

## StandardTest Method for Solidification Point of Benzene<sup>1</sup>

This standard is issued under the fixed designation D852; 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\*

1.1 This test method covers the determination of the solidification point of benzene.

1.2 In determining the 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.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 7.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

- D1015 Test Method for Freezing Points of High-Purity Hydrocarbons
- D1016 Test Method for Purity of Hydrocarbons from Freezing Points
- D1193 Specification for Reagent Water
- D3437 Practice for Sampling and Handling Liquid Cyclic Products
- 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

# E691 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<sup>3</sup>

#### 3. Terminology

3.1 *Definitions:* 

3.1.1 *solidification point, n*—an empirical constant defined as the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase.

3.1.1.1 *Discussion*—Solidification point is distinguished from freezing point which is described in Test Method D1015. An interpretation of mol percent purity in terms of freezing point is given in Test Method D1016.

#### 4. Summary of Test Method

4.1 Solidification point is measured by noting the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase.

### 5. Significance and Use

5.1 This test method may be used as a criteria for determining the purity of benzene. The closer the solidification point reaches that of pure benzene, the purer the sample.

#### 6. Apparatus

6.1 Benzene Container (Air Jacketed):

6.1.1 *Inner Container*, a test tube 15 mm in outside diameter and 125 mm in length.

6.1.2 *Air Jacket*, a standard test tube 25 mm in outside diameter and 150 mm in length.

6.1.3 Insulation—Dry absorbent cotton or glass wool.

6.2 *Benzene Container (thick walled)*, a glass test tube 18 mm in outside diameter, 14 mm in inside diameter and 150 mm in length. The thick walled tube is only compatible with the thermistor.

<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 D16.01 on Benzene, Toluene, Xylenes, Cyclohexane and Their Derivatives.

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<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.

<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.



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FIG. 1 Benzene Solidification Point Apparatus Set Up

6.3 *Ice Bath*, a 1-L beaker, or similar suitable container, having an effective depth of at least 127 mm and filled with chipped or shaved ice.

6.4 *Stirrer*, consisting of a 1-mm wire (copper or stainless steel) or a 2-mm glass rod with one end bent into a circular form at right angles to the shaft so that it will move freely in the annular space between the thermometer stem and the wall of the smaller test tube.

6.5 *Temperature Measurement Device*, either device described below has been found satisfactory.

6.5.1 *Thermometer*, an ASTM Benzene Freezing Point Thermometer having a range from 4.0 to  $6.0^{\circ}$ C and conforming to the requirements for Thermometer 112C as prescribed in Specification E1.

6.5.2 *Thermistor*; in stainless steel housing with resistance greater than 2K ohms at 25°C. Calibration accuracy 0.01°C. Drift in resistance equivalent to less than  $\pm 0.01$ °C/year. Thermistor shall be calibrated to cover the range it is used.<sup>4</sup>

6.6 *Stirring Apparatus (Optional)*, the apparatus illustrated in Fig. 1 has been demonstrated to be an acceptable replacement for manually stirring the benzene solution.

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the apparatus known to the committee at this time is GE Thermometrics, type CSP A 727X-CSP60BA252M, 967 Windfall Rd., St. Mary's PA 15857. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.