Designation: F2622 – 08 (Reapproved 2013)

Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using Various Sensors[1]

This standard is issued under the fixed designation F2622; 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 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 (O_2GTR), *(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 O_2GTR. There may be other methods of O_2GTR determination that use other oxygen sensors and procedures.

1.3 This test method has intentionally been prepared to allow for the use of various sensors, devices, and procedures. The precision and bias of each design needs to be individually established to determine the applicability of that instrument or method to meet the needs of the user.

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

1.5 *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]

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

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

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. The oxygen permeability coefficient 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 O_2GTR 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 15.1) must be stated.

3.1.3 *oxygen transmission rate (O_2GTR)*—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 *Discussion*—A commonly used unit of O_2GTR 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 O_2GTR 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 controlled test environment. Control of carrier gas flow rate (for concentration detectors), relative humidity, temperature, and oxygen concentration in both the carrier gas and permeant (test) gas chambers is critical.

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

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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.

the film into the nitrogen carrier gas, it is transported to the detector where it produces a signal representing the oxygen transmission rate.

5. Significance and Use

5.1 The O_2GTR 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 O_2GTR. 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 Testing which has compared select instruments with other sensors to the instruments specifically described in Test Method D3985 is shown in Section 16, Precision and Bias, of this method.

5.3 The Precision and Bias section of this method shows results using several instruments with non-coulometric and coulometric sensors.

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 carbon monoxide, hydrocarbons, free chlorine, and some strong oxidizing agents. Exposure to carbon dioxide should also be minimized to avoid damage to the sensor through reaction in some sensor types.

7. Apparatus

7.1 *Oxygen Gas Transmission Apparatus,* 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*—Various designs may be included in the diffusion cell design. Some systems may require vacuum grease to form a proper seal. The design will define the test area of the film as it is tested.

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

7.1.1.3 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.

7.1.1.4 Experience has shown that arrangements using multiple diffusion cells are a practical way to increase the number of measurements that can be obtained from a single sensor. Valves connect 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 *Flow Controller*—A flow controller will control the flow of carrier and test gases with sufficient precision to allow determination of the oxygen permeability in instruments which calculate the oxygen permeability based on the oxygen concentration change in the carrier gas stream. In some instruments (such as the Coulometric), the flow rate does not need to be controlled as precisely.

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

7.1.4 *Sensor*—An oxygen-sensitive sensor with sufficient sensitivity and precision to yield meaningful results can use various operating principles including coulometric, electrochemical and zirconium oxide. Different sensors may have different levels of sensitivity. The user should select the instrument/sensor system which will adequately cover the oxygen permeation range and degree of precision of interest.

7.1.5 *Data Recording System*—An appropriate data recording system shall record all pertinent information. Various integrated and external computer systems have been found effective.

8. Reagents and Materials

8.1 *Nitrogen Carrier Gas* shall consist of nitrogen. The carrier gas shall be dry and contain not more than 5 ppm of oxygen. If catalysts or other oxygen absorbers are employed, a higher oxygen level may be found to be acceptable. If other gases are needed to be included in this nitrogen to allow catalysts to function they may be incorporated up to 5 %.

8.2 *Oxygen Test Gas* shall be dry and contain not less than 99 % oxygen (except as provided in 13.8).

8.3 *Sealing Grease*—For some instrument types, a vacuum or stopcock grease may be required to seal the specimen film in the diffusion cell.

8.4 *Water for Humidification*—For humidification of the carrier and permeant gas streams, ultra-high purity water is required for some instrument types to prevent plugging of the humidification system. This water should have a resistivity of at least 18 MΩ. An example of a suitable type is high-pressure liquid chromatography (HPLC) water.

9. Precautions

9.1 Temperature and relative humidity are critical parameters affecting the measurement of O_2GTR. Careful temperature and relative humidity control can help to minimize variations due to environmental fluctuations. During testing, the temperature shall be monitored to the nearest 0.5°C and the relative humidity to the nearest 0.5 percent. The average conditions and range of conditions experienced during the test period shall both be reported.

9.2 The sensor may require a relatively long time to stabilize to a low reading characteristic of a good barrier after it has been used to test a poor barrier such as low-density