Designation: D6701 – 21

Standard Test Method for Determining Water Vapor Transmission Rates Through Nonwoven and Plastic Barriers[1]

This standard is issued under the fixed designation D6701; 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 determining the rate of water vapor transmission ranging between 500 to 100 000 g/(m^2·day) through nonwoven and plastic barrier materials. The method is applicable to films, barriers consisting of single or multilayer synthetic or natural polymers, nonwoven fabric, and nonwoven fabrics coated with films up to 3 mm (0.1 in.) in thickness.

1.2 This test method provides for the determination of (*1*) water vapor transmission rate (WVTR), and (*2*) the permeance to water vapor.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard. The acceptable units for WVTR are usually g/(m^2·day).

1.4 *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.*

1.5 *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]

D123 Terminology Relating to Textiles

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

D4204 Practice for Preparing Plastic Film Specimens for a Round-Robin Study

D5729 Test Method for Thickness of Nonwoven Fabrics (Withdrawn 2008)[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F1249 Test Method for Water Vapor Transmission Rate Through Plastic Film and Sheeting Using a Modulated Infrared Sensor

3. Terminology

3.1 *Definitions:*

3.1.1 *water vapor permeability coefficient, n*—the ratio of the permeance and the thickness.

3.1.1.1 *Discussion*—This quantity should not be used unless the relationship between thickness and permeance has been verified in tests using several thickness' of the material. The water vapor permeability is meaningful only for homogeneous materials, in which case it is a property characteristic of bulk material. An accepted unit of water vapor permeability is the metric perm centimeter, or 1 g per m^2 per day per mm Hg-cm of thickness. The SI unit is the mol/(m^2-s-Pa-mm).

3.1.2 *water vapor permeance, n*—the ratio of a barrier's water vapor transmission rate to the vapor pressure difference between the two surfaces.

3.1.2.1 *Discussion*—An accepted unit of water vapor permeance is the metric perm, or 1 g/m^2 per day per mm Hg. The SI unit is the mol/(m^2-s-Pa). Since the water vapor permeance of a specimen is generally a function of relative humidity and temperature, therefore those conditions must be stated.

3.1.3 *water vapor transmission rate (WVTR), n*—the steady-state time rate of water vapor flow through unit area of a specimen, normal to the surfaces under specific conditions of temperature and humidity at each surface.

3.1.3.1 *Discussion*—A common practice accepted unit of water vapor transmission rate is metric g/m^2 per day. The test

[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.

conditions of relative humidity and temperature where the driving force is the difference in relative humidity across the specimen must be stated.

4. Summary of Test Method

4.1 A dry chamber, guard film, and a wet chamber make up a diffusion cell in which the test film is sealed. A first test is made of the WVTR of the guard film and air gap between an evaporator assembly that generates 100 % relative humidity. A sensor produces an electrical signal, the amplitude of which is proportional to water vapor concentration. The electrical signal is routed to a computer for processing. The computer calculates the transmission rate of the air gap and guard film and stores the value for further use. The barrier is then sealed in the test cell and the apparatus started in the test mode. As before, the electrical signal representing the water vapor is sent to the computer which then calculates the transmission rate of the combination of the air gap, the guard film, and the test barrier. The computer then uses this information to calculate the WVTR of the material being tested. The computer determines when the measured results indicate that the specimens have reached equilibrium values and the testing is considered finished.

5. Significance and Use

5.1 The purpose of this test method is to obtain values for the WVTR of barrier materials.

5.2 WVTR is an important property of materials and can be related to shelf life; product stability, breath-ability, and wearing comfort.

5.3 Data from this test method is suitable as a referee method of testing, provided that the purchaser and seller have agreed on sampling procedures, test conditions, and acceptance criteria.

6. Apparatus

6.1 This method utilizes water vapor transmission apparatus[4] comprised of the following:

6.1.1 *Test Cells,* the apparatus has six test cells within two assemblies. Fig. 1 shows a typical cell cross section. The six cells are formed by metal halves which, when closed upon the test specimens, will accurately define a circular area for each. A typical acceptable diffusion cell area is 10 cm^2. The volume enclosed by each cell half, when clamped, is not critical. It is desirable that the air gap between the water evaporator assembly and the guard film be as small practical, but not so small that an unsupported film which sags or buckles will contact the evaporator assembly. The barrier under test should be in intimate contact with the guard film. A depth of approximately 3.2 mm (0.125 in.) has been found to be satisfactory for the carrier gas side of 10-cm^2 cells.

6.1.1.1 *Test Cell O-Ring,* an appropriately sized groove machined into the humid chamber side of the test cell that retains a chlorprene O-ring. The test area is considered to be

[4] The sole source of supply of the apparatus known to the committee at this time is Mocon, Inc., 7500 Boone Avenue North, Suite 111, Minneapolis, MN 55428. If you are aware of any alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend. Mocon's apparatus is known as the Permatran-W Model 100k.

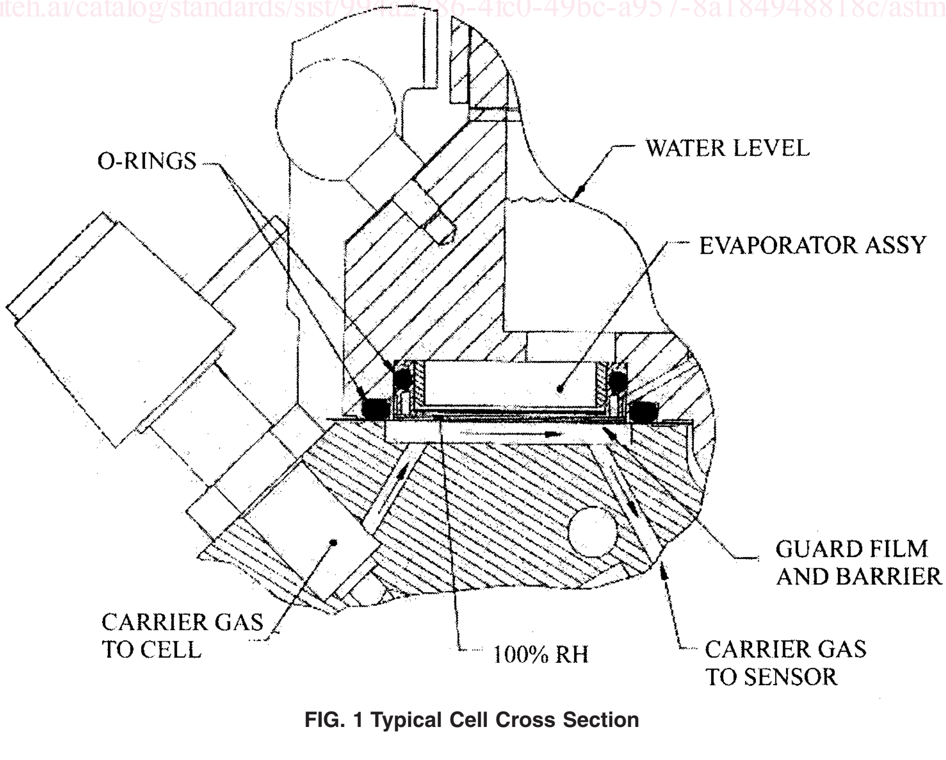

FIG. 1 Typical Cell Cross Section

the area established by the inside contact diameter of the compressed O-ring when the test cell is clamped shut against the test specimen.

6.1.1.2 *Test Cell Sealing Surface,* a flat rim around the dry side of the diffusion cell. This is a critical sealing surface against which the test specimen is pressed; it shall be smooth and without radial scratches.

6.1.1.3 *Test Cell Air Passages,* two holes in the dry half of the diffusion cell that pass carrier gas and water vapor to either exhaust ports or the sensor assembly. One cell at a time can be connected to the sensor assembly by solenoid valves.

6.1.1.4 *Test Cell Guard Film,* a flat film that covers the humid side of each cell. The film is a barrier that stills the air in the gap between the water evaporator and the mounting plane of the specimen. The guard film is a very high transmitter of water vapor. Its transmission rate as well as that of the air gap is accounted for in the apparatus' measurements.

6.1.1.5 *Water Vapor Sensor,* a water vapor detector capable of sensing 0 to 100 % relative humidity with sufficient accuracy so the apparatus can determine transmission rates down to 500 g/m^2 per day.

6.1.1.6 *Post Sensor Dryer,* a no-maintenance dryer that removes water vapor from the measurement gas stream after it passes through the water vapor sensor.

6.1.1.7 *Mass Flowmeter,* a means for regulating the flow of dry air within an operating range of 0 to 200 cc/min.

6.1.1.8 *Flow-Metering Valves,* fine-metering valves capable of controlling the dry-air flow rate to each test.

6.1.2 *Computer System,* a computer provides the main control, calculating, and data storage device for the system.

6.1.3 *Temperature Control,* Temperature of the test specimen is thermostatically controlled by a thermo-electric device (TED) attached to the apparatus that ensures good thermal contact. A thermistor sensor and an appropriate control circuit will serve to regulate the temperature from 20 to 50°C to within 0.1°C.

6.1.4 *Barometric Pressure Sensor,* a sensor that measures the ambient barometric pressure so that variations are automatically corrected in the calculations.

7. Reagents and Materials

7.1 *Desiccant,*[5] for drying air stream.

7.2 *High Purity Level Chromatograph Grade Distilled Water (HPLC),* for producing 100 % relative humidity.

7.3 *Sealing Grease,* a high-viscosity, silicone stopcock grease or other suitable silicone high-vacuum grease is required for lubrication of O-rings.

7.4 *Sample Holder,* a cardboard or metal sample holder is supplied with the apparatus to facilitate loading of specimens.

[5] Linde Molecular Sieve, Type 4A or Type 5A, in the form of 1/8 in. pellets as may be obtained from the Union Carbide Co., Linde Division, Danbury, CT 06817-0001.

8. Sampling and Test Specimens

8.1 Select material for testing in accordance with standard methods of sampling applicable to the material under test. Sampling may be done in accordance with Practice D1898 or by 8.2 below.

8.2 Selection samples considered representative of the material to be tested.

8.2.1 *Primary Sampling Unit*—Consider rolls, bolts, or pieces of the flexible barrier material or nonwoven fabric to be the primary sampling unit, as applicable.

8.2.2 *Laboratory Sampling Unit*—As a laboratory sampling unit, take from the primary sampling unit at least a one full-width piece that is 1 m (1 yd) in length along the (machine direction, after first removing a 1 m (1 yd) length.

8.2.2.1 For primary sampling units less than 1 m (1 yd) in length, use a sufficient number of pieces to prepare the six specimens to the size described in 8.2.3.

8.2.3 *Test Specimen Size*—From each laboratory-sampling unit, cut at least six test specimens using the template supplied with the apparatus or a similar die cutter. The truncated pie shaped template will produce proper size specimens that cover the sample cell.

8.2.4 *Test Specimen Selection*—Select test specimens as follows:

8.2.4.1 Cut specimens representing a broad distribution diagonally across the width of the laboratory-sampling unit.

8.2.4.2 For fabric widths 125 mm (5 in.) or more take no specimen closer than 25 mm (1 in.) from the selvage edge.

8.2.4.3 For fabric widths less than 125 mm (5 in.), use the entire width for specimens.

8.2.4.4 Ensure specimens are free of folds, creases, or wrinkles. Avoid getting oil, water, grease, etc., on the specimens when handling.

9. Conditioning

9.1 No pre-conditioning is necessary before starting a test.

9.2 Any conditioning of the specimens to the water vapor driving force (differential relative humidity) and temperature is carried out during the test within the test apparatus. In general, these materials are high transmitters and the specimens do not require a significant conditioning period; they reach equilibrium in just a few examination periods. (Experience has shown that individual test periods range from 2 to 10 minutes.) The time required for sample conditioning varies as a function of many factors such as barrier composition, thickness, test temperature, etc.

10. Preparation of Test Apparatus and Calibration Pre-Test Sample Considerations

10.1 *Preparation of Apparatus* (Fig. 1):

10.1.1 If preceding tests have exposed the apparatus to high moisture levels, outgas the system to desorb residual moisture. Purge the system with dry air for a period of 3 to 4 hours.

10.2 *Calibrating the System*—Determine the transmission rate of the system including the air gap and the guard film.

CalC is an acronym for the transmission rate of the apparatus hardware, air gap, and guard film without any test specimens present.

10.2.1 Fill the reservoir with HPLC water.

10.2.2 Place a blank six position specimen holder in the system and tighten the clamp.

10.2.3 Adjust the gas flow to each cell for uniform RH reading for all cells.

10.2.4 Set all cells to CalC.

10.2.5 The computer will automatically determine the empty cell transmission rate (CalC) value for each cell.

11. Test Procedure

11.1 Handle the test specimens carefully to avoid altering the state of the material.

11.2 If permeability or permeance are to be calculated after the test, measure specimen thickness at four equally spaced points within the test area and at the center in accordance with guidelines described in Test Method D5729.

11.3 Mount the specimens to the holder noting the material identity in each location. If testing a laminated material, mount the better barrier portion toward the carrier gas side of the test cell and the poorer barrier toward the guard film.

11.4 Because of the type of material that is used for the guard film, grease should not be used on either the lower cell sealing surface or the upper cell O-ring.

11.5 Align the holder over the pins in the bottom portion of the cells of the apparatus, place the upper portion of the cells on the base of the apparatus, and then tighten the clamp.

11.6 Put the specimens into the test mode via the computer keyboard. Enter the global test parameters and individual cell parameters. Place each cell into TEST.

11.6.1 *Conditioning the Specimen*—During the setting of test parameters in the apparatus it is necessary to select to condition or not condition. Experience has shown that most materials will condition in 10 to 20 minutes. Operators often choose to not condition but go directly to test and run an extra cycle through the cells.

11.6.2 *Establish Equilibrium Rate*—After the system has cycled through the cells a few times, the measurements indicate that an equilibrium transmission rate has been reached. This can be determined by manual examination of data or the system can be made to stop further testing of each cell by a setup in the computer. When successive values for any cell are within 1 %, testing will cease; this is known as convergence mode. In most cases, two to ten test cycles per cell are sufficient. Low permeability barriers may require more cycles to come to equilibrium.

NOTE 1—When testing materials for which the operator has no previous history, additional time must be allowed to assure that true equilibrium has been reached and it is best to not use the convergence mode.

11.6.2.1 Note also that the permeation system will require some time to stabilize with materials having low transmission rates after it has been used to test materials with high transmission rates. For this reason it is desirable when testing a number of different samples, sequentially, that materials having similar permeability characteristics should be tested together, or alternatively, the testing time should be extended to insure that the apparatus reaches an equilibrium value consistent with the conditioning state of the specimens. If unfamiliar with the material being tested, investigate the effect of examination time. The preceding precaution is usually not necessary when going from low to higher transmitting specimens.

11.6.3 *System Report*—The system, will indicate DONE when all cells have reached equilibrium (when in convergence mode) and the instrument stops testing. A printed record of the test should be obtained before continuing to test additional barriers.

11.7 *Standby and Shutoff Procedures:*

11.7.1 At the conclusion of a test, but in anticipation of further testing, place the instrument in standby.

11.7.2 When the system is not to be used for an extended period, dry the system, then turn off the electrical power.

12. Calculation

12.1 The test apparatus automatically carries out the calculations given in 12.1.1 and 12.1.2.

12.1.1 The calculation of WVTR values uses the formula:

$$WVTR = \frac{F \cdot \rho sat(T) \cdot RH}{A \cdot (1 - RH)} \text{ in terms of } g/(m^2 \cdot day)$$

where:

F = the flow of carrier gas in terms of cm^3/min,
ρsat(T) = the density of water in saturated air at the analysis temperature, T in terms of g/cm^3,
RH = the relative humidity of the carrier gas sweeping from the test specimen, and
A = exposure area of the sample mounted in the test cell, in cm^2.

NOTE 2—By having the (1 – RH) in the denominator, the above WVTR calculation normalizes the water vapor flux to 100 %RH to be used within the calculations in 12.1.2.

12.1.2 The reported transmission rate is the result of the two measurements described in 4.1 and is calculated according to the equation:

$$(TR_{\text{TEST BARRIER}})^{-1} = (TR_{\text{TEST BARRIER, GUARD FILM, AIR GAP}})^{-1} - (TR_{\text{GUARD FILM, AIR GAP}})^{-1}$$

where:

$TR_{\text{TEST BARRIER}}$ = the transmission rate of the test barrier, in g/(m^2·day),