Designation: F3136 – 15

Standard Test Method for Oxygen Gas Transmission Rate through Plastic Film and Sheeting using a Dynamic Accumulation Method[1]

This standard is issued under the fixed designation F3136; 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 procedure for determination of the transmission rate of oxygen gas through plastics in the form of film, sheeting, laminates, coextrusions, coated or uncoated papers or fabrics.

1.2 This test method is not the only method for measurement of the oxygen transmission rate (OTR). There are other methods of OTR determination that use other oxygen sensors and procedures.

1.3 The values stated in SI units are to be regarded as standard. Commonly used metric units used to report Oxygen Transmission Rate are included in Terminology, Procedure, Precision and Bias sections and in the Calculation section of the Appendix.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3985 Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F2622 Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using Various Sensors

F2714 Test Method for Oxygen Headspace Analysis of Packages Using Fluorescent Decay

3. Terminology

3.1 *Definitions:*

3.1.1 *Oxygen Transmission Rate (OTR)*—the quantity of oxygen gas passing through a unit area of the parallel surfaces of a plastic film per unit time under the conditions of test. The SI unit of transmission rate is the mol/(m^2·s). The test conditions including temperature, relative humidity and oxygen partial pressure on both sides of the film must be stated in the report.

3.1.1.1 *Discussion*—A commonly used unit of OTR is the cm^3 (STP)/(m^2·day) at one atmosphere pressure difference where 1 cm^3 (STP) is 44.62 μmol, 1 atmosphere is 0.1013 MPa, and one day is 86.4×10^3s. The OTR in SI units is obtained by multiplying the value in commonly used units by 5.160×10^{-10}.

4. Summary of Test Method

4.1 The specimen is mounted as a sealed semi-barrier between two chambers, which together make up the permeation apparatus. The sensing well which contains the oxygen sensor is slowly purged by a stream of pure nitrogen or other oxygen deficient gas mixture until the oxygen concentration represents that of the purge gas. A commercial grade of compressed nitrogen containing less than 0.05% oxygen is recommended. A gas of known oxygen concentration, typically air or pure oxygen, is directed into the opposite chamber, the driving well. Oxygen concentration in the sensing well containing the oxygen sensor is measured periodically and the accumulating oxygen concentration recorded. The Oxygen Transmission Rate (OTR) parameter is determined from the slope of the logarithm of accumulated oxygen concentration in the sensing well versus time as described in 14.2.

5. Significance and Use

5.1 The Oxygen Transmission Rate is an important determinant of packaging functionality afforded by packaging materials for a wide variety of packaged products including food, pharmaceuticals and medical devices. In some applications, sufficient oxygen must be allowed to permeate into the package. In others, the oxygen ingress must be minimized to maintain product quality.

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[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and is the direct responsibility of Subcommittee F02.10 on Permeation.

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[2] 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.

5.2 Other ASTM Standard Methods to measure the oxygen transmission rate are described in Standard Test Method D3985 and Standard Test Method F2622.

6. Interferences

6.1 Any leakage within the permeation apparatus or mounted packaging film will affect results. A means to assess leakage is described in paragraph 9.2.

6.2 The condition of the sample film must be noted such as wrinkles or other defects can affect results.

7. Apparatus

7.1 *Oxygen Gas Transmission Apparatus,* as diagrammed in Fig. 1 with the following:

7.1.1 *Permeation Apparatus (diffusion cell)* shall consist of two metal halves, which, when closed upon the test specimen, will define a known gas transmission area. The volume of the sensing well containing the oxygen sensor must be accurately known.

7.1.1.1 *O-ring*—A circular transmission area permits application of a static O-ring in a properly constructed O-ring groove in the side of the permeation apparatus that does not contain the oxygen sensor. The test area of the sensing well is considered to be that area established by the inside contact diameter of the compressed O-ring when the permeation apparatus is clamped shut. The area, A, can be obtained by measuring the inside diameter of the imprint left by the O-ring on the specimen after it has been removed from the permeation apparatus.

7.1.1.2 The sensing well of the permeation apparatus shall have a flat raised rim. Since this rim is a critical sealing surface against which the test specimen is pressed, it shall be smooth and flat without radial scratches.

7.1.1.3 The sensing well of the permeation apparatus shall have a low-permeability window transparent to wavelengths used to activate and read the oxygen sensor which is mounted within the sensing well.

7.1.1.4 The oxygen sensor incorporates a fluorophore that fluoresces in response to a certain wavelength of light, but is quenched in the presence of oxygen. The oxygen quenching effect is calibrated to oxygen concentration. This sensing technology is identical to the sensing technology described in ASTM F2714.

7.1.1.5 The permeability apparatus shall incorporated suitable fittings for the introduction and exhaust of gases without significant loss or leakage.

7.2 It is necessary to control the temperature of the permeability apparatus during the test period. A simple heating/cooling chamber regulated to ±0.5°C, is adequate for this purpose in which the apparatus is housed during the test period.

7.3 Flow meters having an operating range from 1 to 100 cm^3/min are required to monitor the flow rate of the nitrogen purge stream and, if used, the oxygen or compressed air circulation stream. Sufficiently low flow rates and/or balanced pressures on each side of the film are required to avoid stretching the specimen which would modify the effective sensing well volume.

7.4 An external light source provides sufficient light in the appropriate wavelength to activate the oxygen sensor.

7.5 A light detector, and associated electronics, determines the fluorescence decay constant, which is proportional to oxygen concentration.

7.6 A computer is used to calculate the oxygen concentration at specified time intervals based on decay rates. The oxygen transmission rate of the film is calculated from that data.

8. Reagents and Materials

8.1 Nitrogen enriched purge gas shall contain a known concentration of nitrogen. Commercial grade compressed nitrogen (<0.05% oxygen), certified pure nitrogen gas, cryogenically stored nitrogen or nitrogen enriched gas produced using on-site generators may be used.

8.2 Transmission gas shall be of a known oxygen concentration with an oxygen concentration at least 10% greater than the purge gas. Typically, commercial grade compressed oxygen, certified gas or air is used. Also, regulated compressed air can be used or the driving well can simply be left open to ambient air.

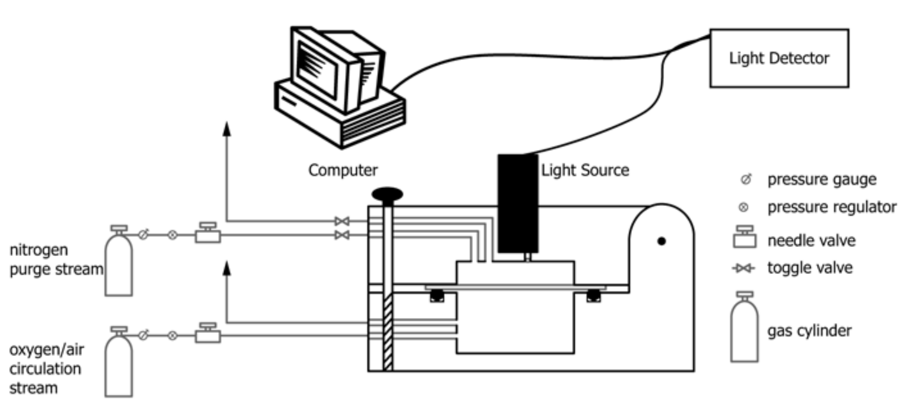
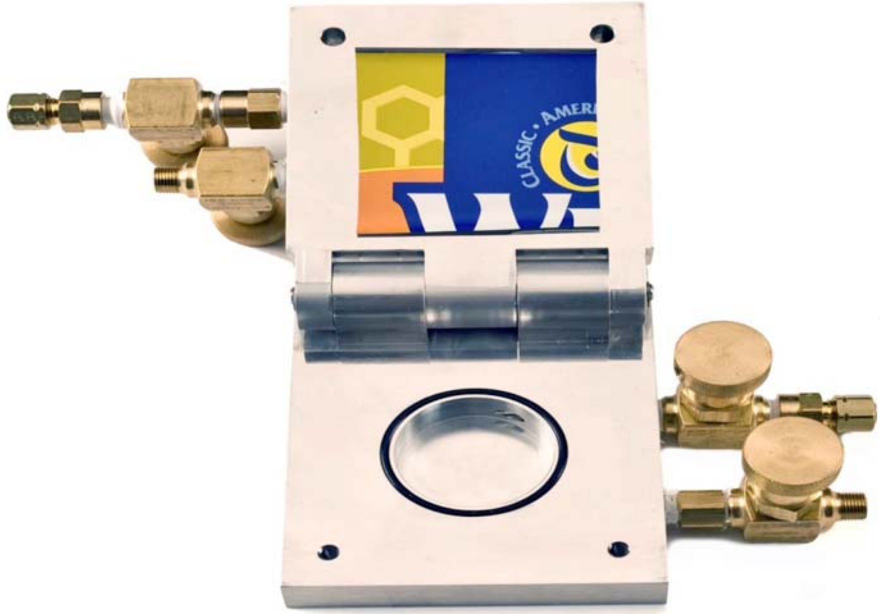


FIG. 1 A Practical Arrangement of Components for the Measurement of Oxygen Transmission Rate Using an Optical Florescent Oxygen Sensor and a Permeation Apparatus



An acceptable Permeation Apparatus (aka Diffusion Cell) is available from OxySense, Inc., 6000 S. Eastern Ave., Suite 14G, Las Vegas, NV 89119, USA.

FIG. 2 Permeation Apparatus as described in this method (film specimen is shown adhered to the sensing well)

8.3 *Sealing Grease*—High-viscosity silicone stop cock grease or high-vacuum grease is required for sealing the specimen film in the diffusion cell.

9. Precautions

9.1 Temperature is a critical parameter affecting the measurement of OTR. Careful temperature control can help to minimize variations due to temperature fluctuations. During testing, the temperature shall be monitored and controlled to ± 0.5°C. Temperature variations should be minimized. The average temperature and a range of temperatures during a test shall both be recorded.

9.2 Oxygen that leaks into the sensing well through faulty valves, fittings, or through an improperly sealed window can significantly affect the accuracy of the measurement. Periodic leak checks using impervious films such as metal foils with thicknesses ≥25 µm (with their expected permeations of zero) should be taken to identify suspected system leaks. Leak check permeation tests should be run at least every 3-6 months and each test should be run for a minimum of 12 hours.

10. Sampling

10.1 Film samples used for the determination of OTR shall be representative of materials for which the data are required. Care shall be taken to ensure that samples are representative of conditions across the width and along the length of a roll of film.

11. Test Specimens

11.1 Test specimens shall be representative of the material and shall be free of defects including wrinkles, creases, and pinholes, unless these are characteristics of the material being tested and included in the material description.

11.2 Average thickness shall be determined ± 3 µm using a calibrated dial gage (or equivalent) at a minimum of five points distributed over the entire test area. Maximum, minimum, and average values shall be recorded. Sample thickness need not be measured for determination of sample OTR only.

11.3 If the test specimen is of an asymmetrical construction, the two surface shall be marked by appropriate distinguishing marks and the orientation of the test specimen within the permeation apparatus shall be reported.

12. Calibration

12.1 *General Approach*—The oxygen sensor fluoresces when exposed to certain wavelengths of light. Oxygen quenches the fluorescent decay response. The sensor apparatus utilizes a light source to deliver light to the oxygen sensor which, in turn, fluoresces. This light is measured by the detector. The detector determines the exponential fluorescent response decay constant, which is calibrated to oxygen concentration.

12.2 *Calibration*—The sensor system is calibrated by measuring oxygen concentration at two known values, typically air (20.9% oxygen) and pure nitrogen (0% oxygen). These values define a calibration curve from which unknown oxygen levels may be determined. Alternatively, 2 gases of known concentrations nearer to the level under test may prove to yield a better calibration value (perhaps 3% and 0% oxygen).

13. Procedure

13.1 Apply a thin layer of sealing grease (see 8.3) around the raised rim of the sensing well opposite the O-ring. Place the specimen on the greased surface, taking care to avoid wrinkles or creases. Close and secure the permeation apparatus.