1. Scope

1.1 These test methods evaluate the torque retention of continuous thread closures on containers, with matching finishes, for predetermined environmental conditions over time.

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:

D 996 Terminology of Packaging and Distribution Environments
D 3198 Test Method for Application and Removal Torque of Threaded or Lug-Style Closures
D 3474 Practice for the Calibration and Use of Torque Meters Used in Packaging Applications
D 4169 Practice for Performance Testing of Shipping Containers and Systems
D 4332 Practice for Conditioning Containers, Packages or Packaging Components for Testing
E 41 Terminology Relating to Conditioning
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions:

3.1.1 For definitions of general packaging and distribution terms, see Terminology D 996.

3.1.2 For definitions of application torque and removal torque, see Test Method D 3198.

3.1.3 For Definitions regarding conditioning, see Terminology E 41.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 **immediate removal torque**—removal torque that is measured at a stated interval, from 1 to 5 min after closure application.

3.2.2 **torque retention**—a comparison between removal torque at the end of a test period and a predetermined immediate removal torque.

4. Summary of Test Methods

4.1 **Test Method A: Static Evaluation**—At predetermined time intervals, the removal torques of representative samples of a container/continuous thread closure system, previously stored at various environmental conditions, are measured.

4.2 **Test Method B: Dynamic Evaluation**—Practice D 4169 is used to develop a uniform system of evaluating the ability of primary packages, in the shipping units, to withstand the distribution environment. At the end of predetermined distribution cycles, the removal torques of representative samples of a container/continuous thread closure system are measured.

5. Significance and Use

5.1 This test method allows for the measurement of the torque retention properties of container/continuous thread closure systems of various designs, materials, and manufacture, and is suitable for packaging development and engineering evaluation.

5.2 This test method can be used for the evaluation of container/continuous thread closure systems under controlled conditions (where the application torque is known and the applied downward force to the closure is zero).

6. Apparatus

6.1 **Torque Meter**, with an appropriate scale that accurately measures within the expected torque range for the particular container/continuous thread closure system to be evaluated.

6.1.1 A spring torque meter, if used, will have a scale where the anticipated torque readings are not less than one-third of the maximum range of the scale for the container/continuous...
thread closure system to be evaluated. Torque results will be available in a visual format.

6.1.2 A digital or automated torque instrument, if used, will have an appropriate design and scale capacity for the container/continuous thread closure system to be evaluated. Torque results will be available in either electronic display or printout formats.

6.1.3 A torque wrench, if used, will have an appropriate design and capacity capable of providing a reading within the anticipated range for the container/continuous thread closure system to be evaluated. Torque results will be available in a visual format.

7. Sampling and Test Specimens

7.1 Measure no less than ten test specimens for each torque measurement point per test variable. See Note 1.

7.2 Select previously unused continuous thread closures and containers as test specimens.

8. Calibration

8.1 Calibrate spring torque meters in accordance with the procedures of Practice D 3474.

8.2 Calibrate digital, automated torque instruments or torque wrenches in accordance with manufacturers recommendations.

9. Conditioning and Preparation of Test Specimens

9.1 Perform test specimen conditioning in accordance with Specification E 171 and Practice D 4332.

9.2 Fill all of the containers with the specified volume or weight of product, or other materials that yield similar weight and thermal characteristics. See Note 2.

NOTE 1—The total quantity of test specimens sampled will depend upon the method selected and the number of environmental storage conditions.

NOTE 2—Given the purpose of the evaluation, empty containers may be used as an option to filled containers.

10. Procedure (See Note 3.)

10.1 Test A: Static Evaluation:

10.1.1 Select the minimum application torque for the container/continuous thread system as recommended by the closure manufacturer. (For example, the U.S. Pharmacopoeia, the Society for the Plastics Industry, the Glass Container Manufacturers Institute, or other sources.)

10.1.2 Firmly position the container or closure in such a manner that the axis of rotation of the closure is concentric with the center of the torque measuring device. See Note 4.

10.1.2.1 Exercise care in positioning the container or closure to prevent distortion of either component.

NOTE 3—Under certain conditions of product-filling, storage and distribution, it may be desirable to combine appropriate segments of Test Method A and Test Method B.

NOTE 4—It is recommended that one operator, and a single torque instrument be used to apply all closures in any one test to reduce possible operator/instrument inconsistencies and variabilities.

10.1.3 Avoiding contact with the fixed component, grip the movable component (normally the continuous thread closure) and rotate it at a constant and uniform rate, in a tightening direction, to the predetermined application torque.

10.1.4 Release the movable component (normally the continuous thread closure) upon reaching the desired torque. See Note 5.

10.1.5 Apply the balance of the closures or containers to the matching components as directed in 10.1.2, 10.1.3, and 10.1.4.

10.1.6 Store the assembled test specimens as appropriate in accordance with Specification E 171 or Practice D 4332.

10.1.6.1 Maintain one group of test specimens as a control by storing this group at ambient laboratory conditions.

NOTE 5—The application torque range is usually determined on the basis of the desired removal torque range.

10.1.6.2 Maintain, as appropriate, one or more groups of test specimens at constant temperatures different than ambient laboratory conditions. If desired, cycling at various temperature and relative humidity conditions may be performed. See Note 6.

10.1.7 At the end of each predetermined time period, determine the removal torque for each test sample.

10.1.7.1 Determine the removal torques either at ambient laboratory conditions or at the alternative temperature and relative humidity conditions.

10.1.7.2 The following test intervals are recommended: immediate (between 1 and 5 minutes), 24 h, 48 h, 7 days, 14 days, and 28 days.

NOTE 6—Sterilization cycles, if applied, using steam, ethylene oxide, gamma radiation, or other methods are known to affect certain plastics; these effects may influence removal torques.

10.1.8 Determine the removal torque for each test specimen by firmly positioning either the container or the continuous thread closure in such a manner that the axis of rotation of the closure is concentric with the center of the measuring device. See Note 7.

10.1.8.1 Exercise care in positioning the container or continuous thread closure to prevent distortion of either component.

10.1.9 Avoiding contact with the fixed component, grip the movable component (normally the continuous thread closure), and rotate it uniformly, at a constant rate, in a loosening direction until the continuous thread closure rotates freely. Note the maximum torque required to loosen the closure and record this value for the test specimen.

NOTE 7—It is recommended that one operator, and a single torque instrument, be used to remove all closures in a given test to reduce possible operator/instrument inconsistencies and variabilities.

10.1.10 Record the maximum torque required to loosen the closure for each remaining specimen.

10.1.11 Remove the balance of the movable components (normally the continuous thread closures) as directed in 10.1.8 and 10.1.9.

10.2 Test Method B: Dynamic Evaluation:

10.2.1 Follow the procedures of Practice D 4169 for recommendations relevant to the number of test specimens, conditioning, and distribution cycles to be used to evaluate these test specimens.
10.2.2 Follow Section 8 on Calibration for test equipment calibration.
10.2.3 Follow Section 10 on Procedure (Test Method A) for recommendations concerning continuous thread closure application.
10.2.4 Begin evaluation of a selected distribution cycle after immediate removal torque has been determined and before any additional long-term storage, if long-term storage is desired. (Testing may be determined following distribution cycling with removal torques noted.)

11. Report

11.1 Report the following information:
11.1.1 Identification of the test method used.
11.1.2 Identification of the continuous thread closure and container manufacturer(s), the material(s) of construction of the closure (including any liner, liner coating, and additional sealing components), and the container (including any coating or annealing agents).
11.1.3 Thread finish and other designation(s) of the continuous thread closure and the container.
11.1.4 Description of test specimen preparation.
11.1.5 Description of storage conditions.
11.1.6 Description of distribution cycles and handling conditions.
11.1.7 Description of the test area environment.
11.1.8 Number of test replicates.
11.1.9 Application torque range and mean value for each test condition variable.
11.1.10 Removal torque range and mean value for each test condition variable.
11.1.11 Description of the torque instruments used.
11.1.12 Description of the rate of rotation for the application torque (if available).
11.1.13 Description of the rate of rotation for the removal torque (if available).
11.1.14 Evaluation of comparative results, if appropriate.
11.1.15 Statement that testing was done in accordance with this test method or a description of the difference(s) from this test method.

12. Precision and Bias 4

12.1 Precision:

NOTE 8—See Research Report RR:D10-1005 for a complete description of the interlaboratory/intralaboratory evaluation of this test method.

NOTE 9—Initial values are expressed in torque inch pounds-force; values in parentheses are expressed in Newton-metres.

12.1.1 The repeatability and reproducibility are in accordance with the definitions of these terms in Practice E 691 (at 95 % probability level). These values are based on an interlaboratory study involving six laboratories each of which measured removal torques of three different closures at the recommended six time intervals. (See Note 8.)

12.1.2 Repeatability— The difference between successive removal torque results, after 28 days of storage, obtained by the same operator with the same equipment unit under constant operating conditions on test packages from a single lot complying with given specifications, within a laboratory, was determined to be as follows (see Note 9):

<table>
<thead>
<tr>
<th>Test Package</th>
<th>Average</th>
<th>Repeatability</th>
<th>Standard deviation, Sr (within a laboratory)</th>
<th>95 % Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.97</td>
<td>0.74</td>
<td>0.08</td>
<td>2.06</td>
</tr>
<tr>
<td>2</td>
<td>8.04</td>
<td>1.06</td>
<td>0.12</td>
<td>5.35</td>
</tr>
<tr>
<td>3</td>
<td>8.09</td>
<td>1.91</td>
<td>0.22</td>
<td>2.97</td>
</tr>
</tbody>
</table>

12.1.3 Reproducibility— The difference between successive removal torque results, after 28 days of storage, obtained by different operators with different equipment units under constant operating conditions on test packages from a single lot complying with given specifications in different laboratories, was determined to be as follows (see Note 9):

<table>
<thead>
<tr>
<th>Test Package</th>
<th>Average</th>
<th>Reproducibility</th>
<th>Standard deviation, SR (between laboratories)</th>
<th>95 % Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.97</td>
<td>1.992</td>
<td>0.23</td>
<td>5.55</td>
</tr>
<tr>
<td>2</td>
<td>8.04</td>
<td>2.932</td>
<td>0.33</td>
<td>7.14</td>
</tr>
<tr>
<td>3</td>
<td>8.09</td>
<td>2.605</td>
<td>0.29</td>
<td>8.17</td>
</tr>
</tbody>
</table>

12.2 Bias—Since there is no accepted reference component suitable for determining the bias for the procedure in this test method for measuring torque retention, no statement on bias is being made.

13. Keywords

13.1 continuous thread closure; removal torque; retention