Designation: D 2561 – 95

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 and Electrical Insulating Materials for Testing[3]

D 1248 Specification for Polyethylene Plastics Molding and Extrusion Materials[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.

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 partly filled and sealed blow-molded standard container to the action of polyoxyethylated nonylphenol,[5] a stress-cracking agent, within the

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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.

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

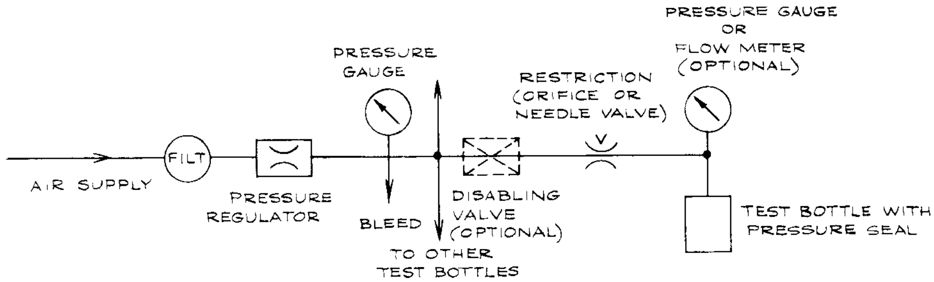

FIG. 1 Apparatus for Procedure C

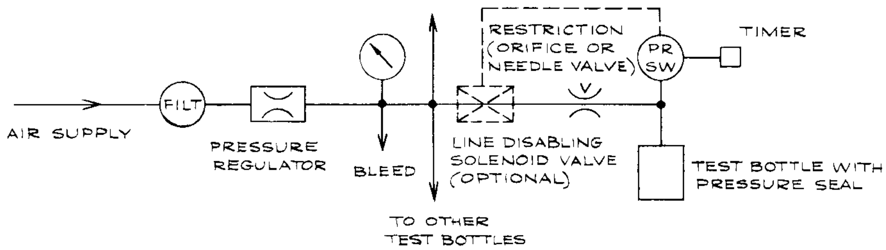

FIG. 2 Apparatus for Procedure C, Including Refinements in Failure Detection

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.

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:

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

[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.

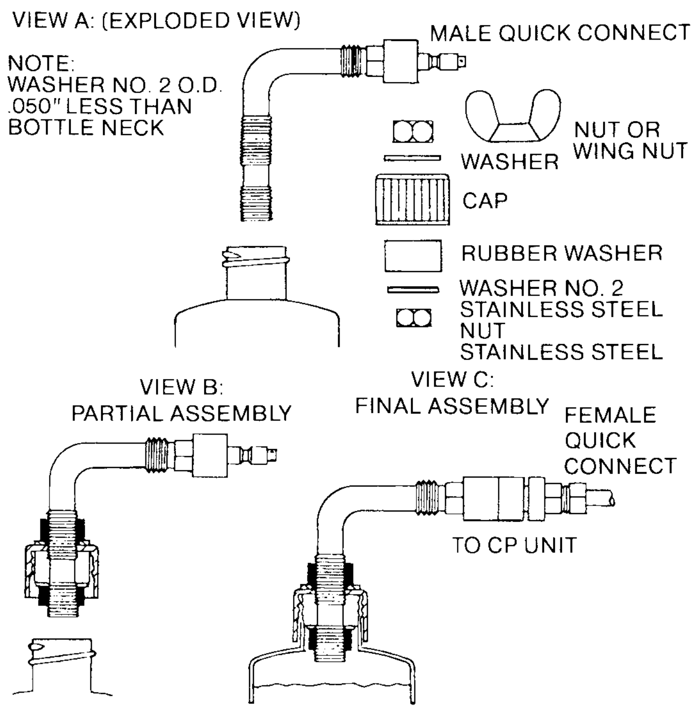

FIG. 3 Bottle Pressure Seal and Tube

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 $\pm$ 1.72 kPa (5.0$\pm$ 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.

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 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

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