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AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103


# Standard Test Method for Purity of Styrene by Freezing Point Method[1]

This standard is issued under the fixed designation D 3799; 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 is used for determining the purity of styrene expressed as weight percent. All impurities are considered to be ethylbenzene.

1.2 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, 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 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* Specific hazard statements are given in Section 5 and 7.5.

## 2. Referenced Documents

2.1 *ASTM Standards:*

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

2.2 *Other Document:*

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

## 3. Summary of Test Method

3.1 The purity of the styrene is determined by a measurement of the freezing point of the sample in equilibrium with air at atmospheric pressure. The presence of small amounts of impurities causes a depression of the freezing point which is proportional to the molal concentration of the contaminating substances. The freezing point is the highest temperature obtained after the supercooling of the liquid. For purities over 99 % it is not necessary to plot a time-temperature curve.

3.2 The freezing point methods, Test Methods D 1015 and D 1016, specified a platinum resistance thermometer for measuring the temperature. For routine work, mercury-in-glass thermometers are used. Other temperature-measuring devices can be utilized in this method provided that they have temperature resolution to 0.01°C or better. They must be calibrated since small differences in the temperature readings are significant. They must be recalibrated about once per week to correct for differences which may develop with age and handling. To simplify the multiple calibrations the freezing point of a large sample of styrene is determined with a platinum resistance thermometer and the mercury-in-glass or other thermometers calibrated against the standard styrene. Styrene may be kept in a deep freeze for several months with no appreciable change in the freezing point.

## 4. Significance and Use

4.1 Purity can be calculated by measuring the freezing point and relating to a freezing point for zero impurities.

4.2 This test method is in wide use for both producer and consumer for determining purity and is suitable for establishing specifications.

4.3 All impurities are considered to be ethylbenzene.

## 5. Hazards

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

## 6. Apparatus

6.1 *Temperature-Measuring Devices*—Temperature-measuring devices can be used, provided they have temperature resolution to 0.01°C or better, are operable in the range from −20 to −40°C, and are calibrated against a platinum resistance thermometer.

6.2 *Styrene Freezing Point Thermometer*[7]—Special design for determination of the freezing point of styrene as shown in Fig. 1 and described in Table 1 specification.

6.3 *Freezing Point Apparatus*—See Fig. 2 which consists of a 665-mL Dewar flask of borosilicate glass, a nest of three

[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 D16.0H on Styrene, Ethylbenzene, and $C_9$ and $C_{10}$ Aromatic Hydrocarbons.

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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 14.02.

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

[7] A thermometer meeting the specification is manufactured by the Precision Thermometer and Instrument Co., Southhampton Industrial Park, Southhampton, PA 18966, catalog No. 0230 (range: −33°C to −30°C). An equivalent may be used.

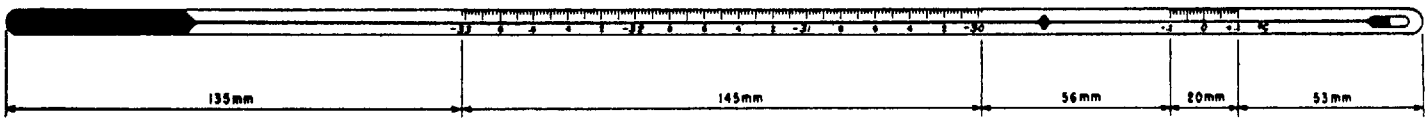


FIG. 1 Special Thermometer for Freezing Point of Styrene

TABLE 1 Thermometer Specification[A]

| | |
|---|---|
| Liquid | mercury |
| Filling above mercury | nitrogen gas |
| Range and subdivision | −33.02 to −29.98°C in 0.02°C |
| intervals with auxiliary scale from | −0.2 to 0.2°C |
| Total length | 405 to 410 mm |
| Immersion | total |
| Stem | plain front, enamel back, suitable thermometer tubing, diameter, 6 to 7 mm. Corning Normal or equally suitable thermometer glass, diameter 6 to 7 mm but not greater than that of stem; length, 50 to 60 mm. |
| Distance from bottom of bulb to −33°C mark | 125 to 140 mm |
| Distance from top of thermometer to −30°C mark | 125 to 135 mm |
| Length of unchanged capillary between the enlargement and the graduation next below | not less than 5 mm |
| Length of unchanged capillary above or below the 0°C point | not less than 15 mm |
| Expansion chamber | to permit heating to at least 50°C |
| Finish (top) | plain |
| Graduation | All lines, figures, and letters to be clear-cut and distinct. The graduation marks to be fine, straight, of uniform width and perpendicular to the axis of the thermometer. Each degree and tenth degree line to be longer than the intermediate ones. Graduations to be numbered in full at each degree mark and in decimals at other multiples of 0.2°C. |
| Special marking | The manufacturer's name or trademark, a serial number, and the words "total immersion" shall be etched on the stem. |
| Scale error | The error at any point on the scale shall not exceed 0.1°C. |

[A] The accuracy attainable with mercury-in-glass thermometers, based on performance when all precautions are taken, for measurements of the kind for which these thermometers were designed, is from 0.01°C to 0.03°C, and calibration corrections are stated to the nearest 0.01°C. Subdivision in 0.02°C intervals, therefore, serves principally for the purpose of facilitating reading.



test tubes (16 by 150, 20 by 150, and 25 by 150 mm), and a mechanically operated stirrer. All corks should fit tightly, and the hole for the stirrer shaft should be as small as practical.

6.4 *Reciprocating-Type Stirrer*—A 1.59 mm (1/16-in.) rod formed into a coil of four turns of diameter to fit inside the 16 by 150-mm test tube and over the thermometer for immersion in styrene is used with either of these mechanical aids.

6.5 *Thermometer*, alcohol-type, used to read the temperature of the cooling bath. It should read as low as −70°C and be graduated in 1°C subdivisions.

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7.3 *Carbon Dioxide*, solid (dry ice), for cooling bath.

7.4 *Cooling Liquid*—1,1,1-trichloroethane is recommended because it is nonflammable.

NOTE—A mechanical cooling device may also be used.

7.5 *Styrene Monomer Standard*—**Caution**: While a standard may be stored for several months in a deep freeze, it should be restandardized on a regular basis in accordance with Test Method D 1015. Standards maintained at 40°C will change the purity in days.

## 8. Sampling

8.1 Sample in accordance with Practice D 3437.

## 9. Procedures

9.1 Add approximately 10 mL of styrene to the sample tube and adjust the liquid level so that the top of the thermometer bulb will be immersed 20 mm when the bottom of the bulb is placed 10 mm above the bottom of the sample tube. Place sample tubes in the Dewar flask, containing 1,1,1-trichloroethane cooled to about −45°C. The 1,1,1-trichloroethane level should be at least 20 mm above the level of the styrene in the cooling cell. Maintain the bath temperature between −42 and −45°C during the determination by cautiously adding dry ice, a small piece at a time.

9.2 Adjust the stirrer to 100 to 150 strokes per minute and

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.