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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Method for Steam Distillation of Bituminous Protective Coatings[1]

This standard is issued under the fixed designation D 255; 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 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 Materials[2]

E 1 Specification for ASTM Thermometers[3]

3. Apparatus

3.1 *Steam Generator*, made of either metal or glass, with a capacity of approximately 2 gal (7600 cm^3), 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:

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

$$1\ mL = 1\ cm^3$$

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).

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-cm^3, 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 ⅜ ± ⅛ 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°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 ± 5 mm
Length of condenser tube	400 ± 25 mm
Outside diameter of condenser tube proper	12.5 ± 0.5 mm
Outside diameter of expanded end of tube	23 ± 1 mm
Length of expanded end of tube	75 ± 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 ± 5° 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 cm^3, 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 cm^3.

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[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.

4. Sampling

4.1 Take the samples for laboratory examination from the original containers immediately after stirring to a uniform condition. Determine the number of containers sampled to represent a shipment in accordance with Practice D 140. Restir this combined sample immediately before taking out portions for individual tests.

5. Procedure

5.1 Assemble the apparatus as shown in Fig. 1. With atmospheric outlet open and steam delivery outlet closed, half-fill the steam generator with water and apply heat. Measure a 500 ± 0.5-cm ^{3} sample into the distilling flask. Calculate the weight of the sample, using its specific gravity at 60°F (15.6°C). Connect the steam generator to the steam delivery tube, the end of which shall be within ⅜ ± ⅛ in. (9.5 ± 3.2 mm) of the bottom of the distilling flask. Connect the outlet from the distilling flask to the condenser and turn on cooling water. Adjust the adapter over the end of the condenser tube to conduct the distillate into the receiver and closely cover the top of the receiver during the distillation with a piece of blotting paper or its equivalent, cut to fit the adapter tightly. Extend the adapter into the receiver at least 1 in. (25.4 mm) but not below the 100-cm^3 mark. Immerse the receiver up to the 100-cm^3 mark in a transparent bath maintained between 55 and 65°F (13 and 18°C). Place the end of the bulb of the thermometer in the distilling flask within ⅜ ± ⅛ in. of the bottom of the flask. Fill the bath with high-flash oil and apply heat. When the temperature of the sample in the distilling flask reaches 266°F (130°C), simultaneously close the atmospheric outlet of the steam generator and open the steam delivery outlet thus directing the steam to pass through the sample. Adjust the flow of steam so that the total condensate is collected at the rate of 6 to 10 cm^3/min. Collect successive 100-cm ^{3} increments of condensate. When 100 cm^3 of total condensate contains approximately 0.5 cm^3 of solvent, or less, raise the sample temperature at a rate of 4 to 6°F (2.2 to 3.3°C)/min, maintaining total condensate rate by controlling steam flow. Stop the distillation when 100 cm^3 of total distillate contains approximately 0.5 cm ^{3} of solvent or at a maximum sample temperature of 400°F (215°C).

5.2 Combine the total condensates in the separatory funnel and separate the water from the solvent by withdrawing the lower aqueous phase. In some cases, the water and solvent may not separate readily; separation can be facilitated by the addition of sodium chloride, which will result in a sufficient difference in gravity to produce a clear separation of the two layers. Transfer the solvent to clean 100-cm^3 graduates. At a solvent temperature of 55 to 65°F (13 to 18°C), measure the total volume of solvent and record. Retain the solvent for any subsequent testing required.

5.3 If solvent and base are required on a mass percent basis, determine the specific gravity of the solvent at 60°F (15.6°C).

6. Calculation

6.1 Calculate the volume percent as follows:

$$A = B/5 \tag{2}$$

$$C = 100 - A$$

where:

A = volume percent of solvent,
B = cubic centimetres of separated solvent, and
C = volume percent of base.

6.2 Calculate the mass percent as follows:

$$D = (BE \times 100)/F \tag{3}$$

$$G = 100 - D$$

where:

D = mass percent of solvent,
B = cubic centimetres of separated solvent,
E = specific gravity of separated solvent at 60°F (15.6°C),
F = grams of sample used, and
G = mass percent of base.

7. Report

7.1 Report the results as volume percent or mass percent, of solvent or of base, as required.

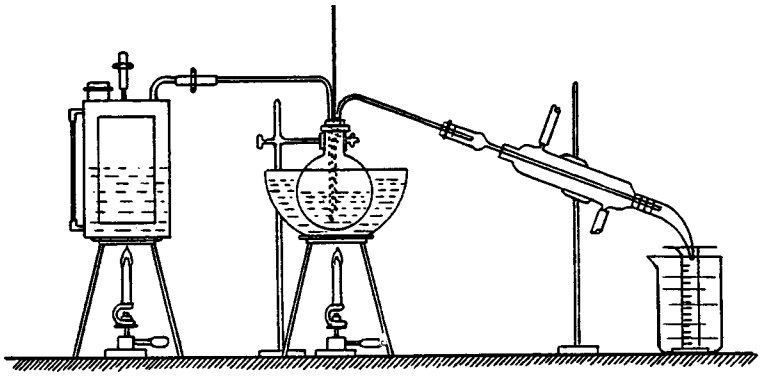

FIG. 1 Assembly of Apparatus for Steam Distillation