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# Standard Test Methods for Gross Leakage of Liquids from Containers with Threaded or Lug-Style Closures<sup>1</sup>

This standard is issued under the fixed designation D 5094; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 These test methods cover the determination of gross leaks in rigid and semi-rigid containers (up to 4 L (1.06 gal)) with threaded or lug-style closures. Such tests may be used to indicate the ability of a liquid container to survive the distribution environment without leaking. These test methods may not be suitable to determine the leak resistance of containers intended for transport of hazardous materials.

1.2 Test Methods:

1.2.1 *Test Method A, Shipping Container Vibration and Storage Test*, covers the ability of a shipping container and its interior packaging to protect the contents from leakage after transportation induced vibration and high-temperature storage.

1.2.2 Test Method B, Shipping Container Vibration and Vacuum Chamber Test, is suitable for individual containers and is usually less severe than Test Method A. The advantage of Test Method B is the shortness of the test.

1.3 This standard does not purport to address the safety conserns 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:
- D 996 Terminology of Packaging and Distribution Environments<sup>2</sup>
- D 999 Test Methods for Vibration Testing of Shipping Containers<sup>2</sup>
- D 3198 Test Method for Application and Removal Torque of Threaded or Lug-Style Closures<sup>2</sup>
- D 3474 Practice for the Calibration and Use of Torque Meters Used in Packaging Applications<sup>2</sup>
- D 4169 Practice for Performance Testing of Shipping Containers and Systems<sup>2</sup>
- D 4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing<sup>2</sup>

### 3. Terminology

3.1 General definitions for packaging are found in Terminology D 996.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *gross leak*—any opening in the container or closure seal that produces visible leakage such that the container would not be suitable for transport or subsequent distribution.

3.2.2 *leak*—any opening in a container which, contrary to intention, either lets contents escape or permits substances to enter.

3.2.3 *leakage*—that which passes through a leak.

### 4. Summary of Test Methods

4.1 Method A, Shipping Container Vibration and Storage Test—Test specimens are filled to their expected fill capacity with product or liquid simulating product and the closure is applied to the container. Closures should be applied with the same torque as would be encountered in production. The specimens are packed into shipping containers and vibrated. The specimens, stored on their sides, are subjected to 40°C (104°F) (or other conditions as appropriate) for four weeks. Each specimen is examined for leakage.

4.2 Method B, Container Vibration and Vacuum Chamber Test—Test specimens are partially filled with product or liquid simulating product and the closure is applied to the container. Closures should be applied with the same torque as would be encountered in production. The specimens are packed into shipping containers and vibrated. Following vibration, the individual specimens are removed from the shipping container and subjected to 10 in. Hg (33.7 kPa) vacuum for 10 min and examined for leakage.

NOTE 1-Other levels of vacuum, or time, or both may be appropriate.

### 5. Significance and Use

5.1 These test methods are used to indicate the integrity of the container and closure system in the distribution environment.

5.2 These test methods measure the ability of the container and closure system to prevent leakage when stored or transported upright, inverted, or on the side.

5.3 These test methods allow for comparison of container/ closure designs of threaded and lug-style closures of similar or

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 15.09.

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different materials, or different manufacturers. These tests are suitable for packaging development, engineering and tooling evaluations.

# 6. Apparatus

6.1 Method A, Shipping Container Vibration and Storage Test:

6.1.1 Drying Oven, constant-temperature, equipped with means for ensuring adequate temperature control,  $40 \pm 1^{\circ}$ C (104  $\pm$  1.8°F) and air circulation, ambient humidity, or other conditions as appropriate.

6.1.2 *Torque Meter*, with an appropriate scale which accurately measures within the expected torque range.

6.1.3 *Vibration Test Machine*, the apparatus described in the Apparatus section of Methods D 999 may be used.

6.2 Method B, Shipping Container Vibration and Vacuum Test:

6.2.1 *Vacuum Chamber*, any suitable chamber capable of withstanding approximately one atmosphere pressure differential, fitted with a vacuum-tight seal. A vacuum gauge, an inlet tube from a source of vacuum, and an outlet tube to the atmosphere shall be sealed to the chamber. The inlet and outlet tubes shall be equipped with shut-off valves. The vacuum gauge shall be laboratory quality with a full scale range of 0 to 30 in. Hg (0 to 100 kPa).

6.2.2 *Torque Meter*, with an appropriate scale which accurately measures within the expected torque range.

6.2.3 *Vibration Test Machine*, the apparatus described in the Apparatus section of Methods D 999 may be used.

#### 7. Sampling

7.1 The complete test unit shall consist of the shipping container, with interior packaging and the actual test specimens (container(s) and closure(s)).

7.2 A minimum of three specimens representing each variable, such as cap or bottle mold from each cavity but not less than ten specimens are to be selected. The total number of specimens may need to be increased to completely fill the shipping container. More than one shipping container may be required.

7.3 Select unused closures and unused containers that are within specifications.

#### 8. Test Specimens

8.1 *Test Method A*—A specimen shall be one container filled to expected fill capacity with product or liquid simulating product. The closure is secured in accordance with 11.1.

8.2 *Test Method B*—A specimen shall be one container one-third filled with product or liquid simulating product. The closure is secured in accordance with 11.1.

8.3 Colorant should be added to the contents when necessary to give permanent indication where there is leakage.

# 9. Calibration

9.1 Calibrate the torque meter in accordance with Practice D 3474.

### **10.** Conditioning

10.1 Store the shipping container components at ambient laboratory conditions for at least 4 h before testing, or other

conditions in accordance with Practice D 4332.

10.2 Store the torqued container/closure test specimens upright at least 24 h at ambient conditions before commencing the test.

### 11. Procedure

11.1 Apply closures to the containers according to the Application Torque Measurement section of the procedure in Test Method D 3198.

11.2 Test Method A, Shipping Container Vibration and Storage Test:

11.2.1 Place the filled specimens in their shipping container or in an appropriate carton so that they will remain firmly in place during vibration.

11.2.2 Proceed as directed according to Methods D 999 (Method A 1 or A 2) 20 min each on bottom, top, and one side, or run an entire shipping cycle in accordance with Practice D 4169.

11.2.3 Remove the specimens from the shipping container or carton. Examine all specimens for visible leakage without removing or disturbing the closure. Note the location(s) of leakage.

11.2.4 Place the test specimens on their sides (liquid in contact with finish/closure) in a container capable of holding the liquid should a leak develop. It is recommended that absorbent blotting paper be placed beneath the samples to more easily detect leakage. Store the test specimens at  $40^{\circ} \pm 1^{\circ}$ C ( $104^{\circ} \pm 1.8^{\circ}$ F), or other appropriate temperature, ambient humidity and inspect for liquid leakage at 24 h and at one-week intervals for four weeks. Note location(s) of leakage.

11.2.5 At the conclusion of the test, remove the closure and inspect the threads for signs of leakage or crusting from partial leakage.

11.3 Test Method B, Shipping Container Vibration and Vacuum Chamber Test:

11.3.1 Place the partially filled specimens in their shipping container or in an appropriate carton so that they will remain firmly in place during vibration.

11.3.2 Proceed as directed according to Methods D 999 (Method A 1 or A 2) 20 min each on bottom, top, and one side, or run an entire shipping cycle in accordance with Practice D 4169.

11.3.3 Remove the specimens from the shipping containers or carton. Examine all specimens for visible leakage without removing or disturbing the closure. Note location(s) of leakage.

11.3.4 Place the partially filled specimens inverted in a beaker or other receptable to contain leakage.

NOTE 2—Other orientations of the specimens may be appropriate. Orientation of the container should be such that suspected leak areas are covered with the liquid inside the container.

11.3.5 Place specimens in the vacuum chamber and close the vacuum chamber.

11.3.6 Close the chamber inlet valve.

11.3.7 Open the chamber outlet valve and turn on vacuum source so that the gauge rises slowly (from 30 s to 1 min) to 10 in. Hg (33.7 kPa) and close the outlet valve.

11.3.8 Maintain the vacuum for 10 min.

11.3.9 Partially open the chamber inlet valve and release the