



Designation: D 3199 – 84 (Reapproved 1994)^{€1}

Standard Test Method for Water Vapor Transmission Through Screw-Cap Closure Liners¹

This standard is issued under the fixed designation D 3199; 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} NOTE—Section 11 was added editorially in August 1994.

1. Scope

1.1 This test method covers the measurement of the barrier efficiency of screw-cap liners against water vapor transmission either into or outward from a container capped under standard conditions.

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 3198 Test Method for Application and Removal Torque of Threaded or Lug-Style Closures²

3. Summary of Test Method

3.1 This test method involves periodic weighing of containers sealed with screw caps containing the liners to be evaluated to determine moisture gain or loss under controlled conditions of temperature and humidity.

4. Significance and Use

4.1 This test method may be used to compare or screen new or existing closure liner materials by their performance in a standard test.

4.2 It also may be used to indicate whether the liner material will be satisfactory for use with moist products, dry products, or both types of products. When this method is used to assist in the choice of liner material for a specific use, the choice of containers and closures should simulate the conditions of use as closely as practicable.

4.3 This test method may be used to establish performance specifications.

5. Apparatus

5.1 *Bottles*, glass, ½ to 1 oz (approximately 15 to 30 cm³) with a 28-mm Glass Packaging Institute (GPI) 400 or 405 finish. Select test bottles for uniformity of flatness of finish, width of lip sealing surface, and freedom from splits, chips, or cracks.

5.2 *Screw Caps*, 28 mm continuous thread (CT) of either metal or plastic.

5.3 *Torque Tester*³, to determine application torque, accurate to within $\pm 1/2$ lbf-in. (0.06 N-m).

5.4 *Humidity Cabinet*, capable of control to $\pm 1.8^\circ\text{F}$ ($\pm 1^\circ\text{C}$) and $\pm 2\%$ relative humidity at 100°F (38°C). Ranges required are 25 and 75 % relative humidity.

5.5 *Analytical Balance*, with an accuracy to at least 0.001 g.

5.6 *Pipet or Graduate*, capable of delivering 10 mL of water with an accuracy of ± 0.1 mL.

6. Reagent

6.1 Activated silica gel (Grade H, Type 46, 16 mesh) anhydrous calcium chloride, or other suitable desiccant material.

7. Test Specimens

7.1 The closure-liner test specimen should be die-cut by either standard manufacturers' screw-cap lining equipment or by a single punch, using care that the liner is made uniform and without distortion of the liner facing.

7.2 No torn liners or wrinkled liners should be used. Prior to use, the liner backing combination must be laminated together and adhered to the cap, if plastic; or forced into the cap, if metal. At least ten test specimens and ten control specimens of each liner to be evaluated should be used for this test.

8. Procedure

8.1 *Water Vapor Transmission Outward from a Container:*

8.1.1 Tare weigh each ½-oz (28-mm) glass container together with its respective lined closure. Use five containers for this portion of the test.

³ Owens-Illinois torque tester, available from Owens-Illinois, Inc., Closure Div., One Seagate, Toledo, OH 43666, or equivalent, has been found satisfactory for this test method.

¹ This test method is under the jurisdiction of ASTM Committee D-10 on Packaging and is the direct responsibility of Subcommittee D10.31 on Child Resistant Packaging.

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² *Annual Book of ASTM Standards*, Vol 15.09.

8.1.2 Place 10.0 mL of distilled water in each tared glass container.

8.1.3 Apply the screw caps carrying the test liners with an application torque of 15 lbf·in. (approximately 1.7 N·m) to the containers.

8.1.4 Assemble with the caps prepared as in accordance with 8.1.1 and 8.1.3 and weigh five empty bottles for use as controls. Correct the test results by subtracting the average mass loss observed in the control containers from the average mass loss in the test containers.

8.1.5 Place the containers upright in the humidity chamber at $100 \pm 1.8^\circ\text{F}$ ($38 \pm 1^\circ\text{C}$) and $25 \pm 2\%$ relative humidity.

8.1.6 Seven days after starting the test, begin weighing each test container to the nearest 0.001 g at weekly intervals for 4 weeks. Allow the containers to come to room temperature before weighing.

8.1.7 Replace the test containers in the humidity cabinet immediately after each weighing.

8.2 Water Vapor Transmission Into a Container:

8.2.1 Tare weigh each ½-oz (28-mm) glass container together with its respective lined closure. Use five containers for this portion of the test.

8.2.2 Place 10.0 g of activated silica gel or other suitable desiccant in each container.

8.2.3 Apply the screw caps carrying the test liners to containers with an application torque of 15 lbf·in. (approximately 1.7 N·m).

8.2.4 Assemble with the caps prepared in accordance with 8.2.1 and 8.2.3 and weigh five empty bottles for use as controls. Correct the test results by subtracting the average mass gain observed in control containers from the average mass gain in the test containers.

8.2.5 Place the containers upright in the humidity chamber at $100 \pm 1.8^\circ\text{F}$ ($38 \pm 1^\circ\text{C}$) and $75 \pm 2\%$ relative humidity.

8.2.6 Seven days after starting the test, begin weighing each test container at weekly intervals for 4 weeks. Allow the packages to come to room temperature before weighing.

8.2.7 Replace the test containers in the humidity cabinet immediately after each weighing.

9. Report

9.1 The report shall include the following:

9.1.1 Material of construction of the liner and whether adhered to or only forced into the cap,

9.1.2 Material of construction of the cap,

9.1.3 Name of the desiccant used,

9.1.4 Number of replicates and controls,

9.1.5 Time interval or intervals of the test,

9.1.6 Temperature and humidity of the test,

9.1.7 Mass losses and gains in tabular form, and their averages,

9.1.8 Average mass gain and loss, the range, and the standard deviation in milligrams corrected for the average mass gain and loss of the controls,

9.1.9 Calculation of daily gain or loss (optional),

9.1.10 Graph on arithmetical paper of mass gain or loss versus time (optional),

9.1.11 Evaluation of comparative results if more than one liner construction was tested,

9.1.12 Statement that this test was done in accordance with Test Method D 3199.

10. Precision and Bias

10.1 The precision of this method is to be determined. Test results that might allow statistical evaluation for this statement are herewith solicited.

10.2 The bias of this method includes quantitative estimates of the uncertainties of the dimensional measuring devices, the calibration of test equipment, and the skill of the operator. At this time, statements on bias should be limited to the developmental performance of particular laboratories.

11. Keywords

11.1 liners; permeability—water vapor; screw-cap closure; water vapor transmission

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