



Standard Test Method for Oxygen Transmission Rate Through Dry Packages Using a Coulometric Sensor¹

This standard is issued under the fixed designation F 1307; 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 the determination of the steady-state rate of transmission of oxygen gas into packages. More specifically, the method is applicable to packages that in normal use will enclose a dry environment.

1.2 *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:

D 1434 Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheet²

D 1898 Practice for Sampling of Plastics³

D 3985 Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheet Using a Coulometric Sensor²

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *oxygen gas transmission rate (O₂GTR)*—as applied to a package, is the quantity of oxygen gas passing through the surface of the package per unit of time.

3.1.1.1 *Discussion*—The SI unit of transmission rate is the mol/pkg·s. The test conditions, including temperature, oxygen partial pressure and humidity on both sides of the package, must be stated. A commonly used unit of O₂GTR is the cm³(STP)/pkg·d, where 1 cm³(STP) is 44.62 × 10⁻⁶ mol, 1 atm is 0.1013 mPa, and one day is 86 400 s.

3.1.2 *oxygen permeability coefficient (P_{O₂})*—the product of the permeance and thickness of film.

3.1.2.1 *Discussion*—The permeability is meaningful only for homogenous 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 in tests using several thicknesses of the material. The SI unit of permeability is the mol/m·s·Pa. The test conditions must be stated.

3.1.3 *oxygen permeance (P_{O₂})*—the ratio of the O₂GTR to the difference between the partial pressure of O₂ on the two sides of the package wall.

3.1.3.1 *Discussion*—The SI unit of permeance is the mol/(pkg·s·Pa). The test conditions (see 4.2) must be stated.

4. Summary of Test Method

4.1 The test method employs a coulometric oxygen sensor and associated equipment in an arrangement similar to that described in Test Method D 3985. Oxygen gas transmission rate (O₂GTR) is determined after the package has been mounted on a test fixture and has equilibrated in the test environment.

4.2 The package is mounted in such a way as to provide that the inside of the package is slowly purged by a stream of nitrogen while the outside of the package is exposed to a known concentration of oxygen. The package may be exposed in ambient room air which contains 20.8 % oxygen, or immersed in an atmosphere of 100 % oxygen. As oxygen permeates through the package walls 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 of time.

5. Significance and Use

5.1 Oxygen gas transmission rate is an important determinant of the protection afforded by barrier materials. It is not, however, the sole determinant, and additional tests, based on experience, must be used to correlate package performance with O₂GTR. This test method is suitable as a referee method of testing, provided that the purchaser and seller have agreed on sampling procedures, standardization procedures, test conditions, and acceptance criteria.

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.

¹ This test method is under the jurisdiction of ASTM Committee F-2 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods.

Current edition approved July 27, 1990. Published September 1990.

² Annual Book of ASTM Standards, Vol 15.09.

³ Annual Book of ASTM Standards, Vol 08.01.

7. Apparatus

7.1 *Oxygen Gas Transmission Apparatus*,⁴ as diagrammed in Fig. 1 with the following:

7.1.1 *Package Test Stations*, providing a means for the introduction and exhaust of the nitrogen carrier gas stream without significant loss or leakage.

7.1.1.1 Experience has shown that arrangements using multiple package test stations are a practical way to increase the number of measurements that can be obtained from a coulometric sensor. A valving manifold connects the carrier gas side of each individual test station to the sensor in a predetermined pattern. Carrier gas is continually purging the carrier gas sides of those packages that are not connected to the sensor. Either test gas (100 % oxygen) or normal room air (20.8 % oxygen), whichever is appropriate, contacts the outside of the package.

7.1.2 *Diffusion Cell*, consisting of two metal halves which, when closed upon the film used for system calibration, will accurately define a circular area of that film. Typical diffusion cell areas are 100 cm² and 50 cm². The volumes inside the cell above and below the enclosed film are not critical; they 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. Means shall be provided for the measurement of cell temperature.

7.1.2.1 *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 the 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.

7.1.2.2 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 must be smooth and flat, without scratches which may promote leakage.

7.1.2.3 *Diffusion Cell Pneumatic Fittings*—Each half of the diffusion cell shall incorporate suitable fittings for the introduction and exhaust of gas without significant loss or leakage.

7.1.2.4 It is desirable to thermostatically control the diffusion cell. A simple resistive heater, attached to the carrier gas side of the cell in such a manner as to ensure good thermal contact, 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 coils to remove some of the heat.

7.1.3 *Catalyst Bed*, a small metal tube with fittings for attachment to the inlet of the nitrogen gas pneumatic fitting containing 3 to 5 g of .5 % platinum or palladium catalyst on alumina⁵ to provide an essentially oxygen-free carrier gas to the diffusion cell and to each package test station.

7.1.4 *Flowmeter*, a metal/glass, ball-in-tube flowmeter having an operating range of 5 to 100 mL/min is required to monitor the flow rate of nitrogen carrier gas through each test station.

7.1.5 *Flow Switching Valves*—Two or more four-port ball valves for the switching of the nitrogen and test gas flow streams.

7.1.6 *Oxygen-Sensitive Coulometric Sensor*, operating at an essentially constant efficiency is employed to monitor the quantity of oxygen transmitted.

7.1.7 *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 load resistors are 5.3Ω and 53 Ω. These values yield a convenient relationship between the output voltage and the oxygen transmission rate as expressed in terms of cm³(STP)/pkg·d.

7.1.8 *Voltage Recorder*—The voltage across the load resistor is measured and recorded using a strip-chart potentiometer, data-logger or other suitable device. The instrument or system should be able to measure a full-scale voltage of 50 mV. It should be able to measure voltages as low as 0.10 mV with a resolution of at least 10μ V. An input impedance of 5000 Ω or higher is acceptable.

8. Reagents and Materials

8.1 *Nitrogen Carrier Gas*, consisting of a nitrogen and hydrogen mixture in which the percentage of hydrogen shall fall between 0.5 and 3.0 volume percent. 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.

⁴ Suitable apparatus such as that embodied in several OXTRAN models, available from MOCON/Modern Controls, Inc., 6820 Shingle Creek Parkway, Minneapolis, MN 55430, or their equivalents, have been found satisfactory.

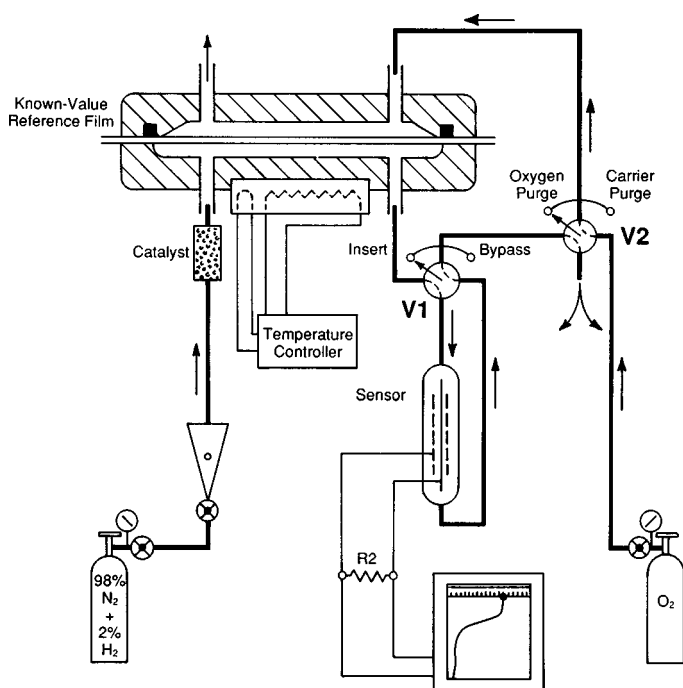


FIG. 1 Arrangement of Components when Reference Film is Used to Calibrate System for Package Testing

⁵ A suitable catalyst which can be obtained from Englehard Industries Division, Chemical Dept., 429 Delancey Street, Newark, NJ 07105, or its equivalent has been found satisfactory.