Designation: D2561 – 17

Standard Test Method for Environmental Stress-Crack Resistance of Blow-Molded Polyethylene Containers[1]

This standard is issued under the fixed designation D2561; 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 Under certain conditions of stress, and in the presence of environments such as soaps, wetting agents, oils, or detergents, blow-molded polyethylene containers exhibit mechanical failure by cracking at stresses appreciably below those that would cause cracking in the absence of these environments.

1.2 This test method measures the environmental stress crack resistance of blow-molded containers, which is the summation of the influence of container design, resin, blow-molding conditions, post treatment, or other factors that can affect this property. Three procedures are provided as follows:

1.2.1 *Procedure A, Stress-Crack Resistance of Containers to Potential Stress-cracking Liquids*—This procedure is particularly useful for determining the effect of container design on stress-crack resistance or the stress-crack resistance of a proposed container that contains a liquid product.

1.2.2 *Procedure B, Stress-Crack Resistance of a Specific Container to Polyoxyethylated Nonylphenol (CAS 68412-54-4), a Stress-Cracking Agent*—The conditions of test described in this procedure are designed for testing containers made from Class 3 polyethylene Specification D4976. Therefore, this procedure is recommended for containers made from Class 3 polyethylene only. This procedure is particularly useful for determining the effect of resin on the stress-crack resistance of the container.

1.2.3 *Procedure C, Controlled Elevated Pressure Stress-Crack Resistance of a Specific Container to Polyoxyethylated Nonylphenol (CAS 68412-54-4), a Stress-Cracking Agent*—The internal pressure is controlled at a constant elevated level.

NOTE 1—There are environmental concerns regarding the disposal of Polyoxyethylated Nonylphenol (Nonylphenoxy poly(ethyleneoxy) ethanol (CAS 68412-54-4), for example, Igepal CO-630). Users are advised to consult their supplier or local environmental office and follow the guidelines provided for the proper disposal of this chemical.

1.3 These procedures are not designed to test the propensity for environmental stress cracking in the neck of containers, such as when the neck is subjected to a controlled strain by inserting a plug.

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

NOTE 2—There is no known ISO equivalent to this standard.

1.5 *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 1.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D4976 Specification for Polyethylene Plastics Molding and Extrusion Materials
D5947 Test Methods for Physical Dimensions of Solid Plastics Specimens
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *failure*—during this test method, the formation of any imperfection, such as a crack, which results in a loss of pressurizing gas or stress-cracking agent.

3.1.1.1 *Discussion*—A container has failed when:

It has lost pressure through any aperture other than heat

[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.

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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

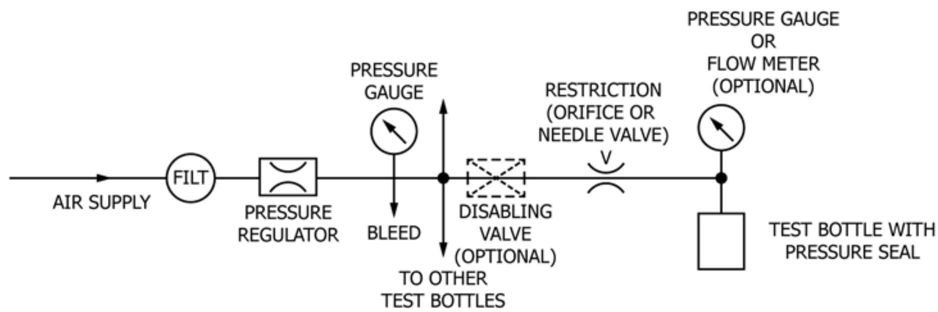


FIG. 1 Apparatus for Procedure C

seal areas; or, in Procedure C, when there is a detectable flow of supply air into the bottle, there is any visible crack completely through the container wall, there is evidence of the contained liquid on the outside of the container through any aperture other than one at the heat-seal area, or the contained liquid volume has been reduced.

3.1.2 *potential stress-cracking liquids*—a liquid that can contain stress-cracking agents, which have the potential to induce a stress crack in the test containers.

3.1.2.1 *Discussion*—Under certain conditions of stress and in the presence of potential stress-cracking liquids such as soaps, wetting agents, oils, or detergents, ethylene plastics may exhibit mechanical failure by cracking.

3.1.3 *stress crack*—defined as a failure.

4. Summary of Test Method

4.1 Procedure A consists of exposing any filled, sealed, blow-molded container to the action of a potential stress-cracking agent within the container, at an elevated temperature. The time to failure is observed.

4.2 Procedure B consists of exposing a sealed blow-molded standard container, partly filled to one third of overflow capacity, to the action of polyoxyethylated nonylphenol, a stress-cracking agent, within the container, as well as to the action of this agent as an external environment, at an elevated temperature. The time to failure is observed.

4.3 Procedure C consists of exposing a blow-molded standard container, partly filled to one fourth of overflow capacity, to the action of polyoxyethylated nonylphenol, a stress-cracking agent, within the container, as well as to a constant elevated pressure internally applied and at an elevated temperature. The time-to-failure can be determined in a tactual-visual manner, or instrumentally.

NOTE 3—Partial filling, that is, one third of nominal capacity, has been found to increase the severity of the test with many test liquids. Thus, the partial fill can be used to accelerate the test. The use of an elevated controlled pressure as in Procedure C can also accelerate the test.

5. Significance and Use

5.1 When properly used, these procedures serve to isolate such factors as material, blow-molding conditions, post-treatment, and so forth, on the stress-crack resistance of the container.

5.2 Environmental stress cracking of blow-molded containers is governed by many factors. Since variance of any of these factors can change the environmental stress-crack resistance of the container, the test results are representative only of a given test performed under defined conditions in the laboratory. The reproducibility of results between laboratories on containers made on more than one machine from more than one mold has not been established.

5.3 Results can be used for estimating the shelf life of blow-molded containers in terms of their resistance to environmental stress cracking provided this is done against a rigorous background of practical field experience and reproducible test data.

6. Apparatus

6.1 *For Procedures A, B, and C:*

6.1.1 *Circulating-Air Oven,* consistent with ovens prescribed in Specification E145, except for size, capable of maintaining a temperature of 60 ± 1°C (140 ± 1.8°F) and an airflow rate of 8.5 to 17 m^3/min (300 to 600 ft^3/min). (**Warning—** A high-temperature safety switch is highly recommended on this oven. Some test liquids can cause extreme pressure buildup upon heating. Under these conditions bottles can rupture with explosive force. This condition can cause injury to the operator as well as damage to the ovens. The override cutoff switch should be set to turn off the oven heat if the test temperature is exceeded by as much as 10°C (18°F).)

6.1.2 *Balance,* accurate to within ±1.0 g (for weighing containers and contents) or a volumetric filling apparatus accurate to ±1 mL.

6.2 *For Procedures A and B Only:*

6.2.1 *Heat-Seal Laminate* for sealing the containers.

6.2.2 *Heat-Sealing Unit.*

6.2.3 *Torque Meter.*

6.2.4 *Glass Beakers,* large enough to hold the contents of one test container.

6.3 *For Procedures A and C Only:*

6.3.1 *Polyethylene Bags,* approximately 0.038-mm (1.5-mil) thick, large enough to enclose completely a test container. The bag should fit loosely around the container and be long enough so that the bag opening can be closed above the container closure.

6.4 *For Procedure C Only:*

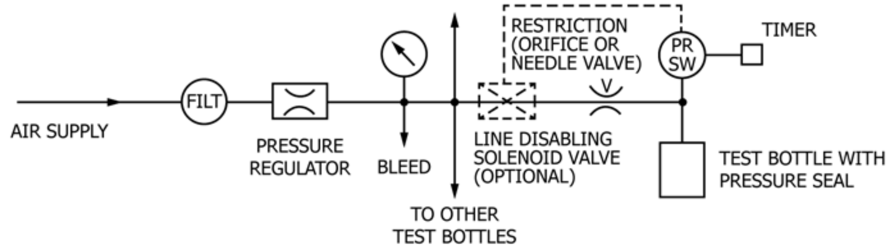


FIG. 2 Apparatus for Procedure C, Including Refinements in Failure Detection

6.4.1 The essential parts of this apparatus are schematically shown in Fig. 1. Additional refinements in failure detection can be added as shown in Fig. 2. The *necessary* equipment is as follows:

6.4.1.1 *Clear Air Supply* of sufficient pressure to operate regulator and maintain regulated pressure to manifold.

6.4.1.2 *Air Filter,* to remove oil, water, dust, and other contaminants.

6.4.1.3 *Pressure Regulator,* to reduce line pressure to 34.5 ± 1.72 kPa (5.0 ± 0.25 psig).

6.4.1.4 *Pressure Gauges,* calibrated to indicate a pressure of 34.5 kPa (5.0 psig) with a precision of 0.34 kPa (0.05 psig).

NOTE 4—A non-mercury manometer is of benefit in calibrating the pressure gauges, and monitoring precise pressure measurements.

6.4.1.5 *Air Valves.*

6.4.1.6 *Restricting Line Orifice or Needle Valve*—This restriction retards the flow of air to the bottle so that supply pressure remains constant after bottle failure, enabling a number of bottles to be pressurized from a single regulated supply. Pressure drop on the bottle side of this restriction is one indication of bottle failure. The orifice size or restriction used will depend upon the sensitivity of the pressure switch or gauge. Orifices that pass 300 cm^3 /min at 6.9 kPa (1 psi) differential pressure have been found satisfactory. Needle valves, which can be adjusted to flow rates as low as 5.0 cm^3/min, can be useful in cases where greater sensitivity to small failures is desired.

6.4.1.7 *Bottle Cap Assemblies*—Each bottle must be securely sealed and attached to the test fixture. Assemblies essentially like those shown in Fig. 3 have been found satisfactory.

7. Reagents

7.1 *For Procedure A*—Any reagent or proprietary liquid that is potentially an environmental stress-cracking agent.

7.2 *For Procedure B:*

7.2.1 *Polyoxyethylated Nonylphenol (CAS 68412-54-4),* a stress-cracking agent.

NOTE 5—Polyoxyethylated nonylphenol is hygroscopic and the undiluted agent should be kept tightly stoppered.

7.2.2 *Polyoxyethylated Nonylphenol Solution*—Prepare a 10 % solution, by volume, of the stress-cracking agent in distilled or deionized water in sufficient volume to fill a minimum of fifteen 473-mL (16-oz) containers to one third of overflow capacity (178 mL).

NOTE 6—It has been found to be helpful due to the viscosity of the stress-cracking agent, to prepare the solution at an elevated temperature. A temperature of 50°C (120°F) has been found suitable.

7.2.3 *Dye Indicator Solution*—Add 0.1 % by weight of a wetting agent (Dioctyl sodium sulfosuccinate—CAS Number 577-11-7) to distilled or deionized water. Dissolve 0.001 % by weight of Gentian Violet in the water.

NOTE 7—Since only about 0.1 mL (2 drops) of this solution is added to each bottle, only a small volume is needed.

7.3 *For Procedure C:*

7.3.1 *Polyoxyethylated Nonylphenol,* a stress-cracking agent. See Note 5.

7.3.2 *Polyoxyethylated Nonylphenol Solution*—Prepare a 33⅓ % solution by volume, of the stress-cracking agent in distilled or deionized water in sufficient volume to fill a minimum of fifteen 473-mL (16-oz) containers to one fourth of the overflow capacity (133 mL). See Note 6.

8. Safety Precautions

8.1 Proper precautions are required to prevent overheating of the containers during testing since some products tested by Procedure A can create an extreme pressure buildup in the container causing the container to rupture explosively. Proper safety measures against over-heating are described in the warning note at the end of 6.1.1.

8.2 A container can also fail by means of a small pinhole. Since the container is under pressure during the test, liquid can be forced out of the opening spraying the inside of the oven and the operator, if an inspection is being made. Precautions to prevent this from happening are described in 11.1.5.

8.3 Care it to be taken in handling the stress-cracking agent since there is a possibility of its causing dermatitis.

8.4 Proper precautions are to be taken in handling compressed air equipment when following Procedure C.

9. Test Specimen

9.1 *For Procedure A*—A minimum of 15 blow-molded containers, representative of the lot to be tested, and fitted with a screw closure affording a leakproof seal, shall be selected.

9.2 *For Procedures B and C*—A standard blow-molded container shall be used for this test. It is a 473-mL (16-oz) cylindrical bottle weighing approximately 20 g, as shown in Fig. 4. A minimum of 15 containers shall be selected as in 9.1. The minimum wall thickness of the container shall be not less than 0.305 mm (12 mil). The pinch-off area of the container shall not extend into the chime radius.

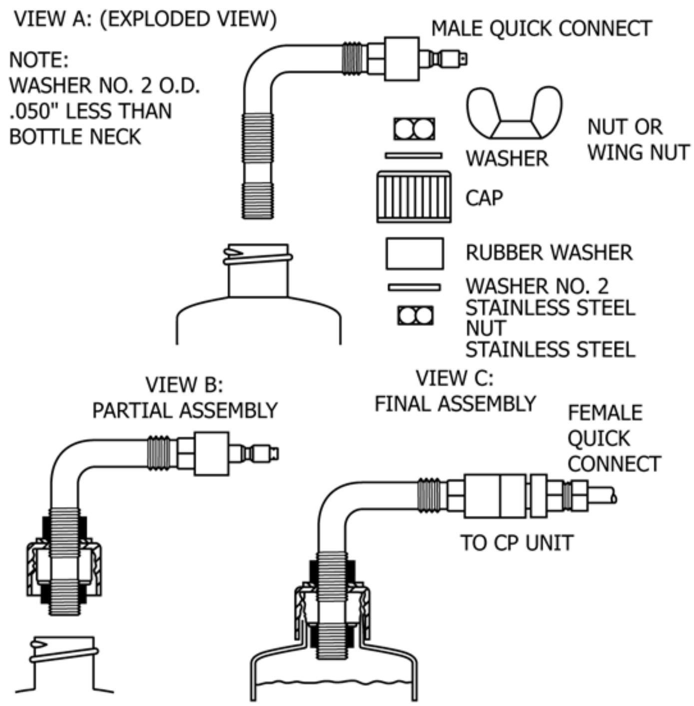
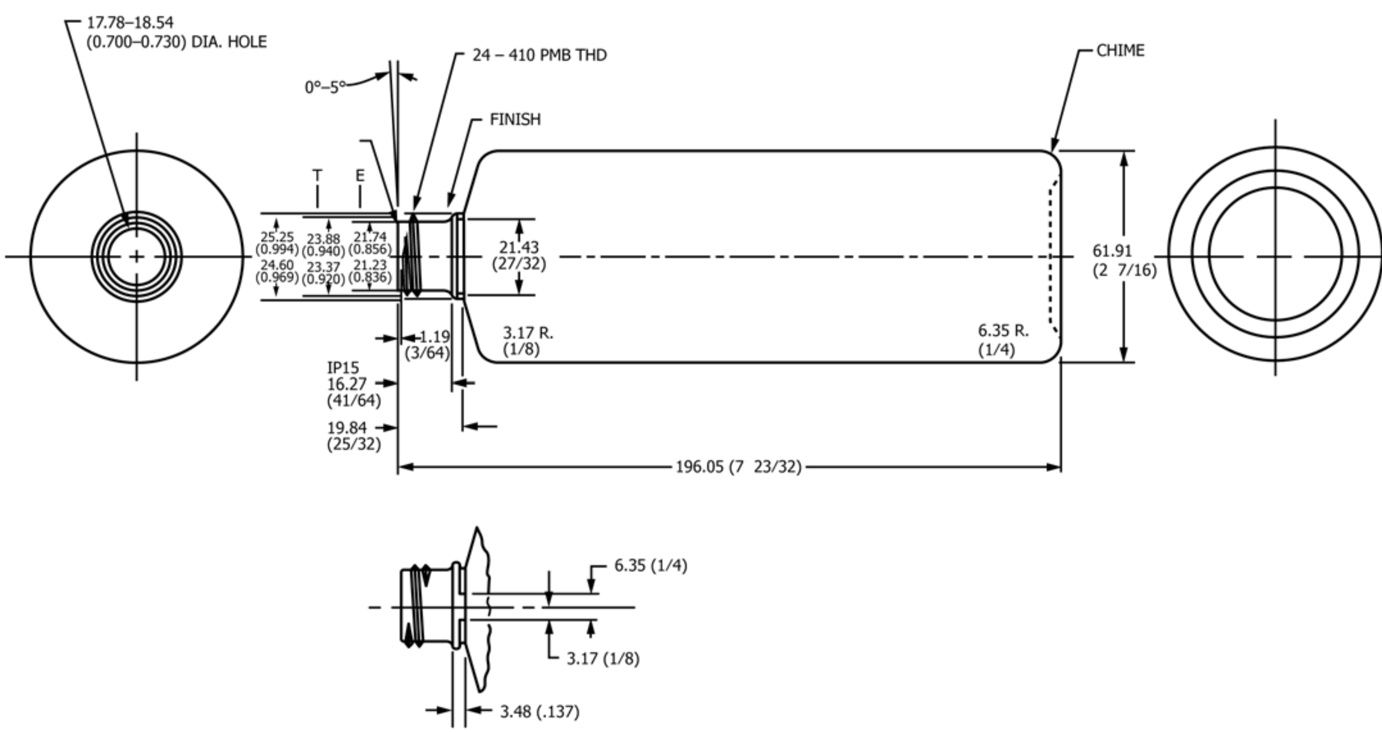


FIG. 3 Bottle Pressure Seal and Tube



NOTE 1—Dimensions are in millimetres with inches in parentheses.

FIG. 4 Standard 473-mL (16-oz) Blown Container

NOTE 8—Test Methods D5947, modified to use a ball tip micrometer, can be used to measure the thickness of the container.

10. Conditioning

10.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) for not less than 40 h prior to test in accordance with Procedure A of Practice D618 unless instructed otherwise. In cases of disagreement, the tolerance shall be ±1°C (±1.8°F).

10.2 *Test Conditions*—Conduct all tests at 60°C, unless instructed otherwise.

11. Procedure

11.1 *Procedure A:*

11.1.1 Obtain a minimum of 15 containers of the type and size chosen for test. Fill each to nominal capacity with the chosen test liquid, usually a potential stress-cracking liquid or an aqueous solution of polyoxyethylated nonylphenol. Avoid spilling the solution on the outside of the container since this might lead to premature failure.

11.1.2 Heat-seal the containers with a suitable triple laminate, polyethylene side to bottle. Apply a polyethylene or suitably lined closure with sufficient torque to ensure a double seal.

11.1.3 Apply minimum pressure during sealing and handling to ensure no deformation of the container.

NOTE 9—Any deformation of the container during sealing could result in a volume change, which will affect the final test pressure. An application torque of 1.7 N·m (15 in·lbf) has been found sufficient for the standard container described in Fig. 4.

11.1.4 After sealing, invert the containers to coat the inside walls with the agent.

11.1.5 It is recommended that, if practical, each container be placed in a polyethylene bag or other suitable containment device and close the bag opening above the closure by folding or by means of a rubber band, string, tape, etc. Do not heat-seal the bag. (**Warning—**A polyethylene bag or other suitable containment device is used to protect the other containers on test from the possibility of one container failing and spraying the other containers. It also protects the operator during inspection of the containers.)

11.1.6 Place the containers in a vertical position with the finish up in the oven at the test temperature of 60 ± 1°C (140 ± 1.8°F). Check the temperature periodically for constancy.

11.1.7 Inspect the containers for environmental stress-crack failure hourly for the first 8 h and thereafter at least once each 24 h. Remove containers that fail and record for each, failure exposure time, position of failure with relation to mold number or other reference point such as a parting line, and type of failure.

NOTE 10—It is not necessary to remove the plastic bag to inspect the containers. Failures are easily detectable with the bag in place.

NOTE 11—During each inspection for failures, it is recommended that the bottles remaining on test be moved in a random manner to new positions in the oven to eliminate any effect due to a static oven temperature profile, if one does exist.

11.1.8 Continue exposure of non-failures until all fail, or to a maximum of 360 h, and record the number of containers still under test at that time.

11.2 *Procedure B:*

11.2.1 Fill a minimum of 15 containers to one third of overflow capacity (178 mL) with the stress-cracking solution described in 7.2.2. Avoid spilling the solution on the outside of the container since this might lead to premature failure.

11.2.2 Put approximately 0.1 mL (2 drops) of the dye solution (7.2.3) in each container.

11.2.3 Heat-seal and invert the filled containers as in Procedure A, 11.1.2 to 11.1.4.

11.2.4 Place the containers in the oven at the test temperature of 60 ± 1°C (140 ± 1.8°F) in a vertical position with the finish up, in beakers containing sufficient undiluted stress-cracking agent (7.2.1) to cover the chime area of the container. Check the temperature periodically for constancy.

11.2.5 Inspect containers for failure to a maximum exposure time of 360 h as detailed in Procedure A, 11.1.7 and 11.1.8.

11.3 *Procedure C:*

11.3.1 Fill a minimum of 15 containers to one fourth of overflow capacity (133 mL) with the stress-cracking solution described in 7.3.2. Avoid spilling the solution on the outside of the container since this might lead to premature failure.

11.3.2 Place the cap assembly (Fig. 3) on each bottle and force the rubber stopper into the bottle far enough to start the cap. Avoid forcing the stopper in too far, or bending the copper tubing.

11.3.3 Invert the container while holding a finger over the opening to coat the inside walls completely with the agent.

11.3.4 Connect the cap assembly to the bottle tester, noting the test position of each bottle. It is recommended that, if practical, each bottle assembly be placed in a polyethylene bag or other suitable containment device as described in 11.1.5. See also the warning note at the end of 11.1.5.

11.3.5 Close the oven door to maintain the proper temperature while the remaining bottles are assembled.

11.3.6 Set the pressure regulator at minimum pressure, that is, unscrew the handle until there is no pressure against the diaphragm.

11.3.7 Adjust the line pressure to 69 kPa (10 psi) and then adjust bottle pressure to 34.5 ± 1.72 kPa (5.0 ± 0.25 psi).

NOTE 12—Since an error exceeding 1.72 kPa (0.25 psi) can be critical, it is essential that the pressure gauge be accurate and that it is read properly. Another critical variable is temperature. The temperature of the air near the bottles should be checked and maintained at 60 ± 1°C (140 ± 1.8°F).

11.3.8 Record the starting time. After 5 min, check to make sure no bottle cap assemblies are leaking. The containers must be connected to the source of pressure during the test in order to ensure that the pressure remains 34.5 ± 1.72 kPa (5.0 ± 0.25 psig).

NOTE 13—Leaking cap assemblies can be checked by immersing the bottles on test in water to a level above the seal. See Fig. 3.

11.3.9 Inspect containers for failure to a maximum exposure time of 360 h as detailed in Procedure A, 11.1.7, and 11.1.8.

NOTE 14—Automatic timing equipment is useful to record the failure time of the containers.

12. Calculation

12.1 Calculate the percentage of the containers that have failed at any given time by the equations in 12.1.1 or in 12.1.2, depending upon the number of containers tested and upon the frequency of inspection.

12.1.1 For 30 or more containers tested or where inspection for failures are made only once every 24 h after the first 8 h, or both:

$$\text{Failures, \%} = (n/N) \times 100 \quad (1)$$

where:

n = cumulative number of containers that have failed at the given time, and

N = number of containers tested.

12.1.2 For less than 30 containers tested *and* where inspections for failures are made more frequently than regular 24-h intervals after the first 8 h, determine the percentage of the containers that have failed at any given time by the equation in 12.1.1, except that the divisor is $N + 1$ instead of N.[3]

12.2 *F_{50} Failure Time:*

12.2.1 Plot the data on a log probability graph with hours on the log scale and percentage failure on the probability scale. Draw the best fitting straight line for the plot. The hours indicated at the intersection of the data line with the 50 % failure level probability line shall be reported as the F_{50} failure time.

12.2.2 At least one half of the containers must have failed before an F_{50} value can be reported.

12.3 If extremes of the distribution need to be studied, additional testing will be necessary.

13. Report

13.1 Report the following information:

13.1.1 Procedure that was used (Procedure A, B, or C) and test temperature, if different from 60°C,

13.1.2 Complete identification and description of the containers tested, including base resin, blow-molding conditions, coloring system, weight, any unusual material distribution, description of geometry, and any other available information,

13.1.3 For Procedure A, description of the test liquid used and the percentage fill of the containers,

13.1.4 Number of bottles tested,

13.1.5 F_i, the time of the first observed failure,

13.1.6 F_{50}, the estimated time at which 50 % of the containers failed as determined from the plot described in Section 12, Calculation.

13.1.7 F_{100}, the observed time in hours at which 100 % of the containers failed, or if all containers did not fail in 360 h, report F_{100} as >360 h,

13.1.8 Locations and types of failure, and

13.1.9 Any unusual occurrences noted during testing.

14. Precision and Bias

14.1 *Procedure B:*

14.1.1 Reproducibility was determined for Procedure B for containers made from a Class 3 polyethylene (Specification D4976). The containers were made from a single laboratory from a single mold and tested in seven different laboratories. The reproducibility that was achieved between laboratories for F_{50} values was $\times\!/\!\div$ 1.81 when expressed as two-sigma limits.

14.1.2 The reproducibility that can be achieved in a single laboratory between groups of containers, from the same mold, and the same raw material, has not been determined.

14.2 *Procedure C*—Reproducibility was determined for Procedure C for containers made from Class 3 polyethylene (Specification D4976). The containers were made in a single laboratory from a single mold and tested in five different laboratories. The reproducibility that was achieved between laboratories for F_{50} values was $\times\!/\!\div$ 1.58 when expressed as two-sigma limits. The repeatability that was achieved in a single laboratory from the same mold and the same raw material for $F_{5\ 0}$ values was $\times\!/\!\div$ 1.58 when expressed as two-sigma limits.

NOTE 15—The within (intra) and between (inter) laboratory two-sigma limits of $\times\!/\!\div$ 1.58 for F_{50} means that if a single laboratory reported a single F_{50} value of 10 h then one expects that repeated evaluations from the same population of bottles at this laboratory would be between 15.8 and 6.3 h, 95 % of the time. If one laboratory reports an F_{50} of 10 h for the average of many specimens, then another laboratory's F_{50} average of many specimens would be between 15.8 and 6.3 h, 95 % of the time. The number 15.8 comes from multiplying 1.58 by 10 and 6.3 comes from dividing 10 by 1.58.

NOTE 16—The precision may be improved by adding automatic recording devices to monitor a specified bottle failure. Such failures may be indicated by a pressure drop or by gas flow to the bottle. See Fig. 1 and Fig. 2.

15. Keywords

15.1 blow-molded containers; environmental stress-crack; plastic bottles; polyethylene containers

[3] E. J. Gumbel, "Statistical Theory of Extreme Values and Some Practical Applications," National Bureau of Standards Applied Mathematics Series 33, Feb. 12, 1954, pp. 13–15.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D2561 - 12) that may impact the use of this standard. (May 1, 2017)

(1) Revised 1.2 to clarify that the method measures the environmental stress crack resistance of blow-molded containers.
(2) Revised 1.2.1 to replace 'commercial liquids' with 'Potential Stress-cracking liquids' and removed reference to commercial package and proprietary liquid products.
(3) Revised 1.5 to make reference to Note 1 instead of Note 2 and removed reference to Note 9.
(4) Revised section 3 terminology to define potential stress-crack liquids and stress cracks.
(5) Revised section 11.1.1 to align it with the modified 1.2.1 section.
(6) Replaced the word 'may' with alternative language where appropriate.