



Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor¹

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^{ε1} 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D1898 Practice for Sampling of Plastics (Withdrawn 1998)³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

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.

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

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/(m²·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/(m²·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 cm³ (STP)/(m²·d) at one atmosphere pressure difference where 1 cm³ (STP) is 44.62 μmol, 1 atm is 0.1013 MPa, and one day is 86.4 × 10³s. 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

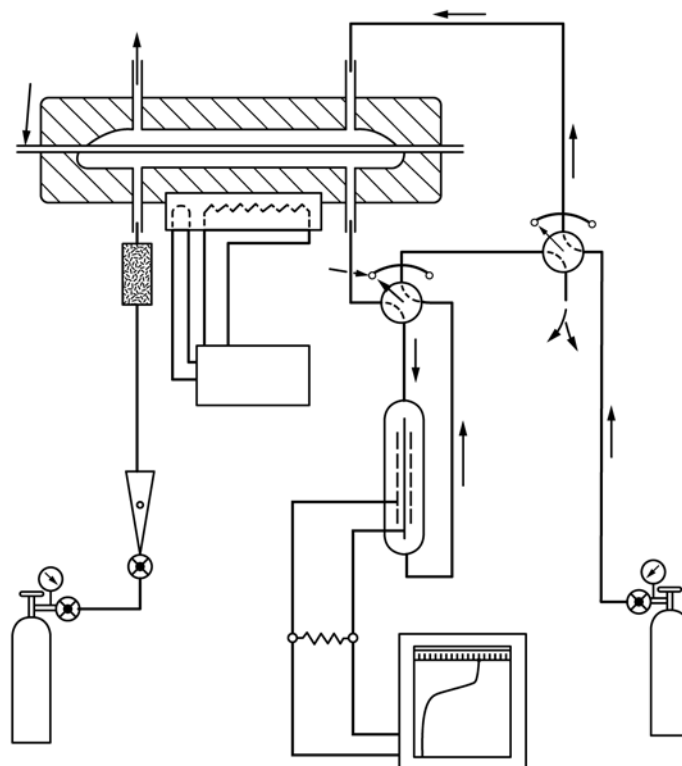


FIG. 1 A Practical Arrangement of Components for the Measurement of Oxygen Transmission Rate Using the Coulometric Method

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 available⁴; 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 $\pm 0.5^\circ\text{C}$, 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 alumina⁵ 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 $\text{cm}^3(\text{STP})/(\text{m}^2 \cdot \text{d})$.

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 $2.5 \mu\text{m}$ (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.

12. Calibration

12.1 *General Approach*—The oxygen sensor used in this test method is a coulometric device that yields a linear output as predicted by Faraday’s Law. In principle, four electrons are produced by the sensor for each molecule of oxygen that passes into it. Considering that the sensor is known to have a basic efficiency of 95 to 98 %, it may be considered an “intrinsic” standard⁶ that does not require calibration, and can thus be used as a reference method.

12.2 Experience has shown, however, that under some circumstances the sensor may become depleted or damaged to the extent that efficiency and response are impaired. For that reason, this test method incorporates means for a periodic sensor evaluation. This evaluation is derived from measurements of a known-value “reference package.” Experience indicates however, that a specimen-to-specimen variability of the reference material⁷ is such that a change should never be made in the calibration factor, as the result of a measurement using a single sheet of the reference material.

12.3 *Establishing a System Calibration Constant (Used only on systems using a chart recorder to determine OTR)*—Determine the exposed area, A , of the calibrating reference film (see 7.1.1.1). Using the permeance value furnished with the reference film, determine the OTR through that film of that area (A). Use this value to determine the calibration constant, Q :

$$Q = \frac{OTR \times R_L}{E_e - E_o} \quad (1)$$

where:

OTR = Oxygen transmission rate through a film of area, A , as calculated from data supplied with the reference film,

R_L = value of load resistance,

E_o = observed steady-state zero-level before oxygen gradient is applied, and,

E_e = observed steady-state voltage with oxygen gradient across test film.

Repeat the calibration using additional sheets of the reference film until the confidence interval for Q defined by the measurements is within acceptable limits.

13. Conditioning

13.1 Trim the test specimen to a size appropriate for the diffusion cell in which it will be mounted. In general, this means that the seal around the edge of the diffusion cell should not be impaired if the specimen bulges or sags slightly. After trimming, condition the specimens in a desiccator over calcium chloride or another suitable desiccator for a minimum of 48 h. This does not imply that 48 h will be sufficient to bring all

materials to a condition where their measured OTRs will be reproducible. Previous experience should serve as the primary guide to the suitability of a given conditioning regimen. If a material is being tested with which the user has no previous experience, the effect of conditioning time should be investigated and a regimen selected such that there is no significant effect due to conditioning time. In any case, the conditioning procedure used should be included in the report section.

13.2 Measure OTR in a temperature-controlled environment with the apparatus placed in a draft-free location.

14. Procedure

14.1 *Preparation of Apparatus*—If preceding tests have exposed the apparatus to high moisture levels, it will be necessary to outgas the system, particularly the catalyst bed, to desorb residual moisture. Water shall be removed from the nitrogen and test gas humidifiers. The system can then be dried by slowly purging overnight using dry carrier gas and with the sensor bypassed. Heating the apparatus will speed the drying and outgassing process.

14.2 *Inserting the Specimen*—With the sensor bypassed, unclamp the diffusion cell and open it. Apply a thin layer of sealing grease (see 8.3) around the raised rim of the lower half of the diffusion cell. Remove the test specimen from the desiccator and place it upon the greased surface, taking care to avoid wrinkles or creases. Set the movable half of the diffusion cell into place and clamp both halves tightly together.

14.3 *Purging the System*—Place the system in the CAR-RIER PURGE mode and purge air from the upper and lower diffusion cell chambers, using a flow rate of 50 to 60 mL. After 3 or 4 min, reduce the flow rate to the desired value between 5 and 15 mL/min. Maintain this configuration for 30 min.

14.4 *Establish E_o* —After the system has been flushed with nitrogen for 30 min, INSERT THE SENSOR so that the carrier gas which has passed through both sides of the diffusion cell is diverted into the sensor.

NOTE 2— E_o often is referred to as “individual zero.”

At this time, the sensor output will usually increase abruptly, indicating that oxygen is entering the sensor with the carrier gas. The most likely sources of this oxygen are (1) outgassing of the sample, (2) leaks in the system, or (3) a combination of (1) and (2). The operator shall observe the recorder trace until the sensor output current stabilizes at a constant value with no significant trend in either direction. Thick samples may require a purge of several hours, or even overnight, before a steady low value of sensor current is obtained. On older systems, the sensor should be bypassed except for brief periods when the zero level is being checked. Once a steady low value of sensor current has been obtained, the sensor may be inserted to monitor the zero level and left there until a stable zero level is obtained. At this time, the zero level is recorded and labelled E_o . It has been found helpful to periodically test the OTR of a piece of brass shim stock in order to ascertain that no leaks or contamination of the carrier gas have developed.

14.5 Once E_o has been established, switch OXYGEN into the test-gas side of the diffusion cell. This action will be automated on newer systems.

⁶ Garner, E. L., and Raspberry, S. D., “What’s new in Traceability,” *Journal of Testing and Evaluation*, Vol 21, No. 6, November 1993, pp. 505–509.

⁷ The sole source of supply of the reference material known to the committee at this time is Mocon, Inc., 7500 Mendelssohn Ave. N, Brooklyn Park, MN 55428. 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.

14.6 The sensor output current should increase gradually, ultimately stabilizing at a constant value. While some thin films with high diffusion coefficients may reach equilibrium in 30 to 60 min, thicker or more complex structures may require a number of hours to reach a steady state of gas transmission. The steady-state voltage value of the oxygen transmission rate shall be recorded and labelled E_e .

NOTE 3—If, after attainment of an apparent steady-state condition, any doubt exists as to whether this is a true steady-state condition, perform a check as follows: (1) bypass the sensor; (2) allow the unit to stabilize for an additional period of time (minimum of 6 h); and, (3) insert the sensor again, and monitor the transmission rate. An increased output indicates that steady-state conditions have not been reached, while the same output (no increase) indicates that steady-state conditions have been initially obtained.

14.7 Temperature shall be obtained by monitoring the temperature as close as possible to the specimen.

14.8 *Standby and Shutoff Procedures*—Follow the manufacturer's instructions in the instrument manual for putting the instrument into standby mode when the system will not be used for an extended period.

14.9 *Tests in a Moist Environment*—This test method is for dry (0 RH) conditions only. Specific procedures for testing in a controlled RH environment are covered by Test Method **F1927**.

14.10 The OTR at temperatures other than ambient may be determined by thermostatically controlling the diffusion cell provided that the temperature of the carrier gas does not adversely affect the operation of the sensor. Experience has shown that the unit can be operated satisfactorily in the range from 4 to 65°C.

14.11 *Testing Poor Barriers*—Films having transmission rates in excess of 200 cm³(STP)/(m²·d) when tested with an oxygen partial pressure difference of one atmosphere are defined as poor barriers. Examples of such materials, depending on thickness, include polyethylene, polycarbonate, and polystyrene. High oxygen concentrations in the carrier gas, from the testing of poor barriers, will tend to produce detector saturation. One way to avoid this problem is to use a test gas that is a mixture with a known concentration of oxygen in nitrogen. The permeance of the film should be calculated using the known value of oxygen partial pressure, and then a transmission rate should be calculated for the appropriate partial pressure difference from the permeance and the desired partial pressure difference. Another way to reduce the oxygen concentration in the carrier gas when testing poor barriers is to mask off most of the area of the test specimen using a mask of thin metal or aluminum foil on both sides of the test specimen by use of a suitable adhesive such as contact cement or epoxy. The specimen area then becomes equal to the open area of the mask. The effect of varying the area of the open hole in the mask should be tested to ensure that the mask is performing properly.

15. Calculation

15.1 If using a chart recorder, determine the OTR as follows:

$$\text{OTR} = \frac{(E_e - E_o) \times Q}{A \times R_L} \quad (2)$$

where:

E_e = steady-state voltage level (see 14.6),

E_o = zero voltage level (see 14.4),

A = specimen area (see 7.1.1.1),

Q = calibration constant (see Section 12), and

R_L = value of load resistance (see 7.1.6).

Newer, computer-controlled systems will automatically record the OTR, and possibly the permeance and permeation coefficient.

15.2 Determine the permeance (PO_2) of the specimen as follows:

$$\text{PO}_2 = \frac{\text{OTR}}{p} \quad (3)$$

where p = partial pressure of oxygen, which is the mol fraction of oxygen multiplied by the total pressure (normally, one atmosphere), in the test gas side of the diffusion cell. The partial pressure of O_2 on the carrier gas side is considered to be zero.

15.3 Determine the oxygen permeability coefficient ($P'\text{O}_2$) as follows:

$$P'\text{O}_2 = \text{PO}_2 \times t \quad (4)$$

where t = average thickness of the specimen (see 11.2). Results should be expressed as permeabilities only in cases where materials have been determined to be homogeneous by investigation of the relationship between specimen thickness and permeance.

16. Report

16.1 Report the following information:

16.1.1 A description of the test specimen, including an identification of the two sides of the material if they are different, a statement as to which side was facing the test gas, the location of the specimen in the lot of material of which it is representative, and the dimensions of the test specimen.

16.1.2 The average thickness of the test specimens as determined in 11.2 and the standard deviation of the thickness values.

16.1.3 The barometric pressure at the time of the test and whether or not barometric pressure compensation is performed on the data, either manually or by computer. Barometric compensation is performed to standardize data to 760 mm Hg (sea level).

NOTE 4—The barometric compensation factor may be calculated as follows:

$$fBP_{760} = \frac{BP_{760}}{BP_a} \quad (5)$$

where:

fBP_{760} = barometric compensation factor

BP_{760} = 760 mm Hg, and

BP_a = ambient barometric pressure in mm Hg.

16.1.4 The partial pressure of the oxygen gas on the test-gas side of the diffusion cell and a statement as to how it was determined.

TABLE 1 Summary of All Test Data

	Sample A		Sample B		Sample C		Sample D		Sample E		Equipment Used
Lab 1	1.120	1.090	0.090	0.062	69.60	73.90	12.20	11.90	0.296	0.293	OxTran Model 2/21 L
Lab 2	1.300	1.260	0.026	0.036	60.50	61.20	12.40	12.30	0.266	0.249	OxTran Model 2/21
Lab 3	1.090	1.050	0.059	0.039	61.23	60.32	12.13	12.02	0.385	0.307	OxTran Model 2/21 H
Lab 4	1.098	1.068	0.048	0.041	70.80	70.50	12.60	12.80	0.310	0.286	OxTran Model 2/21 L
Lab 5	1.140	1.070	0.060	0.060	67.42	75.17	11.82	11.92	0.380	0.350	OxTran Model 2/20
Lab 6	1.090	1.090	0.160	0.180	57.78	56.30	11.07	11.02	0.330	0.340	OxTran Model 702
Lab 7	1.160	1.070	0.050	0.050	58.60	58.30	10.70	11.30	0.260	0.260	OxTran Model 2/21 L
Lab 8	1.12	1.16	0.026	0.025	63.20	65.50	11.80	11.70	0.278	0.285	OxTran Model 2/20 L

TABLE 2 Statistical Summary of Data

Material	Average	Sr	SR	r	R
Sample A (5.5 mil EVOH lamination)	1.124	0.035	0.071	0.098	0.199
Sample B (50 Gage Metalized PET)	0.063	0.010	0.047	0.029	0.130
Sample C (0.92 mil Mylar)	64.395	2.339	6.269	6.549	17.553
Sample D (4.75 mil Mylar)	11.856	0.183	0.605	0.512	1.694
Sample E (5.7 mil EVOH lamination)	0.305	0.022	0.043	0.063	0.120

16.1.5 The rate of flow of the nitrogen carrier gas during the test.

16.1.6 The conditioning procedure used on the test specimens prior to testing.

16.1.7 The temperature of the test specimen (to the nearest 0.5°C) and the method used to determine the temperature.

16.1.8 The values of OTR, permeance (if desired), and the permeability (if desired).

16.1.9 A description of the apparatus used including, if applicable, the manufacturer's model number and serial number.

16.1.10 The calibration factor (Q) and a statement of the means used to obtain the calibration factor (only needed if a voltage recorder is being used to calculate OTR).

16.1.11 The effective area for permeation, A , and a description of how it was obtained.

16.1.12 The time to reach steady-state after introduction of the oxygen gas into the test-gas side of the diffusion cell.

17. Precision and Bias

17.1 A research report (RR:F02-1021 and RR:F02-1022)⁸ describes a round robin conducted in 2004 in accordance with

⁸ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:F02-1021 and RR:F02-1022. Contact ASTM Customer Service at service@astm.org.

Practice E691, involving eight different laboratories measuring five different specimens in duplicate at one test condition. Four models of a commercially available coulometric gas transmission-measuring instrument were used to collect this information. No significant difference was found between results obtained from the four models, and the results from the four models were pooled together. All measurements made were in units of Oxygen Transmission Rate ($cc/(m^2 \cdot day)$) at the conditions of 23°C, 100 % dry Oxygen test gas gradient. All values are normalized to a barometric pressure of one atmosphere. All test data has been summarized in Table 1. Statistical summaries of repeatability (within laboratories) and reproducibility (between laboratories) are listed in Table 2.

18. Keywords

18.1 coulometric; oxygen transmission rate; permeability; permeation; plastic films

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