



Designation: D850 – 25

Standard Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials¹

This standard is issued under the fixed designation D850; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the distillation of industrial aromatic hydrocarbons and related materials of relatively narrow boiling ranges from 30 °C to 250 °C. **Warning**—This test method is not applicable for styrene which will exothermically polymerize during the distillation process.

1.2 The following applies to all specified limits in this test method: for the purposes of determining conformance to this test method, an observed or 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.

1.3 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for details and EPA’s website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 6.5.2, 6.6.1, and Section 7.

1.6 *This international standard was developed in accordance with internationally recognized principles on standard-*

ization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D1078 Test Method for Distillation Range of Volatile Organic Liquids

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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

E1 Specification for ASTM Liquid-in-Glass Thermometers

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

E133 Specification for Distillation Equipment

E220 Test Method for Calibration of Thermocouples by Comparison Techniques

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 Other Document:

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200³

3. Terminology

3.1 Definitions:

3.1.1 See Terminology D4790 for definitions of terms used in this test method.

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty 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 www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

³ 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 distillation of a 100 mL sample of industrial aromatic hydrocarbons and related materials is carried out via a carefully controlled distillation wherein temperature readings are noted for the first drop of distillate and when 5, 10, and each additional 10 up to 90, and 95 % of the sample has distilled over. The temperature corresponding to the dry point is also noted.

5. Significance and Use

5.1 This test method is suitable for setting specifications, for use as an internal quality control tool, and for use in development or research work on industrial aromatic hydrocarbons and related materials.

5.2 This test method gives a broad indication of general purity and can also indicate presence of excessive moisture. It will not differentiate between products of similar boiling range.

6. Apparatus

6.1 *Distillation Flask*—A standard 200 mL side-tube, heat-resistant glass distillation flask as shown in Fig. 1, conforming to the following dimensions:

Diameter of bulb, outside, mm	76 ± 1.5
Diameter of neck, inside, mm	21 ± 1
Height of flask, outside, mm	179 ± 3
Vertical distance from bottom of bulb outside to bottom of vapor-tube opening in neck, mm	120 ± 3
Length of side tube, mm	100 ± 3
Diameter of side tube, outside, mm	7 ± 0.5
Angle of side tube with vertical axis of bulb and neck, °	75 ± 3

The flask does not comply with Flask C of Specification E133.

6.2 Temperature Measurement Devices:

6.2.1 *Manual Distillation Thermometer*—The ASTM Solvents Distillation Thermometer used in the test shall be as prescribed in the specifications for the material being tested. If no thermometer is specified in the material specification, select one from Table 1 with the smallest graduations that will cover the entire distillation range of the material being tested. Table

TABLE 1 ASTM Thermometers for Distillation Test of Industrial Aromatic Hydrocarbons

ASTM Thermometer No.	Name	Range, °C	Subdivision, °C
39C	solvents distillation	48 to 102	0.2
40C	solvents distillation	72 to 126	0.2
41C	solvents distillation	98 to 152	0.2
42C	solvents distillation	95 to 255	0.5
102C	solvents distillation	123 to 177	0.2
103C	solvents distillation	148 to 202	0.2
104C	solvents distillation	173 to 227	0.2
105C	solvents distillation	198 to 252	0.2
106C	solvents distillation	224 to 276	0.2
111C	solvents distillation	170 to 250	0.2

1 lists several ASTM solvents distillation thermometers which are suitable for testing industrial aromatic hydrocarbons, and which meet the requirements of Specification E1.

6.2.2 *Automatic Distillation Temperature Sensor*—Temperature measurement systems using thermocouples or resistance thermometers, otherwise referred to as the “temperature measuring devices,” must exhibit the same temperature lag and accuracy as the equivalent mercury glass thermometer. Confirmation of the calibration of these temperature sensors is to be done at regular intervals. This may be accomplished as prescribed in Test Method E220, or some similar means using a precision resistance decade box. Another technique is to distill pure toluene and compare the temperature indicated by the thermocouple or resistance thermometer with that shown by the thermometer. When installing a new automatic distillation analyzer, a toluene sample with a known distillation range of approximately 1.0 °C must be used to verify the dry point and distillation range.⁴ It is recommended that such a material be used when replacing a temperature measuring device. Alternatively a material of known distillation range and dry point may be used when replacing a temperature measuring device.

6.2.2.1 *Automatic Distillation Temperature Sensor Centering Device*—The temperature sensor shall be mounted through a snug-fitting device designed to mechanically center the sensor in the neck of the flask. The use of a cork or silicone stopper with a hole drilled through the center is not acceptable for this purpose. Examples of acceptable centering devices are shown in Fig. 2.

6.2.2.2 The electronic circuitry or algorithms, or both, used shall include the capability to simulate the temperature lag of a mercury-in-glass thermometer. For that reason the known toluene sample with a distillation range of approximately 1.0 °C must be used to verify the algorithm and dampening software.

6.2.2.3 Alternatively, the sensor can also be placed in a casing with the tip of the sensor covered, so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag similar to that of a mercury-in-glass thermometer.

NOTE 1—In a region where the temperature is changing rapidly during

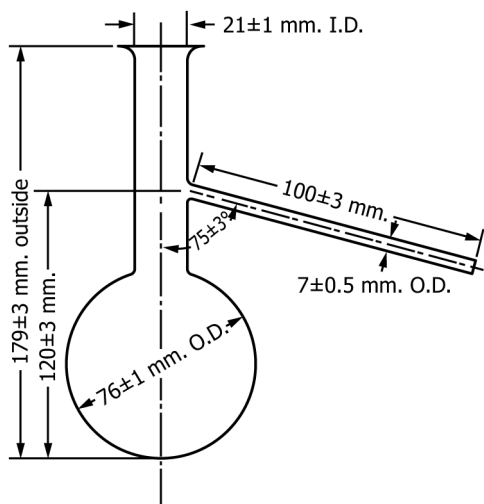


FIG. 1 Distillation Flask

⁴ Such a material is available from Spectrum Quality Standards.