was determined:

14.1.1 The standard deviation of the mean of three replicates (for comparing laboratory-to-laboratory) was 0.4 for materials with an oxygen index below 21 % and 0.7 to 1.4 for materials with an oxygen index above 21 %. The higher value was for a material that exhibits the erratic behavior mentioned in Note 15.

14.1.2 The standard deviation within a laboratory ranged from 0.1 for clean burning materials to 1.0 for erratic materials.

14.2 In a later statistically designed round-robin testing program<sup>9</sup> on Types B, C, and D specimens in which 29 laboratories studied twelve materials, the results in Table 2

**TABLE 2 Precision Results**

| Type | Laboratory-To-Laboratory Standard Deviation | Within Laboratory Standard Deviation |
|------|---|--------------------------------------|
| B    | 0.5 to 1.1                                  | below 0.2                            |
| C    | 0.4 to 1.5                                  | 0.1 to 0.3 (est.)                    |
| D    | 0.5 to 1.4                                  | below 0.6                            |

were found.

14.3 *Bias*—Bias is the systemic error which contributes to the difference between a test result and a true or reference value. There are no recognized standards on which to base an estimate of bias for this test method.

<sup>7</sup> Isaacs, J. L., “The Development, Standardization and Utilization of the Oxygen Index Flammability Test,” *General Electrical TIS Report 69-MAL-13*, August 1969, Louisville, KY.

<sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-0102.

<sup>9</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1031.



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