



Designation: ~~D852-02~~ Designation: D 852 - 08

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

This standard is issued under the fixed designation D 852; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 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 E 29.

~~1.3~~

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>

D 1015 Test Method for Freezing Points of High-Purity Hydrocarbons

D 1016 Test Method for Purity of Hydrocarbons from Freezing Points

D 1193 Specification for Reagent Water

D 3437 Practice for Sampling and Handling Liquid Cyclic Products

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

2.2 *Other Document:*

~~OSHA Regulations, 29CFR~~ Other Document:

OSHA Regulations, 29CFR paragraphs 1910.1000 and 1910.1200<sup>3</sup>

### 3. Terminology

3.1 *Definitions:*

3.1.1 *solidification point* 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 D 1015. An interpretation of mol percent purity in terms of freezing point is given in Test Method D 1016.

### 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.

<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>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards*, Vol 05.01, volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.01.

<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>.

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

## 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.

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°C and conforming to the requirements for Thermometer 112C as prescribed in Specification E 1.

6.5.2 *Thermistor*, Type CSP with accuracy of 0.01°C with a nominal resistance at 2500 s at 25°C. The thermistor shall be equipped with an ohm meter capable of reading resistance to the nearest 0.1 s., 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 ± 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.

6.7 *Ohmometer*, capable of measuring resistance to the nearest 0.1 ohm in the range 1000 to 10 000 ohms with direct temperature readout.<sup>4</sup>

## 7. Hazards

7.1 Consult the latest OSHA regulations **Hazards**

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

7.2 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

## 8. Sampling

8.1 Sample the material in accordance with Practice D 3437.

## 9. Preparation of Apparatus

9.1 Fit the benzene container with a two-hole stopper. Through one hole insert the temperature measurement device. The thermometer should be inserted up to the 4.0°C mark. The thermistor should be inserted, so as to contact the benzene solution. Through the other hole insert the shaft of the stirrer.

9.2 If using the benzene container (air jacketed), place a ~~in. (3.2 mm)~~ **3.2-mm** layer of dry absorbent cotton or glass wool in the bottom of the larger test tube and insert the inner container up to the lip into a stopper or annular ring that just fits into the mouth of the air jacket.

## 10. Calibration of Temperature Measuring Device

10.1 Calibration of ASTM thermometer 112C is accomplished with the small scale etched on the lower portion of the thermometer. Prepare an ice bath by filling a small Dewar flask with crushed ice made from Type I or Type II water (as specified in Specification D 1193) and add just enough chilled Type I or Type II water to make a slurry. Immerse the thermometer in the ice bath, allow 5 min for the system to reach equilibrium and read the thermometer. Solidification point values are subsequently adjusted by adding (or subtracting) the number of degrees the thermometer is below (or above) 0.00°C.

10.2 Calibration of the thermistor is performed by the thermistor manufacturer. Resistance is converted to temperature using an equation supplied by the manufacturer.

<sup>4</sup>Annual Book of ASTM Standards, Vol 06.04.

<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, which you may attend.