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Standard Test Method for Residue by Evaporation of Emulsified Asphalt¹

This standard is issued under the fixed designation D6934; 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 in emulsified asphalts composed principally of a semisolid or liquid asphaltic base, water, and an emulsifying agent.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.4 *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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization 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

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
D6997 Test Method for Distillation of Emulsified Asphalt
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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 Test.

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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.

3. Significance and Use

3.1 The test may be used to indicate compositional characteristics of emulsified asphalt. Evaporation residue may also be subjected to other characterization tests.

3.2 This test method for residue by evaporation tends to give an asphaltic residue lower in penetration and ductility than the distillation test method (D6997). Material may be accepted but shall not be rejected as failing to meet specifications containing requirements for determination of residue by distillation, on data obtained by evaporation. If residue from evaporation fails to meet the requirements for properties specified for residue from distillation, tests shall be rerun using the distillation test method.

NOTE 1—The quality of results produced by this standard is dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guidance provides a means of evaluating and controlling some of those factors.

4. Summary of Method

4.1 A sample of emulsified asphalt in an open top beaker is heated in an oven at 163 ± 3 °C to determine the percentage of asphalt residue. The residue from the evaporation may be tested as required.

5. Sample Conditioning for Testing

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

5.2 All emulsified asphalts with viscosity testing requirements of 50 °C shall be heated to 50 ± 3 °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 ± 3 °C, stir the sample to achieve homogeneity.

5.3 Emulsified asphalts with viscosity testing requirements of 25 °C should be mixed or stirred at 25 ± 3 °C in the original sample container to achieve homogeneity.

NOTE 2—Emulsified asphalts with viscosity testing requirements of 25 °C may be heated and stirred as specified in 5.2 if necessary. For example, if there is settlement at the bottom of the container the sample

should be heated as described in 5.2 followed by stirring to achieve homogeneity. Stirring settled emulsions at $25 \pm 3^\circ\text{C}$ can result in incomplete mixing or in some cases sieve formation.

6. Apparatus

6.1 *Beakers*, low form, 1000 mL capacity, made of glass or metal.

6.2 *Glass Rods*, with flame-polished ends, approximately 6 mm in diameter and approximately 180 mm in length.

6.3 *Balance*, capable of weighing 500 g to within 0.1 g.

6.4 *Oven*, capable of maintaining a temperature of $163 \pm 3^\circ\text{C}$.

6.5 *Sieve*, a 76.2 mm diameter, 300 μm sieve conforming to Specification E11.

7. Procedure A

7.1 Use Procedure A when determination of the percentage of residue only is required.

7.2 Determine the mass of each of three beakers containing a glass rod to 0.1 g. Pour 50 ± 0.1 g of thoroughly mixed, emulsified asphalt into each of three beakers.

7.3 Place the beakers containing the rods and sample in the oven, which has been adjusted to $163 \pm 3^\circ\text{C}$, for 2 h. At the end of this period remove each beaker and stir the residue thoroughly. Replace in the oven for 1 h, then remove the beakers from the oven, allow to cool to ambient temperature, and determine the mass with the rods.

NOTE 3—Exercise care to prevent loss of asphalt from the beaker through foaming or spattering, or both. It is permissible for the technician to observe the beakers periodically and prevent boil-over by stirring the sample. The placing of beakers and emulsified asphalt samples in a cold or warm oven and bringing the oven and sample up to a temperature of 163°C together is also permissible.

NOTE 4—If preferred, preliminary evaporation of water may be accomplished by careful heating on a hot plate, followed by oven treatment at 163°C for 1 h.

NOTE 5—Adding a square of fine mesh screen (20 mesh is suitable) to the beaker tare and placing the screen on top of the beaker prevents loss of residue due to spattering should it occur.

8. Procedure B

8.1 Use Procedure B when tests on the residue from the emulsified asphalts are required.

8.2 Proceed in accordance with 7.2 and 7.3 using four 50 ± 0.1 g samples. After the calculation for percentage of residue,

replace the beakers in the oven until the asphalt residue is sufficiently fluid to pass through a 300 μm sieve (usually requiring 15 to 30 min). Pour the residue of the four beakers through the 300 μm sieve into a single combined container. Mix to achieve homogeneity. Proceed with residue testing by pouring the homogenized residue into test containers or molds.

9. Calculation and Report

9.1 Calculate the percentage of residue on each beaker as follows:

$$\text{residue, \%} = 2(A - B) \quad (1)$$

where:

A = mass of beaker, rod, and residue, g, and

B = tare of beaker and rod, g.

9.2 Report the percentage of residue by evaporation as the average of all the beakers tested provided the maximum difference in the measurements is less than or equal to 0.4 %. If any of the measured values differs by more than 0.4 % from the other measured values, that data point should be rejected in the calculation and the average of the remaining data points should be reported.

10. Precision and Bias

10.1 The following criteria should be used for judging the acceptability of results (95 % probability):

10.1.1 Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amount:

Residue by Evaporation, %	Repeatability, mass %
50 to 70	0.4

10.1.2 The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Residue by Evaporation, %	Reproducibility, mass %
50 to 70	0.8

10.2 The bias of this test method cannot be determined because no material having an accepted reference value is available.

11. Keywords

11.1 asphalt; asphalt emulsion; cationic emulsified asphalt; emulsified asphalt; evaporation; residue

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