Designation: D1434 – 23

Standard Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting[1]

This standard is issued under the fixed designation D1434; 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 utilizes a manometric method to determine the steady-state rate of transmission of a gas through plastics in the form of film, sheeting, laminates, and plastic-coated papers or fabrics. This test method provides for the determination of *(1)* gas transmission rate (GTR), *(2)* permeance, and, in the case of homogeneous materials, *(3)* permeability.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *gas transmission rate, GTR, n*—the quantity of a given gas passing through a unit of the parallel surfaces of a plastic film in unit time under the conditions of test. The SI unit of GTR is mol/(m^2·s). The test conditions, including temperature and partial pressure of the gas on both sides of the film, must be stated. Other factors, such as relative humidity and hydrostatic pressure, that influence the transport of the gas must also be stated. A commonly used unit of GTR is mL (STP)/(m^2·d) at a pressure differential of one atmosphere.

3.1.2 *permeance, P, n*—the ratio of the gas transmission rate to the difference in partial pressure of the gas on the two sides of the film. The SI unit of permeance is mol/ (m^2·s·Pa). The test conditions (see 5.1) must be stated.

3.1.3 *permeability, P', n*—the product of the permeance and the thickness of a film. The permeability is meaningful only for homogeneous materials, in which it is a property characteristic of the bulk material. This quantity should not be used unless the constancy of the permeability has been verified using several different thicknesses of the material. The SI unit of *P* is (mol·m)/(m^2·s·Pa) = mol/(m·s·Pa). The test conditions (see 5.1) must be stated.

NOTE 1—One milliliter (STP) is 44.62 µmol, one atmosphere is 0.1013 MPa, and one day is 86.4×10^3 s. GTR in SI units is obtained by multiplying the value in inch-pound units by 5.160×10^{-10}. Additional units and conversions are shown in Appendix X1.

3.1.4 *steady state*—the state attained when the amount of gas absorbed in the film is in equilibrium with the flux of gas through the film.

4. Summary of Test Method

4.1 The sample is mounted in a gas transmission cell so as to form a sealed semi-barrier between two chambers. One chamber contains the test gas at a specific high pressure, and the other chamber, at a lower pressure, receives the permeating gas.

4.1.1 The lower pressure chamber is initially evacuated. Following the evacuation, the low-pressure chamber is allowed to accumulate the permeating test gas. The transmission of the gas through the test specimen is determined from monitoring the slope of the increasing pressure over time.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Primary 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.

5. Significance and Use

5.1 These measurements give semiquantitative estimates for the gas transmission of single pure gases through film and sheeting. Correlation of measured values with any given use, such as packaged contents protection, must be determined by experience. The gas transmission rate is affected by conditions not specifically provided for in these tests, such as moisture content (Note 2), plasticizer content, and nonhomogeneities. These tests do not include any provision for testing seals that may be involved in packaging applications.

NOTE 2—The tests are run using gas with 0 % moisture changes.

5.2 The historic Interlaboratory testing has revealed that permeances measured by these procedures exhibit a strong dependence on the procedure being used, as well as on the laboratory performing the testing. The historic method relied upon manual calibrations of Hg capillary columns and manual data readings of pressure. The references and use of Hg and capillary columns have been removed from this standard as current D1434 instruments rely upon readily calibrated vacuum gauges and automated recording of data. It is planned that the next revision of this standard includes an updated ILS with modern instrumentation. Additionally, it has been noted that an agreement with other gas transmission rate methods is sometimes poor and may be material-dependent. The materials being tested often affect the between-laboratory precision. The causes of these variations are not precisely known at this time, but is likely due to the fact that this method analyzes ALL gasses from the sample and not just the Test gas. This includes pre-absorbed water vapor within the sample and any free solvents remaining within the specimen. The 48 hr desiccator drying period outlined within the method may not be long enough for all materials. Additionally, other gas transmission rate methods (as those used for oxygen transmission rate, water vapor transmission rate and carbon dioxide transmission rate) often incorporate test gas specific sensors and therefore would minimize influence from other gasses. It is suggested that this method not be used for referee purposes unless purchaser and seller can both establish that they are measuring the same quantity to a mutually agreed upon level of precision.

5.3 Use of the permeability coefficient (involving conversion of the gas transmission rate to a unit thickness basis) is not recommended unless the thickness-to-transmission rate relationship is known from previous studies. Even in essentially homogeneous structures, variations in morphology (as indicated, for example, by density) and thermal history may influence permeability.

6. Test Specimen

6.1 The test specimen shall be representative of the material, free of wrinkles, creases, pinholes, and other imperfections, and shall be of uniform thickness and be capable of being mounted airtight (for example, no edge leakage, which can occur with papers). The test specimen shall be cut to an appropriate size (generally circular) to fit the test cell.

6.2 Use three specimens unless otherwise specified or agreed upon among interested parties.

6.3 Mark the side of the material facing the test gas. Note: It's most common to orientate the sample as it is utilized in real-life (for example, if measuring oxygen passing from the outside of a package to the inside, then the "outside face" of the sample should be exposed to the high pressure oxygen and the "inside face" should be exposed to the vacuum side of the test cell).

6.4 If required for permeability normalization, the thickness of the specimen shall be measured to the nearest 2.5 µm with a calibrated dial gage (or equivalent) at a minimum of five points distributed over the entire test area. Maximum, minimum, and average values should be recorded. An alternative measure of thickness involving the weighing of a known area of specimens having a known density is also suitable for homogeneous materials.

7. Conditioning and Test Temperature

7.1 *Standard Conditioning*—To reduce innate outgassing concerns of the material (such as absorbed water vapor or solvents), condition all test specimens at 23 °C ± 2 °C in a desiccator over calcium chloride or other suitable desiccant for not less than 48 h prior to test in accordance with Practice D618, for those tests where conditioning is required.

7.2 *Alternative Conditioning*—Alternatives to 7.1 may be used for conditioning the specimens provided that these conditions are described in the report. As an example, an alternative conditioning could be placing the sample into a N2 purging box to remove absorbed humidity or solvents.

7.3 *Test Temperature*—For ambient testing, the temperature of the cell should be controlled at 23 °C ± 2 °C. For instruments that can control the cell temperature a tolerance of ± 1 °C shall be utilized.

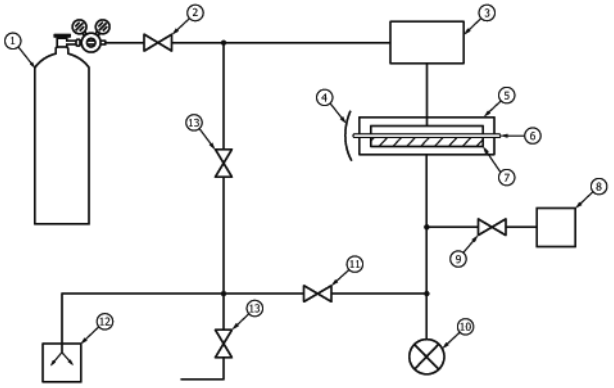
8. Sampling

8.1 The techniques used in sampling a batch of material to be tested by these procedures must depend upon the kind of information that is sought. Care should be taken to ensure that samples represent conditions across the width and along the length of rolls of film. Practice D1898 provides guidelines for deciding what procedures to use in sampling a batch of material. Enough specimens must be tested to ensure that the information obtained is representative of the batch or other lot size being tested.

9. Apparatus

9.1 The apparatus shown in Fig. 1 outlines the components needed for a manometric GTR test.

The apparatus consists of a test cell that clamps a test specimen between two cell halves, creating high-pressure and low-pressure chambers. A pressure differential during the test creates the environment for gas transmission through the sample. On the test gas (high-pressure) side of the sample is a feeder that ensures that the supply and pressure of the test gas remains a constant driving force. On the measurement (low-pressure) side of the sample is a pressure sensor (vacuum gauge) that measures the pressure change of the system as gas permeates through the specimen. Plumbed beneath the low-pressure chamber is a vacuum pump to initially evacuate the



KEY

1 = TEST GAS
2 = NEEDLE VALVE
3 = GAS FEEDER
4 = TEST CELL
5 = HIGH PRESSURE CHAMBER
6 = SAMPLE SPECIMEN
7 = LOW PRESSURE CHAMBER
8 = CELL VOLUME CONTROL ADAPTER
9 = CELL VOLUME VALVE
10 = VACUUM GAUGE
11 = CELL VACUUM VALVE
12 = VACUUM PUMP
13 = ISOLATION & VENTING VALVES

FIG. 1 Components Needed for a Manometric GTR Test

system for the transmission rate measurement. An optional volume control adapter plumbed into the low-pressure chamber can be utilized for extending the range of the GTR test.

9.1.1 *Test Gas*—A tank of known gas with enough volume and pressure to conduct the test.

9.1.2 *Needle Valve,* for slowly admitting and adjusting the pressure of the test gas.

9.1.3 *Gas Feeder*—The gas supplied by the Gas Feeder to the upper chamber is a reservoir (usually a tank gas) that has enough capacity and constant driving force pressure as to not impact the GTR measurement. Additionally, the Gas Feeder incorporates a pressure gauge with a range from 0 kPa to 333 kPa absolute for measuring the supplied test gas pressure.

9.1.4 *Transmission Rate Test Cell*—The transmission test cell shall consist of a high-pressure chamber and a low-pressure chamber, designed so that the gas transmission area is constant for any specimen mounted in the cell. Note: typical transmission rate cell area diameters can range from 10 mm to 150 mm.

9.1.4.1 The high-pressure chamber shall have an inlet for the test gas. The high-pressure chamber's surface that contacts the specimen shall be smooth and flat and incorporates an O-ring for a leak-free seal.

9.1.4.2 The low-pressure chamber shall be connected to a pressure sensor (vacuum gauge) for the transmission rate pressure measurement. The surface of the low-pressure chamber that contacts the test specimen shall be smooth and flat for a leak-free seal. Beneath the sample is a filter paper/support structure. The filter paper/support structure keeps the sample flat as the vacuum is applied and ensures that the test area of the sample is not occluded and allows for gas transmission rate flow.

9.1.5 *Cell Volume-Control Adapter or Fillers*—Optional: Some instruments incorporate a volume control adapter to aid with adjusting the range of the instrument. This adapter can be an additional reservoir plumbed into the system. Alternatively, using filler blocks within the low-pressure chamber reduces the chamber volume resulting in faster pressure accumulation, which is useful for measuring low transmission rate materials.

9.1.6 *Vacuum Gage,* to register the pressure during evacuation of the system to the nearest 13 Pa.

9.1.7 *Cell Vacuum Valve,* capable of maintaining a vacuum-tight seal.

9.1.8 *Vacuum pump,* capable of reducing the pressure in the system to 26 Pa or less.

9.1.9 Additional valving can be utilized to isolate and vent the system as needed.

10. Materials

10.1 *Test Gas*—The test gas shall be dry and pure. As a guidance, ensure that the gas pressure remains constant throughout the test, the ratio of the volume of gas available for transmission to the volume of gas transmitted at the completion of the test shall be at least 100:1.

10.2 *Vacuum Grease*—Low volatility/high vacuum grease.

10.3 *Filter Paper*—Any high-grade, medium retention qualitative nonashing cellulosic filter paper will be satisfactory for this purpose. A thickness of 0.2 mm to 0.3 mm is recommended. As the filter paper supports the specimen, it needs to be cut to the same size as the gas transmission area of the low-pressure chamber.

11. Calibration

11.1 Pressure gauges and temperature sensors within the apparatus should be calibrated per manufacturer's recommended calibration specifications and schedule.

11.2 Some instruments incorporate a film calibration feature. The GTR calculation relies upon the following: the change in pressure over time (dp/dt); the high-pressure value of the test gas; the volume of the low-pressure side of the system; the test temperature; the transmission rate exposure area of the low-pressure chamber and the Ideal Gas Constant (R). By using a known GTR film standard, one can determine a calibration coefficient to adjust for any variances with the calculation inputs. If an instrument does have this feature, follow the manufacture's guidance for film calibration.

12. Procedure

12.1 If utilized, insert the appropriate volume filler into the low-pressure chamber.

12.2 Center a filter paper in the low-pressure chamber.

12.3 Apply a thin and uniform coating of vacuum grease on the sealing surface of the low-pressure chamber that contacts the specimen. Avoid excessive grease as it can overflow onto the filter paper and reduce the transmission rate exposure area.

12.4 Mount the conditioned specimen on the low-pressure chamber ensuring no creasing or slackness occurs. Sample creasing can create leakage from the chamber and a sample mounted with slack will have a larger surface area. Both of these can lead to inaccurate test results.

12.5 Lightly grease the O-ring and sealing surface on the high-pressure chamber and then carefully position it over the specimen and clamp into place with uniform pressure to ensure a vacuum-tight seal.

12.6 Engage the vacuum pump and evacuate ambient air from the system of the low-pressure chamber; followed by the high-pressure chamber.

12.7 When the air has been evacuated to a level of 26 Pa or less, close the related valving and stop the vacuum pump. Note: the time needed to evacuate the system will be dependent upon the system volume and sample outgassing.

12.8 If the pressure within the low-pressure chamber immediately rises, this could be an indication of a leak or a large amount of sample outgassing and the need to repeat the sample mounting or the initial evacuation, or both.

12.9 Introduce the Test Gas to the high-pressure chamber. Shut off the gas supply when the pressure reaches the desired test pressure level (commonly 101 kPa or 1 atm). Record the high-pressure value. With the test gas addition, an increase in pressure on the low-pressure side confirms gas is transmitting through the sample.

12.10 Collect and plot the pressure reading within the low-pressure chamber versus time.

12.11 When the graph indicates a straight line, this indicates that the material has reached equilibrium. The slope of the straight-line portion of the graph relates directly to the GTR. Calculate and report the GTR.

13. Calculations

13.1 Calculate the Gas Transmission Rate (GTR) in SI units from the following relationship:

$$\text{Gas transmission rate GTR} = \frac{Vc}{R \times T \times Pu \times A} \times \frac{dp}{dt}$$

where:

GTR = the gas transmission rate, expressed in mol/(m^2 · s · Pa),
Note: GTR is commonly expressed in terms of $cm^3/(m^2 \cdot day \cdot atm)$
Vc = the volume of the low-pressure chamber, expressed in liters,
T = the test temperature, expressed in K,
Pu = the pressure of the gas on the high-pressure (upper) chamber, expressed in Pa,
A = the transmission rate area, expressed in m^2,
dp/dt = the change in pressure over time in the straight-line portion of the pressure graph, expressed in terms of Pa/s, and
R = the Ideal Gas Constant = 8.31×10^3 (l · Pa)/(K · mol).

13.2 Calculate the gas Permeability coefficient (P') if required.

$$\text{Permeability coefficient } P' = GTR \cdot d$$

where:

P' = the gas permeability coefficient expressed in (mol · m)/(m^2 · s · Pa),
Note: this value is commonly expressed in terms of $(cm^3 \cdot mm)/(m^2 \cdot day \cdot atm)$
GTR = the gas transmission rate, expressed in mol/(m^2 · s · Pa), and
d = the average thickness of the test sample, espressed in m.

13.3 A test result is defined as a single determination of the transmission rate or permeance of an individual sheet of material.

14. Report

14.1 The report shall include the following:

14.1.1 Identification of the procedure utilized.

14.1.2 Description of the sample, including identification of composition, presence of wrinkles, bubbles, or other imperfections, and manufacturer, if known.

14.1.3 Test gas used, and test gas composition, including purity,

14.1.4 Test temperature in degrees Celsius, and the pressure difference used,

14.1.5 If required for Permeability normalization, the sample thickness measurement plus the average for each specimen. When five or more thickness measurements are made per specimen, the average, standard deviation and number of measurements made may be reported instead of each measurement, and

14.1.6 Each GTR measurement obtained, and if required, the appropriate averages in the units of choice. When five or more replicates are obtained the average, standard deviation, and number of replicates may be substituted for the above.

15. Precision

15.1 *General*—An interlaboratory evaluation of this method has been conducted.[4] Ten laboratories participated in determining the permeability, *P*, of four materials to oxygen and carbon dioxide. The results from the round robin are summarized in Table 1. The results demonstrate clearly that the precision of the results obtained depends strongly, but in an unpredictable manner, on the combination of material and gas being tested. Potential users of this method must, therefore, use their own experience in assessing the precision of the results being obtained.

15.1.1 The contribution arising from the between-laboratory component of the variance is larger than that from the within-laboratory component for all materials. This indicates that there are systematic differences between the procedures used in different laboratories. The magnitudes of these differences must be determined whenever two laboratories are comparing results for referee purposes.

[4] Supporting data are available from ASTM Headquarters. Request RR:D20-0049.