Designation: D6875 – 12

Standard Test Method for
Solidification Point of Industrial Organic Chemicals by Thermistor[1]

This standard is issued under the fixed designation D6875; 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 covers a general procedure for determining the solidification point of most organic chemicals having appreciable heats of fusion and solidification points between 4 and 41°C.

NOTE 1—Other test methods for determining freeze point and solidification point of aromatic hydrocarbons include Test Methods D852, D1015, D1016, D3799, D4493, and D6269.

1.2 This test method is applicable to relatively pure compounds only. Solidification point depression is dependent on impurity concentrations.

1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with applicable specifications using this test method, 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.

1.4 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 a specific hazard statement, see Section 8, Hazards.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D852 Test Method for Solidification Point of Benzene

D1015 Test Method for Freezing Points of High-Purity Hydrocarbons

D1016 Test Method for Purity of Hydrocarbons from Freezing Points

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride

D3799 Test Method for Purity of Styrene by Freezing Point Method (Withdrawn 2000)[3]

D3852 Practice for Sampling and Handling Phenol, Cresols, and Cresylic Acid

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

D4493 Test Method for Solidification Point of Bisphenol A (4,4′-Isopropylidenediphenol)

D6269 Test Method for Solidification Point of *p*-Xylene (Withdrawn 2004)[3]

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

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

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*—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 as distinguished from freezing point is described in Test Method D1015. An interpretation of mole percent in terms of freezing point is given in Test Method D1016.

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[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 D16.04 on Instrumental Analysis.

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[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**

4. Summary of Test Method

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

5. Significance and Use

5.1 This test method may be used for process control during the manufacture of organic chemicals described in Section 1, for setting specifications, for development and research work, and to determine if contamination was introduced during shipment.

6. Apparatus

6.1 *Ohmmeter,* capable of measuring resistance to the nearest 0.1 ohm in the range of 1000 to 10 000 ohms with direct temperature readout.[5]

6.2 *Specimen Container,* thick walled test tube with 18 mm outside diameter and 14 mm inside diameter and 150 mm long.

6.3 *Stirrer,* consisting of a 1-mm stainless steel wire bent into a circular form at right angles to the shaft so it will move freely in the annular space between the thermistor and the wall of the test tube.

6.4 *Stirring Apparatus (Optional)*—The apparatus illustrated in Fig. 1 has been demonstrated to be an acceptable replacement for manual stirring.

6.5 *Temperature Bath,* capable of being controlled at 5 ± 1°C below the expected solidification point.

6.6 *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 ±0.01°C/year. Thermistor shall be calibrated to cover the range it is used.[6]

7. Reagents and Materials

7.1 *Cooling Media:*

7.1.1 *Water* is recommended for solidification points between 4 and 30°C.

7.2 *Drying Agents:*

7.2.1 *3A Molecular Sieve,* in the form of a powder or cylindrical granules about 3 mm in diameter.

8. Hazards

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

8.2 Appropriate personal protection equipment such as gloves, safety glasses, a long rubber apron, and a full face shield should be worn when handling hot or corrosive chemicals, or both.

8.3 A fume hood should be used when performing the test method on hazardous chemicals (OSHA 1910.1450 definition).

9. Sampling and Handling

9.1 Sample the material in accordance with Practices D3437, D3438, D3852, and D4297.

10. Preparation of Apparatus

10.1 Fit the sample container with a two-hole stopper. Through one hole insert the thermistor. Through the other hole insert the shaft of the stirrer. (See Fig. 1).

10.2 Set temperature bath at 5 ± 1°C. below the expected solidification point of the sample.

11. Calibration of Thermistor

11.1 Thermistor should be calibrated by the factory at a minimum every two years.

11.2 The thermistor may be checked by determining the ice point of water 0.00°C.

12. Sample Preparation

12.1 *Solids at Room Temperature*—Melt the entire sample by heating it to about 10°C above its solidification point and swirl it for homogeneity.

12.2 *Drying Samples*—If required by specifications or agreement with customer.

12.2.1 *p*-Xylene should be dried before testing by placing about 100 g of the material in a 400 mL Erlenmeyer flask and adding about 50 g of 3A molecular sieve.

12.3 Benzene is a special case and should be saturated with water as follows:

12.3.1 Place 7 to 8 mL of the specimen in the sample container.

12.3.2 Add 1 drop of water to the specimen.

12.3.3 Shake the sample tube vigorously.

13. Procedure

13.1 Place 7 to 8 mL of the specimen from step 12.2 or 12.3 in the sample container.

13.2 Insert the stopper with the thermistor and stirrer into the sample container and adjust the thermistor so that it is about 5 mm from the bottom.

13.3 Place the sample container in the constant temperature bath set at 5 ± 1°C below the expected solidification point. In the case of cyclohexane, the constant temperature bath should be set about 0.3°C below the expected solidification point.

13.4 Stir the specimen continuously and observe the temperature readings closely. The temperature will fall to a minimum (super cooling), then rise to a maximum. Record the maximum temperature.

13.5 If distinct minimum and maximum points are not evident, the determination shall be repeated. If the temperature

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[5] The sole source of supply of the apparatus known to the committee at this time is the Hart Scientific Model 1504, 220 N. 1300 West, P.O. Box 460, Pleasant Grove, UT 84062. 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,[1] which you may attend.

[6] The sole source of supply of the apparatus known to the committee at this time is GE Thermometrics, type CSP A727X-CSP60BA252M, 967 Windfall Rd. St. Marys, 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,[1] which you may attend.

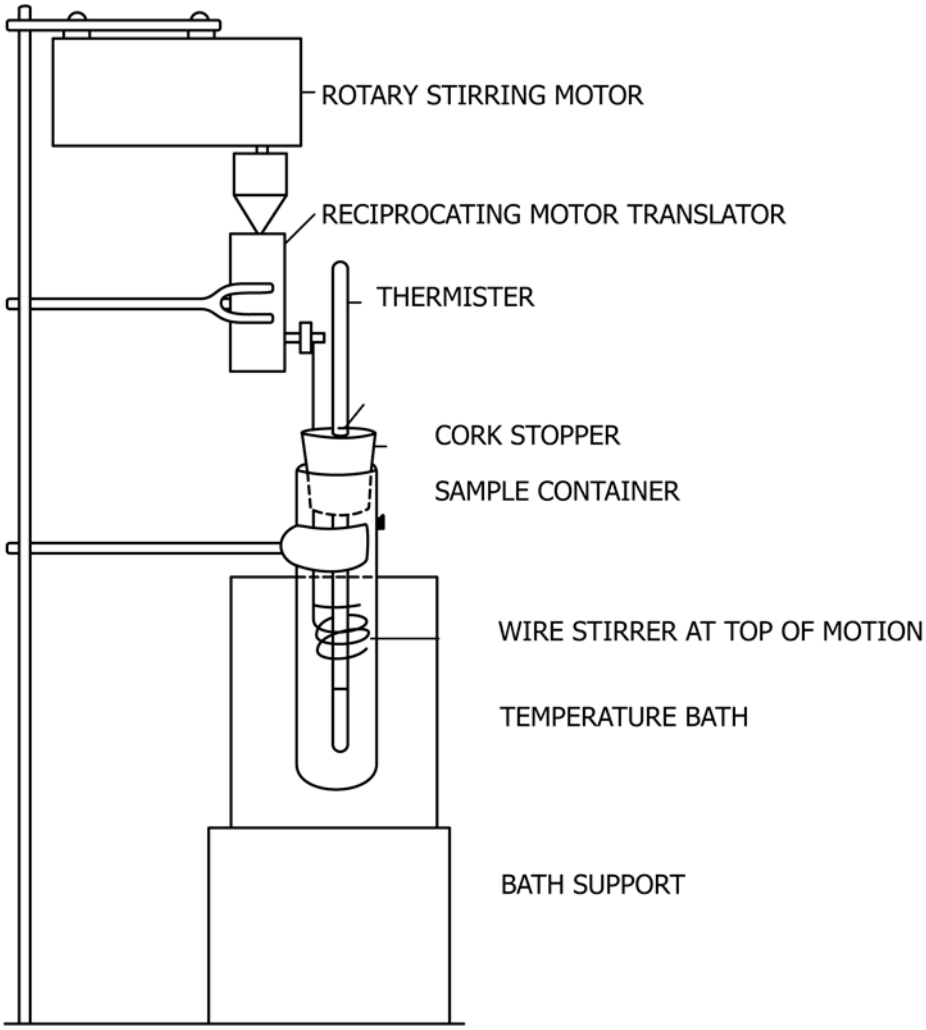

FIG. 1 Solidification Point Apparatus Set Up

does not rise to a maximum, increase the constant temperature bath temperature slightly and repeat the solidification point determination. Repeat increasing the temperature until a minimum temperature is observed and the temperature then rises to a maximum.

14. Calculations

14.1 Benzene results should be reported on an anhydrous basis by adding 0.09°C to the observed maximum temperature following the minimum.

14.2 All other results should be reported as observed.

15. Report

15.1 Results shall be reported to the nearest 0.01°C.

16. Precision and Bias

16.1 *Benzene*[7]—Based on seven labs analyzing three samples on two different days.

16.1.1 *Intermediate Precision (formerly called Repeatability)*—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 the results obtained in the same laboratory on the same material will be expected to exceed this value only 5 % of the time.

16.1.2 *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.

16.2 *Cyclohexane*—Based on a single sample analyzed five times in three different laboratories.

16.2.1 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ more than 0.015°C. On the basis of test error alone, the difference between the results obtained in the same laboratory on the same material will be expected to exceed this value only 5 % of the time.

16.2.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 0.06°C. On the basis of test error alone, the

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1028. Contact ASTM Customer Service at service@astm.org.

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.

16.3 *p-Xylene*—Based on a single sample analyzed five times in six laboratories.

16.3.1 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ more than 0.026°C. On the basis of test error alone, the difference between the results obtained in the same laboratory on the same material will be expected to exceed this value only 5 % of the time.

16.3.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 0.035°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.

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

17. Quality Guidelines

17.1 Laboratories shall have a quality control system in place.

17.1.1 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.

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

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

17.1.4 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.

18. Keywords

18.1 benzene; cyclohexane; *p*-Xylene; solidification point

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D6875 – 08) that may impact the use of this standard. (Approved December 1, 2012.)

(1) Added Guide D6809 to referenced methods in Section 2.
(2) Added Section 17 Quality Control.
(3) Renumbered sections.