Designation: D5419 – 14a

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

1.3 Major factors that influence environmental stress crack resistance (ESCR) of threaded plastic closures 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).

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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 6.2.

NOTE 1—There is no known ISO equivalent to this standard.

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

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.

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.

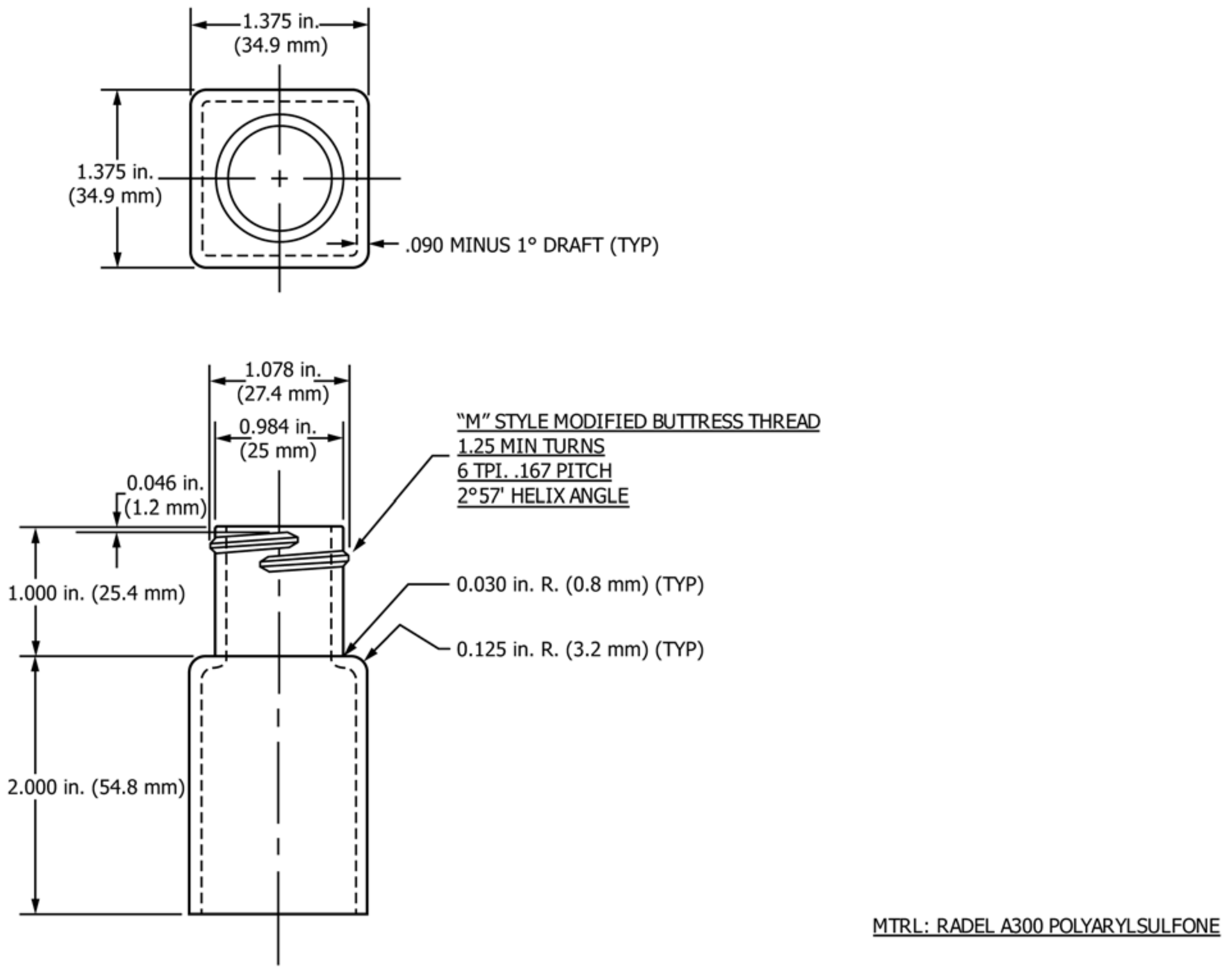
[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film, Sheeting, and Molded Products.

Current edition approved Dec. 1, 2014. Published December 2014. Originally approved in 1993. Last previous edition approved in 2014 as D5419 – 14. DOI: 10.1520/D5419-14A.

[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**

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States



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

FIG. 1 Typical Fixture

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. (**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 it is possible that the test jars will rupture with explosive force. Set the override cutoff switch 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. 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.

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 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

8.1 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 ± 10 % relative humidity for not less than 40 h prior to test, in accordance with Procedure A of Practice D618 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. 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.

11.1.4 Firmly position the bottle finish between the four posts on the torque tester in such a manner that the axis of rotation of the cap is concentric with the center of the movable plate on which the bottle finish rests.

11.1.5 Grip the closure by hand, avoiding any contact with the bottle finish, and twist clockwise while closely watching the gauge. Apply torques smoothly and consistently, at a constant rate consistent with accurate reading of the gauge.

11.1.6 Apply torque continuously until the desired torque as indicated by the needle on the gauge is reached.

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

11.3 Place jars on test at 50 ± 1°C. Check and record the temperature of test area daily and maintain it within the specified limits.

11.4 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—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.

11.5 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.

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

11.7 Any sample (jar) can be 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.

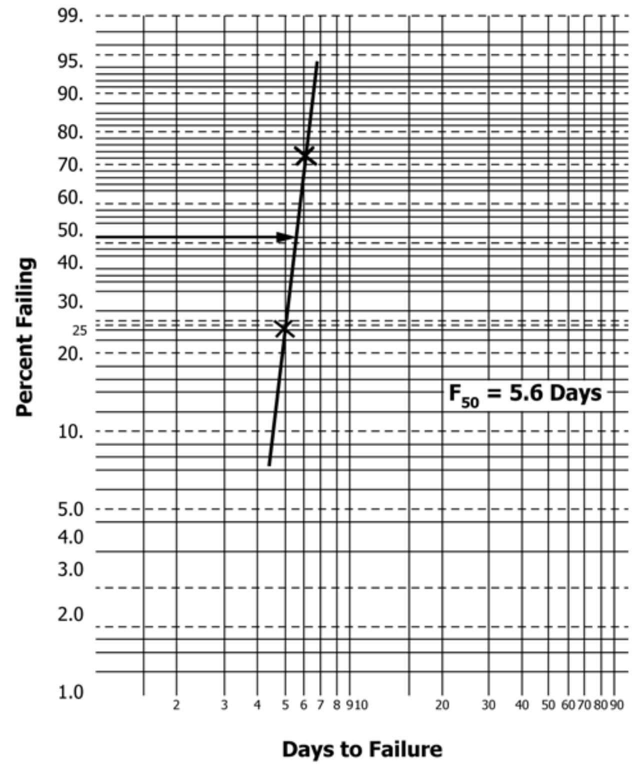


FIG. 2 F_{50} Failure Time

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 plotting position (see the example in Appendix X1). Draw the best fitting straight line for the plot. The days indicated at the intersection of the data line with the 50 % failure level probability line shall be reported as the F_{50} failure time. See Fig. 2 for example.

NOTE 6—The plot or calculations, or both, may be made on computer with suitable software.

13. Report

13.1 Report the following information:

TABLE 1 F_{50}, Days

Closures Tested in Bleach	Average	S_r [A]	S_R [B]	r [C]	R [D]
28 mm-Polypropylene	5.1	0.8	1.5	2.2	4.1

[A] S_r is the within-laboratory standard deviation.
[B] S_R is the between-laboratories standard deviation.
[C] r is the within-laboratory repeatability limit = 2.8 S_r .
[D] R is the between-laboratory reproducibility limit = 2.8 S_R .

13.1.1 Closure identification, specification number (if applicable), manufacturer, molding location, molding date, lot number, liner description (if applicable), molding machine, resin, mold and cavity number(s), process conditions,

13.1.2 Test solution name and composition/analysis,

13.1.3 Application torque,

13.1.4 Date and time of each inspection and location of each failure, and

13.1.5 Failure time (F_{50}).

14. Precision and Bias[3]

14.1 *Precision*—Table 1 is based on a round robin conducted in 1995 in accordance with Practice E691, involving one material tested by 12 laboratories. All of the samples were prepared at one source. Each test result was based on 20 individual determinations. Each laboratory obtained two test results for each material. (**Warning—**The following explanations of r and R (14.1.1.1 – 14.1.1.3) are only intended to present a meaningful way of considering the approximate precision of this test method. Do not apply the data in Table 1 rigorously to the acceptance or rejection of material, as those data are specific to the round robin and are not necessarily representative of other lots, conditions, materials, or laboratories. Users of this test method need to apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials or between specific laboratories. The principles of 14.1.1.1 – 14.1.1.3 would then be valid for such data.)

14.1.1 *Concept of r and R*—if S_r and S_R have been calculated from a large enough body of data,

14.1.1.1 *Repeatability, (r)*—(Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day.) Judge the two test results as not equivalent if they differ by more than the r value for that material.

14.1.1.2 *Reproducibility, (R)*—(Comparing two test results for the same material, obtained by different operators using different equipment on different days.) Judge the two test results as not equivalent if they differ by more than the R value for that material.

14.1.1.3 Any judgment determined in accordance with 14.1.1.1 and 14.1.1.2 would have an approximate 95 % (0.95) probability of being correct.

14.2 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

15. Keywords

15.1 closures; environmental stress crack resistance (ESCR); failure; plastic; stress crack; threaded plastic closures

[3] A research report is currently being written.

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE OF GRAPHICAL DETERMINATION OF F_{50}

TABLE X1.1 Data

No. of Days on Test	No. of Closures Failing on This Date	Total No. of Closures Failed to Date
3	0	0
4	0	0
5	10	10
6	9	19

TABLE X1.2 Plotting Positions for n = 20

No. of Closures Failed	Plotting Position, %
1	2.5
2	7.5
3	12.5
4	17.5
5	22.5
6	27.5
7	32.5
8	37.5
9	42.5
10	47.5
11	52.5
12	57.5
13	62.5
14	67.5
15	72.5
16	77.5
17	82.5
18	87.5
19	92.5
20	97.5

X1.1 *Data (See Table X1.1):*

X1.2 *Plotting Positions for n = 20 (See Table X1.2):*

X1.3 *X-Axis Values for Example:*

X1.3.1 First failures occurred at 5 days, so the first X value is 5 days.

X1.3.2 The second X value is 6 days.

X1.3.3 The test terminated after 6 days (19 failures).

X1.4 *Y-Axis Values for Example:*

X1.4.1 For the first point, the Y value is the average of the plot position for 1 and 10 closures failed

$$= (2.5+47.5)/2 = 25\% \quad (X1.1)$$

X1.4.2 For the second point, the Y value is the average of the plot position for 11 and 19 closures failed

$$= (52.5+92.5)/2 = 72.5\% \quad (X1.2)$$

X1.4.3 *Graph (See Fig. 2):*

X1.5 *Determination of* F_{50} —Interpolate the line at the "percent failing" value of 50 on the Y-axis, and read of f "days to failure" on the X-axis.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D5419 - 14) that may impact the use of this standard. (December 1, 2014)

(1) Deleted reference to ASTM D3198 since it was withdrawn.
(2) Added appropriate directions to Section 11 (Procedure) to address application of closures to bottle finish.

Committee D20 has identified the location of selected changes to this standard since the last issue (D5419 - 09) that may impact the use of this standard. (November 15, 2014)

(1) Editorially changed the wording of the warning note in 6.2.
(2) Clarified the standard units statement in 1.5.
(3) Added units of measurement to Fig. 1.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/