Designation: F3299 − 18 (Reapproved 2023)

Standard Test Method for Water Vapor Transmission Rate Through Plastic Film and Sheeting Using an Electrolytic Detection Sensor (Coulometric P_2O_5 Sensor)[1]

This standard is issued under the fixed designation F3299; 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 through flexible barrier materials. The method is applicable to sheets and films consisting of single or multilayer synthetic or natural polymers and foils, including coated materials. It provides for the determination of water vapor transmission rate (WVTR).

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, 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]

E96/E96M Test Methods for Gravimetric Determination of Water Vapor Transmission Rate of Materials

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

E178 Practice for Dealing With Outlying Observations

E691 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), n*—the time rate of water vapor flow normal to the surfaces, under steady-state conditions, per unit area.

3.1.1.1 *Discussion*—An accepted unit of WVTR is g/m^2 per day ($g/m^2 \cdot 24h$). The test conditions of relative humidity and temperature, where the humidity is the difference in relative humidity across the specimens, 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 dry chamber and the wet chamber make up a test cell in which the test film is sealed. Water vapor permeating through the film mixes with the dry carrier gas in the dry chamber and is carried to an electrolytic sensor. The carrier gas is passed through the sensor where water vapor it holds is absorbed by phosphorous pentoxide (P_2O_5). Direct current, applied to electrodes of the sensor, dissociates the water into hydrogen and oxygen. Two electrons are required to electrolyze each water molecule, and the current in the cell represents the number of molecules dissociated per unit time.

5. Significance and Use

5.1 The purpose of this test method is to obtain reliable values for the WVTR of 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 An example of a suitable apparatus is shown in Fig. 1. It includes a test chamber consisting of two metal halves, an electrolytic cell to determine the amount of water vapor transmitted, a flow meter, a drying device, and switch valves.

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[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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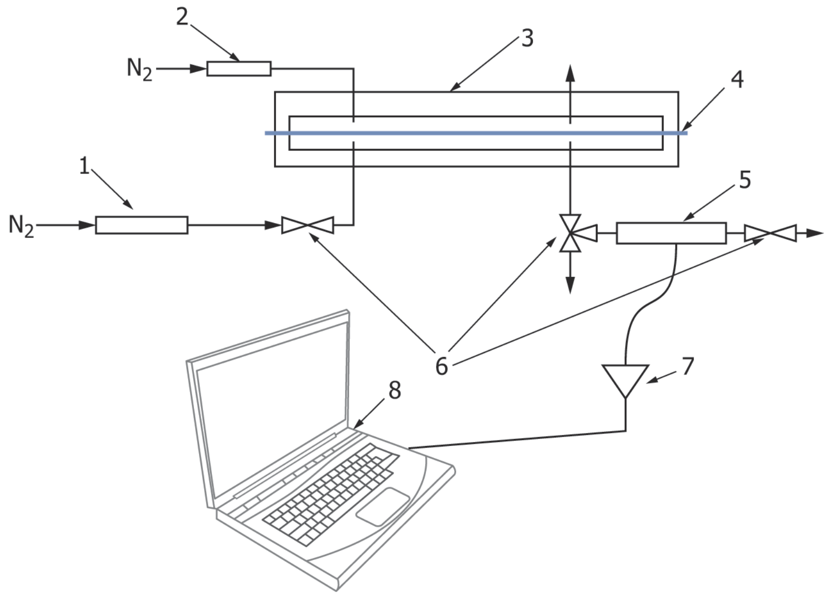
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



Key:
1 – Drying device (containing e.g. a molecular sieve)
2 – RH Generator
3 – Test chamber (temperature controlled)
4 – Specimen
5 – Electrolytic cell and a flow meter
6 – Switch valves
7 – Amplifier
8 – Computer

FIG. 1 Measuring System

6.1.1 *Drying Device,* shall be capable of drying the carrier gas down to the detection limit of the electrolytic cell or lower.

6.1.2 *Relative Humidity (RH) Generator,* should be able to humidify test gas (nitrogen) up to 90 % RH at desired test temperature. Accuracy of measurement of RH should be ±3 % or better.

6.1.3 *Test Chamber,* shall consist of two metal halves (a dry half and a controlled humidity half), which, when closed upon the test specimen, will define a known sample transmission area (typically 50 cm^2). One of the halves (not connected to electrolytic cell) shall incorporate static O-ring in a properly constructed O-ring groove. The second of the halves shall have a flat rim. Since this rim is a critical sealing surface against which the test specimen is pressed, it shall be smooth and flat without radial scratches. It is desirable to thermostatically control the temperature of the test chamber to within ±0.5 °C (±1 °F).

6.1.4 *Flowmeter,* having an operating range from 1 cm^3/min to 100 cm^3/min is required to monitor the flow rate of the nitrogen stream. Sufficiently low flow rates and/or balanced pressures on each side of the film are required to avoid stretching the specimen.

6.1.5 *Electrolytic Cell,* (P_2O_5 moisture sensor) operating at an essentially constant efficiency shall be used to monitor the quantity of water vapor transmitted.

6.1.6 *Flow-Switching Valves,* for the switching of gas flow streams of the water vapor transmission apparatus.

6.1.7 *Amplifier,* and associated electronics, determine electrolytic current, which is proportional to water vapor (moisture) concentration.

6.1.8 *Computer,* used to control apparatus and collect data.

7. Reagents and Materials

7.1 National Institute of Standards and Technology (NIST) traceable calibration gas or reference film (known WVTR material) for system evaluation.

7.2 *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 test chamber.

7.3 *Nitrogen Gas,* shall be dry and contain not less than 99.9 % nitrogen.

7.4 *Distilled Water,* for using in humidity generator.

8. Sampling

8.1 Film samples used for the determination of WVTR shall be representative of materials for which the data are required. Care shall be taken to ensure that samples are representative of conditions across the width and along the length of a roll of film.

8.2 Test specimens shall be representative of the material and shall be free of defects including wrinkles, creases, and pinholes, unless these are characteristics of the material being tested and included in the material description.

8.3 Average thickness shall be determined ±3 µm using a calibrated dial gage (or equivalent) at a minimum of five points distributed over the entire test area. Maximum, minimum, and average values shall be recorded. Sample thickness need not be measured for determination of sample WVTR only.

8.4 If the test specimens of an asymmetrical construction, the two surface shall be marked by appropriate distinguishing marks and the orientation of the test specimen within the permeation apparatus shall be reported.

9. System Evaluation

9.1 The electrolytic sensor used in this method is a coulometric device that is absolute and follows Faraday's Law. A typical sensor utilizes a sensing cell coated with a thin film of phosphorous pentoxide (P_2O_5). Different types of cells with winding or flat electrodes are available. Direct current applied to the electrodes dissociates water, absorbed by the P_2O_5, into hydrogen and oxygen. Two electrons are required to electrolyze each water molecule, and the current in the cell represents the number of molecules dissociated per second. One hundred percent of all water vapor entering this sensor is directly converted to a measurable current. The relationship between measured current and moisture concentration in the electrolytic cell is governed by absolute principles of Faraday's law and no calibration versus a moisture standard is needed.

9.2 Experience has shown, that under some circumstances the sensor may become depleted or damaged. For that reason, this test method incorporates means for a periodic sensor evaluation that should be performed monthly. This evaluation could be derived from measurements of a NIST traceable calibration gas or "reference film" with known value. Follow the manufacturer instructions for system evaluation.

10. Test Procedure

10.1 *Test Conditions:*

10.1.1 Commonly accepted industry test conditions are: 37.8 °C ± 0.5 °C and 90 % ± 3 % RH. Test conditions other than these shall be agreed upon by the interested parties.

10.2 *Preparation of Apparatus:*

10.2.1 If preceding tests have exposed the apparatus to high moisture levels, outgas the system to desorb residual moisture following the manufacturer's instructions.

10.2.2 Measure the WVTR of the film specimen following the manufacturer's instructions.

10.3 *Standby and Shutoff Procedures:*

10.3.1 Follow the manufacturer's instructions for putting the instrument in standby when not being used.

11. Calculation

11.1 Water vapor transmission rate calculated using following equation:

$$WVTR = \frac{I}{A} \times K \qquad (1)$$

where:

$WVTR$ = water vapor transmission rate of the specimen, expressed in grams per square meter per day [$g/m^2 \cdot day$],
A = transmission area, in square meters, of the test specimen,
I = electrolytic current, in amperes, and
K = 8.28 is the conversion factor for converting measured electrolytic current in grams of electrolyzed water at STP.

12. Report

12.1 Report the following information:

12.1.1 A description of the test specimen. If the material is nonsymmetrical (two sides different), include a statement as to which side was facing the high humidity,

12.1.2 The sample thickness,

12.1.3 The humidity environment on each side of the test film,

12.1.4 The test temperature (to nearest 0.5 °C),

12.1.5 The values of WVTR, and

12.1.6 A statement that the instrument was operated in accordance with this standard method and the manufacturer's recommendations including the sensor evaluation process.

13. Precision and Bias

13.1 The precision of this test method is based on an interlaboratory study conducted in 2017. Nine laboratories tested three different film and sheeting samples at 37.8 °C and 90 % RH. Every "test result" represents an individual determination, and all laboratories reported four replicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM RR:F02-1043.[3]

13.1.1 *Repeatability Limit (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

13.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

13.1.1.2 Repeatability limits are listed in Table 1.

13.1.2 *Reproducibility Limit (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1043. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Water Vapor Transmission Rate ($g/m^2 \cdot 24hrs$)

Material	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
1	0.0278	0.0018	0.0054	0.0050	0.0152
2	0.1073	0.0033	0.0121	0.0094	0.0338
3	3.8402	0.0767	0.1934	0.2149	0.5415