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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Test Method for Solidification Point of *p*-Xylene[1]

This standard is issued under the fixed designation D 6269; 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 the determination of the solidification point of *p*-Xylene with purity greater than 99.5 %.

NOTE 1—Other test methods for determining freeze point and solidification point of aromatic hydrocarbons include Test Methods D 852, D 1015, D 1016, and D 1493.

1.2 The following applies to all specified limits in this test method: for purposes of determining conformance with 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 E 29.

1.3 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 852 Test Method for Solidification Point of Benzene[2]

D 1015 Test Method for Freezing Point of High-Purity Hydrocarbons[3]

D 1016 Test Method for Purity of Hydrocarbons from Freezing Points[3]

D 1493 Test Method for Solidification Point of Industrial Organic Chemicals[2]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

2.2 *Other Documents:*

OSHA Regulations, 29 CRFR, paragraphs 1910.1000 and 1910.1200[5]

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 as distinguished from freezing point is described in Test Method D 1015. An interpretation of mole percent in terms of freezing point is given in Test Method D 1016.

3.1.2 *anhydrous*—*p*-xylene that has been treated with 3A molecular sieve to remove water.

4. Summary of Test Method

4.1 P-Xylene is dried with a 3-A molecular sieve. The solidification point is then measured by noting the maximum temperature reached 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 *p*-xylene, for setting specifications, and to determine if contamination was introduced into the *p*-xylene during shipment.

6. Apparatus

6.1 *Temperature Bath*, controlled at 10 ± 1°C.

6.2 *Ohm Meter*[6,7], capable of measuring resistance to the nearest 0.1 Ω in range 1000 to 10 000 Ω with direct temperature readout.

6.3 *Sample Container*, thick walled test tube with 18-mm outside diameter and 14-mm inside diameter, and 150-mm long.

6.4 *Stirrer*, consisting of a 1-mm copper or stainless steel wire (copper or stainless steel) 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.

6.5 *Stirring Apparatus*, (Optional) The apparatus illustrated in Fig. 1 has been demonstrated to be an acceptable replacement for manually stirring the *p*-xylene solution.

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[1] This test method is under the jurisdiction of ASTM Committee D-16 on Aromatic Hydrocarbons and Related Chemicals and are the direct responsibility of Subcommittee D16.0E on Instrumental Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available fro the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[6] The sole source of supply of an ohm meter meeting these specifications is the Hart Scientific Model 1504., 220 N. 1300 West, P.O. Box 460, Pleasant Grove, Utah 84062.

[7] If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, D-16, which you may attend.

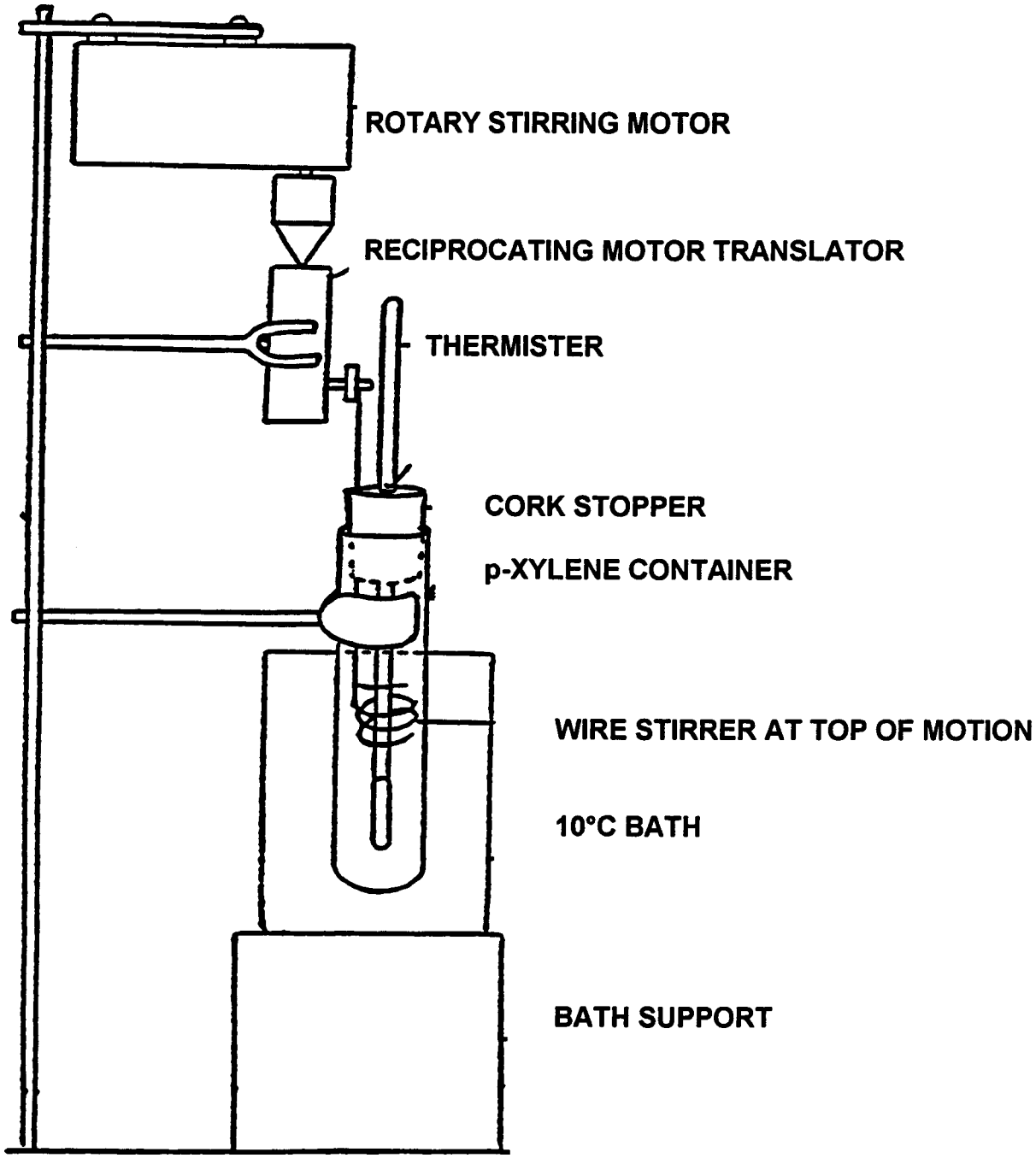

FIG. 1 p-Xylene Solidification Point Apparatus

6.6 *Thermistor*[7,8]—in stainless steel housing with resistance greater than 2 kΩ at 25°C. Calibration accuracy ±0.005°C. from 0 to 25°C. Drift in resistance equivalent to less than ±0.005°C/year.

7. Reagents and Materials

7.1 *Insulation*, dry absorbent cotton or glass wool.

7.2 *3-A Molecular Sieve*, in the form of a powder or cylindrical granules about 3 mm in diameter.

[8] The sole source of supply of a thermistor meeting these requirements is Thermometrics, Type CSP A733V-CSP60BA252M. 808 U.S. Highway #1, Edison, NJ 08817.

8. Hazards

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

9. Sampling and Handling

9.1 Sample the material in accordance with Practice D 3437.

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).

11. Calibration of Thermistor

11.1 Thermistor should be calibrated by the factory.

11.2 The thermistor may be checked by determining the ice point of 0°C.

12. Procedure

12.1 Dry the *p*-xylene by placing about 100 g of *p*-xylene in a 400-mL Erlenmeyer flask. Add about 50 g of 3-A molecular sieve. After fifteen min, decant the material to another flask and repeat the drying step with occasional stirring.

12.2 Place 7 to 8 mL of *p*-xylene from 12.1 in the sample container.

12.3 Insert the stopper with the thermistor and stirrer into the sample container and adjust the thermistor so that it is about 75 mm in the sample.

12.4 Stir the *p*-xylene continuously and observe the temperature readings closely. The temperature will fall to a minimum, then rise to a maximum. Record the maximum temperature to the nearest 0.001°C.

NOTE 2—If distinct minimum and maximum points are not evident, the determination shall be repeated.

13. Report

13.1 Results shall be reported on the anhydrous basis to the nearest 0.001°C.

14. Precision and Bias

14.1 Repeatability is 0.0034°C. based on a single lab analyzing one sample 13 times.

15. Keywords

15.1 p-Xylene; solidification point