Designation: D4493 – 16

Standard Test Method for Solidification Point of Bisphenol A (4,4′-Isopropylidenediphenol)[1]

This standard is issued under the fixed designation D4493; 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 This test method describes the procedure for determination of the solidification point of bisphenol A (4,4′-isopropylidene diphenol).

1.2 The test method has been found 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 E29.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in 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.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1493 Test Method for Solidification Point of Industrial Organic Chemicals (Withdrawn 2004)[3]

D4297 Practice for Sampling and Handling Bisphenol A(4,4′-Isopropylidinediphenol)

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E1 Specification for ASTM Liquid-in-Glass Thermometers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E77 Test Method for Inspection and Verification of Thermometers

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Documents:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200 [4]

3. Terminology

3.1 *Definitions:*

3.1.1 *solidification point, n*—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.

4. Summary of Test Method

4.1 Bisphenol A is melted, and then cooled slowly with constant agitation. When crystallization begins, and supercooling 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 absorb moisture. Absorbed moisture will lower the solidification point.

[1] 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.

Current edition approved June 1, 2016. Published June 2016. Originally approved in 1985. Last previous edition approved in 2012 as D4493 – 12. DOI: 10.1520/D4493-16.

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] 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.

***A Summary of Changes section appears at the end of this standard**

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. A suitable block is 100 by 110 by 175 mm high, and made of aluminum. Alternatively, a thermostatically controlled hot oil bath may be used.

7.3 *Thermometer*—ASTM 102C, having a range from 123 to 177°C and conforming to the requirements for thermometer 102C as prescribed in Specification E1. Thermometers should be calibrated in accordance with Test Method E77 or 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.

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

7.5 *Magnetic Spinner,* starhead. A wire stirrer, as specified in Test Method D1493, may be used as well.

7.6 *Chloroprene Rubber Stopper,* number 6, with hole to fit thermometer. If wire stirrer is used, an additional hole is needed. Stoppers made of cork or other materials should not be used.

8. Reagents and Materials

8.1 *Methyl Silicone Oil,* suitable for continuous use at 200°C.

9. Hazards

9.1 Consult current OSHA Regulations, supplier's Safety Data Sheets, and local regulations for all materials used in this test method.

9.2 When handling molten solids in open tubes, adequate ventilation must be provided and proper protection should be used to prevent thermal burns. It is preferable to perform this test in a fume hood.

10. Sampling

10.1 Sample the material in accordance with Practice D4297.

11. Procedure

11.1 Place a Nessler tube, filled with bisphenol A, and containing the magnetic spinner, the stopper, and thermometer, in an electric heat block, preheated to 170 ± 5°C, to melt bisphenol A. The solidification point is determined on the specimen as received, with no drying procedure.

11.2 As the bisphenol A melts, add more to the Nessler tube, if necessary, so that the immersion requirement of the thermometer will be met. It takes approximately 30 to 45 min to melt enough bisphenol A to run the test. In order to minimize the loss of volatile components, it is advisable to begin the solidification point determination within 5 min after the bisphenol A is molten.

11.3 After the bisphenol A has melted, remove the Nessler tube from the heat block and place in a 500 mL Erlenmeyer flask, which has been clamped to a ring stand (see Fig. 1). It

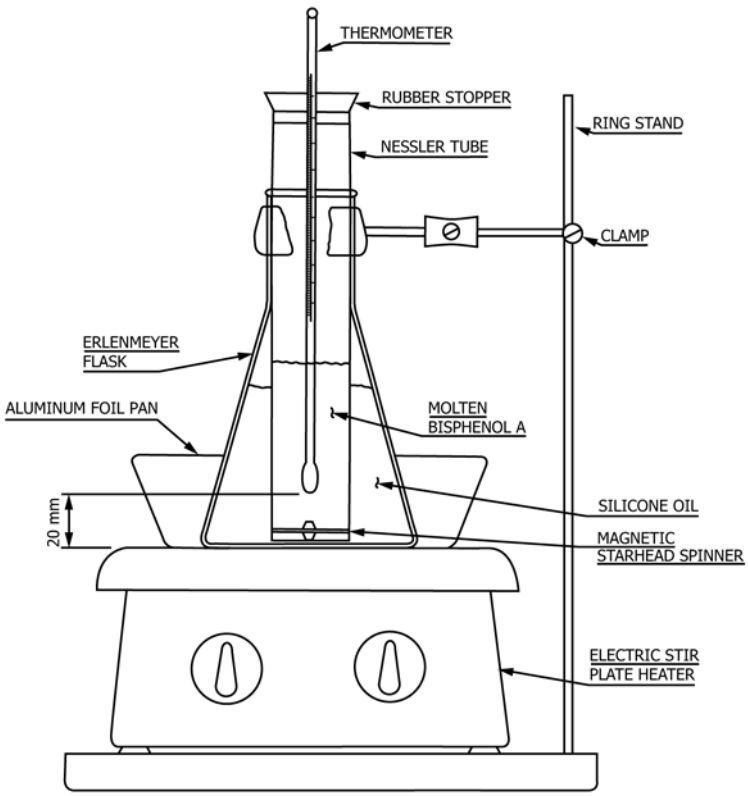

FIG. 1 Bisphenol A Solidification Point Apparatus

may be necessary to wrap aluminum foil around the top portion of the Nessler tube before placing in the Erlenmeyer flask to prevent the tube from turning in the flask. The flask contains 400 mL of silicone oil that has been preheated to 140 ± 2°C, and is on a heater-stir plate. It is advisable to set the flask in a small aluminum foil pan to catch the oil in the event of a flask failure. Alternatively, the melted sample may be cooled in an air jacket-cooling bath, as specified in Test Method D1493.

11.4 Submerge the thermometer to the immersion mark and ensure that the bulb of the thermometer is approximately 20 mm above the bottom of the Nessler tube, clearing the magnetic spinner.

11.5 Start the magnetic spinner stirring at a rate to create a vortex, and continue stirring until the liquid becomes solid enough to prevent the spinner from stirring. The cooling rate should be adjusted to maintain a constant temperature for about 3 min. The cooling rate may or may not be critical, depending upon the product purity.

11.6 Observe and record the thermometer readings at 30-s intervals to the nearest 0.1°C until the temperature rises from the minimum, due to super cooling, to a maximum, and finally begins to drop. Taking temperature readings 30 s apart will ensure against mistaking a temporary plateau for the maximum temperature. Plotting the temperature readings against time will also help to identify a temporary plateau. Further stirring will be impossible at this point. The maximum temperature after crystallization begins is the solidification point.

11.7 Correct the observed solidification point for the calibration of the thermometer.

12. Report

12.1 Report the solidification point as the maximum temperature attained after crystallization begins. Report to the nearest 0.1°C.

13. Precision and Bias[5]

13.1 *Precision*—The following criteria should be used to judge the acceptability at the 95 % probability level of the results obtained by this test method. The criteria were derived from an interlaboratory study conducted by four laboratories to determine solidification points on three separate materials. Duplicates were run by each laboratory on two different days. The details are given in ASTM Research Report RR:D16-1008. Results of the interlaboratory study were calculated using Practice E691, although it should be noted the size of the sample set did not meet the minimum requirements of Practice E691.

13.1.1 *Repeatability*—Results should not be suspect unless they differ by more than 0.5°C. Results differing by less than 0.5°C have a 95 % probability of being correct.

13.1.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 1.8°C. Results differing by less than 1.8°C have a 95 % probability of being correct.

13.2 *Bias*—Since there is no accepted reference material or alternate test method suitable for determining the bias in this test method, bias has not been determined.

14. Quality Guidelines

14.1 Laboratories shall have a quality control system in place.

14.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

14.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

14.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

14.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

15. Keywords

15.1 bisphenol-A; 4,4′-isopropylidenediphenol; solidification point

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1008. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D4493–12) that may impact the use of this standard. (Approved June 1, 2016.)

(1) Section 6: Used absorb instead of adsorb.

(2) Section 9: Changed MSDS to SDS.