

Designation: D4493 - 08 Designation: D4493 - 08

# Standard Test Method for Solidification Point of Bisphenol A (4,4'-Isopropylidenediphenol)<sup>1</sup>

This standard is issued under the fixed designation D 4493; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

#### 1. Scope

- 1.1This test method describes the procedure for determination of the solidification point of 4,4'-isopropylidene diphenol, commercially known as bisphenol A, between 150 and 157°C.
- 1.2The following applies to all specified limits in this standard: For purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E29\*
- 1.1 This test method describes the procedure for determination of the solidification point of bisphenol A (4,4'-isopropylidene diphenol).
  - 1.2 The method is applicable for determination of the solidification point between 150 and 157°C.
- 1.3 In determining conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E 29.
- 1.3This 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 test method. For specific hazard statements, see Section
- 1.4 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. For specific hazard statements, see Section 9.

## 2. Referenced Documents

- 2.1 ASTM Standards: <sup>2</sup>
- D 1493 Test Method for Solidification Point of Industrial Organic Chemicals
- D 4297 Practice for Sampling and Handling Bisphenol A (4,4'-Isopropylidenediphenol)<sup>2</sup> (4,4 -Isopropylidinediphenol)
- D 6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials
- E 1 Specification for ASTM Liquid-in-Glass Thermometers
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E 77 Test Method for Inspection and Verification of Thermometers
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- 2.2 Other Document:
- OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200 Other Documents:
- OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200 <sup>3</sup>

## 3. Terminology

- 3.1 Definitions:
- 3.1.1 *solidification point*—the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small amount of the same substance in its solid phase.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 06.04.

<sup>&</sup>lt;sup>2</sup> 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.

Annual Book of ASTM Standards, Vol 14.03.

<sup>&</sup>lt;sup>3</sup> Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.



### 4. Summary of Test Method

4.1 Bisphenol A is melted, and then cooled slowly with constant agitation. When crystallization begins, and super-cooling occurs, the temperature falls to a minimum, rises to a maximum, and then falls again. The maximum temperature attained after crystallization begins is the solidification point of bisphenol A.

#### 5. Significance and Use

- 5.1 The solidification point of bisphenol A is a direct indication of its purity, although it gives no information as to the nature of any impurities present.
  - 5.2 High purity bisphenol A has a solidification point of approximately 157°C.
  - 5.3 This test method can be used for internal quality control or for setting specifications.

#### 6. Interference

6.1 Bisphenol A that is not stored or packaged properly may adsorb moisture. Adsorbed moisture will lower the solidification point.

#### 7. Apparatus

- 7.1 Nessler Tubes, borosilicate, 100 mL, short form, 32-mm diameter.
- 7.2 *Electric Heat Block*, thermostatically controlled, capable of reaching 170°C; having flat-bottom holes 34 mm in diameter by 172 mm deep. Note1—AA suitable size-block is 100 by 110 by 175 mm high, and made of aluminum. Note2—AAlternatively, a thermostatically controlled hot oil bath may be used.
  - 7.3 Erlenmeyer Flask, 500-mL.

Note3—The melted sample may be cooled in an air jacket-cooling bath, as specified in Test Method D1493.

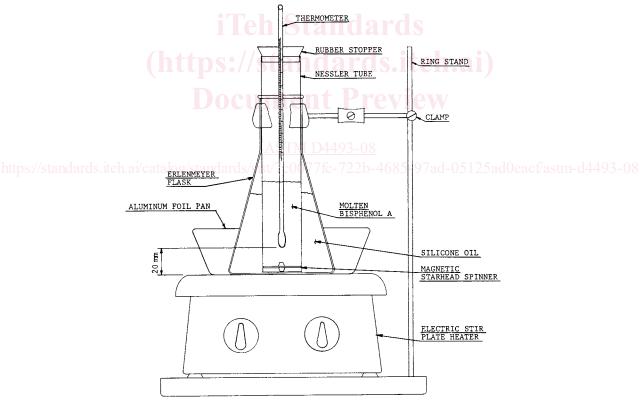


FIG. 1 Bisphenol A Solidification Point Apparatus

7.4Thermometer—ASTM 102C, having a range from 123 to 177°C and conforming to the requirements for thermometer 102C as prescribed in Specification E 1. Note4—Thermometers Thermometers should be calibrated in accordance with Test Method E 77or calibrated from 154 to 157°C versus an NBS thermometer or platinum resistance thermometer. Preferably, thermometers should be calibrated and certified by a thermometer manufacturer. An alternative thermometer is a platinum resistance thermometer with digital read-out.

7.5

7.4 Electric Heater, stir plate, capable of reaching 150°C.