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

This standard is issued under the fixed designation D 2561; 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 may 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 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 Commercial 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 commercial package containing a proprietary liquid product.

1.2.2 *Procedure B, Stress-Crack Resistance of a Specific Container to Polyoxyethylated Nonylphenol, a Stress-Cracking Agent*—The conditions of test described in this procedure are designed for testing containers made from Type III polyethylene Specification D 1248. Therefore, this procedure is recommended for containers made from Type III 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, a Stress-Cracking Agent*—The internal pressure is controlled at a constant elevated level.

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 the standard.

NOTE 1—There is no similar or equivalent ISO 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 and Note 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 374 Test Methods for Thickness of Solid Electrical Insulation[2]

D 618 Practice for Conditioning Plastics for Testing[3]

D 1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.2 *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. A container has failed when:

3.2.1 It has lost pressure through any aperture other than heat seal areas; or, in Procedure C, when there is a detectable flow of supply air into the bottle,

3.2.2 There is visible to an observer with normal eyesight any crack completely through the container wall, or

3.2.3 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.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.20 on Plastic Products.

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[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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.

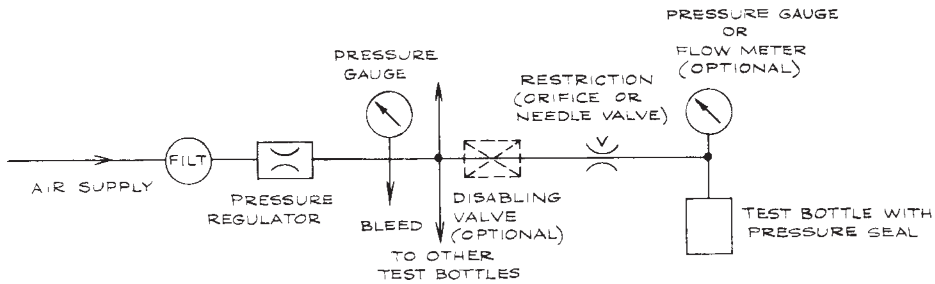


FIG. 1 Apparatus for Procedure C

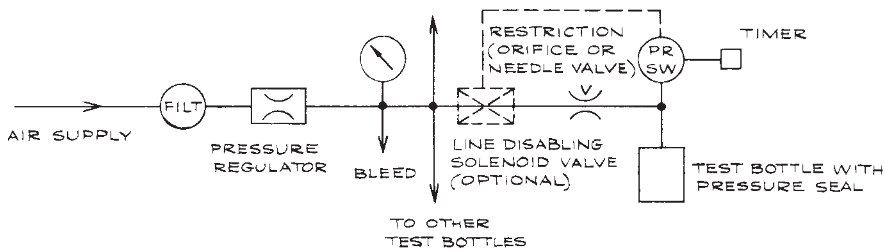


FIG. 2 Apparatus for Procedure C, Including Refinements in Failure Detection

4.2 Procedure B consists of exposing a partly filled and sealed blow-molded standard container to the action of polyoxyethylated nonylphenol,[5] 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 partly filled blow-molded standard container 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.

5. Significance and Use

5.1 When properly used, these procedures may serve to isolate such factors as material, blow-molding conditions, post-treatment, etc., 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.

5.4 Before proceeding with this test method, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or a combination thereof, covered in the materials specification shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply.

6. Apparatus

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

6.1.1 *Circulating-Air Oven*, consistent with ovens prescribed in Specification E 145, 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).

NOTE 2—**Caution:** 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*[6] for sealing the containers.

[5] Igepal CO-630 (Antarox CO-630) obtained from GAF Corp., Dyestuff and Chemical Div., 140 W. 51 St., New York, NY 10020.

[6] A suitable polyethylene-foil laminate (aluminum seal) is available from Berlin Packaging, 111 North Canal St., Suite 300, Chicago, IL 60606.

6.2.2 *Heat-Sealing Unit.*[7]

6.2.3 *Torque Meter.*[8]

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*:

6.4.1 The essential parts of this apparatus are schematically shown in Fig. 1. Additional refinements in fail 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 Gages*, calibrated to indicate a pressure of 34.5 kPa (5.0 psig) with a precision of 0.34 kPa (0.05 psig).

NOTE 3—A mercury manometer is of benefit in calibrating the pressure gages, 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 gage. Orifices that pass 300 cm^3 /min at 6.9 kPa (1 psi) differential pressure have been found satisfactory. Needle valves, which may be adjusted to flow rates as low as 5.0 cm^3 /min, may 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.

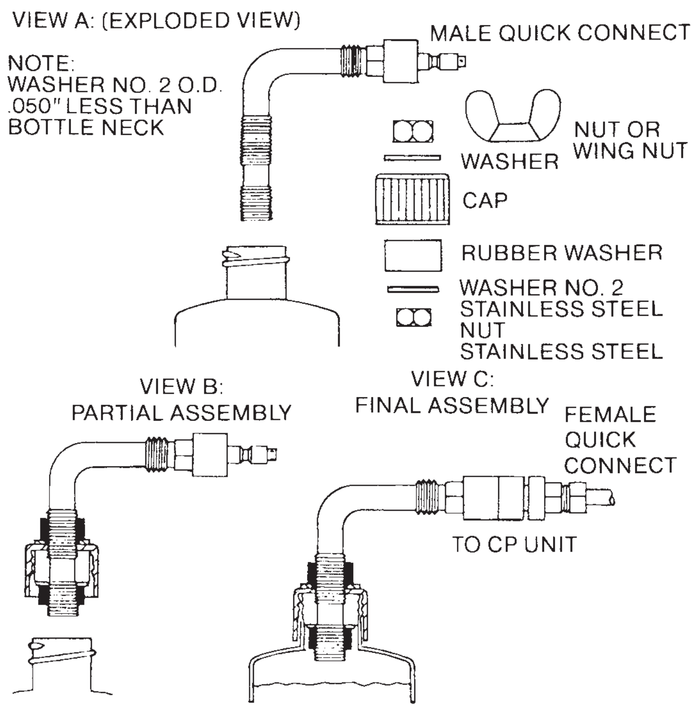


FIG. 3 Bottle Pressure Seal and Tube

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*, a stress-cracking agent.[5]

NOTE 4—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 water in sufficient volume to fill a minimum of fifteen 473-mL (16-oz) containers to one third of overflow capacity (178 mL).

NOTE 5—It has been found to be helpful due to the viscosity of the

[7] Selector Hand Sealing Iron (165 W) made by Selector of Shelton, CT, or Super Sealer made by Clamco Div., Cleveland Detroit Corp., 5400 Brookpark Rd., Cleveland, OH, is suitable.

[8] Model 25 Owens-Illinois Spring Torque Tester, Owens-Illinois Glass Co., Toledo, OH, is suitable.

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[9] to distilled water. Dissolve 0.001% by weight of Gentian Violet in the water.

NOTE 6—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.[5] See Note 4.

7.3.2 *Polyoxyethylated Nonylphenol Solution*—Prepare a 33⅓ % solution by volume, of the stress-cracking agent in distilled 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 5.

8. Safety Precautions

8.1 Proper precautions should be taken to prevent overheating of the containers during testing since some products which may be tested by Procedure A may cause an extreme pressure buildup in the container and could cause the container to rupture explosively. Proper safety measures against overheating are described in Note 2.

8.2 Sometimes a container will fail by means of a small pinhole. Since the container is under pressure during the test, liquid may 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 11.1.4 and Note 10.

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

8.4 Proper precautions should 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 9.1.1. 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.

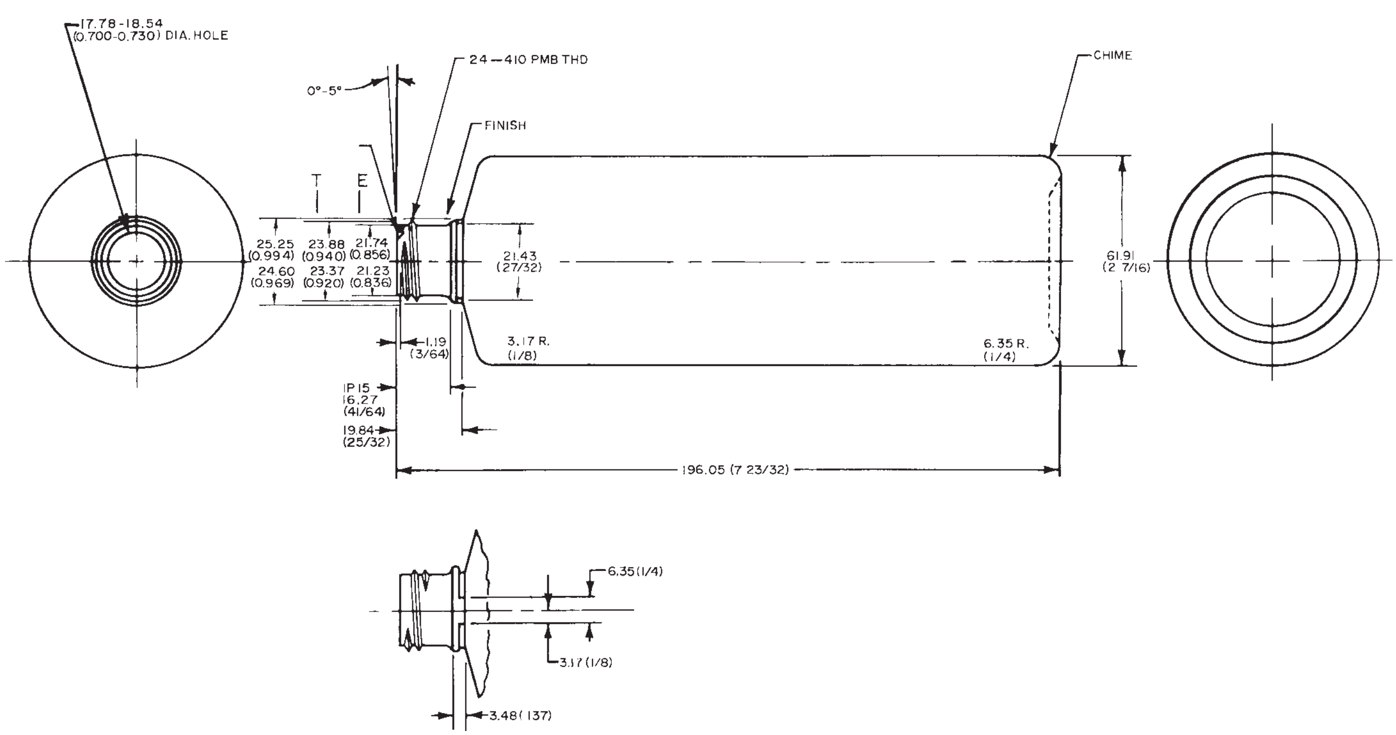
NOTE 7—Test Methods D 374, modified to use a ball tip micrometer, may be used to measure the thickness of the container.

10. Conditioning

10.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5% relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ±1°C (±1.8°F) and ±2% relative humidity.

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

[9] Aerosol OT Solution from Fisher Scientific Corp., 203 Fisher Building, Pittsburgh, PA 15219, has been found suitable.



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

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

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 proprietary liquid product or an aqueous solution of polyoxyethylated nonylphenol.

NOTE 8—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 may be used to accelerate the test. The use of an elevated controlled pressure as in Procedure C may also accelerate the test.

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.

NOTE 9—The pressure applied during sealing should be a minimum to ensure no deformation of the container. The container should not be handled in such a manner as to deform the walls during sealing. Any deformation of the container during sealing may 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.

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

11.1.4 Place each container in a plastic bag 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.

NOTE 10—**Caution:** A plastic bag 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.

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

11.1.6 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 parting line, and type of failure.

NOTE 11—It is not necessary to remove the plastic bag to inspect the containers. Failures are easily detectable with the bag in place.

NOTE 12—During each inspection for failures, the bottles remaining on test may 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.

11.1.7 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.

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 and 11.1.3.

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, 11.1.6 and 11.1.7.

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. Enclose each bottle assembly in a plastic bag as described in 11.1.4. See also Note 10.

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 13—Since an error exceeding 1.72 kPa (0.25 psi) can be critical, it is essential that the pressure gage 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 14—Leaking cap assemblies may 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, 11.1.6, and 11.1.7.

NOTE 15—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:

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

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.[10]

12.2 F_{50} *Failure Time*:

12.2.1 Plot the data on log probability graph paper[11] 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 Type III polyethylene (Specification D 1248). 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 Type III polyethylene (Specification D 1248). 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 16—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 17—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

[10] 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.

[11] Probability paper is available from Keuffel & Esser Co., No. 359-22G (probability × logarithmic).