Designation: D3985 – 05 (Reapproved 2010)$^{\varepsilon 1}$

Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor[1]

This standard is issued under the fixed designation D3985; 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.

ε^1 NOTE—Editorial changes were made throughout in November 2010.

1. Scope

1.1 This test method covers a procedure for determination of the steady-state rate of transmission of oxygen gas through plastics in the form of film, sheeting, laminates, coextrusions, or plastic-coated papers or fabrics. It provides for the determination of (*1*) oxygen gas transmission rate (OTR), (*2*) the permeance of the film to oxygen gas (PO_2), and (*3*) oxygen permeability coefficient ($P'O_2$) in the case of homogeneous materials.

1.2 This test method does not purport to be the only method for measurement of OTR. There may be 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. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety problems, 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]

D1434 Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

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

F1927 Test Method for Determination of Oxygen Gas Transmission Rate, Permeability and Permeance at Controlled Relative Humidity Through Barrier Materials Using a Coulometric Detector

3. Terminology

3.1 *Definitions:*

3.1.1 *oxygen permeability coefficient* ($P'O_2$)—the product of the permeance and the thickness of film. The permeability is meaningful only for homogeneous materials, in which case it is a property characteristic of the bulk material. This quantity should not be used, unless the relationship between thickness and permeance has been verified on tests using several different thicknesses of the material. The SI unit of oxygen permeability is the mol/(m·s·Pa). The test conditions (see 3.1.3) must be stated.

3.1.2 *oxygen permeance* (PO_2)—the ratio of the OTR to the difference between the partial pressure of O_2 on the two sides of the film. The SI unit of permeance is the mol/(m^2·s·Pa). The test conditions (see 5.1) must be stated.

3.1.3 *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 and oxygen partial pressure on both sides of the film must be stated.

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

4. Summary of Test Method

4.1 The oxygen gas transmission rate is determined after the sample has equilibrated in a dry test environment. In this context, a "dry" environment is considered to be one in which the relative humidity is less than 1 %.

4.2 The specimen is mounted as a sealed semi-barrier between two chambers at ambient atmospheric pressure. One chamber is slowly purged by a stream of nitrogen and the other chamber contains oxygen. As oxygen gas permeates through

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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.

Current edition approved Oct. 1, 2010. Published November 2010. Originally approved in 1981. Last previous edition approved in 2005 as D3985 – 05$^{\varepsilon 1}$. DOI: 10.1520/D3985-05R10E01.

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

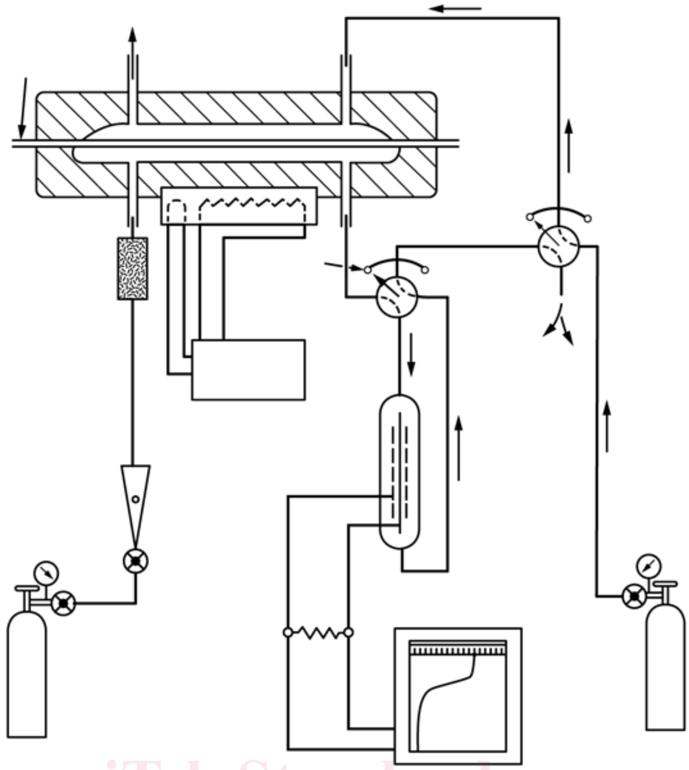

FIG. 1 A Practical Arrangement of Components for the Measurement of Oxygen Transmission Rate Using the Coulometric Method

the film into the nitrogen carrier gas, it is transported to the coulometric detector where it produces an electrical current, the magnitude of which is proportional to the amount of oxygen flowing into the detector per unit time.

5. Significance and Use

5.1 The OTR is an important determinant of the packaging protection afforded by barrier materials. It is not, however, the sole determinant, and additional tests, based on experience, must be used to correlate packaging performance with OTR. It is suitable as a referee method of testing, provided that the purchaser and the seller have agreed on sampling procedures, standardization procedures, test conditions, and acceptance criteria.

5.2 Limited statistical data on correlations with Test Method D1434 methods are available[4]; however, the oxygen transmission rate of a standard reference material (see 12.1) as determined manometrically by NIST, is in good agreement with the values obtained in the coulometric interlaboratory test using material from the same manufacturing lot. Thus, this test method may be used as a referee method.

6. Interferences

6.1 The presence of certain interfering substances in the carrier gas stream may give rise to unwanted electrical outputs and error factors. Interfering substances include free chlorine and some strong oxidizing agents. Exposure to carbon dioxide should also be minimized to avoid damage to the sensor through reaction with the potassium hydroxide electrolyte.

7. Apparatus

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

7.1.1 *Diffusion Cell* shall consist of two metal halves, which, when closed upon the test specimen, will accurately define a circular area. The volume enclosed by each cell half, when clamped, is not critical; it should be small enough to allow for rapid gas exchange, but not so small that an unsupported film which happens to sag or bulge will contact the top or bottom of the cell. The diffusion cell shall be provided with a thermometer well for measuring temperature.

7.1.1.1 *O-Ring*—An appropriately sized groove, machined into the oxygen (or test gas) side of the diffusion cell, retains a neoprene O-ring. The test area is considered to be that area established by the inside contact diameter of the compressed O-ring when the diffusion cell is clamped shut against the test specimen. 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 diffusion cell.

7.1.1.2 The nitrogen (or carrier gas) side of the diffusion cell 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 *Diffusion Cell Pneumatic Fittings*—The diffusion cell shall incorporate suitable fittings for the introduction and exhaust of gases without significant loss or leakage.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1085. Contact ASTM Customer Service at service@astm.org.

7.1.1.4 It is desirable to thermostatically control the diffusion cell. A simple heating or heating/cooling system regulated to ±0.5°C, is adequate for this purpose. A thermistor sensor and an appropriate control circuit will serve to regulate the cell temperature unless measurements are being made close to ambient temperature. In this case, it is desirable to provide cooling capability to remove some of the heat.

7.1.1.5 Experience has shown that arrangements using multiple diffusion cells are a practical way to increase the number of measurements which can be obtained from a coulometric sensor. Valving connects the carrier gas side of each individual diffusion cell to the sensor in a predetermined pattern. Carrier gas is continually purging the carrier gas sides of those cells that are not connected to the sensor. Either test gas or carrier gas, as is appropriate, purges the test gas chamber of any individual cell.

7.1.2 *Catalyst Bed*—A small metal tube with fittings for attachment to the inlet on the nitrogen side of the diffusion cell shall contain 3 to 5 g of 0.5 % platinum or palladium catalysts on alumina[5] to provide an essentially oxygen-free carrier gas.

7.1.3 *Flowmeter*—A flowmeter having an operating range from 5 to 100 mL/min is required to monitor the flow rate of the nitrogen carrier gas.

7.1.4 *Flow Switching Valves*—Valves for the switching of the nitrogen and test gas flow streams.

7.1.5 *Coulometric Sensor*—An oxygen-sensitive coulometric sensor (see Note 1) operating at an essentially constant efficiency shall be used to monitor the quantity of oxygen transmitted.

NOTE 1—It is deemed advisable upon initial setup of the voltage recorder and periodically thereafter to check the response of the recorder on all ranges to a suitable voltage input.

7.1.6 *Load Resistor*—The current generated by the coulometric cell shall pass through a resistive load across which the output voltage is measured. Typical values for the load resistor are such that the values yield a convenient relationship between the output voltage and the oxygen transmission rate in standard units cm^3(STP)/(m^2·d).

7.1.7 *Voltage Recorder*—A multirange, potentiometer strip chart recorder may be used for measuring the voltage developed across the load resistor. The recorder should be capable of measuring a full-scale voltage of 50 mV. It should be capable of measuring voltages as low as 0.100 mV and have a resolution of at least 10 µV. An input impedance of 1 megohm or higher is acceptable.

8. Reagents and Materials

8.1 *Nitrogen Carrier Gas* shall consist of a nitrogen and hydrogen mixture in which the percentage of hydrogen shall fall between 0.5 and 3.0 volume %. The carrier gas shall be dry and contain not more than 100 ppm of oxygen. A commercially available mixture known as "forming gas" is suitable.

8.2 *Oxygen Test Gas* shall be dry and contain not less than 99.5 % oxygen (except as provided in 14.11).

8.3 *Sealing Grease*—A high-viscosity silicone stopcock grease or a high-vacuum grease is required for sealing the specimen film in the diffusion cell.

9. Precautions

9.1 Extended use of the test unit, with no moisture in the gas stream, may in some older systems result in a noticeable decrease in output and response time from the sensor (equivalent to an increase in the calibration factor, Q). This condition is due to drying out of the sensor.

9.2 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 to the nearest 0.5°C. The average temperature and the range of temperatures found during a test shall both be reported.

9.3 The sensor will require a relatively long time to stabilize to a low reading characteristic of a good barrier after it has been used to test a barrier such as low-density polyethylene. For this reason, materials of comparable gas transmission qualities should be tested together.

9.4 Back diffusion of air into the unit is undesirable. Care should therefore be taken to ensure that there is a flow of nitrogen through the system at all times. This flow can be low when the instrument is not being used.

9.5 Elevated temperatures can be used to hasten specimen outgassing, provided that the treatment does not alter the basic structure of the specimen (crystallinity, density, and so forth). This can be accomplished by the use of the heaters in the diffusion cells.

10. Sampling

10.1 The sampling units used for the determination of OTR shall be representative of the quantity of product for which the data are required, in accordance with Practice D1898. 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 being tested and shall be free of defects, including wrinkles, creases, and pinholes, unless these are a characteristic of the material being tested.

11.2 Average thickness shall be determined to the nearest 2.5 µm (0.0001 in.), 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.

11.3 If the test specimen is of an asymmetrical construction, the two surfaces shall be marked by appropriate distinguishing marks and the orientation of the test specimen in the diffusion cell shall be reported (for example, "side II was mounted facing the oxygen side of the diffusion cell").

[5] The sole source of supply of the catalyst known to the committee at this time is Englehard Industries Division, Chemical Dept., 429 Delancey Street, Newark, NJ 07105. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.