Designation: D6936 – 09

Standard Test Method for Determining Demulsibility of Emulsified Asphalt[1]

This standard is issued under the fixed designation D6936; 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, applicable to both anionic and cationic emulsified asphalts of the RS and MS type, measures the chemical breaking of the emulsified asphalt.

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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D511 Test Methods for Calcium and Magnesium In Water

D6934 Test Method for Residue by Evaporation of Emulsified Asphalt

D6997 Test Method for Distillation of Emulsified Asphalt

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 This test method is used to identify or classify an emulsified asphalt as an RS or MS by measuring the amount of available asphalt that is broken from the emulsified asphalt by utilizing specified amounts and concentrations of calcium chloride solution for anionic emulsified asphalts and dioctyl sodium sulfosuccinate for cationic emulsified asphalts.

4. Sample Conditioning for Testing

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

4.2 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.

4.3 Emulsified asphalts with viscosity testing requirements of 25°C should be mixed or stirred at 25 ± 1°C in the original sample container to achieve homogeneity.

NOTE 1—Emulsified asphalts with viscosity testing requirements of 25°C may be heated and stirred as specified in 4.2, if necessary. In the event the 4.2 method is used, the sample should be cooled to 25 ± 1°C before testing.

5. Apparatus and Reagents

5.1 *Wire Cloth*—1.40-mm wire cloth that is at least 125 mm square, unframed, and having wire diameters and openings that conform to Specification E11.

5.2 *Beaker*—metal beaker or other suitable metal container with a minimum 300-mL capacity.

5.3 *Stirring Rod*—metal rod with rounded ends, approximately 10 mm in diameter.

5.4 *Buret*—50-mL glass buret graduated in 0.1 mL intervals.

5.5 *Calcium Chloride Solution* (1.11 g/L)—1.11 g of calcium chloride ($CaCl_2$) dissolved in distilled or deionized water and diluted to 1 L. The 1.11 g/L calcium chloride solution may be standardized to be a 0.02 ± 0.001 N solution of calcium chloride in water. Although this solution will remain stable, it shall be stored in an airtight container when not in use.

5.6 *Calcium Chloride Solution* (5.55 g/L)—5.55 g of calcium chloride ($CaCl_2$) dissolved in distilled or deionized water and diluted to 1 L. The 5.55 g/L calcium chloride solution may be standardized to be a 0.10 ± 0.001 N solution of calcium chloride in water. Although this solution will remain stable, it shall be stored in an airtight container when not in use.

NOTE 2—Test Methods D511 is a method that could be used to standardize the solutions in 5.5 and 5.6.

5.7 *Dioctyl Sodium Sulfosuccinate Solution* (0.80 %)—8.00 g of dioctyl sodium sulfosuccinate dissolved in 992 g of distilled or deionized water. This solution will degrade over time and shall be stored in a cool dark location when not in use, in a dark glass or dark impermeable plastic air-tight container.

[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.

Current edition approved June 1, 2009. Published July 2009. Originally approved in 2004. Last previous edition approved in 2004 as D6936 – 04. DOI: 10.1520/D6936-09.

[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.

It shall not be used for testing purposes if more than 90 days have elapsed since it was prepared.

NOTE 3—A test method that could be used to standardize this solution can be found in "The United States Pharmacopeia, The National Formulary" under the section on Docusate Sodium (444.56) as recommended by Cytec Industries (a manufacturer of this material).

5.8 *Balance,* capable of weighing 500 ± 0.1 g.

5.9 *Oven,* capable of maintaining a temperature of 163 ± 3°C

6. Procedure

6.1 Determine the percentage of residue as described in Test Method D6997 or Test Method D6934.

6.2 Record the weight of the assembly consisting of: the metal beaker or metal container with a 300 mL minimum capacity, the metal stirring rod, and the wire cloth.

6.3 Weigh 100 ± 0.1 g of the 25 ± 1.0°C emulsified asphalt into the 300-mL beaker in the weighed assembly. Bring the weighed sample of emulsion and the proper reagent to a temperature of 25 ± 1.0°C.

NOTE 4—While conditioning the emulsified asphalt sample to the test temperature, it should be kept covered to avoid the evaporation of water, which will affect the residue of the test sample and consequently the test result.

6.4 Over a period of approximately 2 min, add the appropriate reagent (at room temperature) to the metal beaker or metal container, from a buret:

6.4.1 35 mL of CaCl2 0.02 N solution (1.11 g/L) (5.5) for anionic rapid setting emulsified asphalts, or

6.4.2 50 mL of 0.10 N CaCl2 solution (5.55 g/L) (5.6) for anionic medium setting, or mixing-type, emulsified asphalts, or

6.4.3 35 mL of dioctyl sodium sulfosuccinate solution (0.80 %) (5.7) for cationic rapid setting emulsified asphalts.

6.5 While adding the solution (6.4.1, 6.4.2, or 6.4.3), stir the contents of the metal beaker or container continuously and vigorously, kneading any lumps against the sides of the metal beaker or container to ensure thorough mixing of the reagent with the emulsified asphalt.

6.6 Continue kneading any lumps for an additional 2 min after all the solution has been added.

6.7 Decant the mixture of any unbroken emulsified asphalt and reagent onto the wire cloth. Rinse the metal beaker or container containing the sample and metal rod with distilled water, pouring the rinse water through the wire cloth. Knead and break up all lumps, and continue washing the metal beaker or container, rod, and wire cloth until the rinse water drains clear.

6.8 Place the wire cloth enclosing the asphalt residue in the metal beaker or container with the metal rod. Place the assembly in a 163 ± 3°C drying oven for 1 h. Allow the beaker and contents to cool and weigh. Repeat the heating and weighing until successive weights differ by no more than 0.1 g.

7. Calculation

7.1 Subtract the tare weight of the assembly from the weight of the dried assembly to obtain the demulsibility residue. Calculate the demulsibility as follows:

$$\text{Demulsibility, \%} = (A/B) \times 100 \quad (1)$$

where:

A = weight of demulsibility residue from the test of the sample of emulsified asphalt, and

B = weight of residue in 100 g of the emulsified asphalt.

8. Precision and Bias

8.1 The following criteria should be used for judging the acceptability of results of tests on RS emulsified asphalt (95 % probability):

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

Demulsibility, weight %	Repeatability, % of the mean
30 to 100	5

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

Demulsibility, weight %	Reproducibility, % of the mean
30 to 100	30

8.2 Precision does not apply when using dioctyl sodium sulfosuccinate solution in the testing of cationic emulsified asphalt for demulsibility.

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

9. Keywords

9.1 asphalt emulsion; cationic emulsified asphalt; demulsibility; emulsified asphalt; emulsion