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Standard Test Methods and Practices for Emulsified Asphalts¹

This standard is issued under the fixed designation D244; 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 U.S. Department of Defense.

1. Scope

1.1 These test methods and practices, given under the headings titled Composition, Consistency, Stability, and Examination of Residue, cover the examination of asphalt emulsions composed principally of a semisolid or liquid asphaltic base, water, and an emulsifying agent. The test methods cover the following tests and practices:

Test	Sections
Composition:	
Water Content	4 – 10
Residue and Oil Distillate by Distillation	See Test Method D6997
Residue by Evaporation	See Test Method D6934
Particle Charge of Cationic Emulsified Asphalts	See Practice D7402
Consistency:	
Viscosity (Saybolt Furol)	See Test Method D7496
Stability:	
Demulsibility	See Test Method D6936
Settlement	See Test Method D6930
Cement Mixing	See Test Method D6935
Sieve Test	See Test Method D6933
Aggregate Coating	See Practice D6998
Miscibility with Water	See Practice D6999
Freezing	See Practice D6929
Coating Ability and Water Resistance	11 – 18
Storage Stability of Asphalt Emulsion	See Test Method D6930
Examination of Residue	19 – 26
Identification Test for Rapid Setting Cationic Emulsified Asphalt	27 – 34
Identification of Cationic Slow Set Emulsions	See Practice D7402

¹ These test methods and practices are under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and are the direct responsibility of Subcommittee D04.42 on Emulsified Asphalt Test.

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Field Coating Test on Emulsified Asphalts
 Emulsified Asphalt/Job Aggregate Coating Test
 Density of Emulsified Asphalt

Residue by Low-Temperature Vacuum Distillation

35 – 40
 41 – 46
 See Test Method
 D6937
 See Test Method
 D7403

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.*

1.4 *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

~~E778 Specification for Standard Sand~~

~~D5D5/D5M Test Method for Penetration of Bituminous Materials~~

~~D70D70/D70M Test Method for Specific Gravity and Density of Semi-Solid Asphalt Binder (Pycnometer Method)~~

~~D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure~~

~~D113 Test Method for Ductility of Asphalt Materials~~

~~D128 Test Methods for Analysis of Lubricating Grease~~

~~D139 Test Method for Float Test for Bituminous Materials~~

~~D140D140/D140M Practice for Sampling Asphalt Materials~~

~~D977 Specification for Emulsified Asphalt~~

~~D2042 Test Method for Solubility of Asphalt Materials in Trichloroethylene or Toluene~~

~~D2397 Specification for Cationic Emulsified Asphalt~~

~~D3289 Test Method for Density of Semi-Solid and Solid Asphalt Materials (Nickel Crucible Method)~~

~~D6929 Practice for Evaluating the Effect of Freezing on Emulsified Asphalts~~

~~D6930 Test Method for Settlement and Storage Stability of Emulsified Asphalts~~

~~D6933 Test Method for Oversized Particles in Emulsified Asphalts (Sieve Test)~~

~~D6934 Test Method for Residue by Evaporation of Emulsified Asphalt~~

~~D6935 Test Method for Determining Cement Mixing of Emulsified Asphalt~~

~~D6936 Test Method for Determining Demulsibility of Emulsified Asphalt~~

~~D6937 Test Method for Determining Density of Emulsified Asphalt~~

~~D6997 Test Method for Distillation of Emulsified Asphalt~~

~~D6998 Practice for Evaluating Aggregate Coating using Emulsified Asphalts~~

~~D6999 Practice for Miscibility of Emulsified Asphalts~~

~~D7226 Test Method for Determining the Viscosity of Emulsified Asphalts Using a Rotational Paddle Viscometer~~

~~D7402 Practice for Identifying Cationic Emulsified Asphalts~~

~~D7403 Test Method for Determination of Residue of Emulsified Asphalt by Low Temperature Vacuum Distillation~~

~~D7404 Test Method for Determination of Emulsified Asphalt Residue by Moisture Balance Analyzer~~

~~D7496 Test Method for Viscosity of Emulsified Asphalt by Saybolt Furol Viscometer~~

~~E1 Specification for ASTM Liquid-in-Glass Thermometers~~

~~E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves~~

~~E145 Specification for Gravity-Convection and Forced-Ventilation Ovens~~

3. Sample Conditioning for Testing

3.1 All emulsions with viscosity requirements of 50 °C should be heated to 50 ± 3 °C in the original sample container in a 71 °C 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.

COMPOSITION

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.

WATER CONTENT

4. Scope

4.1 This test method covers the procedure for determining the water content of an emulsified asphalt by reflux distillation using a water trap.

5. Significance and Use

5.1 This test method measures the amount of water present in the emulsified asphalt, as distinguished from either bitumen or petroleum solvent.

6. Apparatus and Materials

6.1 *Metal Still*—The metal still (Fig. 1(a)) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the

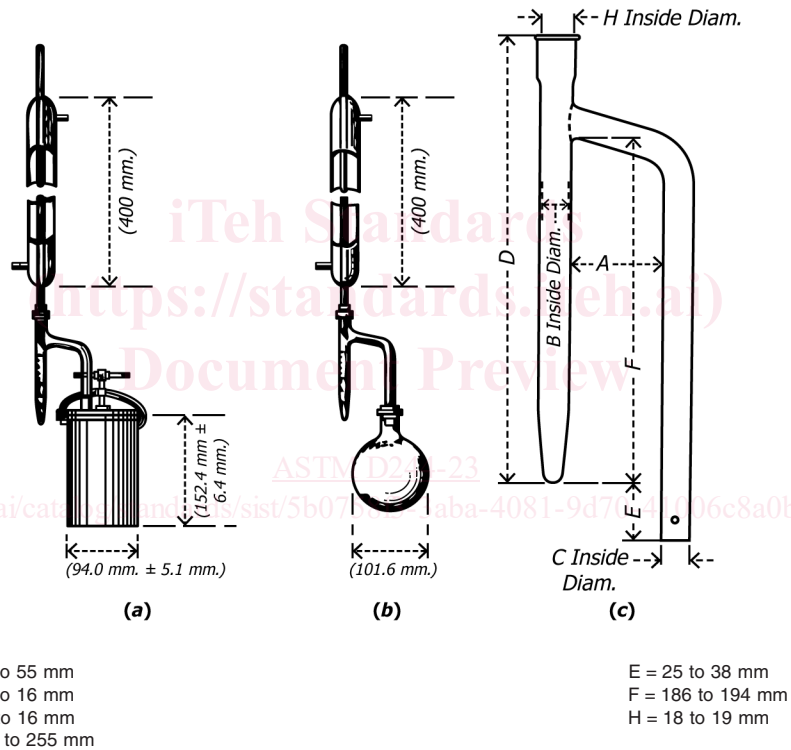


FIG. 1 Apparatus for Determining Water Content

top to which the head is tightly attached by means of a clamp. The head shall be made of metal, preferably brass or copper, and shall be provided with a tubulation 25.4 mm (1 in.) in inside diameter.

6.2 *Glass Still*—The glass still (Fig. 1(b)) shall be a short-neck, round-bottom flask, made of well-annealed glass, and having an approximate capacity of 500 mL.

6.3 *Heat Source*—The heat source used with the metal still shall be a ring gas burner of 100 mm (4 in.) inside diameter or an electric mantle heater. The heat source for the glass still shall be either an ordinary gas burner or an electric heater.

6.4 *Condenser*—The condenser shall be a water-cooled reflux glass-tube type, having a jacket not less than 400 mm in length, with an inner tube 9.5 to 12.7 mm in outside diameter. The end of the condenser shall be ground to an angle of $30 \pm 5^\circ$ from the vertical axis of the condenser.

6.5 *Trap*—The trap shall be made of annealed glass constructed in accordance with Fig. 1(c) and shall be graduated in ~~0.10 mL~~ 0.10 mL divisions from 0 to 2 mL, and in ~~0.20 mL~~ 0.20 mL divisions from 2 to 25 mL.

6.6 *Solvent*—Xylol or other petroleum distillate conforming to the following distillation requirements: 98 % distills between 120 and 250 °C. This distillation shall be conducted in accordance with Test Method **D86**.

7. Sample

7.1 Obtain a representative sample of the material for test using standard procedures as specified in Practice ~~D140~~D140/D140M.

NOTE 1—The difficulties in obtaining representative samples for this determination are unusually great, so ~~that~~ the importance of sampling cannot be too strongly emphasized.

8. Procedure

8.1 When the material to be tested contains less than 25 % water, place 100 ± 0.1 g of sample in the still. When the material contains more than 25 % water, use a 50 ± 0.1 ~~g~~ 0.1 g sample. Thoroughly mix the sample to be tested with 200 mL of solvent by swirling, taking proper care to avoid any loss of material.

8.2 Connect the still, trap, and condenser by means of tight-fitting corks as shown in Fig. 1(a) or (b). Adjust the end of the condenser in the trap to a position which will allow the end to be submerged to a depth of not more than 1 mm below the surface of the liquid in the trap after distillation conditions have been established. When using the metal still, insert a heavy paper gasket, moistened with the solvent, between the lid and flange before attaching the clamp.

8.3 When the ring burner is used with the metal still, place it about 76.2 mm above the bottom of the still at the beginning of the distillation, and gradually lower it as the distillation proceeds. Regulate the heat so that the condensate falls from the end of the condenser at a rate of from 2 to 5 drops per second. Continue the distillation at the specified rate until no water is visible on any part of the apparatus and a constant volume of water is obtained in the trap. Remove any persistent ring of condensed water in the condenser tube by increasing the rate of distillation for a few minutes.

9. Calculation and Report

9.1 Calculate the water content as follows:

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

where:

A = volume of water in trap, mL, and
 B = original weight of sample, g.

9.2 Report the result as “... water weight percent, ASTM D244.”

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:

Water Content, weight % 30 to 50	Repeatability, weight % 0.8
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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:

COATING ABILITY AND WATER RESISTANCE

11. Scope

11.1 This test method is intended to aid in the identification of asphalt emulsions suitable for mixing with coarse-graded calcareous aggregates. It can be applied to other aggregates. (See [Note 2](#).)

12. Significance and Use

12.1 This test method covers the determination of the ability of an asphalt emulsion to (1) coat an aggregate thoroughly, (2) withstand a mixing action while remaining as a film on the aggregate, and (3) resist the washing action of water after completion of the mixing.

13. Apparatus

13.1 *Mixing Pan*—A white-enameled kitchen saucepan with handle, of approximately ~~3-L~~ 3 L capacity.

13.2 *Mixing Blade*—A putty knife with a 31.8 by ~~88.9-mm~~ 88.9 mm steel blade with rounded corners. A ~~254.0-mm~~ 254.0 mm kitchen mixing spoon may be used as an alternative.

13.3 *Sieves*—Standard ~~19.0-mm and 4.75-mm~~ 19.0 mm and 4.75 mm sieves conforming to Specification [E11](#).

13.4 *Constant-Head Water-Spraying Apparatus*—An apparatus for applying tap water in a spray under a constant head of 774.7 mm ([Figs. 2 and 3](#)). The water shall issue from the apparatus in a low-velocity spray.

13.5 *Thermometer*—An ASTM low softening point thermometer 15F (or 15C), having a range from -2 to 80 °C and conforming to the requirements in Specification [E1](#).

13.6 *Balance*, capable of weighing 1000 g to within ± 0.1 g.

13.7 *Pipet*, of ~~10-mL~~ 10 mL capacity.

14. Materials

14.1 *Aggregate*—Standard reference aggregate³ shall be a laboratory-washed and air-dried limestone aggregate graded to pass the ~~19.0-mm~~ 19.0 mm sieve and be retained on the ~~4.75-mm~~ 4.75 mm sieve.

NOTE 2—Aggregates other than limestone may be used, provided calcium carbonate is omitted throughout the test method. Laboratory washing and air drying of such aggregates shall also be omitted.

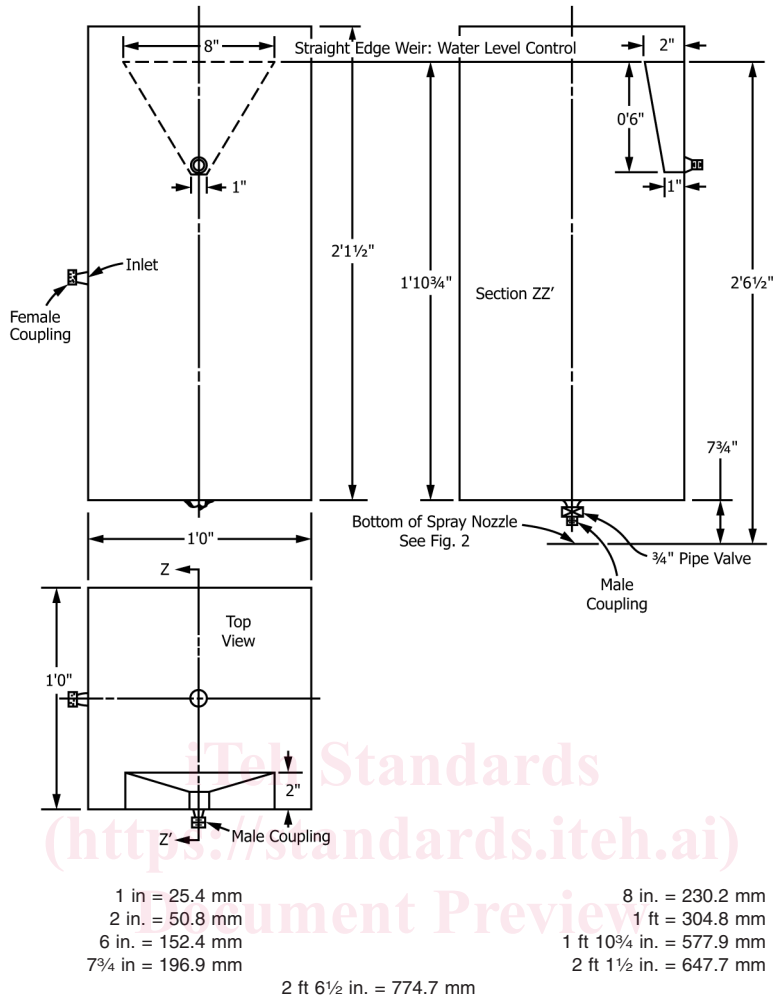
14.2 *Calcium Carbonate*—Chemically pure, precipitated calcium carbonate (CaCO_3) shall be used as a dust to be mixed with the standard reference aggregate.

14.3 *Water*—Tap water of not over 250 ppm CaCO_3 hardness for spraying over the sample.

15. Sample

15.1 The sample shall be representative of the asphalt emulsion to be tested.

³ Limestone from the Monon Stone Co. of Monon, IN, has been found suitable as reference aggregate.



NOTE 1—Use galvanized steel sheeting for the tank. All joints and fitting attachments shall be soldered and shall be watertight. All couplings shall be standard brass garden hose fittings. The 19 mm pipe valve shall be placed as close as possible to the bottom of the tank, allowing space to shut off the valve. The tank shall be placed on a suitable stand, so that the distance from the bottom of the spray head to the top of the test sample is 0.914 m ± 25.4 mm.

FIG. 2 Constant-Head Flow Tank

16. Procedure for Tests with Dry Aggregate

16.1 Carry out the test at 23.9 ± 5.5 °C.

16.2 Weigh 461 g of the air-dried, graded reference aggregate in the mixing pan.

16.3 Weigh 4.0 g of CaCO₃ dust in the mixing pan and mix with the 461 g of aggregate for approximately 1 min by means of a mixing blade to obtain a uniform film of dust on the aggregate particles.

NOTE 3—The total weight of aggregate and dust shall equal 465 g. If no calcium carbonate is included, the weight of aggregate alone shall be 465 g.

16.4 Weigh 35 g of the asphalt emulsion into the aggregate in the pan and mix vigorously with the mixing blade for 5 min using a tossing action created by a back-and-forth motion in an elliptical path of the mixing blade or spoon. At the end of the mixing period, tilt the pan and permit any excess emulsion not on the aggregate to drain from the pan.

16.5 Remove approximately one half of the mixture from the pan and place it on absorbent paper and evaluate the coating.