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Standard Test Method for Environmental Stress Crack Resistance (ESCR) of Threaded Plastic Closures[1]

This standard is issued under the fixed designation D5419; 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 determines the susceptibility of threaded plastic closures to failure due to environmental stress cracking (ESC).

1.2 ~~T~~In use, threaded plastic closures ~~in use may~~ can contact agents that appreciably reduce the stress at which cracks form. Examples of such agents are: soaps, detergents, oils, and liquid bleaches.

~~1.3Other major~~ 1.3 Major factors that influence environmental stress crack resistance (ESCR) of threaded plastic closures ~~are~~include the closure material(s), closure design, molded-in stress, and applied stress.

1.4 This procedure can be applied to all closures, but is particularly applicable to closures made from plastics based on polypropylene (PP) or polystyrene (PS). ~~It may also apply to other polymers.~~

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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.* Specific precautionary statements are given in Section 8 ~~and Note 2~~and 6.2.

NOTE~~1—There are no ISO standards covering the primary subject of this test method.~~ 1—There is no known ISO equivalent to this test method.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1600 Terminology for Abbreviated Terms Relating to Plastics
D2911 Specification for Dimensions and Tolerances for Plastic Bottles
D3198 Test Method for Application and Removal Torque of Threaded or Lug-Style Closures
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—Except for those terms below, see Terminologies D883 and D1600.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *assembly*—closure applied to a bottle finish.

3.2.2 *failure*—during this test, any visible crack.

3.2.2.1 *Discussion*—A crack does not have to penetrate the closure wall to be considered a failure.

3.2.3 *finish*—fixture representing the threaded portion of the bottle.

3.2.4 *threaded closure*—part applied to seal bottle as specified in Specification D2911.

4. Summary of Test Method

4.1 This test method consists of applying closures at a specified application torque to rigid finishes (of polysulfone or other appropriate resin), immersing the assembly in a potential stress-cracking agent, and observing and reporting time-to-failure.

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[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film and Sheeting.

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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.

***A Summary of Changes section appears at the end of this standard.**

5. Significance and Use

5.1 This test method compares closures for ESCR. Suitable variables are: closure materials, closure designs, processes, applied torque, and stress-crack agents.

5.2 Results can be used for estimating shelf life of closures in terms of ESCR. This requires that the user has calibrated failure time in this test to failure time in the field for actual packaging systems.

6. Apparatus

6.1 *Wide-Mount Gallon Jars*, glass, PET, or other suitable material. Must have lined closures to ensure air-tight seal. Use one jar per sample.

6.2 *Circulating-Air Oven*, capable of maintaining a temperature of 50 ± 1°C (critical in this application). See Specification E145 for a procedure for confirming satisfactory uniformity of temperature within the oven. There is no air-flow requirement in this application. An environmental room with these properties is also suitable.

~~NOTE 2—~~ (~~**Caution:**~~**Warning—**A high-temperature safety switch is highly recommended on this oven. Some test liquids can cause extreme pressure to build up upon heating. Under these conditions the test jars may rupture with explosive force. The override cutoff switch should be set to turn off the oven if the test temperature is exceeded by 10°C or more.)

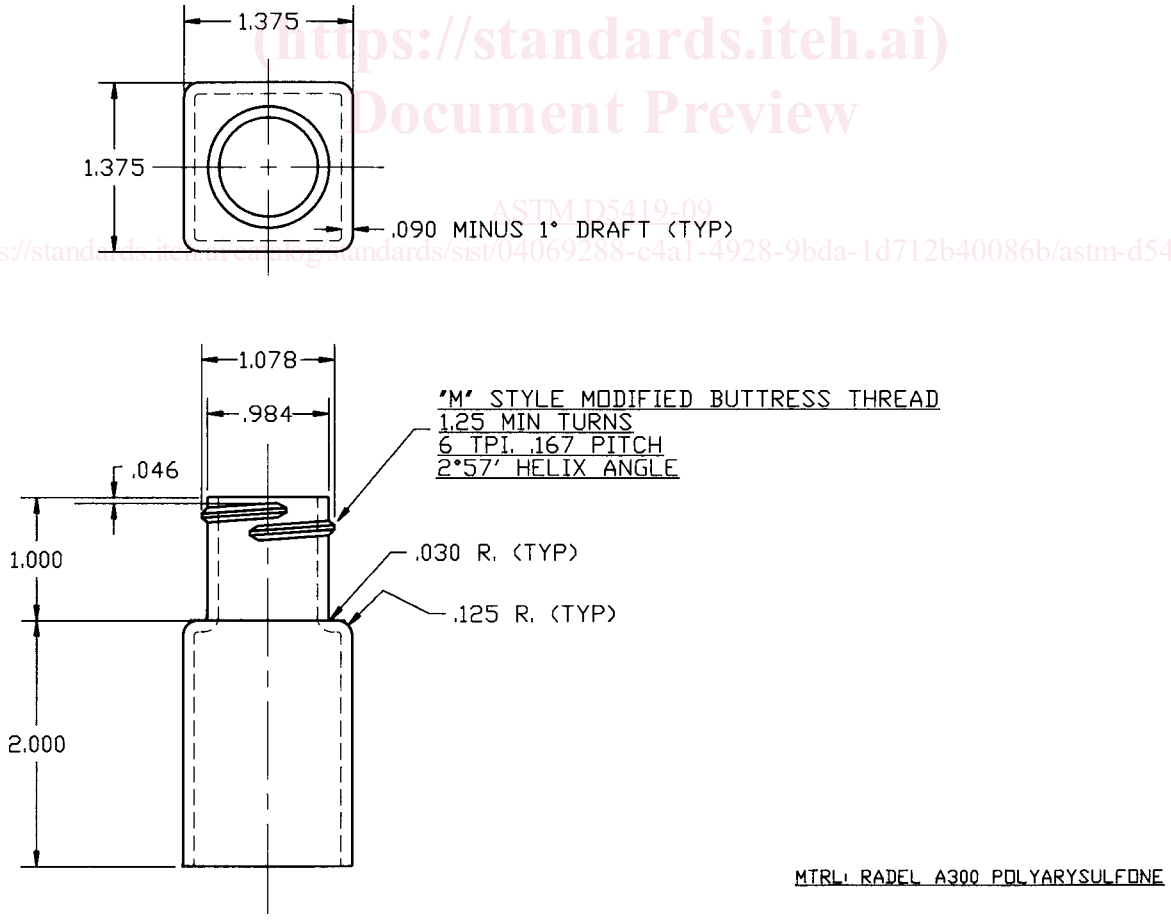
6.3 *Tongs*, for sample removal and inspection.

6.4 *Bottle Finishes*, polysulfone or other material of equivalent stiffness and thermal coefficient of expansion, to which closures are applied. ~~May~~These can be made by injection molding or by machining rod stock. See Fig. 1 for a drawing of a typical fixture. Use an appropriate size based on closure and bottle specifications.

6.5 *Torque Meter*, with capacity of at least 5 torque Nm, calibrated or verified within the past 12 months.

6.6 *Plastic Test Closures*~~, lined or unlined closure based on specifications. PP-based closures should be at least 3 weeks old before testing, and PS closures at least 16 h old. This is to ensure that full crystallization has essentially been achieved.~~ , lined or unlined closure based on specifications.

NOTE 2—To ensure that full crystallization has essentially been achieved, PP-based closures should condition for at least three weeks before testing and PS closures for at least 16 h.



NOTE 1—Tolerances for Dimensions *T*, *E*, and *S* shall be in accordance with Specification D2911.

FIG. 1 Typical Fixture

NOTE 3—To convert lbf-in. torque to Nm torque, multiply by 0.113.

7. Reagents and Materials

7.1 *Test Solution*—Use solution for which the closure is intended.

8. Hazards

~~8.1Always wear protective equipment appropriate to the product hazard when setting up or inspecting closures. This may include goggles, gloves, and aprons.~~

8.1 Always wear protective equipment, such as goggles, gloves, and aprons, appropriate to the product hazard when setting up or inspecting closures.

9. Test Specimens

9.1 Normal sample size is 20 closures, typical of lots to be tested. It is strongly advisable to run the test in duplicate (two sets of 20) or to sample more than one lot.

9.2 Visually inspect each closure to be tested. Replace any that appear defective or irregular.

10. Conditioning

10.1 *Conditioning*—After aging in accordance with 6.6, condition closures and bottle finishes at 23 ± 2°C and 50 ± ~~5%~~10 % relative humidity for not less than 40 h prior to test, in accordance with Procedure A of Practice D618 ~~. Condition test solution at 50 ± 2°C until it reaches 50 ± 2°C (16 h normally required).~~ unless otherwise specified by agreement or the relevant ASTM material specification. Condition test solution at 50 ± 1°C until it reaches 50 ± 1°C (16 h normally required).

10.2 *Test Conditions*—Conduct all tests at 50 ± 2°C, unless instructed otherwise.

11. Procedure

11.1 Apply closures to bottle finishes (see Test Method D3198~~). Application torque should be either of the following:~~

~~11.1.1That corresponding to the upper limit of immediate removal torque in production (rule of thumb: application torque=1.05×immediate removal torque), or~~). Unless otherwise specified, apply torque to a tolerance of ±5 % using one of the following values:

11.1.1 A value corresponding to the upper limit of immediate removal torque in production (rule of thumb: application torque = 1.05 × immediate removal torque), or

11.1.2 A nominal value based on the closure diameter (rule of thumb: torque, Nm = closure diameter, mm × 0.08), or

11.1.3 A value agreed upon between the laboratory and the customer.

~~11.2Tolerance for the torque should be ±5%.~~

~~11.3Place~~ 11.2 Place 20 assemblies in jars. Fill jars with enough test solution at 50 ± ~~2°C~~1°C to cover all assemblies. Wipe any test solution from jar-finish area. Cap jars and hand tighten.

11.~~4~~3 Place jars on test at 50 ± ~~2°C.~~1°C. Check and record the temperature of test area daily and maintain it within ~~limits. Record~~ the ~~temperature of the oven or room.~~ specified limits.

11.~~5~~4 Inspect the assemblies daily except on weekends. Move jars from test condition to inspection area. Do not allow to be off test more than 60 min. Remove the assemblies individually, using tongs on the bottle finishes, not the closures.

NOTE~~4—Inspection frequency may~~ 4—It is recommended that the inspection frequency be increased during periods of known high-failure rates. Delaying the start of the second duplicate sample facilitates this; however, frequency of torque reapplication should remain at daily intervals except on weekends.

11.~~6~~5 After inspection, set aside failures. Reapply initial torque to nonfailing closures, return them to jars and move jars back to test condition. Do not remove or loosen closures. If test solution degrades with age, replace it often enough that the failure rate is not significantly reduced. If closure cracks on retorqueing, count this as a failure on the next inspection.

~~11.7For~~11.6 For each failure, note the time, description, and location of failure.

~~11.8Any~~11.7 Any sample (jar) ~~may~~can be ~~taken off~~ removed and the test terminated when there have been failures on at least two inspections, and a total of at least 11 out of 20 closures have failed. Record inspections even on days when there are no failures.

NOTE 5—If testing against an F_{50} specification, the test may be terminated if no more than 1 closure out of 20 fails by the specification time.

12. Calculation

12.1 Calculate closures predicted to fail at any given time by the following equation:

$$\text{failures, \%} = [(n - 0.5)/N] \times 100 \quad (1)$$

where:

n = cumulative number of closures that have failed as of the given time, and

N = number of closures tested (20 unless otherwise stated).

12.2 F_{25} *Failure Time*—Plot the data on Weibull probability graph paper with days on the log scale and percent failure on the probability scale. When more than one closure fails on a given inspection, use the average % failing on that inspection for the