Water vapor transmission rate through plastic film and sheeting using a modulated infrared sensor

1. Scope

1.1 This test method covers a procedure for determining the rate of water vapor transmission through flexible barrier materials. The method is applicable to sheets and films up to 2.54 mm (0.1 in.) in thickness, consisting of single or multilayer synthetic or natural polymers and foils, including coated materials. It provides for the determination of (1) water vapor transmission rate (WVTR), (2) the permeance of the film to water vapor and (3), for homogeneous materials, water vapor permeability coefficient.

NOTE 1: Values for water vapor permeance and water vapor permeability must be used with caution. The inverse relationship of WVTR to thickness and the direct relationship of WVTR to the partial pressure differential of water vapor may not always apply.

1.2 This method does not purport to address the safety problems associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable documents


3. Terminology

3.1 Water vapor permeability coefficient, The product of the permeance and the thickness of the film. The permeability is meaningful only for homogeneous materials, in which case it is a property characteristic of bulk material.

NOTE 2: This quantity should not be used unless the relationship between thickness and permeance has been verified in tests using several thicknesses of the material. An accepted unit of permeability is the metric perm centimeter, or 1 g/m²·d·mmHg·cm of thickness. The SI unit is the mol/m²·s·Pa·mm. The test environment (relative humidity gradient across the film and temperature) must be stated.

3.2 Water vapor permeance, The ratio of a barrier’s WVTR to the vapor pressure difference between the two surfaces.

NOTE 3: An accepted unit of permeance is the metric perm, or 1 g/m²·d·mmHg. The SI unit is the mol/m²·s·Pa. Since the permeance of a specimen is generally a function of relative humidity and temperature, those test conditions must be stated.
3.3 Water vapor transmission rate (WVTR), The time rate of water vapor flow normal to the surfaces, under steady-state conditions, per unit area.

NOTE 4: An accepted unit of WVTR is g/m²·d. The test conditions (see Note 2) must be stated.

4. Summary

A dry chamber is separated from a wet chamber of known temperature and humidity by the barrier material to be tested. The dry chamber and the wet chamber make up a diffusion cell in which the test film is sealed. The diffusion cell is placed in a test station where the dry chamber and the top of the film are swept with desiccated air or desiccated nitrogen. Water vapor diffusing through the film mixes with the air and is carried into a pressure-modulated infrared sensor. This sensor measures the fraction of infrared energy absorbed by the water vapor and produces an electrical signal, the amplitude of which is proportional to water vapor concentration. The amplitude of the electrical signal produced by the test film is then compared to the signal produced by measurement of a calibration film of known transmission rate. This information is then used to calculate the WVTR.

5. Significance and use

5.1 The purpose of this test is to obtain reliable values for the WVTR of barrier materials.
5.2 WVTR is an important property of packaging materials and can be directly related to shelf life and packaged product stability.
5.3 Data from this method are suitable as a referee method of testing, provided that the purchaser and seller have agreed on sampling procedures, standardization procedures, test conditions, and acceptance criteria.

6. Apparatus

6.1 This method utilizes water vapor transmission apparatus (Fig. 2) comprised of the following:
6.1.1 Diffusion cell, an assembly consisting of two metal halves which, when closed upon the test specimen, will accurately define a circular area. A typical acceptable diffusion cell area is 50 cm². 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 buckle will contact the top or bottom of the cell. A depth of approximately 6 mm (.25 in.) has been found to be satisfactory for 50-cm² cells.
6.1.1.1 Diffusion cell O-ring, An appropriately sized groove machined into the humid chamber side of the diffusion cell retains a neoprene O-ring. The test area is considered to be the area established by the inside contact diameter of the compressed O-ring when the diffusion cell is clamped shut against the test specimen.
6.1.1.2 Diffusion 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 Diffusion cell air passages, two holes in the dry half of the diffusion cell. These shall incorporate O-rings suitable for sealing the diffusion cell to the test chamber pneumatic fittings for the introduction and exhaust of air without significant loss or leakage.

NOTE 5: Use of multiple diffusion cells: Experience has shown that arrangements using multiple diffusion cells are a practical way to increase the number of measurements which can be obtained in a given time. A separate conditioning rack (Fig. 2) contains a manifold which connects the dry-chamber side of each individual diffusion cell to a dry-air source. Dry air is continually purging the dry chamber of those cells that are connected to the conditioning rack while the humid chamber side is held at a specific relative humidity by distilled water or a saturated-salt solution. It is desirable to thermostatically control the temperature of the conditioning rack as described in 6.1.3.

6.1.2 Test chamber, a cavity into which the diffusion cell is inserted. The test chamber shall incorporate means for clamping the diffusion cell in accurate registration with pneumatic system operating to the dry-air source and the infrared detector. The chamber shall also provide a thermometer well for the measurement of temperature.

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1 Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the bound set of TAPPI Test Methods, or may be available from the Information Resources Center.
6.1.3 **Test station temperature control.** It is desirable to thermostatically control the temperature of the test station. A simple resistive heater attached to the station in such a manner as to ensure good thermal contact is adequate for this purpose. A thermistor sensor and an appropriate control circuit will serve to regulate the temperature unless measurements are being made close to ambient temperature. In that case it may be necessary to provide cooling coils to remove some of the heat.

6.1.4 **Flowmeter.** A means for regulating the flow of dry air within an operating range of 5 to 100 cc/min is required.

6.1.5 **Flow switching valves,** for the switching of dry-air flow streams of the water vapor transmission apparatus and the conditioning rack.

6.1.6 **Infrared sensor,** a water vapor detector capable of sensing 1µg/L of water, or, in other terms, 1 ppm by volume, or 0.002% relative humidity at 37.8°C.

6.1.7 **Recording device,** a multi-range strip chart recorder or other appropriate instrument for measuring the voltage developed by the signal amplifier.

6.1.8 **Test cell desiccant drying system,** shall be capable of reducing the concentration of water vapor from the test cell air source down to less than 0.5 ppm by volume or 0.001% relative humidity at 37.8°C.

6.1.9 **Conditioning rack desiccant drying system,** capable of reducing the concentration of water vapor flowing into the diffusion cells in the conditioning rack to less than 0.5 ppm by volume or 0.001% relative humidity at 37.8°C.

6.1.10 **Flow-metering valve,** a fine-metering valve capable of controlling the dry-air flow rate to the test cell when the apparatus is in the "measure" mode of operation.

### 7. Reagents and materials

7.1 **Desiccant,** for drying air stream.

7.2 **Absorbent pads (not critical),** such as filter pads of 30-75 mm dia.

7.3 **Distilled water,** for producing 100% relative humidity, or various saturated salt solutions to produce other relative humidities as described in Practice E 104.

**NOTE 6:** Fiber based barriers (wax or PVDC coated paper) require a 90% solution or lower to prevent fiber swelling that ruins barrier.

7.4 **Reference film,** known WVTR material for system calibration.

7.5 **Sealing grease,** a high-viscosity, silicone stopcock grease or other suitable high-vacuum grease is required for lubrication of O-rings and to seal the specimen film in the diffusion cell.

### 8. Sampling

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 D-1898. Select samples considered representative of the material to be tested. If the material is of non-symmetrical construction, the orientation should be noted.

### 9. Pre-test sample conditioning

Mount the reference film and the test samples in their individual diffusion cells and place them in an environment that duplicates the test environment. Condition the reference film and the test samples for a suitable period of time. While samples can be conditioned in the system test chamber, use of a multi-head conditioning rack (Fig. 1) is recommended. The time required for pre-test sample conditioning varies greatly, as a function of many factors such as barrier composition, thickness, test temperature, etc. Note also that the permeation system will require a relatively long 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. If unfamiliar with the material being tested, the operator should investigate the effect of conditioning time. Twenty-four-hour-interval check points are suggested for materials having long equilibration times. The conditioning procedure used should be described in the test report.
10. System calibration with reference film

10.1 Set the operating temperature in the test station to within 1°F (0.5°C) of the desired temperature. Check the filter pads in the diffusion cell and, if necessary, add distilled water (for 100% relative humidity) or a selected saturated salt solution to achieve other desired relative humidities. Practice E 104 suggests salt solutions to be used for a range of relative humidities.

10.2 Zero the recording device. This is accomplished with a shorting bar across the recorder input terminals while the recorder is temporarily disconnected from the permeation system.

10.3 Remove the recorder shorting bar and reconnect the recorder input terminals to the signal output from the permeation system. With the test chamber in BYPASS, the air flow valve in MEASURE, and the flow rate set at approximately 60 mL/min, observe the recorder trace for a period of time until it has stabilized at a constant-voltage level with no discernible drift up or down.

10.4 Using the permeation system “Zero” control (not recorder zero), make adjustments to set the recorder trace at some convenient level above chart zero; (for example, the first major division above chart zero).

10.5 Install the reference film diffusion cell in the test chamber. Using procedures described in Section 11, initiate air flow through the cell.

10.6 Observe the voltage produced by the reference film until it stabilizes at a constant value.

10.7 Calculate the calibration factor, C, of the reference film as follows:
Where:

\[ TR = \text{reference film transmission rate at the test conditions, g/m}^2 \cdot \text{d}, \]

\[ EO = \text{steady-state voltage produced by dry air (see 10.4)}, \]

\[ ER = \text{steady-state voltage produced by vapor transmitted through the reference film (see 10.6)}, \]

\[ C = \text{the reference-film calibration factor, g/volt} \cdot \text{m}^2 \cdot \text{d}. \]

Alternatively, a microprocessor or computer-based system may be used to calculate the calibration factor and equilibrium transmission rates.

11. Test procedure

11.1 Preparation of apparatus (Fig. 2), If preceding tests have exposed the apparatus to high moisture levels, outgas the system to desorb residual moisture. Clamp an empty diffusion cell in the test chamber and purge the system with dry air for a period of 3 to 4 h.

11.2 Number of specimens tested, test enough specimens to obtain the desired result, but never less than three per sample.

11.3 Preparation of test samples:

11.3.1 Cut the test specimen to approximately 10 cm by 10 cm (4 in. by 4 in.).

11.3.2 Measure specimen thickness at four or more equally spaced points within the test area and at the center after the WVTR testing has been completed, in accordance with guidelines described in Test Method D 374.

11.3.3 Lightly grease the upper cell sealing surface and the lower cell O-ring.

11.3.4 Insert one to three absorbent pads into the lower half-cell and saturate with one to three cc's of distilled water or a desired salt solution.

11.3.5 Align the test film against the greased upper sealing surface and then clamp both halves together using the locating pins (Fig. 3).

Fig. 3. Film diffusion cell.

11.3.6 Clamp the assembled cell in the conditioning rack. Allow to condition as described in Section 9.

11.4 Insert the specimen, Place valve V1 (Fig. 1) in the BYPASS position to avoid swamping the system with moist room air. Unclamp and remove the test sample diffusion cell from the conditioning rack; insert and clamp it into the test chamber.

11.5 Purge the sample, Place valve V2 (Fig. 1) in the PURGE position and valve V1 in the INSERT position. Purge the sample for 3 to 6 minutes to remove moist air admitted into the system when inserting the specimen.

11.6 Establish equilibrium rate, After the system has been flushed with dry air, move the valve V2 to the MEASURE position. The dry air now passes through the diffusion cell into the sensor at the rate determined by valve V3 and as displayed by the flowmeter. Do not make any adjustments of valve V3 from the setting determined at calibration (See 10.3). Observe the recorder trace until the sensor output voltage stabilizes at a constant value with no significant trend in either direction. Record the observed final reading in terms of volts or millivolts.
NOTE 7: When testing materials for which the operator has no previous history, additional time must be allowed to assure that true equilibrium has been reached. When in doubt, retest after an additional conditioning interval of several hours.

11.7 Record the temperature of each test with reference to a thermometer or thermocouple installed in the test chamber thermometer well. Temperature is a critical parameter affecting the measurement of WVTR. During testing, monitor the temperature, periodically, to the nearest 0.5°C. Report the average temperature and the range of temperatures observed during the test.

11.8 Standby and shut-off procedures.
11.8.1 At the conclusion of a test, but in anticipation of further testing, place the instrument in standby as follows:
11.8.1.1 Insert and clamp a standard test film diffusion cell into the test station and then turn V1 (Fig. 1) to INSERT.
11.8.1.2 When the system is not to be used for an extended period and there are no films that require conditioning, the electrical power may be turned off.

12. Calculations

12.1 \( WVTR \), Calculate water vapor transmission rate using the formula:

\[
WVTR = C \times (ES - EO)
\]

Where:
- \( C \) = a calibration factor expressing rate as a function of voltage (or mV). The value of \( C \) is derived from tests of a known reference film (Section 10).
- \( EO \) = system zero level voltage (10.4), and
- \( ES \) = equilibrium voltage obtained with the test specimen (Section 11.6).

12.2 \( Permeance \), Calculate sample permeance (if required) using the following relationship:

\[
\text{Metric Perms} = \frac{WVTR}{P_w} = \frac{g/m^2 \cdot d \cdot mm Hg}{P_w}
\]

Where:
- \( WVTR \) = specimen water vapor transmission rate, g/m²·d,
- \( P_w \) = water vapor partial pressure gradient across the test specimen, mm Hg

12.3 \( Permeability coefficient \), Calculate the water vapor permeability coefficient (if required) using the following relationship:

Permeability = metric perms \( \times t \)

Where:
- \( t \) = the average thickness of the specimen, cm. Note that permeability calculations are meaningful only in cases where materials have been determined to be homogeneous.

13. Report

13.1 Report the following information:
13.1.1 A description of the test specimen. If the material is non-symmetrical (two sides different), include a statement as to which side was facing the high humidity.
13.1.2 The humidity environment on each side of the test film and means by which it was obtained.
13.1.3 The test temperature (to nearest 0.5°C).
13.1.4 The values of WVTR and, if desired, values of permeance and permeability. These entries should be rounded-off to three significant figures or less, as may be consistent with the operator's estimate of precision or bias.
13.1.5 A statement of the means used to obtain the calibration factor.
13.1.6 The effective area exposed to permeation and a description of how it was defined.
13.1.7 The time to reach steady-state after introduction of the diffusion cell into the test chamber, and
13.1.8 A description of the conditioning procedure.

14. Precision and bias

14.1 Precision.
14.1.1 Four different film materials cut and distributed in accordance with Standard Practice D 4204 were evaluated by 11 laboratories using modulated infrared sensors:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Material</th>
<th>Average WVTR, g/m²/d</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Saran (1-mil)</td>
<td>0.75</td>
</tr>
<tr>
<td>B</td>
<td>Polyester (3-mil)</td>
<td>7.69</td>
</tr>
<tr>
<td>C</td>
<td>Polyester (1-mil)</td>
<td>24.0</td>
</tr>
<tr>
<td>D</td>
<td>Barex (1-mil)</td>
<td>66.2</td>
</tr>
</tbody>
</table>

14.1.2 The tests utilized two known-value reference films for system calibration. These were cut from master rolls and distributed to round robin participants with the test samples.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Material</th>
<th>Average WVTR, g/m²/d</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>Polyester (5-mil)</td>
<td>4.4</td>
</tr>
<tr>
<td>Y</td>
<td>Polyester (1-mil)</td>
<td>23.3</td>
</tr>
</tbody>
</table>

14.1.3 In all cases, values for samples A and B were derived from comparisons with reference film X; values for C and D were obtained using reference film Y. The measurement precision is indicated as follows:

<table>
<thead>
<tr>
<th>Sample tested</th>
<th>Within lab precision</th>
<th>Between lab precision</th>
<th>% Error within lab</th>
<th>% Error between lab</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.071</td>
<td>0.137</td>
<td>9.5</td>
<td>18.3</td>
</tr>
<tr>
<td>B</td>
<td>0.234</td>
<td>0.365</td>
<td>3.0</td>
<td>4.8</td>
</tr>
<tr>
<td>C</td>
<td>0.478</td>
<td>0.823</td>
<td>2.0</td>
<td>3.4</td>
</tr>
<tr>
<td>D</td>
<td>4.066</td>
<td>8.430</td>
<td>6.1</td>
<td>12.7</td>
</tr>
</tbody>
</table>

14.1.4 It will be noted that the coefficient of variation (the % error) is proportional to the proximity of the measured value of the calibration standard. Increased precision can therefore be achieved by use of a calibration (reference) standard as close to the measurement value as possible.

14.2 Bias, measured values are derived from comparisons with known-value reference films. Method accuracy is dependent upon the validity of values assigned to these reference films.

15. Additional information

Effective date of issue: March 14, 1995.

Appendix (nonmandatory information)

A1.1 Normal procedures as described for the modulated infrared permeation system are considered suitable for testing barrier materials having rates up to 100g/m²/d. Above this level, a different approach may be required in order to keep the sensor output within design limits.
A1.2 In general, the testing of a “high transmitter” requires that means be employed to reduce the concentration of water vapor in the sensor. This may be accomplished in two ways:

A1.2.1 By increasing the flow of dry air, or
A1.2.2 By reducing the area of the test film.
A1.2.3 By reducing the humidity (changing) the saturated salt solution that is placed in the lower half of the diffusion cell.

A1.3 The flow rate can be increased by opening the flow-rate metering valve (V3) to its maximum (the flowmeter will be off-scale). The calibration film and the test film are then run at this rate.

A1.4 Alternatively, apply foil masks with die-cut apertures. These may be applied to both sides of a barrier to reduce the test area.

A1.5 Each of these methods, when used alone or in combination, serve to reduce the vapor concentration of the air stream.

NOTE 8: The precision and bias of results obtained with reduced-area masked samples have not been established.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Divisions Administrator.