Designation: D 852 – 00ε1 2

Standard Test Method for Solidification Point of Benzene[1]

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

ε[1] NOTE—RR: D16-1028 was added editorially in March 2002.

1. Scope*

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

1.2 The following applies to all specified limits in this test method: for purposes of

1.2 In determining the conformance with this of the test method, an observed value or a calculated value results using this method to applicable specifications, results 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 E 29.

1.3 *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:*

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

Current edition approved June Aug. 10, 2002. Published August 2000. October 2002. Originally published as D 852 – 45. Last previous edition D 852 – 97 00ε1.

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

D 1015 Test Method for Freezing Points of High-Purity Hydrocarbons[2]
D 1016 Test Method for Purity of Hydrocarbons from Freezing Points[2]
D 1193 Specification for Reagent Water[3]
D 3437 Practice for Sampling and Handling Liquid Cyclic Products[4]
E 1 Specification for ASTM Thermometers[5]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[6]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

2.2 *Other Document:*
OSHA Regulations, 29CFR, paragraphs 1910.1000 and 1910.1200[7]

3. Terminology

3.1 *Definitions:*
3.1.1 *solidification point*—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.

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 ~~Ω~~ s at 25°C. The thermistor shall be equipped with an ohm meter capable of reading resistance to the nearest 0.1 ~~Ω.~~ s.
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.

7. Hazards

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

8. Sampling

8.1 Sample the material in accordance with Practice D 3437.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.
[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] *Annual Book of ASTM Standards*, Vol 06.04.
[5] *Annual Book of ASTM Standards*, Vol 14.03.
[6] *Annual Book of ASTM Standards*, Vol 14.02.
[7] Available from ~~Superintendent of Documents,~~ U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20402~~1~~1.

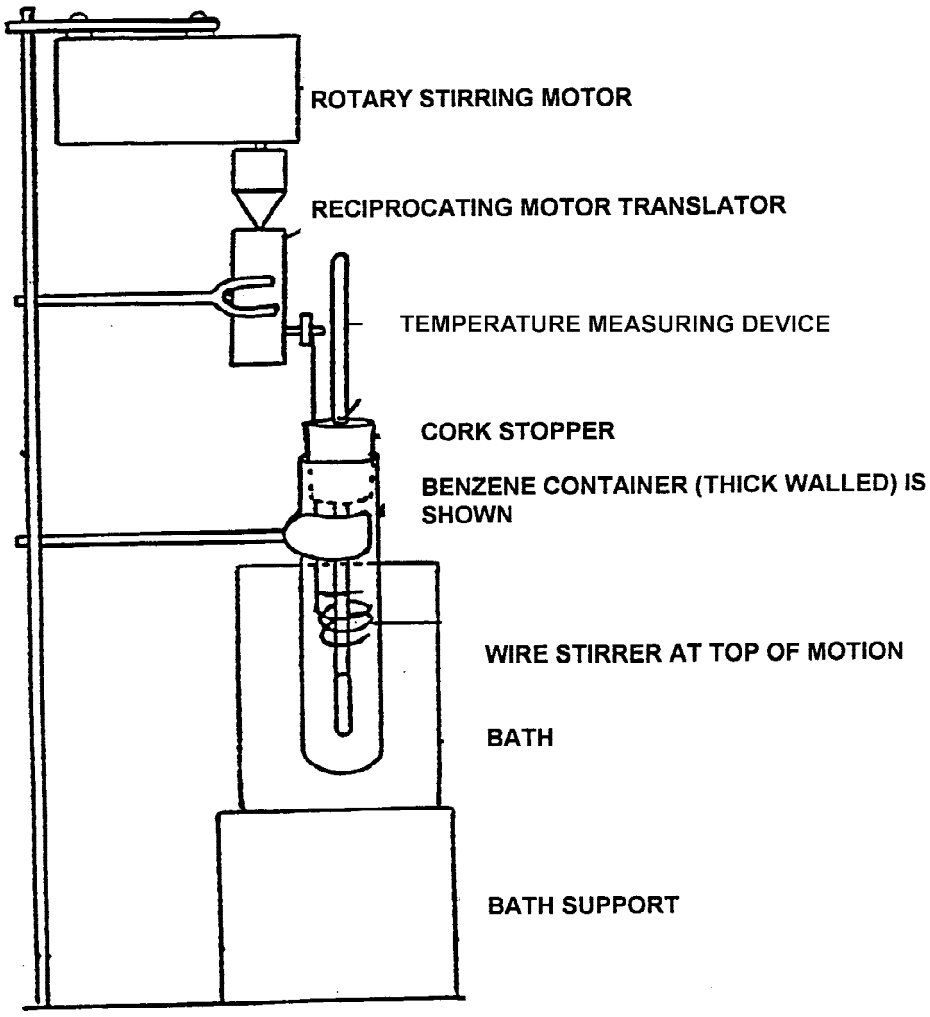

FIG. 1 Benzene Solidification Point Apparatus Set Up

9. Preparation of Apparatus

9.1 Fit the benzene container with a two-hole ~~cork~~ 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 ⅛-in. (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 ~~cork~~ stopper or annular ring ~~of cork~~ 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. ~~When using a thermistor, the ohmic value is read and converted to temperature by using the calibration data supplied by the manufacturer.~~ 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.

11. Procedure

11.1 Saturate the sample of benzene with water as follows: Place 7 to 8 mL of the sample in the benzene container, add 1 drop

of water, and shake the tube and contents vigorously.

11.2 Place the ~~cork~~ stopper ~~onto the benzene container~~ and ~~onto the~~ stirring ~~apparatus, if available.~~ apparatus (if any) into the benzene container.

11.3 When using the benzene container (air jacket), the operator may cool the smaller test tube and contents rapidly to about 6°C in the ice bath, while stirring. Wipe dry the outside of the smaller test tube and insert it into the larger test tube. Place the assembled tubes in the ice bath.

11.4 Stir the benzene continuously and observe the ~~thermometer reading~~ temperature closely. The temperature will fall to a minimum, then rise to a maximum, remain constant at this maximum for approximately 15 s, and then fall again (Note 1). The minimum temperature is due to super-cooling before solidification starts and shall not be more than 0.7°C below the maximum when using a thermometer. Record the maximum constant temperature observed to the nearest 0.01°C and designate it as "wet" (Note ~~2).~~ 1).

NOTE 1—If distinct minimum and maximum points are not evident, or if the temperature does not remain constant at the maximum for at least 15 s, the determination shall be ~~repeated.~~

~~NOTE 2—The precision can be increased to ±0.01°C by using a magnifying glass that ensures a~~ repeated. The thermistor reading ~~perpendicular~~ should remain constant to at least two places to the ~~stem~~ right of the ~~thermometer. In such cases it may be necessary to correct for stem exposure, that under ordinary conditions this correction will be less than 0.01°C.~~ decimal.

12. Report

12.1 Results shall be reported on the anhydrous basis. Since the determination is actually made on water-saturated benzene, the solidification point shall be corrected to the anhydrous basis by adding 0.09°C to the observed maximum temperature following the minimum. Corrections for accuracy of the thermometer shall be made.

13. Precision and Bias [8]

13.1 *Thermometer Precision*—The following criteria should be used to judge the acceptability ~~(95 % probability level)~~ of results obtained by this test method when using a thermometer. The criteria were ~~derived from an interlaboratory study between six laboratories. One~~ determined by measuring the solidification point of a sample ~~was analyzed in triplicate in~~ twelve times at one ~~day~~ laboratory using a ~~thermistor and a thick walled glass test tube.~~

~~13.1.1 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ more than 0.03 °C. On the basis of test error alone, the difference between two~~ thermometer. Duplicate results ~~obtained~~ in the same laboratory ~~on the same material will be expected to exceed this value only 5 % of the time.~~

~~13.1.2 *Reproducibility*—Results submitted by each of two laboratories~~ should not be considered suspect unless they differ by more than ~~005 °C. On the basis of test error alone, the difference between two test results obtained in different laboratories on the same material will be expected to exceed this value only 5 % of the time.~~ 0.04°C.

13.2 *Thermistor Precision*—The following criteria should be used to judge the acceptability (95 % probability level) of results obtained by this test method when using a thermistor. The criteria were derived from an interlaboratory study between six laboratories. Three different samples were analyzed in triplicate on two different days using a thermistor and a thick walled glass test tube.

13.2.1 *Intermediate Precision* —Results in the same laboratory should not be considered suspect unless they differ more than 0.04°C. On the basis of test error alone, the difference between two results obtained in the same laboratory on the same material will be expected to exceed this value only 5 % of the time.

13.2.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 0.05°C. On the basis of test error alone, the difference between two test results obtained in different laboratories on the same material will be expected to exceed this value only 5 % of the time.

13.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure, bias has not been determined.

14. Keywords

14.1 benzene; solidification point

[8] Supporting data are available for ASTM International Headquarters. Request RR: D16–1028.

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last date of issue (D 852 - 00ε1) that may impact the use of this standard.

(*1*) Correction of bath temperature and generalization of temperature measuring device in Fig. 1.
2) Addition of new Precision statements for thermometer and ~~Bias statement.~~ thermistor.