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D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]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

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 polyethylene. For this reason, materials of comparable gas transmission qualities should be tested together.

9.3 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 lowered when the instrument is not being used.

9.4 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 O_2GTR 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. If this measurement may damage the specimen, it can be done after permeation has been tested.

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 (test gas) side of the diffusion cell").

12. Conditioning

12.1 After the sample has been mounted in the diffusion cell, a sufficient length of time must be allowed for the film to reach equilibrium. No conditioning prior to mounting the film sample in the diffusion cell is needed.

13. Procedure

13.1 Various instruments will have somewhat different operating procedures and each instrument's specifics are beyond the scope of this test method.

13.2 Regardless of the specific instrument employed, there are some steps common to any system.

13.2.1 *Instrument Warm-Up Period*—Depending on the sensor and specific system involved some period of time should be allowed to insure stability.

13.2.2 *Diffusion Cell Preparation*—In many systems, the seal separating the test area of the film and the surrounding environment requires O-rings or finished surfaces. If recommended by the instrument manufacturer, apply sealing grease evenly.

13.2.3 *Specimen Preparation*—The size of the specimen obviously depends on the diffusion cell design. The sample is placed carefully in the diffusion cell taking care to avoid wrinkles and creases. Clamp the halves of the cell together tightly.

13.2.4 *Purging the System*—Allow the gases to flow to purge the system of ambient air before taking any measurements

13.3 The following three flow alternative configurations for the carrier gas are made using various valves and controls. The oxygen transmission rate in the carrier gas is measured in each configuration. Typically, the background oxygen transmission rate levels are measured first, followed by the measured level of oxygen transmission rate through the film.

13.3.1 *Background Gas Cylinder Oxygen Transmission Rate*—The gas is flowed directly from the carrier gas source, through an oxygen reducing catalyst or other oxygen absorber, if desired, and then to the sensor.

13.3.2 *Background Diffusion Cell Oxygen Transmission Rate*—A stream of carrier gas is directed through the upper (test gas) side of the diffusion cell) and another stream flows through the lower (carrier gas) side of the diffusion cell chambers. The oxygen transmission rate going through the carrier side of the cell is measured.

13.3.3 *Measured Diffusion Cell Oxygen Transmission Rate*—The carrier gas is directed to flow through the carrier side of the cell while oxygen (in whatever concentration is desired) is directed through the oxygen (test gas) side of the diffusion cell and then to the sensor.

13.4 *Temperature* shall be obtained by monitoring the temperature as closely as possible to the specimen.

13.5 *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.

13.6 *Tests in a Moist Environment*—This test method can be conducted with test and carrier gases at any controlled temperature and relative humidity. Provision to control and monitor environmental conditions must be made and validated. Design details to accomplish temperature and humidity control are beyond the scope of this test method.

13.7 The O_2GTR 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. Follow the manufacturer's recommendations for temperature operating ranges for any specific instrument.

13.8 *Testing Poor Barriers*—Films having transmission rates in excess of 200 cm^3 (STP) / (m^2·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 for some detector types. One way to avoid this problem is to use a test gas that is a mixture with a known concentration of oxygen in nitrogen instead of nominally 100 % oxygen. 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 a portion 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.

14. Calculation

14.1 The Background Oxygen Transmission Rate is determined by subtracting the Background Gas Cylinder Oxygen Transmission Rate (13.3.1) from the Background Diffusion Cell Oxygen Transmission Rate (13.3.2). This value can be thought of as a system leak rate. This Background Oxygen Transmission Rate is OTR_B.

14.2 The Measured Oxygen Transmission Rate is determined by subtracting the Background Gas Cylinder Oxygen Transmission Rate (13.3.1) from the Measured Diffusion Cell Oxygen Transmission Rate (13.3.3). This measured Oxygen Transmission Rate is OTR_M.

14.3 The Final Measured Oxygen Transmission Rate is determined by subtracting the Background Oxygen Transmission Rate (14.1) from the Measured Oxygen Transmission Rate (14.2). The final measured oxygen transmission rate OTR_F.

14.4 The Final Measured Permeation Concentration is converted to Oxygen Transmission Rate as follows:

$$OTR_F = OTR_M - OTR_B \text{ where units are cm}^3\text{/day}$$

$$\frac{OTR_F}{A_{ts}} = OTR$$

where:

OTR = Oxygen Transmission Rate, ml or cm^3 of O_2/m^2/day, and
A_{ts} = Surface Area of Test Sample, m^2.

For Concentration Detectors:
OTR_F = Q(Cal Factor)*{$conc_F$[ppm(v/v)]/1E6}*flow(cm^3/min)*{1440 min/day}
OTR_M = Q(Cal Factor)*{{$conc_M$[ppm(v/v)]/1E6}*flow(cm^3/min)*{1440 min/day}
OTR_B = Q(Cal Factor)*{{$conc_B$[ppm(v/v)]/1E6}*flow(cm^3/min)*{1440 min/day}
Note: Cal Factor calculated based on known Tank Gas or Reference Film
For Coulometric Detectors:
OTR_F = 5.3*Sensor $Current_F$(mA)
OTR_M = 5.3*Sensor $Current_M$(mA)
OTR_B = 5.3*Sensor $Current_B$(mA)
Note: No Cal Factor required for Coulometric

15. Report

15.1 Report the following information:

15.1.1 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.

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

15.1.3 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 mmHg (sea level).

NOTE 1—The barometric compensation factor may be calculated as follows:

$$fBP_{760} = \frac{BPa}{BP_{760}} \quad (1)$$

where:

fBP_{760} = barometric compensation factor,
BP_{760} = 760 mmHg, and
BPa = ambient barometric pressure in mmHg.

15.1.4 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.

15.1.5 The rate of flow of the nitrogen carrier gas during the test for flow sensitive instruments such as those using concentration detectors.

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

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

15.1.8 The values of O_2GTR, permeance (if desired), and the permeability (if desired).

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

15.1.10 The calibration factor (*Q*) and a statement of the means used to obtain the calibration factor (only needed for concentration detection systems).

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

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

16. Precision and Bias

16.1 An interlaboratory test program was performed on the proposed standard during 2005 and 2006. Twenty-two laboratories participated using 3 different sensor systems from Mocon, PBI-Dansensor, and Illinois Instruments, respectively. The number of laboratories using each instrument type were 9, 6, and 7, respectively.

16.2 Five different materials were tested. Each lab was provided with two samples of each material. Several laboratories did not perform all replicates. The data was analyzed for each material both overall and also by each instrument type. Table 1 provides a summary of the precision results to show repeatability (sr and r) and reproducibility (sR and R).

16.3 "s xbar" is the standard deviation among lab averages. "sr" is standard deviation of repeatability and "sR" is standard deviation for reproducibility.

TABLE 1 Summary of ILS Results

Instrument Manufacturers	Number of Labs Participating	Number of Labs Providing Duplicate Data	Average	s xbar	sr	sR	r	R
				Material 2				
Overall	22	18	0.058	0.047	0.015	0.048	0.042	0.134
Mocon	9	9	0.061	0.043	0.011	0.044	0.030	0.123
PBI	6	6	0.061	0.032	0.023	0.035	0.064	0.099
Illinois	7	3[A,B]	0.050	0.069	0.010	0.069	0.028	0.194
				Material 5				
Overall	22	20	0.314	0.118	0.035	0.121	0.098	0.339
Mocon	9	9	0.299	0.043	0.022	0.046	0.060	0.129
PBI	6	6	0.376	0.064	0.054	0.074	0.152	0.208
Illinois	7	5[A]	0.281	0.194	0.023	0.194	0.065	0.544
				Material 1				
Overall	22	20	1.052	0.166	0.067	0.173	0.188	0.484
Mocon	9	9	1.108	0.079	0.036	0.083	0.101	0.231
PBI	6	6	1.025	0.122	0.108	0.144	0.301	0.403
Illinois	7	5[A,C]	0.995	0.274	0.045	0.276	0.126	0.772
				Material 4				
Overall	22	19	12.28	1.01	0.22	1.02	0.616	2.856
Mocon	9	8[D]	11.85	0.57	0.18	0.60	0.51	1.694
PBI	6	6	12.21	1.15	0.18	1.16	0.518	3.250
Illinois	7	5[A]	12.82	1.14	0.30	1.16	0.842	3.260
				Material 3				
Overall	22	20	63.49	5.23	1.58	5.35	4.418	14.980
Mocon	9	9	64.09	5.73	2.22	5.94	6.203	16.632
PBI	6	6	62.55	3.82	0.65	3.85	1.818	10.776
Illinois	7	5[A]	63.52	6.19	0.79	6.22	2.201	17.413

[A] With each material tested, two or three labs conducted only one test. The single results were included in estimating average and reproducibility standard deviations only.
[B] One laboratory was dropped due to significantly negative results which were also deemed to be outliers as the results were about 24 standard deviations outside the norm, which is essentially statistically impossible.
[C] One laboratory dropped from calculations because of a single very low results which was deemed to be an outlier as the result was about 6 standard deviations from the norm, which would be highly unusual.
[D] One laboratory did not report any values.

16.4 The following materials were used in the interlaboratory study:

Material	Material Identification Type	Nominal Thickness	Approximate OTR (cm^3/m^2-day)
1	EVOH	5.5 mil	1.10
2	Metalized PET	50 gauge	0.06
3	PET	0.92 mil	64.00
4	PET	4.75 mil	11.90
5	EVOH Lamination	5.7 mil	0.30

16.5 A Research Report[4] is available from ASTM Headquarters listing all individual data and a complete statistical analysis of results.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1025.

17. Keywords

17.1 oxygen transmission rate; permeability; permeation; plastic films