Designation: D 1493 – 97

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

This standard is issued under the fixed designation D 1493; 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 −20 and +150°C.

1.2 This test method is applicable only to relatively pure compounds. Values obtained for grossly impure compounds can be low because of the freezing out of one component during the determination.

1.3 In order that the test method may be used on many materials, a choice of certain alternatives and a selection of apparatus are permitted. The report (Section 12) requires that the selected alternatives must be stated. Materials to which the test method is applicable in particular detail include phenol, naphthalene, phthalic anhydride, and maleic anhydride.

1.4 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 right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29E 29.

NOTE 1—A companion test method is Test Method D 852D 852.

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 specific hazard statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials[2]

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

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

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

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

D 3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride[2]

D 3852 Practice for Sampling and Handling Phenol and Cresyl Acid[2]

E 1 Specification for ASTM Thermometers[4]

E 29 Practice for Using Significant Digit in Test Data to Determine Conformance with Specifications[5]

E 77 Test Method for Inspection and Verification of Thermometers[4]

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[6]

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 amount of the same substance in its solid phase.

3.1.1.1 *Discussion*—Solidification point is distinguished from freezing point, which is described in Test Method D 1015D 1015. An interpretation of mole percent purity in terms of freezing point is given in Test Method D 1016D 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 in a liquid sample.

5. Significance and Use

5.1 This test method is suitable for setting specifications on compounds of the type described in Section 1. It is also suitable for use as an intense quality-control tool and in development and research work involving these compounds.

[1] This test method is under the jurisdiction of ASTM Committee D-16 on Aromatic Hydrocarbons and Related Chemicals and is 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 14.03.

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

[6] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

6. Apparatus

6.1 *Thermometer*—An etched-stem liquid-in-glass thermometer having a range of no more than 30°C shall be used. It shall be graduated in subdivisions no greater than 0.1°C. Unless otherwise specified, it shall be calibrated, for 76-mm immersion, at no fewer than three points. These shall include the approximate solidification point of the material being tested and two other points, respectively, about 5°C above and below the solidification point. A total immersion thermometer can be used if it is specified for testing a particular material, as, for example, benzene. (See Test Method D 852D 852.) The thermometer shall conform to Specification E 1E 1. It should be so chosen that the graduation at which the solidification point is to be observed is not hidden from view when the thermometer is inserted in the sample container. Suitable thermometers are ASTM Thermometers 89C to 96C, inclusive. Table 1 lists several of these thermometers and the materials for which, respectively, they are useful in particular in accordance with this test method. A thermometer, for example ASTM Thermometer 17C, 18C, or 91C, is needed when the average temperature of the emergent mercury column of the solidification-point thermometer is measured.

TABLE 1 ASTM Thermometers for Solidification Point

ASTM Number	Range of Temperature, °C	Selected Standardization Temperature,° C	Average Temperature of Emergent Mercury Column, °C	Material for Test
91C	20 to 50	40	25	phenol
92C	40 to 70	52	25	maleic anhydride
93C	60 to 90	80	30	naphthalene
96C	120 to 150	130	35	phthalic anhydride

6.2 *Specimen Container*—A standard heat-resistant glass test tube with lip shall be used. The test tube shall measure 25 mm in outside diameter and 150 mm in length.

6.3 *Stirrer (see Fig. 1)*—The stirrer shall consist of a 1-mm diameter (B&S gage 18), corrosion-resistant wire bent into a series of three circular loops about 25 mm apart at right angles to the shaft, the circle of each loop being about 20 mm in diameter, so that the stirrer can move freely in the annular space between the inner wall of the specimen container and the thermometer stem when the latter is inserted in the container. The shaft of the stirrer may be of any convenient length not less than 150 mm, and shall pass through an off-center hole in a two-hole cork stopper, the center hole of which holds the thermometer. The upper end of the shaft may be attached to a reciprocating device for mechanical stirring, or may be formed into a loop to facilitate lifting it for stirring by hand.

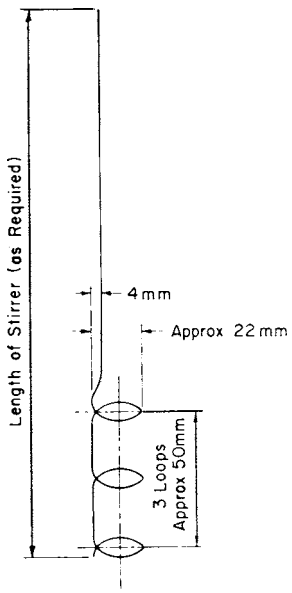

FIG. 1 Stirrer

6.4 *Flasks*:

6.4.1 A 200-mL, side-tube, heat-resistant glass distillation flask as described in Test Method D 850D 850 and

6.4.2 Two narrow-neck, heat-resistant glass Erlenmeyer flasks, 400-mL capacity each.

6.5 *Heaters*:

6.5.1 A hot plate, and

6.5.2 An electric heater that is fully adjustable as described in Test Method D 850D 850, or a bunsen or similar gas burner (see Section 9.2), or both.

6.6 *Insulation Board*—A sheet of hard insulation board 3 to 6-mm thick and 15 cm square, with a circular hole 50 mm in diameter in the center of it, is needed if the drying procedure given in 9.2 is used.

NOTE 2—Items described in 6.7, 6.8, and 6.9, are not essential for routinely testing materials that have solidification points substantially above room temperature, for example above 30°C; but, for referee testing, these items shall be used always, regardless of the solidification points of the materials being tested.

6.7 *Air Jacket*—A standard heat-resistant glass tube with lip, 38 mm in outside diameter and 200 mm in length, shall be fitted with a cork stopper bored with a hole of 25 to 26-mm diameter and into which the specimen container is to be inserted up to its lip.

6.8 *Cooling Bath*—A 2-L beaker or similar suitable container having an effective depth of at least 175 mm shall be filled with a cooling medium, which shall be glycerin for operating at temperatures between 145 and 25°C, water and ice between 25 and 0°C, and alcohol and dry ice between 0 and −25°C. A thermostatically controlled agitated bath may be used optionally. (See Fig. 2 for assembly of apparatus.)

6.9 *Clamp and Ring Stand*—A clamp, attached to a stand, holds the air jacket rigidly just below its lip when it is immersed in the cooling bath to a depth between 160 and 200 mm.

NOTE 3—Items listed in 6.10-6.15 inclusive, are required only for checking the accuracy of the thermometer at the solidification point of the material being tested. This check is desirable to detect changes that occur