Designation: E133 – 92 (Reapproved 2016)

Standard Specification for Distillation Equipment[1]

This standard is issued under the fixed designation E133; 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 specification covers distillation equipment used in the following ASTM test methods: D86, D850, and (for approved alternative requirements only) Test Method D1078.

1.2 Some items of equipment included in this specification are common to all methods; other items are not. Therefore, those portions of this specification pertinent to the need must be selected.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials

D1078 Test Method for Distillation Range of Volatile Organic Liquids

E1 Specification for ASTM Liquid-in-Glass Thermometers

E1272 Specification for Laboratory Glass Graduated Cylinders

E1405 Specification for Laboratory Glass Distillation Flasks

3. Apparatus Assembly

3.1 Typical assemblies of the apparatus are shown in Fig. 1 and Fig. 2.

4. Distillation Flasks

4.1 Flasks of heat-resistant glass shall be provided as indicated in Fig. 3 and shall comply with the requirements of Specification E1405.

5. Condenser and Cooling Bath

5.1 Typical approved types of condenser and cooling bath are included in Fig. 1 and Fig. 2. These items are common to all methods covered by this specification.

5.2 The condenser shall be made of seamless brass tubing, 22 in. (55.88 cm) in length. It shall be 9⁄16 in. (14.29 mm) in outside diameter, and shall have a wall thickness of 0.031 to 0.036 in. (0.08 to 0.09 cm).

5.3 The condenser shall be set so that approximately 15.5 in. (39.4 cm) of the tube will be in contact with the cooling medium, with about 2 in. (5 cm) outside the cooling bath at the upper end, and about 4½ in. (11 cm) outside at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75° with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.26 in. (0.66 cm)/linear in. (2.54 cm) of condenser tube (sine of angle of 15°), and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. (0.61 cm) nor more than 0.28 in. (0.71 cm-)/linear in. of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm) and slightly backward so as to ensure contact with the wall of the receiving graduate at a point approximately 1 to 1¼ in. (2.54 to 3.18 cm) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle so that the tip may be brought into contact with the wall of the cylinder.

5.4 The capacity of the cooling bath shall be not less than 340 in.^{3} (5.55 L) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than 1¼ in. (3.18 cm) below the plane of the top of the bath at its point of entrance and not less than ¾ in. (1.91 cm) above the floor of the bath at its exit.

5.5 Clearances between the condenser tube and the walls of the bath shall be at least ½ in. (1.27 cm) except for the sections

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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.

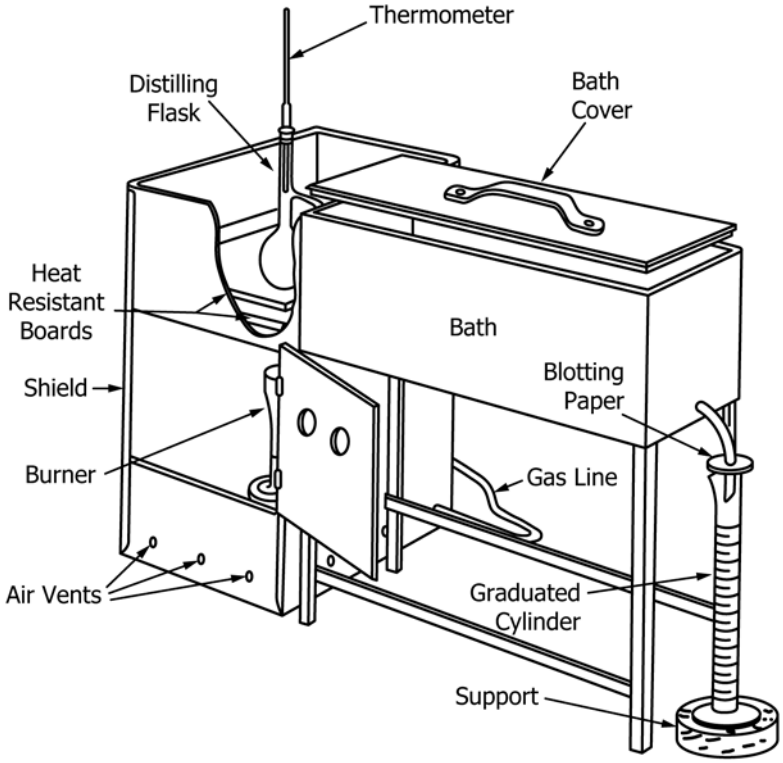

FIG. 1 Apparatus Assembly Using Gas Burner

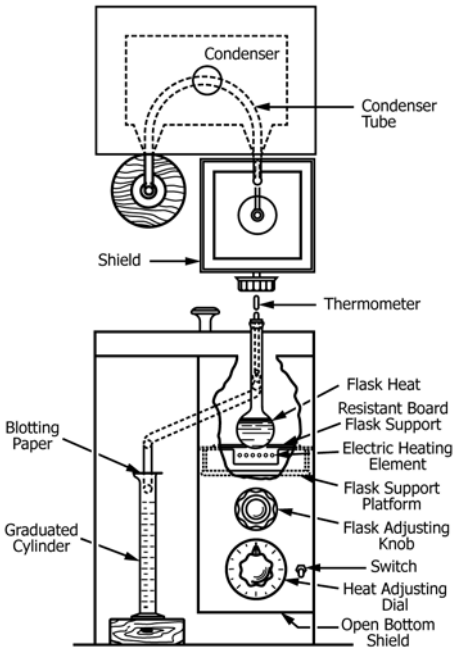

FIG. 2 Apparatus Assembly Using Electric Heater

adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 in.^{3} (5.55 L)/tube.

6. Metal Shield or Enclosure for Flask

6.1 *Type 1 Shield* (Fig. 1) is 19 in. (48.26 cm) high, 11 in. (27.94 cm) long, and 8 in. (20.32 cm) wide, made of sheet metal of approximately 22 gauge. It shall have a door on one narrow side, and two openings 1 in. (2.54 cm) in diameter, equally spaced in each of the two narrow sides, with a slot cut in one side for the vapor tube. The centers of these four openings shall be 8½ in. (21.59 cm) below the top of the shield. There shall be three ¼-in. (1.27 cm) holes in each of the four sides, with their centers 1 in. above the base of the shield.

6.2 *Type 2 Shield* (Fig. 2) is 17¼ in. (44.45 cm) high, 8 in. (20.32 cm) long, and 8 in. wide, made of sheet metal of approximately 22 gauge, with a window on the front side. The open bottom of the shield shall be spaced approximately 2 in. (5 cm) from the base of the unit. The rear of the shield shall have an elliptical hole for the vapor tube. A flask-adjusting knob shall be located on front of the shield for adjusting the flask support. Also, a heat-adjusting indicating dial shall be used to provide stepless heat control when the electric heater is used. The entire mechanism shall be built into the bottom portion of the shield. When an electric heater is employed, the portion of the shield above the board shall be the same as with the gas burner, but the part below may be omitted.

7. Heat Source

7.1 *Gas Burner* (Fig. 1), so constructed that sufficient heat from the available gas can be obtained to distill the product at the specified rate. A sensitive regulating valve and gas pressure governor to give complete control of heating may be provided.

7.2 *Electric Heater* (Fig. 2), may be used instead of a gas burner, provided it is capable of bringing over the first drop from a cold start within the time specified and of continuing the