INTERNATIONAL

Designation:F1249–90(Reapproved 1995) Designation: F 1249 – 06

Standard Test Method for Water Vapor Transmission Rate Through Plastic Film and Sheeting Using a Modulated Infrared Sensor[1]

This standard is issued under the fixed designation F 1249; 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.

Note—Paragraph 13.1.1 was editorially corrected and the year date was changed on June 22, 2006.

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 3 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 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]

D 374 Test Methods for Thickness of Solid Electrical Insulation

D 1898 Practice for Sampling of PlasticsD4204Practice for Preparing Plastic Film Specimens for a Round-Robin Study [3]

E 96/E 96M Test Methods for Water Vapor Transmission of Materials

E 104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions

E 178 Practice for Dealing With Outlying Observations

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *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.

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 thicknesses of the material. An accepted unit of permeability is the metric perm centimeter, or 1 g/m^2 per day per mm Hg·cm of thickness. The SI unit is the $mol/m^2 \cdot s \cdot Pa \cdot mm$. The test conditions (see 3.1) must be stated.

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

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

3.1.3 *water vapor transmission rate (WVTR)*—the time rate of water vapor flow normal to the surfaces, under steady-state conditions, per unit area.

3.1.3.1 *Discussion*—An accepted unit of WVTR is g/m^2 per day. The test conditions of relative humidity and temperature where the humidity is the difference in relative humidity across the specimens, must be stated.

[1] This test method is under the jurisdiction of ASTM Committee F-2 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods. Current edition approved July 27, 1990. Published September 1990. Originally published as F1249–89. Last previous edition F1249–89.F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.10 on Permeation.

Current edition approved June 22, 2006. Published June 2006. Originally approved in 1989. Last previous edition approved in 2005 as F 1249 – 05.

[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* , Vol 10.01.volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

4. Summary of Test Method

4.1 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 dry air.~~ Water vapor diffusing through the film mixes with the gas in the dry chamber and is carried ~~into~~to 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 water vapor transmission rate. This information is then used to calculate the rate at which moisture is transmitted through the material being tested.

5. Significance and Use

5.1 The purpose of this test method is to obtain reliable values for the WVTR of ~~barrier materials.~~ plastic film and sheeting.

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 test method is 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[4] (Fig. 1) comprised of the following:

6.1.1 *Diffusion Cell*~~, an~~— 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 cm^2. 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~~that happens to sag or buckle will contact the top or bottom of the cell. A depth of approximately 6 mm (0.250 in.) has been found to be satisfactory for 50-cm^2 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~~—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.~~ —Two holes in the dry half of the diffusion cell. This is necessary only in the earlier model WVTR instruments that have a separate conditioning rack and testing chamber. 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.

~~[4] Annual Book of ASTM Standards, Vol 08.02.~~

[4] The sole source of supply of the apparatus known to the committee at this time is Mocon/Modern Controls, Inc., 7500 Boone Avenue North, Minneapolis, MN 55428. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

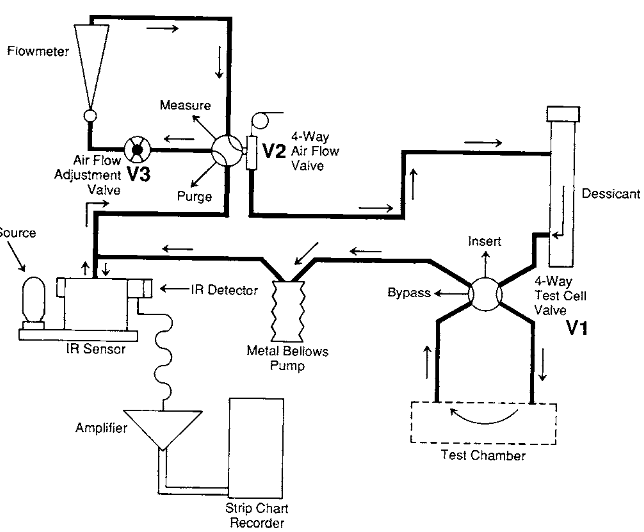

FIG. 1 Measuring System

NOTE 2—*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~~that can be obtained in a given time. A separate conditioning rack (Fig. 2) may be used that 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 openings to the dry-air source and the infrared detector. The chamber shall also provide a thermometer well for the measurement of temperature.~~ — A cavity into which the diffusion cell is inserted. Again, this is necessary only in the earlier model WVTR instruments that have a separate conditioning rack and testing chamber. The test chamber shall incorporate means for clamping the diffusion cell in accurate registration with pneumatic system openings to the dry-air source and the infrared detector. The chamber shall also provide a thermometer well for the measurement of temperature.

6.1.3 ~~*Test Station*~~*Diffusion Cell Temperature Control*—It is desirable to thermostatically control the temperature of the ~~test station.~~diffusion cell to within ±1°F. 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.~~ , for the switching of dry-gas flow streams of the water vapor transmission apparatus.

6.1.6 *Infrared Sensor*~~, a~~— A water vapor detector capable of sensing 1 µg/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~~— A multirange 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.~~ Desiccant Drying System, shall be capable of reducing the concentration of water vapor in the gas source down to less than 0.5 ppm by volume or 0.001 % relative humidity at 37.8°C. In earlier model WVTR equipment, a separate desiccant drying system is needed for the conditioning rack and test chamber.

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 .001% relative humidity at 37.8°C.~~

~~6.1.10~~*Flow-Metering Valve*~~, a~~ —A fine-metering valve capable of controlling the ~~dry-air~~dry-gas 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.~~ Desiccant,[4] [,5] for drying gas stream.

7.2 *Absorbent Pads* ~~(not critical), such as filter pads of 30 to 75 mm in diameter.~~ (not critical), such as filter pads of 30 to 75 mm in diameter. Necessary only in earlier model WVTR equipment that utilizes distilled water or saturated salt solutions to generate the desired relative humidity.

~~[5] Annual Book of ASTM Standards, Vol 04.06.~~

[5] Linde Molecular Sieve, Type 4A or Type 5A, in the form of ⅛ in. pellets as may be obtained from the Union Carbide Co., Linde Division, Danbury, CT 06817-0001.

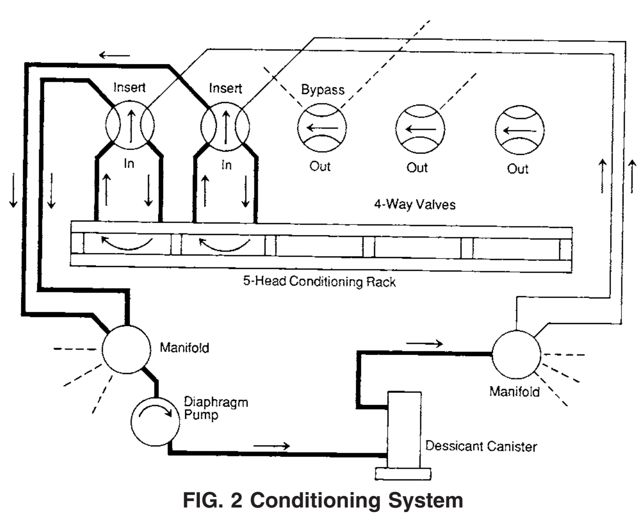

FIG. 2 Conditioning System

7.3 *Distilled Water*, for producing 100% relative humidity, or various saturated salt solutions to produce other relative humidities as described in Practice E104. , for producing 100 % relative humidity, or various saturated salt solutions to produce other relative humidities as described in Practice E 104. Newer WVTR equipment does not require saturated salt solutions. Refer to the manufacturer's instructions for generating relative humidity.

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.

7.6 *Nitrogen Gas*, shall be dry and contain not less than 99.5 % nitrogen. Needed only with certain WVTR instruments.

8. Sampling

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 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. System Calibration With Reference Film

9.1Set 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 E104 suggests salt solutions to be used for a range of relative humidities.

9.2Zero 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.

9.3Remove 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.

9.4Using 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.)

9.5Install the reference film diffusion cell in the test chamber. Using procedures described in Section 11, initiate air flow through the cell.

9.6Observe the voltage produced by the reference film until it stabilizes at a constant value.

9.7Calculate the calibration factor, *C*, of the reference film as follows:

$$C = \frac{TR}{ER - EO}$$

where:

TR = reference film transmission rate at the test conditions g/m^2 per day,
EO = steady-state voltage produced by dry air (see 9.4),
ER = steady-state voltage produced by vapor transmitted through the reference film (see 9.6), and
C = the reference-film calibration factor, g/volt·m^2 per day.

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

9.1 Follow the manufacturer's instructions for calibrating the WVTR instrument with a reference film.

10. Pre-Test Sample Conditioning

10.1Mount 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. 2) 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.

11.Test Procedure

11.1

10.1 *Preparation of Apparatus (*Fig. 1*)*—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—*If preceding tests have exposed the apparatus to high moisture levels, outgas the system to desorb residual moisture.*