



Standard Method for Steam Distillation of Bituminous Protective Coatings¹

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

1. Scope

1.1 This is a general method for the separation by steam distillation and the recovery of solvent and base from bituminous mixtures.

1.2 The following safety hazards caveat pertains only to the test methods described in this specification. *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 140 Practice for Sampling Bituminous Materials²

E 1 Specification for ASTM Thermometers³

3. Apparatus

3.1 *Steam Generator*, made of either metal or glass, with a capacity of approximately 2 gal (7600 cm³), suitable for continued use in the production of steam. If of glass, it shall be fitted with two outlets having suitable connections for rubber tubing. In the case of a metal generator, a large opening for filling and a water gage shall be additional parts of the apparatus. The generator shall be supplied with suitable pinch-cocks or valves so that steam may be blown off to the atmosphere until the test is ready.

NOTE 1—The following conversion factors are given for volumetric glassware graduated in litres:

$$1 \text{ L} = 1 \text{ dm}^3 \text{ or } 1000 \text{ cm}^3 \quad (1)$$

$$1 \text{ mL} = 1 \text{ cm}^3$$

3.2 *Bath*, made of metal and having sufficient capacity to permit immersion of the distilling flask to a depth of not less than 4 in. (101.6 mm).

¹ This method is under the jurisdiction of ASTM Committee D-8 on Roofing, Waterproofing, and Bituminous Materials and is the direct responsibility of Subcommittee D08.05 on Solvent-Bearing Bituminous Compounds for Roofing and Waterproofing.

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² *Annual Book of ASTM Standards*, Vol 04.03.

³ *Annual Book of ASTM Standards*, Vol 14.01.

3.3 *Source of Heat* for the bath, such as a gas burner, electric hot plate, or the equivalent. An electric mantle type heater, equipped with a variable resistance transformer of suitable size and heat capacity, is a suitable replacement for the bath and heat source.

3.4 *Distilling Flask*, 1000-cm³, heat-resistant glass, with a short ring neck and round bottom. It shall be fitted with a three-hole rubber stopper, a steam delivery tube that will reach to within $\frac{3}{8} \pm \frac{1}{8}$ in. (9.5 ± 4.7 mm) of the bottom of the flask and project from the top to a convenient distance for connection to the generator, a vapor outlet tube extending from beneath the rubber stopper to a point sufficiently above the distilling flask to permit convenient connection to the condenser, and a thermometer. The steam tubing shall be not less than 2 nor more than 4 mm in internal diameter, and the vapor outlet tube shall be not less than 5 mm in internal diameter.

3.5 *Thermometer*, ASTM Low Distillation, having a range of -2 to $+300^\circ\text{C}$ (30 to 580°F), as specified, and conforming to the requirements for thermometer 7C or 7F, respectively, as prescribed in Specification E 1.

3.6 *Condenser*—A glass-jacketed condenser. The following dimensions are recommended:

Length of jacket, excluding the necks	250 \pm 5 mm
Length of condenser tube	400 \pm 25 mm
Outside diameter of condenser tube proper	12.5 \pm 0.5 mm
Outside diameter of expanded end of tube	23 \pm 1 mm
Length of expanded end of tube	75 \pm 1 mm

3.7 *Adapter*—A heavy-wall (1 mm) glass adapter, with reinforced top, having an angle of approximately 105° . The inside diameter at the large end shall be approximately 18 mm and at the small end, not less than 5 mm. The lower surface of the adapter shall be on a smooth descending curve from the larger end to the smaller. The inside line of the outlet end shall be vertical and the outlet shall be cut or ground (not fire-polished) at an angle of $45 \pm 5^\circ$ to the inside line.

3.8 *Receiver*—Graduated cylinders of uniform diameter, with a pressed or molded base and a lipped top. The over-all height shall be 10 ± 0.250 in. (254 ± 6.35 mm). The cylinder shall be graduated in single millilitres to contain 100 cm³, and the graduated portion shall be 7.5 ± 0.500 in. (190.5 ± 12.70 mm) in length.

3.9 *Separatory Funnel*—A separatory funnel having a capacity of not less than 500 cm³.