



Designation: D2561 – 12

Standard Test Method for Environmental Stress-Crack Resistance of Blow-Molded Polyethylene Containers¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 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 (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 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 Aug. 1, 2012. Published September 2012. Originally approved in 1966. Last previous edition approved in 2005 as D2561 - 95 (2005). DOI: 10.1520/D2561-12.

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 2** and **Note 9**.

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*:

3.2 *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.

3.2.1 *Discussion*—A container has failed when:

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, there is any visible crack completely through the container wall, there is evidence of the contained liquid on the outside of the container through

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

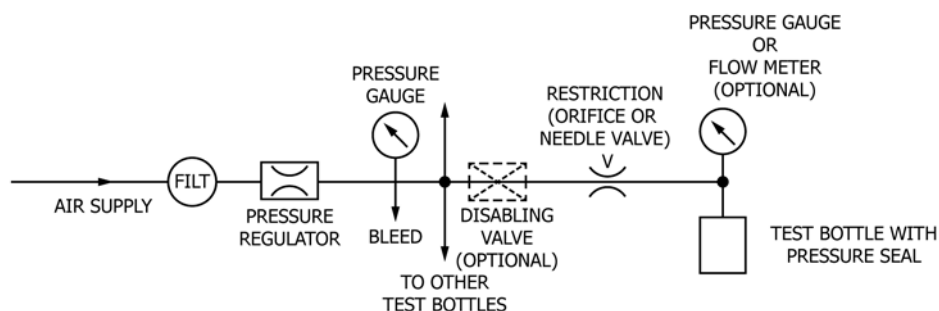


FIG. 1 Apparatus for Procedure C

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 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 $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$) 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:

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 Gages*, 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 gages, and monitoring precise pressure measurements.

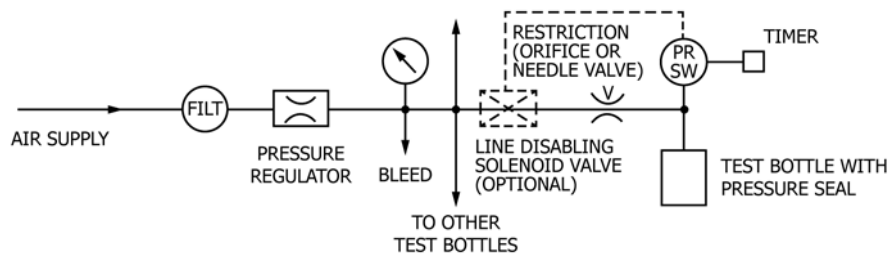


FIG. 2 Apparatus for Procedure C, Including Refinements in Failure Detection

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 cm³/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 cm³/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 (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 33 1/3 % 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.

NOTE 8—Test Methods D5947, 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) 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: