



Standard Test Method for Water Vapor Transmission Rate of Flexible Barrier Materials Using an Infrared Detection Technique¹

This standard is issued under the fixed designation F 372; 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 rapid procedure for determining the rate of water vapor transmission of flexible barrier materials in film or sheet form. This test method is applicable to sheets and films up to 3 mm in thickness, consisting of single-layer or multilayer synthetic or natural polymers and metal foils including coated materials.

1.2 *This standard does not purport to address all of the safety problems, 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 Plastics

E 96 Test Methods for Water Vapor Transmission of Materials

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

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 transmission rate (WVTR)* (of a body between two specified parallel surfaces)—the time rate of water vapor flow normal to the surfaces, under steady conditions, through unit area, under the conditions of test (for example, temperature and relative humidity). An accepted unit of WVTR is $\text{g/day}\cdot\text{m}^2$. The test conditions must be stated.

3.1.2 *water vapor permeance* (of a body between two specified parallel surfaces)—the ratio of its WVTR to the vapor pressure difference between the two surfaces. An accepted unit of permeance is a metric perm, or $1 \text{ g/day}\cdot\text{m}^2\cdot\text{mm Hg}$. Since the permeance of a specimen is generally a function of relative humidity and temperature, the test conditions must be stated.

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 time for a given increase in water vapor concentration of the dry chamber is measured by monitoring the differential between two bands in the infrared spectral region, one in which water molecules absorb and the other where they do not. This information is then used to calculate the water vapor movement through a known area of barrier material.

5. Significance and Use

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

5.2 The WVTR is an important property of packaging materials, which can be related to shelf life and product stability.

5.3 In any application, the WVTR of a barrier material is determined by its permeance under the given conditions. In most cases, the WVTR will increase when the absolute humidity is increased on the wet side of the barrier and when the temperature of the environment increases, particularly if a polymeric material is the primary water vapor barrier in the construction.

5.4 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 the water vapor are not always linear.

6. Apparatus

6.1 *Water Vapor Transmission Apparatus*,³ (Fig. 1) with the following:

¹ This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods.

Current edition approved May 10, 1999. Published July 1999. Originally published as F 372 – 73. Last previous edition F 372 – 94.

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 Modern Controls, Inc. no longer supplies the IRD-2, but some are still in use.

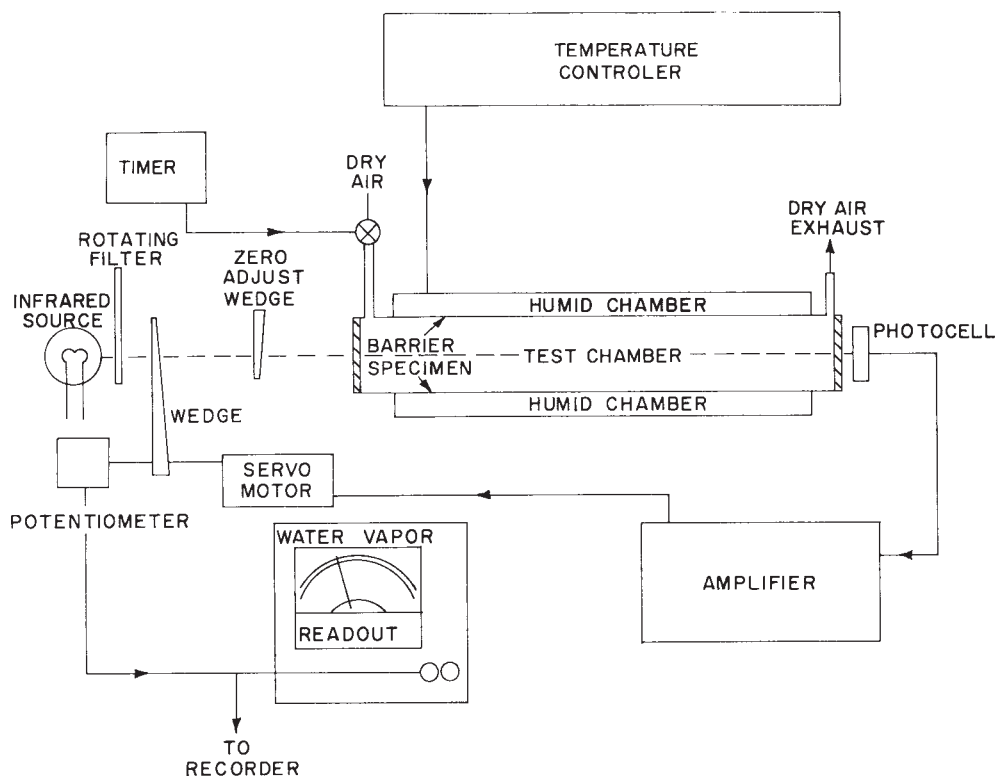


FIG. 1 Functional Diagram of Diffusometer

6.1.1 *Temperature Control*, capable of maintaining humid chambers and test material at $37.8 \pm 0.1^\circ\text{C}$ ($100 \pm 0.2^\circ\text{F}$).

6.1.2 *Infrared Detection System*, capable of detecting changes of $1 \mu\text{g}$ of water vapor per litre or in other terms 1 ppm by volume or 0.002 % relative humidity at 37.8°C .

6.2 *Micrometer*, graduated to 0.0025 mm (0.10 mil) or better, and accurate to 0.00125 mm (0.05 mil) to measure specimen thickness.

6.3 *Analytical Balance*, accurate to 0.0001 g.

6.4 *Clock or Watch*.

6.5 *Schwartz U-Shaped Drying Tube*.

6.6 *Calcium Chloride Drying Tube*.

6.7 *Sponges*.

7. Reagent and Materials

7.1 *Desiccant for Drying Air Stream*.⁴

7.2 *Desiccant for Drying Tubes*.⁵

7.3 *Distilled Water*, for controlling humidity at 100 % relative humidity at 37.8°C .

7.4 *Zinc Sulfate Solution*, saturated at 37.8°C .

7.5 *Ammonium Sulfate Solution*, saturated at 37.8°C .

7.6 *Sealing Grease*.

8. Sampling

8.1 Practice D 1898 provides guidance in developing sampling procedures.

8.2 The material shall be sampled in accordance with standard methods of sampling applicable to the material under test. The sample shall be selected as representative of the material to be tested, and shall be of uniform thickness. If the material is of nonsymmetrical construction, the two faces shall be designated by distinguishing marks (for example, on a one-side-coated sample, "I" for the coated side and "II" for the uncoated side).

9. Test Specimens

9.1 Test specimens shall be representative of the sample. Test at least three specimens by the same method. If the surfaces of the sample differ, ensure that they are positioned properly relative to the wet and test chambers. Take care not to contaminate the test area of the specimen.

9.2 Determine the average thickness from measurements taken to the nearest 0.0025 mm (0.10 mil) on the surface area interfacing the humid chamber(s) and the test chamber. The number of measurements will depend upon the size of the total interfacing area.

10. Procedure for Gravimetric Calibration of Instrument and Preparation of Standard Film

10.1 Set the operating temperature in the humid chambers within 1°F (0.5°C) of the desired temperature. Check the sponges and dampen if necessary with distilled water for 100 % relative humidity, saturated zinc sulfate for 90 % relative humidity, or saturated ammonium sulfate for 81 % relative humidity.

10.2 Purge the test chamber with dry air and then measure the time for a given increase in water vapor concentration as

⁴ Linde Molecular Sieve, Type 4A, 1/8-in. (3.2-mm) pellets from Union Carbide Corp., Linde Div., Molecular Sieve Products, 120 Riverside Plaza, Chicago, IL 60605, have been found suitable.

⁵ Anhydron, available from J. T. Baker Chemical Co., Phillipsburg, NJ, has been found satisfactory.

the water transmits from the humid chamber(s) through the film into the test chamber.

10.3 Repeat 10.2 until time measured remains constant.

10.4 Attach a charged and weighed Schwartz drying tube to the air exit tube of the test chamber. Follow it with a charged calcium chloride tube to prevent absorption of water vapor from the laboratory atmosphere.

10.5 Record the time of day (t_1) and allow the unit to either cycle or continually purge until sufficient water is absorbed in the Schwartz tube to obtain a reliable weight, 100 mg or more.

10.6 Record the time of day upon completion (t_2), immediately close the stopcocks, remove the drying tubes and weigh the Schwartz tube. The increase in weight (W) is due to the water vapor absorbed in the time (t) elapsed between t_1 and t_2 . Calculate the weight of water vapor absorbed per hour (W_t).

10.7 Insert a “perfect” barrier such as polytetrafluoroethylene-coated aluminum foil into the unit, aluminum to the humid side.

10.8 Repeat 10.4 through 10.6 allowing the test chamber to purge for at least 48 h. Determine the weight of water vapor absorbed (W_A) from the dry air stream. This water vapor originates either from leaks in the system or incomplete drying of the air stream. W_A should not exceed a few tenths of a milligram per hour.

10.9 Calculate the weight of water vapor transmitted by the standard barrier per hour (W_S) by subtracting the water vapor absorbed from the dry air stream per hour (W_A) from the total water vapor absorbed per hour (W_t).

10.10 Calculate the WVTR for the standard as follows:

$$WVTR = 24 W_S/A$$

where:

$WVTR$ = water vapor transmission rate at the temperature and humidity used, $g/m^2 \cdot \text{day}$,

W_S = weight of water vapor transmitted, g/h , and

A = transmitting surface area of the standard, m^2 .

10.11 Repeat several times and average the results. The standard deviation should not be greater than $\pm 5\%$ of the average.

10.12 Calibrate each range of the readout device for the water vapor sensor using the calibrated barrier standard in conjunction with its WVTR as determined in 10.11.

10.12.1 This can be accomplished in a number of ways depending upon the readout device. If the readout device is an ammeter, one of the methods that can be employed is to determine the time for the meter reading to increase a specific increment for each range.

10.12.2 Calculate the weight of water vapor transmitted through the barrier during that time as follows:

$$g = (WVTR) (A) (t)$$

where:

$WVTR$ = WVTR of standard barrier under same temperature and relative humidity condition as determined in 10.11,

A = surface area of sample interfacing test and humid chamber(s).

t = time for instrument to span the increment, and

g = weight of water vapor transmitted in time t .

11. Procedure for Measuring WVTR of Samples

11.1 Control the temperatures and humidity in the humid chambers in accordance with 10.1.

11.2 Place the sample in the instrument and purge a few minutes with dry air.

11.3 Discontinue purging and follow the increase in the water vapor concentration of the test chamber with time. Record, purge, and repeat until time measured remains constant, that is, when the change being recorded for repetitive cycles differs by no more than $\pm 5\%$. Calculate WVTR.

11.3.1 If the readout calibration procedure in accordance with 10.12.1 and 10.12.2 was employed, determine the time t for the readout to span the calibrated increment corresponding to g for that range.

11.3.2 Record, purge, and repeat until time measured remains constant, that is, when t for repetitive cycles differs by no more than $\pm 5\%$.

11.3.3 Determine the WVTR of the sample from the equation in 10.12.2.

11.4 Determine the average thickness in accordance with 9.2.

12. Report

12.1 Report the following information:

12.1.1 Description of the barrier tested including identification of the two sides (if the barrier is homogeneous, so state),

12.1.2 Thickness of the specimen as determined in 11.4,

12.1.3 Temperature of the test and humid chambers,

12.1.4 Relative humidity of the humid chambers, and

12.1.5 WVTR of each specimen referencing which side of the barrier, if heterogeneous, was in contact with the humid chamber.

13. Precision and Bias

13.1 The precision of this test method was estimated from the results of a round robin conducted in the early 1970's. Eleven laboratories participated, six used Test Methods E 96 and five used this test method, MoCon IRD-2 (see Annex). Seven films between 1.0 and 3.6 mils thick were tested, as follows:

No. 1—Single-side polyethylene-coated glassine

No. 2—Two-side saran-coated glassine

No. 3—Nylon/polyethylene-nylon to the humid side of chamber

No. 4—Nylon/polyethylene-polyethylene to the humid side of chamber

No. 5—Polyethylene

No. 6—Polyethylene terephthalate

No. 7—Rubber hydrochloride

NOTE 1—Glassine is a paper material.

13.1.1 The data indicated that this test method and Test Methods E 96 yielded the same results with the new method providing measurements in far less time and with better precision. This study and the data obtained are recorded in

TABLE 1 Practice E 691 – 79 Precision Summary for WVTR by IRD-2 (g/100 in.², 24 h at 100°F and 90 % Relative Humidity)

NOTE 1—The data is taken from ASTM STP 548.

NOTE 2—Material 2 appears to be nonuniform.

Material Number	\bar{X}^A	Sr ^B	SL ^C	SR ^D	Vr ^E	VR ^F	Ir ^G	IR ^H
1	0.928	0.0641	0.1148	0.1315	6.90	12.38	0.181	0.372
2	0.350	0.0504	0.1354	0.1445	14.40	38.69	0.143	0.409
3	0.334	0.0172	0.0380	0.0417	5.15	11.38	0.0487	0.118
4	0.290	0.0170	0.0185	0.0251	5.86	6.37	0.0481	0.0710
5	0.436	0.0137	0.0326	0.0353	3.14	7.48	0.0387	0.0999
6	1.348	0.0498	0.0954	0.1076	3.69	7.08	0.141	0.305
7	0.450	0.0383	0.0175	0.0421	8.51	3.89	0.108	0.119
Averages					6.81	12.47		
Averages omitting Material 2					4.75	6.94		

^A Laboratory averages, $n = 5$.

^B Within-laboratory pooled standard deviation.

^C Between-laboratory standard deviation.

^D Between-laboratory variability estimate of precision.

^E Within-laboratory coefficient of variation.

^F Between-laboratory coefficient of variation.

^G 95 % within-laboratory repeatability interval.

^H 95 % between-laboratory reproducibility interval.

detail in STP 548 (1). These data were treated by the procedures in accordance with⁶ Practice E 691 and presented at a symposium in 1987. This treatment is recorded in STP1039. Table 1 is the currently recommended precision summary for this test method.

13.2 *Bias*—Since Test Methods E 96 were the accepted test methods for measuring WVTR when this test method was approved and is still the test methods that use a fundamental

(gravimetric) approach, bias was determined by comparing data on the same materials from the two test methods. A precision summary for Test Methods E 96 and a comparison of average WVTRs from the two test methods are in STP 1039. (2) They show no significant differences between average rates obtained with the two test methods. Therefore, this test method exhibits no bias with respect to Test Methods E 96.

14. Keywords

14.1 films; infrared detection; measurement; packaging; water vapor transmission rate; WVTR

⁶ The boldface numbers in parentheses refer to the list of references at the end of this test method.

ANNEX

(Mandatory Information)

A1. OPERATING INSTRUCTIONS FOR THE MOCON IRD-2 INFRARED DIFFUSOMETER (Refs 3, 4, 5)

A1.1 Calibration Procedure:

A1.1.1 Open humid chambers and check thermometers for correct operating temperatures, normally 38°C (100°F). If the difference between them is greater than $\pm 0.5^\circ\text{C}$ (1°F), or if either chamber differs by more than 0.5°C from the desired operating temperature, adjust the indicated potentiometers on the amplifier chassis to obtain temperatures within the above ranges. Check the sponges and dampen if necessary with either distilled water for 100 % relative humidity, saturated zinc sulfate for 90 % relative humidity, or saturated ammonium sulfate for 81 % relative humidity.

A1.1.2 Insert barrier sample to be used as a standard and close chambers.

A1.1.3 Turn the dry air switch on and the solenoid valve switch to PURGE. Check the airflow indicator, rotameter, and set between 2 and 5 if necessary. Check the desiccant and replace if necessary.

A1.1.4 Turn the range switch to CAL and adjust vapor penetration meter reading to 5.0 with the CAL control if necessary.

A1.1.5 Turn the range switch to 5 or other range to be checked and adjust the vapor penetration meter to near 1.5 with the balance wedge control.

A1.1.6 Turn the solenoid valve switch to TEST and plug in the printer.

A1.1.7 Check the setpoint markers and adjust to 3.0 and 7.0 if necessary.

A1.1.8 When the cycle time becomes constant, that is, cycle times differ by no more than $\pm 5\%$, attach the charged and weighed Schwartz drying tube to the air-exit tube of the IRD-2. Follow it with the charged calcium chloride tube to prevent absorption of water vapor from the laboratory air by the Schwartz tube, record the time of day (t_1) and the range and cycle time (Ct_R) of the IRD-2.

A1.1.9 Allow the instrument to either cycle or purge until sufficient water is absorbed to obtain a reliable weight, 100 mg or more. A 0.0635 mm (2.5-mil) low-density polyethylene film requires approximately 25 h. The time in hours required to absorb 100 mg of water vapor can be estimated by dividing the expected WVTR in $\text{g/m}^2\cdot\text{day}$ into 224.13.

A1.1.10 At the end of the estimated time interval, record the time of day (t_2) and the IRD-2 range and cycle time (Ct_R), close the stopcocks and remove the drying tubes. Weigh the Schwartz tube. The increase in weight (W) is due to the water vapor absorbed in the time (t) elapsed between t_1 and t_2 . Calculate the total grams of water vapor absorbed per hour (W_T) by dividing the increase in weight (W) of the Schwartz tube in grams by the elapsed time (t) in hours.

A1.1.11 Insert an impenetrable barrier such as polytetrafluoroethylene-coated aluminum foil, aluminum to humid side, in the IRD-2 and purge until the dry chamber is thoroughly equilibrated with the dry air stream. Equilibration is indicated by long and constant cycle times, at least 2000 s on R5. Install the charged and weighed Schwartz tube followed by the calcium chloride tube and purge for 48 h. Determine the weight of water vapor absorbed per hour from the dry air stream (W_A). It is due to incomplete drying of the air stream or leaks, or both, in the system and should be no greater than a few tenths of a milligram per hour.

A1.1.12 Calculate the weight of water vapor transmitted by the standard film per hour (W_S) by subtracting the water vapor absorbed from the dry air stream per hour (W_A) from the total water vapor absorbed per hour (W_T).

A1.1.13 Calculate the WVTR of the standard film as follows:

$$WVTR = 2241.3 \times W_S$$

where:

$WVTR$ = water vapor transmission rate at the temperature and humidity used, $\text{g/m}^2\cdot\text{day}$, and
 2241.3 = constant for converting the sample area from 16.6 in.^2 to m^2 and the time from 1 to 24 h.

A1.1.14 Repeat several times and average the results. The standard deviation should *not* be greater than $\pm 5\%$ of the average.

A1.1.15 Determine IRD-2 cycle times for each range to be used (Ct_R) and calculate a calibration constant for each range as follows:

$$K_R = Ct_R \times WVTR$$

where:

K_R = IRD-2 calibration constant for range R, and
 Ct_R = IRD-2 cycle time for Range R on the standard film, s. It should repeat to within $\pm 5\%$ or 4 s, whichever is larger.

A1.1.16 Prepare a secondary barrier standard that can be used in the event that unusual values are obtained with the primary standard. The employment of the secondary standard will verify whether the discrepancy is due to the instrument or morphological changes in the barrier. The primary standard should be used for routine checking of the IRD-2 (A1.2).

A1.2 Procedure for Checking Instrument Performance:

A1.2.1 Follow the procedure in accordance with A1.1.1.

A1.2.2 Insert the primary standard barrier film and close the chambers.

A1.2.3 Follow A1.1.2 through A1.1.7.

A1.2.4 If the constant cycle times printed are within the established range for the standard film, proceed to measure the samples. Otherwise find and eliminate the cause of difference. It is usually due to either moisture in the dry-air stream or electronic drift.

A1.3 Procedure for Measuring WVTR:

A1.3.1 Make sure the range switch is turned to zero and the solenoid switch to PURGE. Insert the sample to be measured and close the chambers. Allow the instrument to purge for the required time interval determined by past experience, or a few minutes for unknown materials.

A1.3.2 Turn the range switch to the position determined by past experience for the material, or 4 for an unknown material.

A1.3.3 Adjust the vapor penetration meter to near 1.5 with the balance wedge control and turn the solenoid valve switch to TEST.

A1.3.4 Allow the instrument to cycle until constant times, with $\pm 5\%$, are recorded (Ct_R). Change ranges if necessary to increase measurement speed or precision. Cycle times near 40 s are ideal. Some hygroscopic materials equilibrate slowly and yield misleading results if the test is stopped before the sample has thoroughly equilibrated.

A1.3.5 Calculate the WVTR of the sample as follows:

$$WVTR = K_R/Ct_R$$

where:

$WVTR$ = water vapor transmission rate (units are determined by the calibration constant K_R and conditions of operation, usually $\text{g/m}^2\cdot\text{day}$ at 38°C (100°F) at either 100 % or 90 % relative humidity),
 K_R = calibration constant for R used for the sample involved, and
 Ct_R = constant cycle time of the IRD-2 for the sample involved at Range R.

A1.3.6 Remove the sample and obtain the average thickness of the sample from at least five measurements taken across each of the surfaces interfacing the humid chambers and the test chamber.

A1.4 Standby Setting for Overnight or Brief Intervals of Non-Operation:

A1.4.1 Place a good water vapor barrier such as a polytetrafluoroethylene-coated aluminum foil in the instrument.

A1.4.2 Leave the power switch ON, turn the range switch to zero, the dry air switch to ON, and the solenoid switch to PURGE. Alternatively, to save wear on the pump and extend the time periods for drying the sieve, the dry-air switch may be turned to OFF and the solenoid switch to TEST. This, however, will require a 30 to 120-min drying period when reemploying the unit to remove water absorbed from the laboratory atmosphere.

A1.4.3 Unplug the printer.

REFERENCES

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- (2) Pike, L., "Review of Correlation of Infrared Detection Techniques for Water Vapor Transmission Rates with Test Methods E 96 and Some Approaches to Gravimetric Calibration," *ASTM STP 1039*, 1989, p. 134.
- (3) Wood, R. C., "Rapid Evaluation of Moisture Barriers," *Modern Converter*, April 1972.
- (4) Wood, R. C., "Changes in WVTR of Wax Papers at Test Conditions Near 100°F and 90 % RH," *Paper, Film and Foil Converter*, July 1971, p. 29.
- (5) Wood, R. C., "Evaluation of Packaging Materials," Parts I and II *Modern Converter*, September and October 1970.

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