



Designation: D6997 – 04

Standard Test Method for Distillation of Emulsified Asphalt¹

This standard is issued under the fixed designation D6997; 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 covers the quantitative determination of residue and oil distillate in emulsified asphalts composed principally of a semisolid or liquid asphaltic base, water, and an emulsifying agent.

1.2 *This test method 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:*²

E1 Specification for ASTM Liquid-in-Glass Thermometers

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Method

3.1 A sample of emulsified asphalt is heated in an aluminum-alloy still to $260 \pm 5^\circ\text{C}$ and the volumes of distillate and water obtained are measured. The residue from the distillation, and also any oil portion of the distillate may be tested as required.

4. Significance and Use

4.1 This test method can be used for quantitative determination of residue and oil distillates in emulsified asphalts for specification acceptance, service evaluation, control, and re-

search. This test method can also be used to obtain residue and oil distillate for further testing.

5. Apparatus

5.1 *Aluminum-Alloy Still*—approximately 240 mm in height by 95 mm in inside diameter with cover and clamp as shown in Fig. 1. A variety of gasket materials may be used to seal the still providing they withstand the maximum temperature reached during distillation.

NOTE 1—Residue by distillation results obtained with iron stills are acceptable.

5.2 *Heat Sources*—one ring burner, approximately 125 mm inside diameter, having holes on the inner periphery and having three spacers, to ensure centering of burner around the still (see Fig. 2); one Bunsen burner equipped with optional wing tip as shown in Fig. 3

5.3 *Connection Apparatus*—consisting of a 12.5 ± 0.5 mm glass or metal connecting tube, tin shield (as detailed in Fig. 3), and a water-cooled glass condenser tube with a metal or borosilicate glass jacket. The metal connecting tube may be secured to the lid by a threaded compressing fitting. Glass connecting tube may be secured to the lid by means of a cork or silicone stopper. Other condensers of 12.5 ± 0.5 mm glass or metal tubing may be used, providing that the wetted length is 400 to 550 mm.

5.4 *Graduated Cylinder*—100-mL, with graduation intervals of 1.0 mL.

5.5 *Thermometer*—ASTM 7C thermometer, as prescribed in Specification E1, or any other thermometric device of equal accuracy.

5.6 *Balance*—capable of weighing 3500 g to within ± 0.1 g.

5.7 *Stoppers*—Cork (wrapped in foil) or silicone stoppers to seal holes in still cover, maintain temperature measuring device position, and secure connecting tube to still cover.

5.8 *Rubber Tubing*—heat resistant tubing of adequate size to secure glass to glass joint of connecting tube and condenser.

¹ This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.42 on Emulsified Asphalt Tests.

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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.

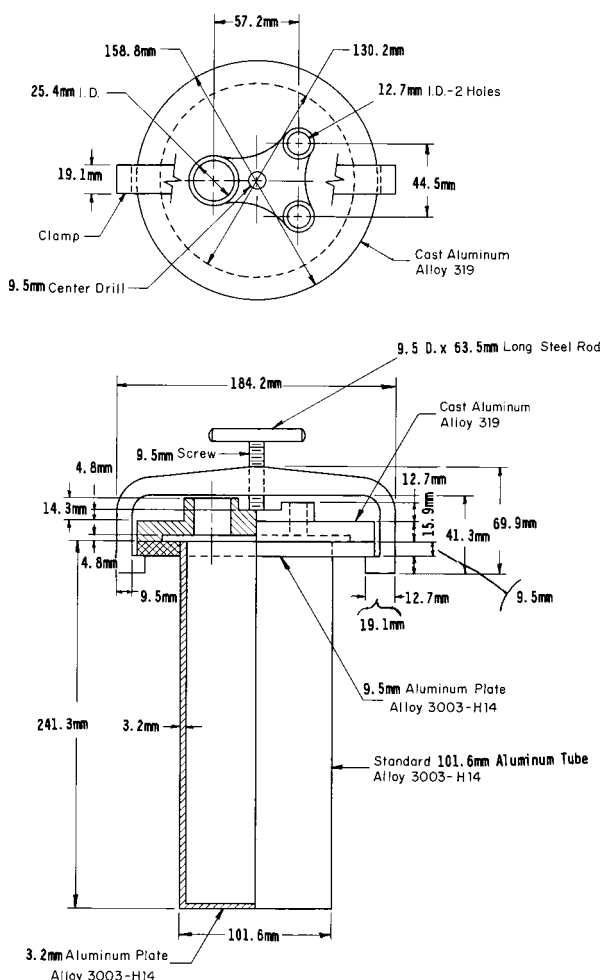


FIG. 1 Aluminum-Alloy Still

5.9 *Sieve*—A sieve having a 76.2-mm frame conforming to Specification E11, and having a 300- μ m wire sieve cloth.

NOTE 2—For details of the assembly of apparatus for the distillation test, see Fig. 3.

6. Sample Conditioning for Testing

6.1 All emulsified asphalts shall be properly stirred to achieve homogeneity before testing.

6.2 All emulsified asphalts with viscosity testing requirements of 50°C shall be heated to $50 \pm 3^\circ\text{C}$ in the original sample container in a water bath or oven. The container should be vented to relieve pressure. After the sample reaches $50 \pm 3^\circ\text{C}$, stir the sample to achieve homogeneity.

6.3 Emulsified asphalts with viscosity testing requirements of 25°C should be mixed or stirred at $25 \pm 3^\circ\text{C}$ in the original sample container to achieve homogeneity.

NOTE 3—Emulsified asphalts with viscosity testing requirements of 25°C may be heated and stirred as specified in 6.2, if necessary. In the event the 6.2 method is used, the sample should be cooled to $25 \pm 3^\circ\text{C}$ before testing.

7. Procedure

7.1 Weigh 200 ± 0.1 g of a representative sample of the emulsified asphalt in the previously weighed aluminum-alloy

still (including lid, clamp, temperature measuring device, small stopper, and gasket, if gasket is used).

7.2 Use a gasket of suitable material between the still and its cover, or grind the joint to a tight fit. Securely clamp the cover on the still.

7.3 Insert a thermometer, or other thermometric device, through a cork or silicone stopper, in one of the small holes provided in the cover, seal the other hole with a cork or silicone stopper. Adjust the temperature measuring device so that the end of the bulb is approximately 6 mm from the bottom of the still.

NOTE 4—A second temperature measuring device may be used in the second small hole, with the bulb placed approximately 165 mm from the bottom of the still. A sudden change in temperature reading of upper temperature measuring device indicates foam on bulb. Remove heat until foaming ceases.

7.4 Place the ring burner around the still about 150 mm from the bottom of the still. Apply heat by lighting this burner and adjusting to low flame. Also apply just enough heat from a bunsen burner to the connecting tube to prevent condensation of water in this tube.

7.5 When the temperature can be read on the thermometer, or at approximately 215°C , move the ring burner approximately level with the bottom of the still. Increase the temperature to $260 \pm 5^\circ\text{C}$, maintaining it at this temperature for 15 min. Complete the total distillation in 60 ± 15 min from the first application of heat.

NOTE 5—The location of the burner at the start of the test is flexible. It may be raised to decrease chance of foam-over or lowered to middle of still for emulsion containing no solvent. The ring burner may be gradually lowered as the distillation proceeds to ensure that the time requirements of this test method are satisfied.

7.6 Immediately at the expiration of the heating period, again weigh the still and accessories as described in 7.1. The aluminum-alloy still at ambient temperature (7.1) weighs 1.5 g more than at 260°C . Correct for this error by adding 1.5 g to gross weight obtained prior to calculating the percentage of residue by distillation. Calculate and report the percentage residue by distillation. Record the volume of oil distillate to the nearest 0.5 ml. Calculate and report the oil distillate as a volume percentage of the total emulsified asphalt.

NOTE 6—For purposes of this test result calculation, assume a 1:1 mass to volume relationship of distillate to emulsified asphalt.

7.7 Remove the cover from the still, stir, and immediately pour suitable portions of the residue into a container or suitable molds for making any required tests. If there is foreign matter in the residue, the material shall be poured through a 300-mm sieve prior to pouring into the test molds and containers.

8. Calculation

8.1 Calculate the percentage of residue in the sample as follows:

$$\text{Sample residue, \%} = \left(\frac{B - A}{C} \right) \times 100$$