Designation: ~~D1434 – 82 (Reapproved 2015)$^{\varepsilon 1}$~~ 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.

~~ε^1 NOTE—Editorial corrections were made in September 2015.~~

1. Scope

1.1 This test method ~~covers the estimation of the~~ 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 Two procedures are provided:~~

~~1.2.1 *Procedure M*—Manometric.~~

~~1.2.2 *Procedure V*—Volumetric.~~

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 and health~~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D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]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:*

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

3.1.1 *gas transmission rate, ~~GTR—~~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 ~~1~~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. ~~The inch-pound unit of GTR, a~~ A commonly used unit of ~~GTR,~~GTR is ~~1~~mL (STP)/(m^2·d) at a pressure differential of one atmosphere.

3.1.2 *permeance, ~~P—~~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 ~~1~~mol/ (m^2·s·Pa). The test conditions (see 5.1) must be stated.

3.1.3 *permeability, ~~PP',—~~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 ~~1~~(mol·m)/(m^2·s·Pa) = mol ~~mol/(m·s·Pa). /~~(m·s·Pa). The test conditions (see ~~3.1~~5.1) must be stated.

NOTE 1—One ~~millilitre~~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. ~~For Method V, this is obtained when the GTR is constant.~~

4. Summary of Test Method

4.1 The sample is mounted in a gas transmission cell so as to form a sealed ~~semibarrier~~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. ~~Either of the following procedures is used:~~

4.1.1 ~~*Procedure M*—In Procedure M the~~ The lower pressure chamber is initially ~~evacuated and the~~ 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 ~~indicated by an increase in pressure.~~determined from monitoring the slope of the increasing pressure over time.

~~4.1.2 *Procedure V*—In Procedure V the lower pressure chamber is maintained near atmospheric pressure and the transmission of the gas through the test specimen is indicated by a change in volume.~~

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. ~~Agreement with other~~ 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.~~ 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.~~ 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 ~~The~~ 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*—~~Condition~~ To reduce innate outgassing concerns of the material (such as absorbed water vapor or solvents), condition all test specimens at ~~23 ± 2°C~~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. ~~In cases of disagreement, the tolerances shall be ±1°C.~~

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.

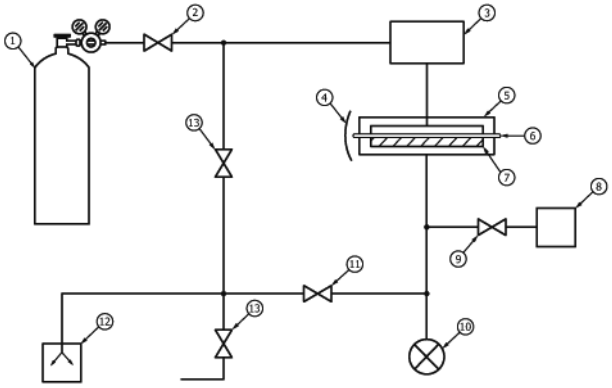
~~PROCEDURE M~~

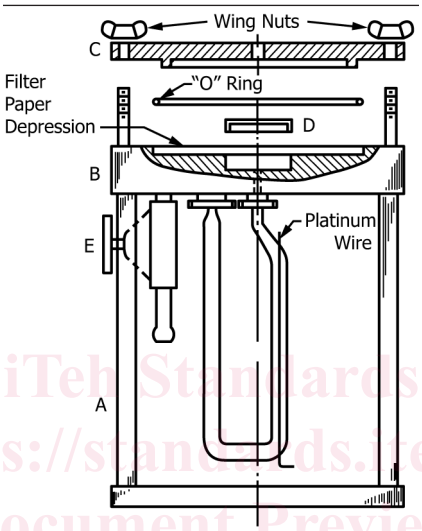
~~(Pressure changes in the manometric cell may be determined by either visual or automatic recording.)~~

~~MANOMETRIC VISUAL DETERMINATION~~

9. Apparatus

9.1 The apparatus shown in Fig. 1 ~~and~~outlines ~~Fig. 2 consists of the following items:~~the components needed for a manometric GTR test.





~~*A*—Supporting Legs~~
~~*B*—Lower Plate~~
~~*C*—Upper Plate~~
~~*D*—Adapter~~
~~*E*—Vacuum Valve~~

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. 21 ~~Schematic View of Gas Transmission Cell~~Components Needed for a Manometric GTR Test
~~FIG. 3 Cell Manometer with Test Specimen in Place~~

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 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 ~~*Cell Manometer System—*~~*Needle Valve,* ~~The calibrated cell manometer leg, which indicates~~ for slowly admitting and adjusting the pressure of ~~transmitted gas, shall consist of precision-bore glass capillary tubing at least 65 mm long with an inside diameter of 1.5 mm.~~the test gas.

9.1.3 ~~*Cell Reservoir System,*~~ *Gas Feeder*—~~consisting of a glass reservoir of sufficient size to contain all the mercury required in the cell.~~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 ~~*Adapters—*~~*Transmission Rate Test Cell*—~~Solid and hollow adapters for measurement of widely varying gas transmission rates. The solid adapter provides a minimum void volume for slow transmission rates. The hollow adapter increases the void volume by about a factor of eight for faster transmission rates.~~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.

~~9.1.4 *Cell Vacuum Valve,* capable of maintaining a vacuum-tight seal.~~[5]

~~9.1.5 *Plate Surfaces,* that contact the specimen and filter paper shall be smooth and flat.~~

~~9.1.6 *O-Ring,* for sealing the upper and lower plates.~~

~~9.1.7 *Pressure Gage,* mechanical or electrical type with a range from 0 to 333 kPa absolute. Used for measuring upstream gas pressure.~~

9.1.5 ~~*Barometer,*~~ *Cell Volume-Control Adapter or Fillers*—~~suitable for measuring the pressure of the atmosphere to the nearest 133 Pa.~~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,*~~*pump,* capable of reducing the pressure in the system to 26 Pa or less.

~~9.1.11 *Needle Valve,* for slowly admitting and adjusting the pressure of the test gas.~~

9.1.9 ~~*Cathetometer,* to measure the height of mercury in the cell manometer leg accurately. This instrument should be capable of measuring changes to the nearest 0.5 mm.~~Additional valving can be utilized to isolate and vent the system as needed.

~~9.1.13 *Micrometer,* to measure specimen thickness, graduated to 2.5 µm (0.1 mil) or better.~~

~~9.1.14 *Elevated-Temperature Fittings*—Special cell fittings are required for high-temperature testing.~~

10. Materials

10.1 *Test Gas*—The test gas shall be dry and pure. ~~The~~ 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 ~~*Mercury*~~—*Filter Paper*—~~Mercury used in the cell shall be triple distilled, checked regularly for purity, and replaced with clean mercury when necessary.~~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.

~~10.2.1 *Warning*—Very low concentrations of mercury vapor in the air are known to be hazardous. Guidelines for using mercury in the laboratory have been published by Steere.[6] Be sure to collect all spilled mercury in a closed container. Transfers of mercury should be made over a large plastic tray. Under normal daily laboratory-use conditions, the cells should be cleaned about every 3 months. Dirty mercury is indicated when the drop of the capillary becomes erratic or when mercury clings to the side of the capillary, or both. Whenever such discontinuities occur, the mercury should be removed and the cell cleaned as follows:~~

~~*(1)* Wash with toluene (to remove greases and oils).~~

~~*(2)* Wash with acetone (to remove toluene).~~

~~*(3)* Wash with distilled water (to remove acetone).~~

~~*(4)* Wash with a 1 + 1 mixture of nitric acid and distilled water (to remove any mercury salts that may be present). This operation may be repeated if necessary in order to ensure complete cleaning of glassware.~~

~~*(5)* Wash with distilled water (to remove nitric acid).~~

~~*(6)* Wash with acetone (to remove water).~~

~~*(7)* Dry the cell at room temperature or by blowing a small amount of clean dry air through it.~~

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 ~~Each cell should be calibrated at the test temperature as follows (~~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~~Fig. 3~~): 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.

~~11.1.1 Determine the void volume of the filter paper from the absolute density of its fiber content (Note 3), the weight of the filter paper, and its apparent volume (Note 4). Express the void volume determined in this way in microlitres and designate as V_{CD}.~~

~~NOTE 3—Any high-grade, medium-retention qualitative nonashing cellulosic filter paper, 90 mm in diameter will be satisfactory for this purpose. Cellulose fiber has an approximate density of 1.45 g/mL.~~

~~NOTE 4—The apparent volume may be calculated from the thickness and diameter of the filter paper.~~

~~11.1.2 Determine the volume of the cell manometer leg from *B* to *C*, Fig. 3, by mercury displacement. (Since the void volume of the adapters is included in this part of the calibration, the volume from *B* to *C* should be determined twice, once with the solid adapter in place, and once with the hollow.) This volume is obtained by dividing the weight of the mercury displaced by its density (Note 5). Determine this volume to nearest 1 µL and designate as V_{BC}.~~

~~NOTE 5—The density of mercury at 23°C is 13.54 g/mL.~~

~~11.1.3 Determine the volume, in microlitres, of the cell manometer leg from *A* to *B*, Fig. 3, by mercury displacement. Determine~~

~~the average cross-sectional area of the capillary by dividing this volume by the length (expressed to the nearest 0.1 mm) from *A* to *B*.Determine this area to the nearest 0.01 mm^2 and designate as a_c.~~

~~11.1.4 Determine the area of the filter paper cavity to the nearest 1 mm^2. Designate this area as *A*, the area of transmission.~~

~~11.1.5 Pour the mercury from the reservoir into the manometer of the cell by carefully tipping the cell. Record the distance from the datum plane to the upper calibration line *B* in the capillary leg as h_B. Record the distance from the datum plane to the top of the mercury meniscus in the reservoir leg as h_L. Determine h_B and h_L to the nearest 0.5 mm.~~

~~11.2 NBS Standard Reference Material 1470[7] is a polyester film whose permeance to oxygen gas has been certified for a range of experimental conditions. The calibration steps in 11.1 can be verified by comparing measurements obtained using this method of test in the user's laboratory with the values provided on the certificate accompanying the SRM.~~

12. Procedure

12.1 ~~Transfer all the mercury into the reservoir of the cell manometer system by carefully tipping the cell in such a way that the mercury pours into the reservoir.~~If utilized, insert the appropriate volume filler into the low-pressure chamber.

~~12.2 Insert the appropriate adapter in the cell body.~~

12.2 Center a filter paper in the ~~lower plate cavity.~~low-pressure chamber.

12.3 Apply a ~~light~~ thin and uniform coating of vacuum grease on the ~~flat metal that the~~ sealing surface of the ~~specimen will contact. Avoid excessive grease.~~ 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 ~~Place~~Mount the conditioned specimen ~~smoothly on the lower lightly greased plate so that it covers the filter paper and the entire exposed face of the lower plate.~~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 ~~Locate~~ Lightly grease the O-ring ~~on the upper plate;~~ and sealing surface on the high-pressure chamber and then carefully position ~~this plate~~ it over the specimen and ~~fix the plate~~clamp into place with uniform pressure to ensure a vacuum-tight seal.

~~12.7 Connect the line in which the test gas will be subsequently admitted to the upper plate. (The entire cell is now directly connected to the test gas line.)~~

~~12.8 Connect the vacuum source to the nipple attached to the cell vacuum valve. Evacuate the bottom of the cell; then, with the bottom still being evacuated, evacuate the top of the cell. Close off the vacuum line to the top of the cell; then close the line to the bottom (Fig. 4).~~

~~12.9 Flush the connecting line and the top of the chamber with test gas.~~

~~12.10 Reevacuate the system in the same manner as 12.8. The cell manometer system should be evacuated to a pressure of 26 Pa or less, as indicated on the vacuum gage.~~

12.6 ~~Pour mercury from the reservoir into the manometer~~ Engage the vacuum pump and evacuate ambient air from the system of the ~~cell by carefully tipping the cell. The height of the mercury in the capillary leg should be at approximately the same level as line~~ low-pressure chamber; followed by the high-pressure chamber.~~B~~(~~Fig. 3~~) ~~and stationary.~~

~~NOTE 6—A leak is indicated if the height of the mercury does not remain stationary. If such a leak occurs, discontinue the test and repeat the entire procedure. (If a leak occurs on a second trial, this may indicate a mechanical failure of the equipment.)~~