Standard Method of Test for

Emulsified Asphalts

AASHTO Designation: T 59-22

Technically Revised: 2022

Technical Subcommittee: 2a, Emulsified Asphalts

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1. SCOPE

1.1. These test methods, given under the headings titled Composition, Consistency, Stability, Examination of Residue, Identification Tests, Coating Tests on Emulsified Asphalts, and Density of Emulsified Asphalts, cover the examination of Emulsified Asphalt composed principally of a semisolid or liquid asphalt base, water, and an emulsifying agent. The methods cover the following tests:

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1.2. The values stated in SI units are to be regarded as the standard. The values stated in parentheses are provided 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 and health practices and determine the applicability of regulatory limitations prior to use.*

1.4. *The quality of the results produced by this standard are 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 R 18 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with R 18 alone does not completely assure reliable results. Reliable results depend on many factors; following the suggestions of R 18 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.*

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:

- M 85, Portland Cement
- M 140, Emulsified Asphalt
- M 208, Cationic Emulsified Asphalt
- M 231, Weighing Devices Used in the Testing of Materials
- M 316, Polymer-Modified Cationic Emulsified Asphalt
- M 339M/M 339, Thermometers Used in the Testing of Construction Materials
- R 18, Establishing and Implementing a Quality Management System for Construction Materials Testing Laboratories
- R 61, Establishing Requirements for Equipment Calibrations, Standardizations, and Checks
- R 66, Sampling Asphalt Materials
- T 44, Solubility of Bituminous Materials
- T 49, Penetration of Bituminous Materials
- T 50, Float Test for Bituminous Materials
- T 51, Ductility of Asphalt Materials
- T 72, Test for Saybolt Viscosity
- T 98M/T 98, Fineness of Portland Cement by the Turbidimeter
- T 111, Mineral Matter or Ash in Asphalt Materials
- T 153, Fineness of Hydraulic Cement by Air Permeability
- T 228, Specific Gravity of Semi-Solid Asphalt Materials
- T 382, Viscosity of Emulsified Asphalt by a Rotational Paddle Viscometer

2.2. *ASTM Standards*:

- C778, Standard Specification for Standard Sand
- D86, Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
- D3289, Standard Test Method for Density of Semi-Solid and Solid Asphalt Materials (Nickel Crucible Method)
- E1, Standard Specification for ASTM Liquid-in-Glass Thermometers
- E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E230/E230M, Standard Specification for Temperature Electromotive Force (emf) Tables for Standardized Thermocouples
- E879, Standard Specification for Thermistor Sensors for General Purpose and Laboratory Temperature Measurements
- E1137/E1137M, Standard Specification for Industrial Platinum Resistance Thermometers
- E2251, Standard Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
- E2877, Standard Guide for Digital Contact Thermometers

2.3. *International Electrotechnical Commission Standards*:

- IEC 60584 1: 2013 Thermocouples Part 1: EMF Specifications and Tolerances
- IEC 60751: 2008 Industrial Platinum Resistance Thermometers and Platinum Temperature Sensors

3. CALIBRATION, STANDARDIZATION, AND CHECK

3.1. Unless otherwise specified, follow the requirements and intervals for equipment calibration, standardizations, and checks in R 18.

3.2. Follow the procedures for performing equipment calibration, standardization, and checks found in R 61.

4. SAMPLING AND SAMPLE CONDITIONING FOR TESTING

4.1. *Scope*:

4.1.1. This practice describes how to sample and prepare the emulsified asphalt for testing.

4.2. *Significance and Use*:

4.2.1. To obtain a representative sample and adequate quantity of emulsified asphalt for testing, sample the emulsified asphalt in accordance with R 66, obtaining 3.8 L (1 gallon) of material, or quantity as specified by the agency.

4.2.2. Before performing the tests listed in this standard, the test samples must be conditioned in accordance with M 140, M 208, or M 316 to ensure that the emulsified asphalt is in its optimal state for determining its true properties. Failure to condition the test samples according to the requirements of this standard will result in a nonconforming test situation.

4.3. *Apparatus*:

4.3.1. *Thermometers*—Thermometers for measuring sample conditioning shall meet the requirements of M 339M/M 339 with a temperature range of at least 20 to 80°C (68 to 176°F), and an accuracy of ±0.5°C (±0.9°F) (see Note 1).

Note 1—Thermometer types suitable for use include ASTM E1 mercury thermometers as listed in Table 1; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

Table 1—Conditioning Temperatures and Suitable Ranges for Thermometers

Initial Sample Temperature	Required Viscosity Temperature	Conditioning Temperature Adjustment	Maximum Bath or Oven Temperature	Minimum Thermometer Temperature Range
Below 21°C (70°F)	25°C (77°F)	Bring sample to at least 21°C (70°F)	74°C (165°F), if used	19 to 27°C (66 to 80°F)
Above 25°C (77°F)	25°C (77°F)	Cool sample to appropriate test temperature identified in the test section of this standard	n/a	19 to 27°C (66 to 80°F)
Below 50°C (122°F)	50°C (122°F)	Bring sample to at least 50°C (122°F)	74°C (165°F)	49 to 57°C (120 to 134°F) or 19 to 27°C (66 to 80°F)
Above 50°C (122°F)	50°C (122°F)	Cool sample to appropriate test temperature identified in the test section of this standard	n/a	49 to 57°C (120 to 134°F)

4.3.2. *Water Bath or Oven*—A vessel for heating or maintaining the temperature of the samples shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 20 to 80°C (68 to 176°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 2).

Note 2—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

4.3.3. *Stirring Rod*—A glass or metal rod for stirring the sample. Alternatively, a non-mercury thermometer may be used as the stirring rod.

4.4. *Procedure*:

4.4.1. Condition the sample to the appropriate temperature in Table 1 based on the initial temperature and the viscosity temperature of the sample as noted.

4.4.2. Stir the sample before testing.

Note 3—Stirring prematurely can damage the emulsion and possibly alter test results. Some higher viscosity emulsions may require periodic stirring after reaching 21°C (70°F).

Note 4—The container may be vented to relieve pressure.

COMPOSITION

5. WATER CONTENT

5.1. *Scope*:

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

5.2. *Significance and Use*:

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

5.3. *Apparatus and Materials*:

5.3.1. *Metal Distillation Pot*—The metal distillation pot [Figure 1(a)] shall be a vertical cylindrical vessel, preferably made of copper, having a faced flange at the 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 (ID).

5.3.2. *Glass Distillation Pot*—The glass distillation pot [Figure 1(b)] shall be a short-neck, round-bottom flask, made of well-annealed glass, and having an approximate capacity of 500 mL.

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

5.3.4. *Condenser*—The condenser shall be a water-cooled, reflux glass-tube type, having a jacket not less than 400 mm ($15^{3}/_{4}$ in.) in length, with an inner tube 9.5 to 12.7 mm ($^{3}/_{8}$ to $^{1}/_{2}$ in.) in outside diameter (OD). The end of the condenser shall be ground to an angle of 30 degrees ±5 degrees from the vertical axis of the condenser.

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

5.3.6. *Solvent*—Xylol or other petroleum distillate conforming to the following distillation requirements: 98 percent distills between 120 and 250°C (248 and 482°F). This distillation shall be conducted in accordance with ASTM D86.

5.3.7. *Balance*—Conforming to the requirements of M 231, Class G 2.

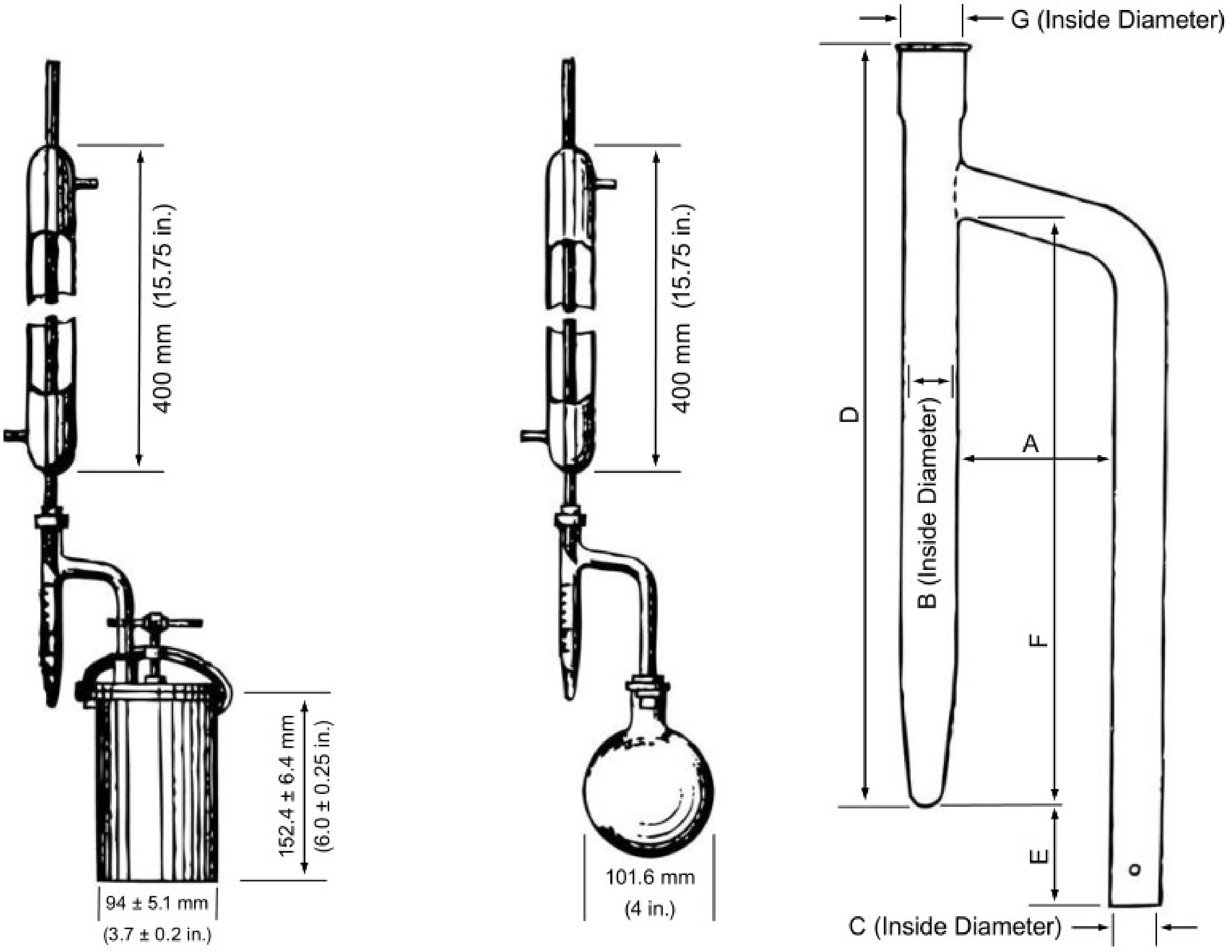
5.4. *Sample*:

5.4.1. Obtain a representative sample of the material as specified in R 66.

Note 5—The difficulties in obtaining representative samples for this determination are unusually great, so the importance of proper sampling cannot be too strongly emphasized.

5.5. *Procedure*:

5.5.1. When the material to be tested contains 25 percent or less water, place 100 ± 0.1 g of the sample in the distillation pot. When the material contains more than 25 percent water, use a 50 ± 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.



(a) (b) (c)

Metric Equivalents

A = 45 to 55 mm (1.8 to 2.2 in.)
B = 14 to 16 mm (0.55 to 0.62 in.)
C = 12 to 16 mm (0.47 to 0.62 in.)
D = 235 to 255 mm (9.3 to 10.0 in.)
E = 25 to 38 mm (1.0 to 1.5 in.)
F = 186 to 194 mm (7.3 to 7.6 in.)
G = 18 to 19 mm (0.71 to 0.75 in.)

Figure 1—Apparatus for Determining Water Content

5.5.2. Connect the distillation pot, trap, and condenser by means of tight-fitting corks as shown in Figure 1(a) or 1(b). Adjust the end of the condenser in the trap to a position that will allow the end to be submerged to a depth of not more than 1 mm (0.04 in.) below the surface of the liquid in the trap after distillation conditions have been established. When using the metal distillation pot, insert a heavy paper gasket, moistened with the solvent, between the lid and flange before attaching the clamp. Insert a loose cotton plug in the top of the condenser tube to prevent condensation of atmospheric moisture.

5.5.3. When the ring burner is used with the metal distillation pot, place it about 75 mm (3 in.) above the bottom of the distillation pot 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 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 (V_{wt}). Remove any persistent ring of condensed water in the condenser tube by increasing the rate of distillation for a few minutes.

5.6. *Calculation and Report*:

5.6.1. Calculate the water content as follows:

$$\text{Water Content, \%} = (V_{wt} / M_s) \times 100 \quad (1)$$

where:

V_{wt} = volume of water in the trap, mL; and

M_s = original mass of the sample, g.

5.6.2. Report the result as "water mass percent."

5.7. *Precision and Bias*:

5.7.1. The following criteria should be used for judging the acceptability of results (95 percent probability):

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

Water Content, mass %	Repeatability, mass %
30 to 50	0.8

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

Water Content, mass %	Reproducibility, mass %
30 to 50	2.0

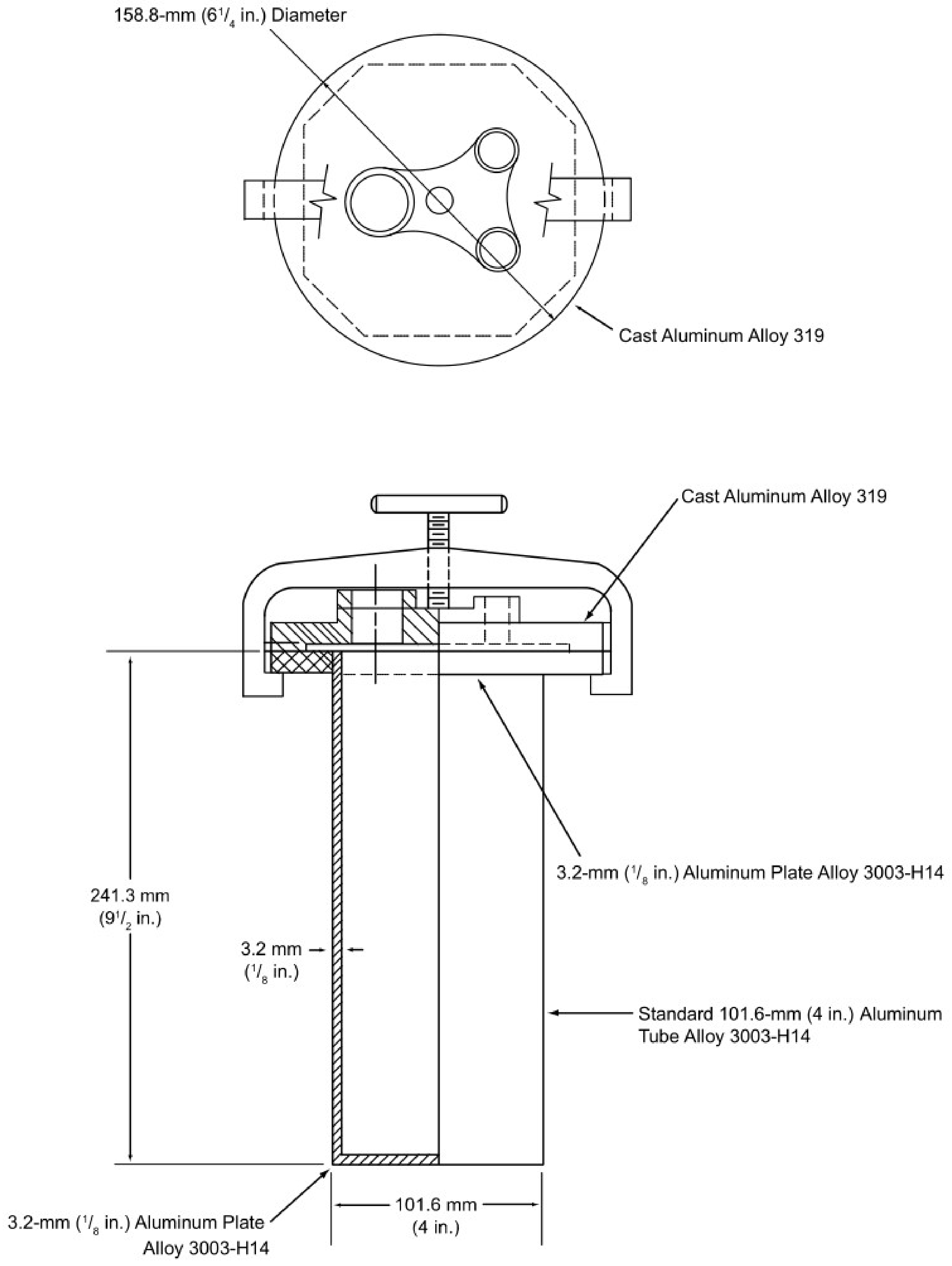
6. RESIDUE AND OIL DISTILLATE BY DISTILLATION

6.1. *Scope*:

6.1.1. This test method covers the quantitative determination of residue and oil distillate in emulsified asphalt composed principally of a semisolid or liquid asphalt base, water, and an emulsifying agent.

6.2. *Significance and Use*:

6.2.1. This test method can be used for quantitative determination of residue and oil distillate in emulsified asphalt for specification acceptance, service evaluation, control, and research. This method can also be used to obtain residue and oil distillate for further testing.



Note: The distillation pot cover may be machined from Rolled Aluminum Plate Alloy 3003-H14.

Figure 2—Example of an Aluminum-Alloy Distillation Pot (Approximate dimensions, see Section 6.3.1.)

6.3. *Apparatus*:

6.3.1. *Aluminum-Alloy Distillation Pot*[1] *and Heat Source*—Approximately 240 mm ($9^{1}/_{2}$ in.) in height by 95 mm ($3^{3}/_{4}$ in.) in inside diameter (Figure 2) with one 121-mm ($4^{3}/_{4}$-in.) inside diameter ring burner,[2] having holes on the inner periphery and having three spacers to ensure centering of the burner around the distillation pot (Figure 3).

Note 6—Residue by distillation obtained with iron stills in accordance with T 59-65 is acceptable. Similarly, results obtained with a 127-mm (5-in.) ring burner as in subsequent issues of T 59 are acceptable.

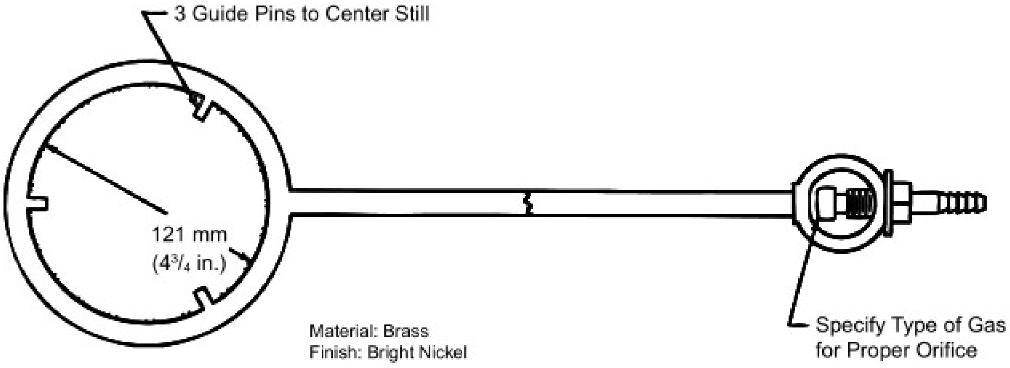


Figure 3—Ring Burner with 121-mm ($4^{3}/_{4}$-in.) Inside Diameter

6.3.2. *Connection Apparatus*—Consisting of a glass connecting tube, tin shield, and water-cooled glass condenser of the West or Liebig type with a borosilicate glass or metal jacket and a suitable adapter between the condenser and a 100-mL graduated cylinder, all with dimensions as shown in Figure 4.

6.3.3. *Graduated Cylinder*—100-mL capacity with graduation intervals of 1.0 mL.

Note 7—Larger cylinders may be used if a product's distillate exceeds 100 mL.

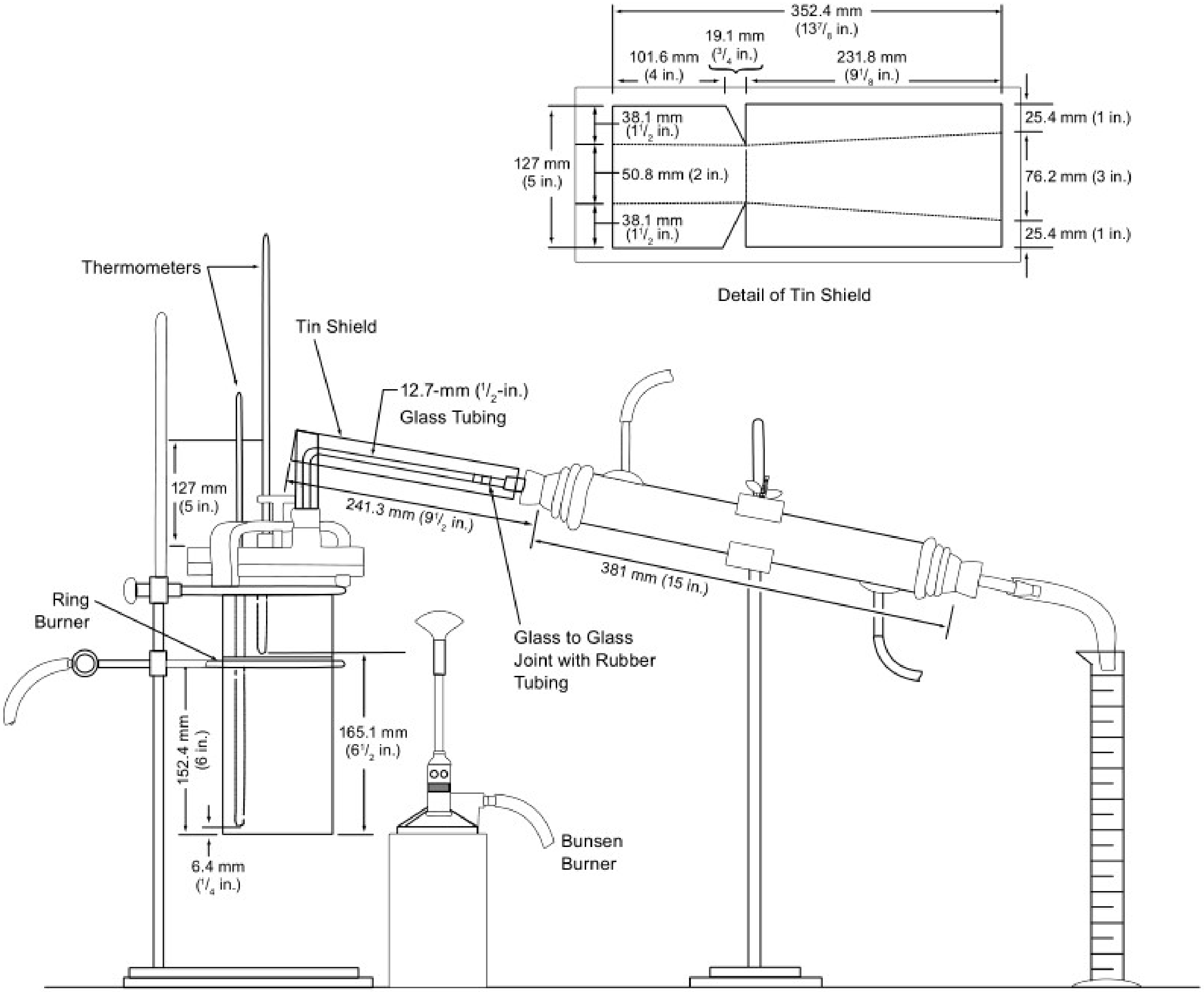
6.3.4. *Thermometers*—Two thermometers shall be constructed such that they can be positioned in the manner described in Section 6.4.3. The thermometers shall meet the requirements of M 339M/M 339 with a temperature range of at least -2 to 300°C (30 to 580°F), and an accuracy of ±0.5°C (±0.9°F) (see Note 8). For materials that distill at lower temperature ranges, the range of the thermometers shall include the range of temperature expected to be encountered during the distillation (see Table 2). The thermometers shall be constructed such that they can be positioned in the manner described in Section 6.4.3.

Note 8—Thermometer types suitable for use include ASTM E1 mercury thermometer No 7C (7F); ASTM E1137/E1137M Pt-100 RTD platinum resistance thermometer, Special order; or IEC 60751: 2008 Pt-100 RTD platinum resistance thermometer, Special order.

6.3.5. *Balance*—Conforming to the requirements of M 231, Class G 2.

6.3.6. *Sieve*—A 0.300-mm (No. 50) sieve conforming to ASTM E11.

6.3.7. *Bunsen Burner.*



Metric Equivalents															
mm	6.5	13	19	25	38	51	76	102	127	152	165	232	241	352	381
in.	1/4	1/2	3/4	1	1 1/2	2	3	4	5	6	6 1/2	9 1/8	9 1/2	13 7/8	15

Figure 4—Apparatus Assembly for Distillation Test of Emulsified Asphalt

6.4. *Procedure*:

6.4.1. Determine the mass of the aluminum-alloy distillation pot (including the lid, clamp, cork, thermometers, and gaskets, if a gasket is used.) Pour 200 ± 0.1 g of a representative sample of the emulsified asphalt into the distillation pot.

6.4.2. Use a gasket of oiled paper between the distillation pot and its cover or grind the joint to a tight fit. Securely clamp the cover on the distillation pot.

6.4.3. Insert a thermometer, through a cork, in each of the small holes provided in the cover. Adjust these thermometers so that the end of the bulb of one is approximately 6 mm (1/4 in.) from the bottom of the distillation pot and the bulb of the other is approximately 165 mm (6 1/2 in.) from the bottom of the distillation pot.

6.4.4. Place the ring burner around the distillation pot about 150 mm (6 in.) from the bottom of the distillation pot. Apply heat by lighting this burner and adjusting it to a low flame. Also apply just enough heat from a burner to the connecting tube to prevent condensation of water in this tube.

6.4.5. Move the ring burner approximately level with the bottom of the distillation pot when the temperature on the lower thermometer is approximately 215°C (420°F). Increase the temperature to 260 ± 5°C (500 ± 10°F), as read on the lower thermometer, maintaining it at this temperature for 15 min. Complete the total distillation in 60 ± 15 min from the first application of heat. Alternative distillation temperature ranges specified by the manufacturer should be used when testing polymer modified samples, but this shall be approved by the purchaser. When lower distillation temperatures are used, replace the "temperature to lower the ring" in this section and the "correction factor to add to the mass of the still" in Equation 2 according to Table 2.

Table 2—Alternative Distillation Requirements for Testing Polymer-Modified Samples

Distillation Temperature, °C (°F)	Temperature to Lower the Ring, °C (°F)	Correction Factor
260 (500)	215 (420)	1.5
230 (450)	195 (380)	1.4
205 (400)	170 (340)	1.2
175 (350)	150 (300)	1.1

Note 9—The location of the burner at the start of the test is flexible. It may be raised to decrease the chance of "foam-over" or lowered to the middle of the distillation pot for emulsified asphalt containing no solvent. A sudden drop in the temperature reading of the upper thermometer indicates that excess foaming is occurring. Quickly remove the heat from the pot by immersion in a pan of cold water, or other appropriate means, until the foaming ceases. (The ring burner may be gradually lowered as the distillation proceeds to ensure that the time requirements of this test are satisfied.)

6.4.6. Immediately at the expiration of the heating period, determine the mass of the distillation pot and accessories again as described in Section 6.4.1. Calculate and report the percentage of residue by distillation according to Equation 2. Record the volume of oil distillate to the nearest $^1/_2$ mL (V_{od}). Calculate and report the oil distillate as a volume percentage of the total emulsified asphalt according to Equation 3. Save this oil distillate if identification is desired.

Note 10—The mass of the aluminum-alloy distillation pot at room temperature (Section 6.4.1) is 1.5 g higher than at 260°C (500°F). Correct for this error by adding 1.5 g (or the other correction factor value in Table 2 that corresponds to the distillation temperature chosen) to the gross mass obtained in Section 6.4.6 prior to calculating the percentage of residue by distillation (M_{pr} as provided in Equation 2).

6.4.7. Remove the cover from the distillation pot; stir, and immediately pour appropriate portions of the residue into a 236-mL (8-oz) tin or other suitable molds and containers for performing the required tests. If the residue does not readily pour from the still, use a scraper or spatula to remove the remaining material. Handle or condition molds and containers for the desired examination of the residue as described in Sections 21 to 26 and proceed as required by the appropriate method from the points that follow the pouring stage. If there is foreign matter in the residue, and the residue will be used for further testing, the material shall be poured through a 300-µm (No. 50) sieve prior to pouring it into the test molds and containers.

6.5. *Calculation and Report*:

6.5.1. Calculate the percentage of residue in the sample as follows:

$$P_{or} = (M_{pr} - M_p)/2 \qquad (2)$$

where:

P_{or} = percentage of oil residue;

M_{pr} = mass of the distillation pot, accessories, and residue after the test (+ 1.5 g or other appropriate correction factor identified in Table 2), g; and

M_p = mass of the distillation pot and accessories before the test, g.

6.5.2. Calculate the percentage of oil distillate as follows:

$$P_{od} = V_{od}/2 \qquad (3)$$

where:

P_{od} = percentage of oil distillate (by volume of emulsified asphalt); and

V_{od} = volume of oil distillate, to the nearest 0.5 mL.

6.5.3. Report both the percentage of asphalt residue and percentage of asphalt distillate to the nearest 0.1 percent.

6.6. *Precision and Bias*:

6.6.1. The following criteria should be used for judging the acceptability of results (95 percent probability):

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

Residue by Distillation, mass %	Repeatability, mass %
50 to 70	1.0

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

Residue by Distillation, mass %	Reproducibility, mass %
50 to 70	2.0

7. EMULSIFIED ASPHALT RESIDUE BY EVAPORATION

7.1. *Scope*:

7.1.1. This test method covers the quantitative determination of emulsified asphalt residue in emulsified asphalt composed principally of a semisolid or liquid asphalt base, water, and an emulsifying agent.

7.2. *Significance and Use*:

7.2.1. The test may be used to indicate compositional characteristics of emulsified asphalt. Evaporation residue may also be subjected to other characterization tests outlined under the "examination of residue" portions of this method (Sections 21 to 26). However, properties of the residue from the evaporation procedure may differ from those from the distillation residue (see Section 7.4.2.1).

7.2.2. This test is to be conducted using four sets of beakers and rods.

Note 11—At the discretion of the specifier of the material the test may be conducted using two sets of beakers and rods. If there is any doubt about the validity of the test results using two sets of beakers and rods, the test shall be conducted using four sets of beakers and rods. The precision and bias statement was developed using four sets of beakers.

7.3. *Apparatus*:

7.3.1. *Beakers*—Low form, l000-mL capacity, made of glass or metal, or a container of similar capacity.

7.3.2. *Glass Rods*—With flame-polished ends, approximately 6 mm ($^1/_4$ in.) in diameter and at least 180 mm (7 in.) in length.

7.3.3. *Balance*—Conforming to the requirements of M 231, Class G 2.

7.3.4. *Oven*—A convection oven that can maintain a temperature of 163 ± 3°C (325 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 150 to 175°C (302 to 347°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 12).

Note 12—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

7.3.5. *Screen (optional)*—A wire mesh screen such as window screen or other suitable perforated sheet that can be placed on top of the beaker to reduce sample loss from splatter.

7.4. *Procedure*:

7.4.1. Determine the mass of each beaker containing a glass rod and screen (if used) to the nearest 0.1 g (M_{br}). Pour 50 ± 0.1 g of thoroughly mixed emulsified asphalt into each of four beakers (see Section 7.2.2). Place the beakers containing the rods and the sample in the oven, the temperature of which has been adjusted to 163 ± 3°C (325 ± 5°F), for 2 h. At the end of this period, remove each beaker, and stir the residue thoroughly. Replace the beakers in the oven for 1 h; then remove the beakers from the oven. Allow them to cool to room temperature and determine the mass of each beaker (containing a rod and screen (if used), and the residue from the sample). Designate this mass as M_{brr}.

Note 13—Care must be taken to prevent the loss of asphalt from the beaker through foaming or spattering. The placing of the beakers and emulsified asphalt samples in the oven and bringing the oven and sample to a temperature of 163°C (325°F) together is permissible. If preferred and when the physical testing of the residue is not being performed, preliminary evaporation of water may be accomplished by careful heating, to avoid loss of asphalt, on a hot plate, followed by oven heating at 163°C (325°F) for 1 h.

7.4.2. When tests on the residue from the emulsified asphalt are required, replace the beakers in the oven until the asphalt residue is sufficiently fluid to pour (usually requiring 15 to 30 min). Pour the residue into suitable containers and molds for performing such tests as desired.

7.4.2.1. The method for residue by evaporation described in Section 7 tends to give an asphalt residue lower in penetration and ductility than the distillation method described in Section 6. 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 by evaporation fails to meet the requirements for properties specified for residue by distillation, the tests shall be repeated using the distillation method.

7.5. *Calculation and Report*:

7.5.1. Calculate the percentage of residue for each beaker as follows:

$$\text{Residue, \%} = 2(M_{brr} - M_{br}) \qquad (4)$$

where:

M_{brr} = mass of the beaker, rod, screen (if used) and residue, g; and

M_{br} = tare mass of the beaker, rod, and screen (if used), g.

7.5.2. Report the percentage of residue by evaporation as the average of the four results (see Section 7.2.2).

7.6. *Precision and Bias*:

7.6.1. The following criteria should be used for judging the acceptability of results (95 percent probability):

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

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

7.6.1.2. 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, mass %	Reproducibility, mass %
50 to 70	0.8

8. PARTICLE CHARGE OF CATIONIC EMULSIFIED ASPHALTS

8.1. *Scope*:

8.1.1. This test method is used to identify cationic emulsified asphalt. Positively charged particles are classified as cationic.

8.2. *Significance and Use*:

8.2.1. Cationic emulsified asphalt is identified by the migration of the particles to a negatively charged electrode (cathode) by means of a direct current.

8.3. *Apparatus*:

8.3.1. *Current Source*—Consisting of 12-V DC, a milliammeter, and a variable resistor (Figures 5 and 6).

8.3.2. *Electrodes*—Two stainless steel plates, 25.4 mm by 101.6 mm (1 in. by 4 in.) insulated from each other and rigidly held parallel 12.7 mm ($^1/_2$ in.) apart (Figure 7).

8.3.3. *Insulator*—Polytetrafluoroethylene resin square rod, virgin electrical grade, 12.7 mm ($^1/_2$ in.) thick (see Figure 7). An insulator made from other suitable material may be used.

8.3.4. *Beaker*—250 mL capacity.

8.3.5. *Glass Rod*—101.6 mm (4 in.) long and 6.35 mm ($^1/_4$ in.) thick or other suitable material or device that is capable of insulating and suspending the electrode assembly in the emulsified asphalt.

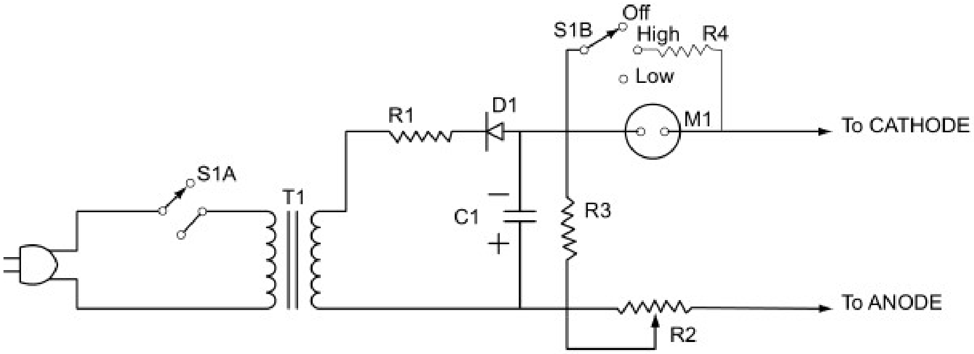
8.3.6. *Oven or Water Bath*—Shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 45 to 80°C (113 to 176°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 14).

Note 14—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

8.3.7. *Thermometer*—The thermometer for measuring the temperature of the materials in the water bath shall meet the requirements of M 339M/M 339 with a temperature range of at least 45 to 80°C (113 to 176°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 15).

Note 15—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

Figure 5—Particle Charge Tester



C1—500-μF, 25-V capacitor
D1—silicon diode
M1—0- to 10-mA milliammeter
R1—47-Ω, 1-W resistor
R2—5000-Ω potentiometer
R3—6800-Ω ,$^{1}/_{4}$-W resistor

R4—meter shunt (determined by type of meter used)
S1—2-pole, 3-position rotary switch
T1—12.6-V filament transformer

Figure 6—Particle Charge Tester Circuit Diagram

8.3.8. *Timer*—Graduated in 0.1 s and accurate within 0.1 percent when tested over a 15-min interval.

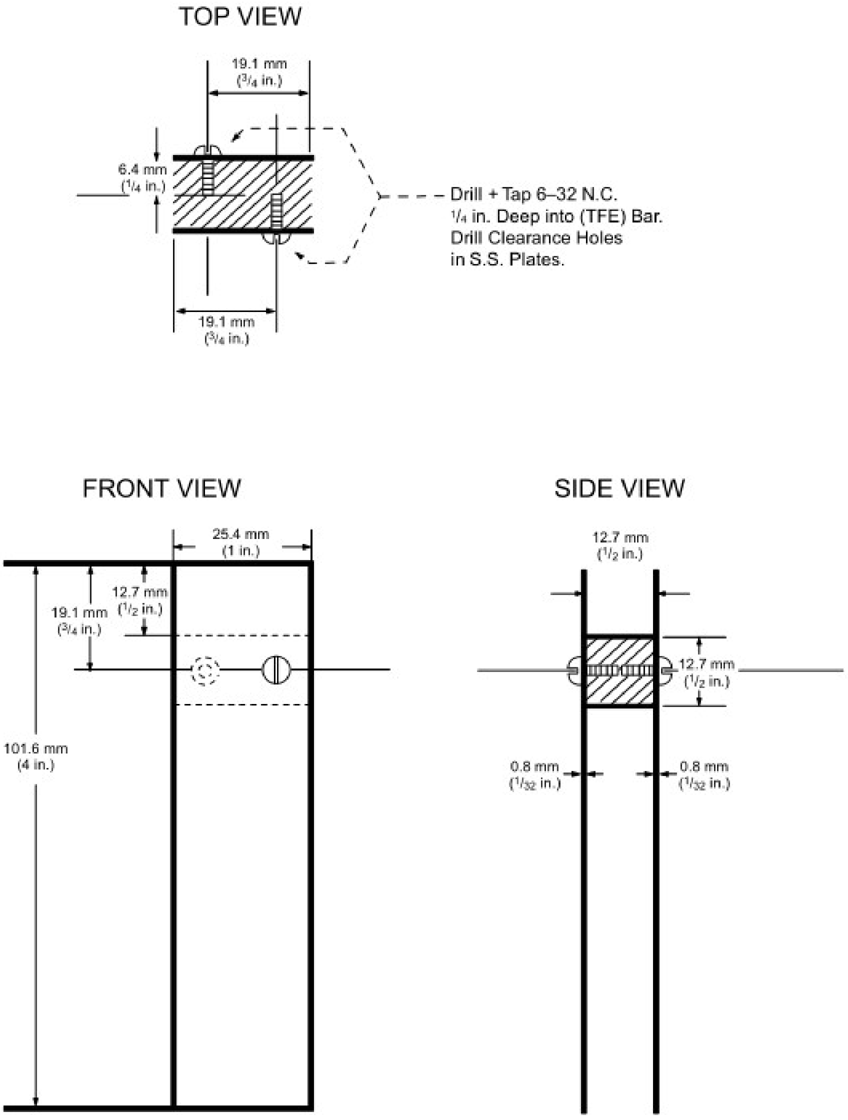


Figure 7—Insulator

8.4. *Procedure*:

8.4.1. Heat the emulsified asphalt to be tested to 50 ± 3°C (122 ± 5°F) in a water bath or oven with a temperature not to exceed 74°C (165°F). Stir the emulsified asphalt thoroughly to ensure uniformity of temperature.

8.4.2. Pour the emulsified asphalt to be tested into the 250-mL beaker to a height that will allow the electrodes to be suspended 25.4 mm (1 in.) in the emulsified asphalt. To facilitate suspension of the electrodes, insert the glass rod or equivalent between the two electrodes under the insulator. Place the ends of the glass rod or equivalent on the two opposite top edges of the beaker. An apparatus capable of manual height adjustment to insulate and suspend the electrode assembly in the emulsified asphalt may be used if desired.

8.4.3. Connect the electrodes, properly cleaned according to Section 8.4.3.1, to the DC source.

8.4.3.1. New electrodes and electrodes to be reused should be cleaned in the following sequence:

1. Wash with distilled water;
2. Wash with a suitable asphalt solvent;
3. Wash with isopropyl or ethyl alcohol; and
4. Wash with distilled water.

8.4.4. Adjust the current to a minimum of 8 mA with the variable resistor and start timing with a suitable timing device.

Note 16—Higher current levels may be specified, but the current used shall be reported.

8.4.5. When the current drops to 2 mA or at the end of 30 min, whichever occurs first, disconnect the current source and gently wash the electrodes with a smooth, thin stream of distilled water.

8.4.6. Observe the asphalt deposit on the electrodes. A cationic emulsified asphalt will deposit a discernible amount of asphalt on the cathode (negative electrode), while the anode (positive electrode) will be relatively clean. Any evidence of a clearly discernible asphalt deposit on the cathode when compared with the anode is passing. If the particle charge test does not produce conclusive results for cationic slow-setting (CSS) emulsified asphalt, proceed to Section 28.

8.5. *Report*:

8.5.1. Report the following information:

8.5.1.1. Level of current used; and

8.5.1.2. Whether the tested emulsified asphalt passes or fails as defined in Section 8.4.6.

8.6. *Precision and Bias*:

8.6.1. This test method, requiring subjective evaluation of test results and reporting of only two possible conditions, does not lend itself readily to a conventional statistical round-robin exercise. At present, there is no precision and bias statement for this test method.

CONSISTENCY

9. VISCOSITY

9.1. Determine viscosity in accordance with test methods T 72 (Saybolt Furol) or T 382 (Rotational Paddle Viscometer) to measure the consistency of emulsified asphalt. It is applicable to all the emulsified asphalt specified in M 140, M 208, and M 316.

9.2. Viscosity has significance in the use of emulsified asphalt because it is a property that affects utility. When used in application types of construction, the material must be thin enough to be uniformly applied through the spray bar of a distributor, yet thick enough so that it will not flow from the crown or grade of the road. For mixing-grade emulsified asphalt, the viscosity may affect the mixability and resulting thickness of film on the aggregate. The viscosity of emulsified asphalt may be affected by shear. Therefore, strict adherence to test procedures is necessary to achieve precision.

STABILITY

10. DEMULSIBILITY

10.1. *Scope*:

10.1.1. This test method, applicable to anionic and cationic emulsified asphalt of the rapid-setting (RS) and medium-setting (MS) types, measures the chemical breaking of the emulsified asphalt.

10.2. *Significance and Use*:

10.2.1. This test method is used to identify or classify an emulsified asphalt as an RS or MS type 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 asphalt and dioctyl sodium sulfosuccinate for cationic emulsified asphalt.

10.3. *Apparatus and Reagents*:

10.3.1. *Wire Cloth*—Three pieces of 1.40-mm (No. 14) wire cloth approximately 125 mm (5 in.) square, unframed, having wire diameters and openings that conform to ASTM E11.

10.3.2. *Beakers*—Three metal beakers of 600-mL capacity each.

10.3.3. *Rods*—Three metal rods with rounded ends, approximately 8 mm ($^5/_{16}$ in.) in diameter.

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

10.3.5. *Calcium Chloride Solution (0.02 N)*—Dissolve 1.11 g of calcium chloride ($CaCl_2$) in distilled water and dilute it to 1 L. The 1.11 g/L calcium chloride solution shall 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.

10.3.6. *Calcium Chloride Solution (0.10 N)*—Dissolve 5.55 g of $CaCl_2$, in distilled water and dilute it to 1 L. The 5.55 g/L calcium chloride solution shall be standardized to be a 0.1 ± 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.

10.3.7. *Dioctyl Sodium Sulfosuccinate Solution (0.8 percent)*—Dissolve 8.00 g of dioctyl sodium sulfosuccinate in 992 mL of distilled water. This solution will degrade over time; it shall be stored in a dark glass, airtight container in a cool, dark location when not in use. This solution shall not be used for testing purposes if more than 90 days have elapsed since it was prepared.

10.3.8. *Balance*—Conforming to the requirements of M 231, Class G 2.

10.3.9. *Oven*—Capable of maintaining a temperature of 163 ± 3°C (325 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 150 to 175°C (302 to 347°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 17).

Note 17—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

10.3.10. *Timer*—Graduated in 0.1 s and accurate within 0.1 percent when tested over a 15-min interval.

10.4. *Procedure*:

10.4.1. Determine the percentage of residue by distillation as described in Section 6 or by evaporation as described in Section 7.

10.4.2. Record the mass of each assembly of beaker, rod, and wire cloth.

10.4.3. Pour 100 ± 0.1 g of the emulsified asphalt into each of three 600-mL beakers in the assemblies. Bring the sample of emulsified asphalt and proper reagent to a temperature of 25 ± 0.5°C (77 ± 1.0°F).

10.4.4. *Over a period of approximately 2 min, add to each beaker, from a buret, one of the following*:

10.4.4.1. 35 mL of $CaCl_2$ solution (0.02 N) for anionic RS emulsified asphalt:

10.4.4.2. 50 mL of $CaC1_2$ solution (0.10 N) for MS or mixing-type emulsified asphalt; or

10.4.4.3. 35 mL of dioctyl sodium sulfosuccinate solution (0.8 percent) for cationic RS emulsified asphalt.

10.4.5. While adding the appropriate solution (Section 10.4.4.1, 10.4.4.2, or 10.4.4.3), stir the contents of the beaker continuously and vigorously, kneading any lumps against the sides of the beaker to ensure thorough mixing of the reagent with the emulsified asphalt.

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

10.4.7. Decant the mixture of any unbroken emulsified asphalt and reagent onto the wire cloth. Rinse the beaker 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 beaker, rod, and wire cloth until the rinse water drains clear.

10.4.8. Place the wire cloth containing the asphalt residue in the beaker with the metal rod. Place the assembly in a 163 ± 3°C (325 ± 5°F) drying oven for 1 h. Preliminary heating at lower temperatures to prevent spattering is permissible. Allow the beaker and contents to cool and determine the mass. Repeat the heating and mass determination until successive masses differ by no more than 0.1 g.

10.5. *Calculation*:

10.5.1. Subtract the tare mass of the beaker, rod, and wire cloth from the mass of the dried assembly to obtain the demulsibility residue. Calculate the demulsibility as follows:

$$\text{Demulsibility, \%} = (M_{der} / M_{dir}) \times 100 \qquad (6)$$

where:

M_{der} = average mass of demulsibility residue from the three tests of each sample of emulsified asphalt, g (Section 10.4.8); and

M_{dir} = mass of residue by distillation (or evaporation) in 100 g of the emulsified asphalt, g (Section 10.4.1).

10.6. *Precision and Bias*:

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

Note 18—Precision does not apply when using dioctyl sodium sulfosuccinate solution in the testing of cationic emulsified asphalt for demulsibility or when the residue is determined using evaporation.

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

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

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

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

11. SETTLEMENT

11.1. *Scope*:

11.1.1. This test method measures the settlement of emulsified asphalt that occurs in a cylindrical container.

11.2. *Significance and Use*:

11.2.1. This test method is a measure of the uniformity of emulsified asphalt dispersion in storage over a period of time.

11.3. *Apparatus*:

11.3.1. *Cylinders*—Two 500-mL glass cylinders, with pressed or molded glass bases and cork or glass stoppers, having an outside diameter of 50 ± 5 mm (2 ± 0.2 in.). The cylinders should have 5-mL graduations.

Note 19—Cylinders with side arms may be used so that samples can be withdrawn without the use of pipets.

11.3.2. *Glass Pipet*—A glass tube pipet or siphon assembly capable of removing and delivering 55 mL of sample.

11.3.3. *Balance*—Conforming to the requirements of M 231, Class G 2.

11.3.4. *Beakers*—Low-form, 1000-mL capacity, made of glass or metal, or a container of similar capacity.

11.4. *Procedure*:

11.4.1. Place a 500-mL representative sample in each of the two glass cylinders. Stopper the cylinders and allow them to stand undisturbed at laboratory air temperature for 5 days on a work surface that is free of vibration. After standing for this period, remove approximately the top 55 mL of emulsified asphalt by means of a pipet or siphon without disturbing the balance. Mix each portion thoroughly. Pour 50 g of each sample into separate 1000-mL beakers and determine the asphalt residue by evaporation in accordance with Section 7.

11.4.2. After removal of the top portion of the sample, siphon off approximately the next 390 mL from each of the cylinders. Thoroughly mix the emulsified asphalt remaining in the cylinders and pour

50 g into separate 1000-mL beakers. Determine the asphalt residue by evaporation of these samples in accordance with Section 7.

Note 20—If the emulsified asphalt contains appreciable amounts of oil distillate as determined by distillation (see Section 6), the settlement value may be calculated from the difference in the percentage of water content between the top and bottom portion of the samples as determined by the procedure described in Section 5.

Note 21—If cylinders with side arms are used, siphoning is not required. Obtain a 55-mL sample from the upper arm. Drain off 390 mL from the lower arm.

11.5. *Calculation and Report*:

11.5.1. Calculate the settlement for each cylinder as follows:

$$\text{Settlement, \% (5 days)} = P_{rb} - P_{rt} \quad (7)$$

where:

P_{rb} = the percentage of residue from the bottom portion of the sample; and
P_{rt} = the percentage of residue from the top portion of the sample

Note 22—If the settlement values between the two cylinders differ by more than the stated repeatability, the result is considered suspect and the test shall be repeated. If for individual cylinders the percent residue of both the top and bottom samples is less than the percent residue of the emulsified asphalt, the result is considered suspect and the test shall be repeated. If for individual cylinders the percent residue of both the top and bottom portion of the samples is greater than the percent residue of the emulsified asphalt, the result is considered suspect and the test shall be repeated.

11.5.2. Report the settlement as the average of the two individual cylinder results.

11.6. *Precision and Bias*:

11.6.1. The following criteria should be used for judging the acceptability of results (95 percent probability):

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

Settlement, mass %	Repeatability
0 to 1.0	0.4 mass %
Above 1.0	5% of the mean

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

Settlement, mass %	Reproducibility
0 to 1.0	0.8 mass %
Above 1.0	10% of the mean

12. CEMENT MIXING

12.1. *Scope*:

12.1.1. This test method is a mixing test used to identify or classify a slow-setting, SS or CSS, type of emulsified asphalt.

12.2. *Significance and Use*:

12.2.1. The result of this test indicates the ability of an SS emulsified asphalt to mix with a finely divided, high surface area material (i.e., high early strength, Type III, portland cement) without breaking the emulsified asphalt.

12.3. *Apparatus and Material*:

12.3.1. *Sieves*—A 0.180-mm (No. 80) sieve and a 76.2-mm (3-in.) diameter 1.40-mm (No. 14) sieve, made of wire cloth conforming to ASTM E11.

12.3.2. *Dish*—A round-bottom iron dish or kitchen saucepan of approximately 500-mL capacity.

12.3.3. *Stirring Rod*—A steel rod with rounded ends, approximately 13 mm ($^1/_2$ in.) in diameter.

12.3.4. *Graduate*—A 100-mL graduated cylinder.

12.3.5. *Balance*—Conforming to the requirements of M 231, Class G 2.

12.3.6. *Oven*—Capable of maintaining temperatures of 110 ± 3°C (230 ± 5°F) or 163 ± 3°C (325 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 100 to 175°C (212 to 347°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 23).

Note 23—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

12.3.7. *Cement*:

12.3.7.1. High-early-strength portland cement conforming to the requirements for Type III portland cement in M 85 and having a minimum specific surface area of 1900 cm^2/g (928 ft^2/lb) as measured by the Wagner Turbidimeter according to T98M/T 98 or 345 m^2/kg (1685 ft^2/lb) as measured by the Blaine air permeability apparatus according to T 153.

12.4. *Procedure*:

12.4.1. Dilute the emulsified asphalt with distilled water to a residue of 55 percent, as determined by distillation or by evaporation for 3 h at 163°C (325°F), according to Section 6 or 7, as appropriate.

12.4.2. Sieve a portion of the cement through the 0.180-mm (No. 80) sieve. Add 50 ± 0.1 g of the cement passing the 0.180-mm (No. 80) sieve into the dish.

12.4.3. Ensure that the ingredients and apparatus are at a temperature of approximately 25°C (77°F) before mixing. Add 100 mL of the diluted emulsified asphalt to the cement and stir the mixture at once with the steel rod, using a circular motion at a rate of 60 rpm. At the end of the 1-min mixing period, add 150 mL of distilled water, and continue stirring for 3 min.

12.4.4. Pour the mixture through a weighed 1.40-mm (No. 14) sieve. Use repeated washings to completely remove the material from the mixing bowl. Pour these washings over the sieve, and rinse the sieve using distilled water held at a height of approximately 150 mm (6 in.) until the water running through the sieve is clear. Place the sieve in a shallow pan; heat it at 110 ± 3°C (230 ± 5°F) in an oven for 2 h and determine the mass. Repeat the heating and mass determination until successive masses differ by no more than 0.1 g.

12.5. *Calculation and Report*:

12.5.1. Calculate the mass of the sample retained on the sieve and in the pan as follows:

$$\text{Mass Retained} = M_{spr} - M_{sp} \qquad (8)$$

where:

M_{spr} = mass of the sieve, pan, and residue, g; and

M_{sp} = mass of the sieve and pan, g.

12.5.2. Report the mass, in grams, of the material retained on the sieve and in the pan as the percentage of break in the cement mixing test.

12.6. *Precision and Bias*:

12.6.1. *The following criteria should be used for judging the acceptability of results (95 percent probability)*:

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

Cement Mixing, mass %	Repeatability, mass %
0 to 2	0.2

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

Cement Mixing, mass %	Reproducibility, mass %
0 to 2	0.4

13. SIEVE TEST

13.1. *Scope*:

13.1.1. This test method measures the degree to which an emulsified asphalt may contain particles of asphalt or other discrete solids retained on a 0.850-mm (No. 20) mesh sieve.

13.2. *Significance and Use*:

13.2.1. The retention of an excessive amount of particles on the sieve indicates that problems may occur in handling and application of the material. Particles of asphalt retained on the sieve often are caused by agglomeration of the dispersed phase. Storage, pumping, handling, and temperature can all contribute to the formation of particles. Contamination from the tank, transport, or hose is another factor affecting particle formation.

13.3. *Apparatus and Reagents*:

13.3.1. *Sieve*—A sieve having a 76.2-mm (3-in.) frame and a 0.850-mm (No. 20) wire sieve cloth conforming to ASTM E11.

13.3.2. *Pan*—A tin box cover or shallow metal pan of appropriate size to fit over the bottom of the standard sieve.

13.3.3. *Distilled Water*—For use with cationic emulsions.

13.3.4. *Sodium Oleate Solution (2 percent)*—Dissolve 20 g of pure sodium oleate in distilled water and dilute it to 1 L. This solution is to be used instead of distilled water for determining if anionic or nonionic emulsions break on the sieve.

13.3.5. *Balances*—Conforming to the requirements of M 231, Class G 5 for determining the mass of the emulsified asphalt, and M 231, Class G 2 for determining the mass of the sieve and residue.

13.3.6. *Oven*—Capable of maintaining a temperature of 110 ± 3°C (230 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 100 to 125°C (212 to 257°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 24).

Note 24—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

13.3.7. *Desiccator.*

13.3.8. *Oven or Water Bath*—Capable of maintaining a temperature of no more than 74°C (165°F) if it is necessary to heat the emulsified asphalt before testing. The oven or water bath shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 20 to 80°C (68 to 176°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 25).

Note 25—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

13.4. *Procedure*:

13.4.1. The temperature at which the sieve test should be performed is related to the viscosity of the emulsified asphalt. For those materials whose viscosity is 100 s or less at 25°C (77°F), perform the test at room temperature. For those materials whose viscosity is more than 100 s at 25°C (77°F) and those whose viscosity is specified at 50°C (122°F), use a test temperature of 50 ± 3°C (122 ± 5°F). If heating is necessary, the emulsified asphalt may be placed in a closed container in an oven or water bath, followed by stirring to achieve homogeneity.

13.4.2. Record the mass of the sieve and pan. Wet the wire cloth with distilled water. Transfer 1000 g of the emulsified asphalt into a suitable container and pour it through the sieve. Wash the container and residue on the sieve with distilled water until the washings run clear. Place the pan under the sieve and heat the pan and sieve for 2 h in a 110 ± 3°C (230 ± 5°F) drying oven. Cool them in a desiccator and determine the mass of the sieve, pan, and residue. A 2-percent sodium oleate solution shall be used instead of distilled water for determining if anionic or nonionic emulsions break on the sieve.

13.5. *Calculation*:

13.5.1. Calculate the percentage of sample retained on the sieve as follows:

$$\text{Sample retained, \%} = (M_{spr} - M_{sp})/10 \qquad (9)$$

where:

M_{spr} = mass of the sieve, pan, and residue, g; and

M_{sp} = mass of the sieve and pan, g.

13.6. *Precision*:

13.6.1. *The following criteria should be used for judging the acceptability of results (95 percent probability)*:

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

Sieve Test, mass %	Repeatability, mass %
0 to 0.1	0.03

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

Sieve Test, mass %	Reproducibility, mass %
0 to 0.1	0.08

14. COATING TEST

14.1. *Scope*:

14.1.1. This test method is applicable to those emulsified asphalts intended for use by mixing with aggregate. It is not applicable to RS types or diluted materials used for tack coats, priming, or mulch treatments.

14.2. *Significance and Use*:

14.2.1. The intent of the coating test is to ensure that a given mixing-grade emulsified asphalt is adequately stable emulsified asphalt and otherwise possesses the ability to mix with a reference stone for the prescribed time and coat it uniformly and thoroughly.

14.3. *Apparatus and Material*:

14.3.1. *Sieves*—19.0-mm ($^3/_4$-in.) and 6.4-mm ($^1/_4$-in.) sieves conforming to ASTM E11.

14.3.2. *Spatula*—A steel spatula or its equivalent, having a blade approximately 200 mm (8 in.) in length.

14.3.3. *Dish*—A round-bottom iron dish or a kitchen saucepan, of approximately 1-L (1-qt) capacity.

14.3.4. *Stone*—A supply of reference stone (hard limestone, trap rock, or other similar type) that has been washed with water and dried before using. All stone shall pass through a 19.0-mm ($^3/_4$-in.) screen and not more than 5 percent shall pass through the 6.4-mm ($^1/_4$-in.) screen.

Note 26—Each laboratory shall select its own reference stone supply, the source of which is not apt to change; this policy is to avoid rapid changes in the character of reference stone used in any one laboratory.

14.3.5. *Balance*—Conforming to the requirements of M 231, Class G 2.

14.4. *Procedure*:

14.4.1. Add 465 ± 0.1 g of stone into the dish. Add 35 ± 0.1 g of the emulsified asphalt to the stone in the dish and mix vigorously with the spatula for 3 min.

14.4.2. Record whether there is appreciable separation of the asphalt base from the water in the emulsified asphalt and whether the stone is uniformly and thoroughly coated with the emulsified asphalt.

15. COATING ABILITY AND WATER RESISTANCE

15.1. *Scope*:

15.1.1. This test method is intended to aid in the identification of emulsified asphalt suitable for mixing with coarse-graded calcareous aggregates.

Note 27—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.

15.2. *Significance and Use*:

15.2.1. This test method covers the determination of the ability of an emulsified asphalt 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.

15.3. *Apparatus:*

15.3.1. *Mixing Pan*—An enameled kitchen saucepan with handle, of approximately 3-L (3-qt) capacity.

15.3.2. *Spatula*—A steel spatula or its equivalent, having a blade approximately 200 mm (8 in.) in length.

15.3.3. *Sieves*—19.0-mm ($^3/_4$-in.) and 4.75-mm (No. 4) sieves conforming to ASTM E11.

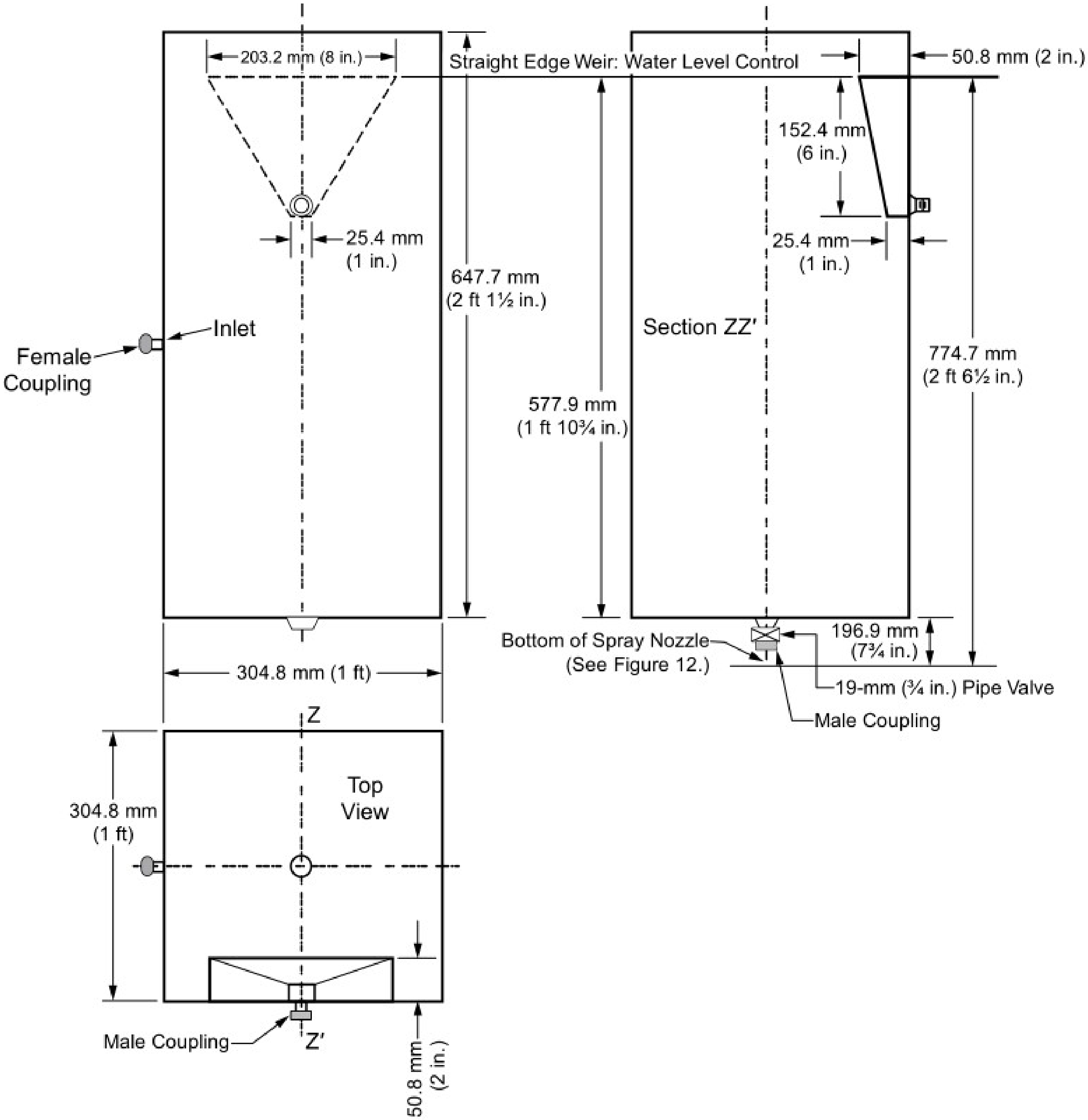
15.3.4. *Constant-Head Water-Spraying Apparatus*—An apparatus for applying tap water in a spray under a constant head of 774.7 mm (2.54 ft) (Figures 11 and 12). The water shall issue from the apparatus in a low-velocity spray.

15.3.5. *Thermometer*—A thermometer for measuring asphalt temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 0 to 50°C (32 to 122°F), and an accuracy of ±0.5°C (±0.9°F) (see Note 28).

Note 28—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E2877 digital metal stem thermometer; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

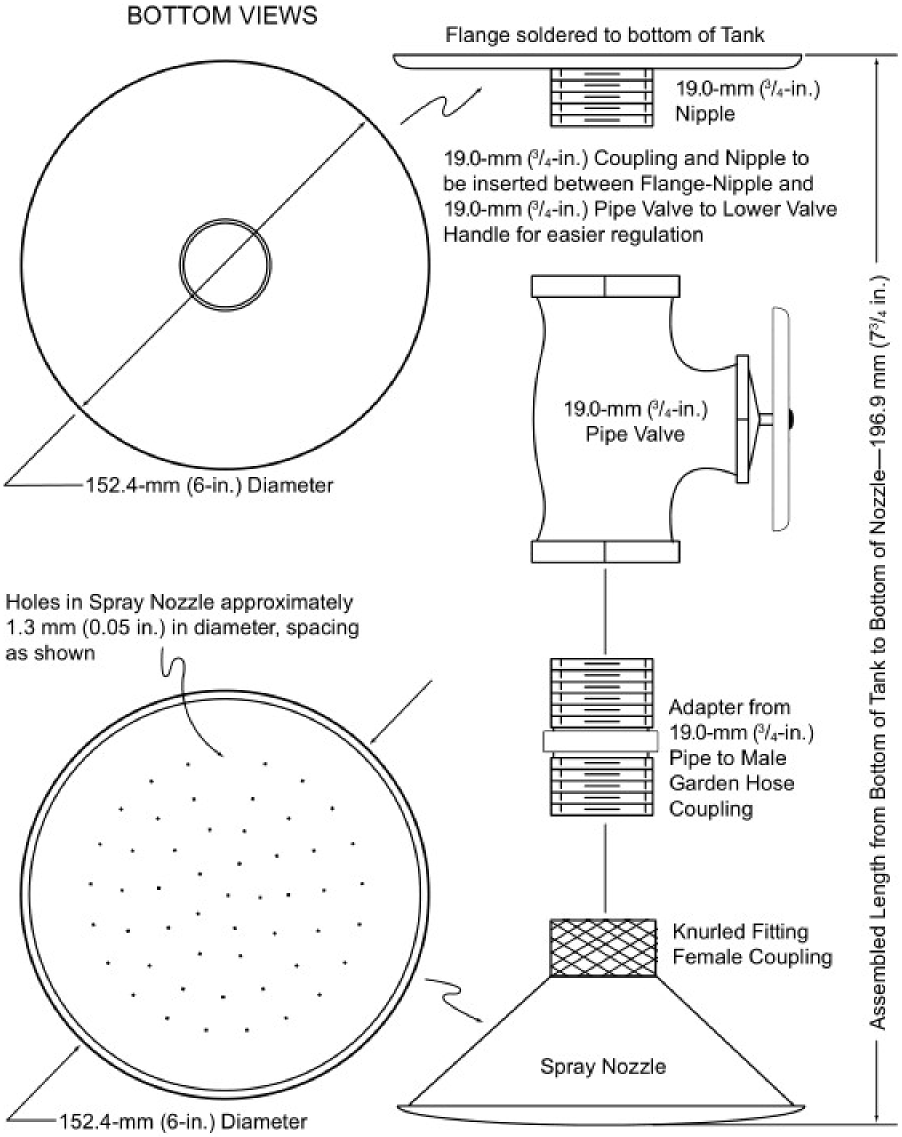
15.3.6. *Balance*—Conforming to the requirements of M 231, Class G 2.

15.3.7. *Pipet*—10-mL capacity.



Note: 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 19.0-mm ($^3/_4$-in.) 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 914.4 ± 25.4 mm (3 ft ± 1 in.).

Figure 8—Constant-Head Flow Tank



Note 1: A Speakman, Model 235S, all brass, fixed showerhead has been found acceptable.

Note 2: Existing 101.6-mm (4-in.) diameter shower heads may continue to be used.

Figure 9—Valve and Nozzle Assembly for Constant-Head Flow Tank

15.4. *Materials*:

15.4.1. *Aggregate*—Reference aggregate shall be a laboratory-washed and air-dried limestone aggregate graded to pass the 19.0-mm ($^3/_4$-in.) sieve and be retained on the 4.75-mm (No. 4) sieve[3] (Note 18).

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

15.4.3. *Water*—Tap water with a hardness not over 250 ppm $CaCO_3$ for spraying over the sample.

15.4.4. *Absorbent Paper.*

15.5. *Procedure for Tests with Dry Aggregate*:

15.5.1. Conduct the test at 23.9 ± 5.5°C (75 ± 10°F).

15.5.2. Add 461 g of the air-dried, graded reference aggregate in the mixing pan.

15.5.3. Add 4.0 g of $CaCO_3$ dust in the mixing pan and mix it with the 461 g of aggregate for approximately 1 min with a spatula to obtain a uniform film of dust on the aggregate particles.

Note 29—The total mass of aggregate and dust shall equal 465 g. If no calcium carbonate is included, the mass of aggregate alone shall be 465 g.

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

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

15.5.6. Immediately spray the mixture remaining in the pan with tap water from the constant-head water-spraying apparatus to cover the mixture. The distance from the spray head to the sample shall be 305 ± 75 mm (12 ± 3 in.). Then carefully pour off the water. Continue spraying and pouring off the water until the overflow water runs clear. Carefully drain the water in the pan. Transfer the mixture from the mixing pan onto absorbent paper for evaluation of coating retention.

15.5.7. Evaluate the mixture immediately by visual estimation as to the total aggregate surface area that is coated with asphalt.

15.5.8. Repeat the evaluation by visual estimation of the coating of aggregate surface area with asphalt after the mixture has been air dried in the laboratory at room temperature. A fan may be used for drying if desired.

15.6. *Procedure for Tests with Wet Aggregate*:

15.6.1. Proceed in accordance with Sections 17.5.1 to 17.5.3.

15.6.2. Pipet 9.3 mL of water into the aggregate and $CaCO_3$ dust mixture in the mixing pan and mix thoroughly to obtain uniform wetting.

15.6.3. Continue in accordance with Sections 17.5.4 to 17.5.8.

15.7. *Interpretation of Results*:

15.7.1. Evaluate and report the following information for tests with both dry and wet aggregate:

15.7.1.1. At the end of the mixing period, record the coating of the total aggregate surface area with emulsified asphalt as "good," "fair," or "poor," where a rating of "good" means fully coated with emulsified asphalt (exclusive of pinholes and sharp edges of the aggregate), a rating of "fair" applies to the condition of more area of coated than uncoated, and a rating of "poor" applies to the condition of more area uncoated than coated.

15.7.1.2. After spraying with water, record the coating of the total aggregate surface area with the asphalt as "good," "fair," or "poor," according to the definitions for those terms in Section 17.7.1.1.

15.7.1.3. After air-drying in the laboratory, record the coating of the total aggregate surface area by the asphalt as “good,” “fair,” or “poor.”

Note 30—Comments about the results of the test may be included in the evaluation.

16. STORAGE STABILITY OF EMULSIFIED ASPHALT

16.1. *Scope*:

16.1.1. This test method relates to the ability of emulsified asphalt to remain as a uniform dispersion during storage. It is applicable to emulsified asphalt composed principally of a semisolid or liquid asphalt base, water, and an emulsifying agent.

16.2. *Summary of Test Method*:

16.2.1. This test method determines the difference in percent residue of samples taken from the top and bottom of material placed in undisturbed simulated storage for 24 h. The result is expressed as the average of the two individual values obtained by determining the difference between the percent residue of the top and bottom samples for each storage cylinder.

16.3. *Significance and Use*:

16.3.1. This test method is useful for determining the storage stability of emulsified asphalt in a comparatively short amount of time. It is a measure of the permanence of the dispersion as related to time, but it is not to be construed to have significance as a measure of other stability aspects involved in use of emulsified asphalt.

16.4. *Apparatus*:

16.4.1. *Cylinders*—Two 500-mL glass cylinders, with pressed or molded glass bases and cork or glass stoppers, having an outside diameter of 50 ± 5 mm (2 ± 0.2 in.), and having 5-mL graduations.

Note 31—Cylinders with side arms may be used so that samples can be withdrawn without the use of pipets.

16.4.2. *Glass Pipet*—A 60-mL siphon glass-tube pipet of optional form.

16.4.3. *Balance*—Conforming to the requirements of M 231, Class G 2.

16.4.4. *Beakers*—Low form, 1000-mL capacity, made of glass or metal, or a container of similar capacity.

16.4.5. *Oven*—Capable of maintaining a temperature of 163 ± 3°C (325 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 150 to 175°C (302 to 347°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 32).

Note 32—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

16.5. *Procedure*:

16.5.1. Place a 500-mL representative sample in each of the two glass cylinders. Stopper the cylinders and allow them to stand undisturbed at laboratory air temperature for 24 h on a work surface that is free of vibration. After standing for this period, remove approximately 55 mL from the top of the emulsified asphalt by means of the pipet or siphon without disturbing the balance (Note 22). Thoroughly mix each portion.

16.5.2. Pour 50 ± 0.1 g of each sample into separate 1000-mL beakers; the mass of each beaker along with a 6-mm (1/4-in.) diameter by 178-mm (7-in.) glass rod shall previously have been determined. Determine the asphalt residue by evaporation of these samples in accordance with Section 7.

16.5.3. After removal of the top portion of the sample, siphon off approximately the next 390 mL from each of the cylinders (Note 22). Thoroughly mix the emulsified asphalt remaining in the cylinders and pour 50 g into separate 1000-mL beakers. Determine the asphalt residue of these samples in accordance with Section 16.5.2.

16.6. *Calculation and Report*:

16.6.1. Calculate and report the storage stability in accordance with Section 11.5 and observe Note 17.

16.7. *Precision and Bias*:

16.7.1. *Repeatability*—The repeatability standard deviation is 0.2 percent. The average difference between two results, obtained by the same operator with the same equipment, but not concurrently, will be approximately 0.2 percent. Two such values should be considered suspect (95 percent confidence level) if they differ by more than 0.5 percent.

16.7.2. *Reproducibility*—The reproducibility standard deviation is 0.2 percent. The average difference between two results obtained by operators in different laboratories will be approximately 0.3 percent. Two such values should be considered suspect (95 percent confidence level) if they differ by more than 0.6 percent.

Note 33—If this test method is performed using only one cylinder instead of two for each determination as specified in the method, then the following precision criteria should be used:

	Repeatability, %
Standard deviation	0.2
Average difference	0.3
Suspect criterion	0.5

	Reproducibility, %
Standard deviation	0.2
Average difference	0.3
Suspect criterion	0.6

EXAMINATION OF RESIDUE

17. SCOPE

17.1. Tests for specific gravity, ash content, solubility in trichloroethylene, penetration, ductility, and the float test are suggested for examination of the emulsified asphalt residue obtained by distillation (Section 6) or evaporation (Section 7).

18. SIGNIFICANCE AND USE

18.1. The suggested procedures are used to characterize and evaluate the properties of emulsified asphalt residues.

19. SPECIFIC GRAVITY

19.1. Determine the specific gravity on a representative portion of the residue in accordance with T 228 or ASTM D3289.

20. ASH CONTENT

20.1. Determine the ash content on a representative portion of the residue in accordance with T 111.

21. SOLUBILITY IN TRICHLOROETHYLENE

21.1. Determine the solubility in trichloroethylene on a representative portion of the residue in accordance with T 44.

22. PENETRATION

22.1. Determine the penetration on a representative portion of the residue in accordance with T 49.

22.2. *Precision*—The following criteria should be used for judging the acceptability of results (95 percent probability):

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

Penetration of Residue, range	Repeatability, points
80 to 200	15

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

Penetration of Residue, range	Reproducibility, points
80 to 200	30

23. DUCTILITY

23.1. Determine the ductility on a representative portion of the residue in accordance with T 51.

24. FLOAT TEST

24.1. Perform the float test on a representative portion of the residue in accordance with T 50.

IDENTIFICATION TESTS

25. IDENTIFICATION TEST FOR CATIONIC RAPID-SETTING (CRS) EMULSIFIED ASPHALTS

25.1. *Scope*:

25.1.1. This test method covers a procedure for identifying CRS emulsified asphalt. The inability to coat a specific silica sand distinguishes CRS emulsified asphalt from MS and SS cationic grades.

25.2. *Significance and Use*:

25.2.1. This test method is based upon the rapid asphalt deposition properties of CRS emulsified asphalt as a function of sand surface area and surface charge. The test method differs from the usual coating test because the material passes the requirement when it fails to coat the specified silica sand.

25.2.2. Impurities in the silica sand often prevent the emulsified asphalt from passing the test. Washing the sand to remove impurities prior to the test is required. Impurities affecting test results are normally due to the presence of iron (Fe^{+++}) and organic dewatering agents that can alter the sand surface charge.

25.3. *Apparatus*:

25.3.1. *Mixing Bowl*—Metal or porcelain saucepan, $2^1/_2$ to 3 L ($2^1/_2$ to 3 qt), equipped with a handle.

25.3.2. *Washing Container*—Beaker made from borosilicate glass, 1000-mL capacity.

25.3.3. *Stainless Steel Spatula or Glass Rod.*

25.3.4. *Oven*—Convection or forced-draft, capable of maintaining 120 to 150 ± 3°C (248 to 302 ± 5°F). Oven(s) shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 110 to 160°C (230 to 320°F), and an accuracy of ±0.75°C (±1.35°F) (see Note 34).

Note 34—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E230/E230M thermocouple thermometer, Type T, Special Class; or IEC 60584 thermocouple thermometer, Type T, Class 1.

25.3.5. *Balance*—Conforming to the requirements of M 231, Class G 2.

25.3.6. *Drying Pan*—Stainless steel or glass, 150 mm (6 in.) by 225 mm (9 in.) or larger.

25.3.7. *Graduated Cylinder*—Made from borosilicate glass, 500-mL capacity.

25.3.8. *Thermometer*—A thermometer for measuring materials temperatures shall meet the requirements of M 339M/M 339 with a temperature range of at least 45 to 155°C (113 to 311°F), and an accuracy of ±0.1°C (±0.18°F) (see Note 35).

Note 35—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E1137/E1137M Pt-100 RTD platinum resistance thermometer, Special order; or IEC 60751: 2008 Pt-100 RTD platinum resistance thermometer, Special order.

25.3.9. *Eye Protection*—Suitable safety glasses or other appropriate eye protection.

25.3.10. *Hand Protection*—Sufficiently acid and alcohol resistant.

25.3.11. *Timer*—Graduated in 0.1 s and accurate within 0.1 percent when tested over a 15-min interval.

25.4. *Material*:

25.4.1. *Hydrochloric Acid*—Reagent, 36.5 to 38.0 percent.

25.4.2. *Isopropyl Alcohol*—Reagent, 100 percent.

25.4.3. *Distilled Water.*

25.4.4. *Absorbent Paper.*

25.4.5. *Silica Sand*—F-95, whole grain, with a gradation similar to that in the table below.[4]

Typical Gradation								
U.S. sieve no.	30	40	50	70	100	140	200	270
Millimeters	0.850	0.425	0.300	0.212	0.150	0.106	0.075	0.053
% passing	100	99	97	89	56	17	2	Trace

25.5. *Emulsified Asphalt Sample*:

25.5.1. Obtain a representative sample of the CRS emulsified asphalt for testing in accordance with R 66.

25.6. *Sand Preparation*:

25.6.1. Prepare 400 mL of 5-percent hydrochloric acid, by volume, in isopropyl alcohol by mixing 20 mL of hydrochloric acid into a blend of 80 mL of distilled water and 300 mL of isopropyl alcohol in a 500-mL graduated cylinder.

25.6.2. Add 500 g of the F-95 silica sand into a 1000-mL beaker. Add 400 mL of the "5-percent hydrochloric acid in isopropyl alcohol/water" mixture into the beaker containing the silica sand and stir for 5 min with a stainless-steel spatula or glass rod.

25.6.3. Decant the acid/alcohol/water mixture carefully without a loss of sand.

25.6.4. Wash the sand with at least 400 mL of hot distilled water, 50 to 70°C (122 to 158°F) and stir for approximately 1 min. Decant the water without a loss of sand. Repeat water-washing the sand two more times.

25.6.5. Scrape the washed sand onto the drying pan (avoiding as much loss of sand as possible) and spread the sand over the surface of the drying pan. Place the pan with the sand in an oven and dry it to a constant mass at a temperature of 120 to 150°C (248 to 302°F).

25.6.6. Allow the sand to cool and transfer the dry sand to a suitable container. Mix the sand for approximately 30 s to obtain uniformity.

25.7. *Procedure*:

25.7.1. Perform the following operations at 25 ± 5°C (77 ± 9°F).

25.7.2. Add 465 g of the washed silica sand blend into a mixing bowl. Add 35 g of the emulsified asphalt to be tested and mix vigorously with a spatula for approximately 2 min using a combined stirring and kneading action. At the end of the mixing period, tilt the bowl and allow any excess emulsified asphalt, which is not coating the sand, to drain from the bowl.

25.7.3. Place the mix on absorbent paper. Visually estimate the amount of uncoated and coated area in the mixture.

Note 36—Visual estimation is less accurate at moderate levels of coating than at the two extremes. If the results are inconclusive, the washing and mixing procedure should be repeated.

25.8. *Interpretation of Results*:

25.8.1. From the visual estimation of the amount of uncoated area and coated area in the mixture, record the coating of the total sand surface area by the emulsified asphalt. More uncoated area than coated area shall be considered as a passing rating for identification of CRS emulsified asphalt (Note 36).

25.9. *Precision and Bias*:

25.10. This test method, which requires a subjective evaluation of test results and reports only two possible conditions, does not lend itself readily to a statistical round-robin exercise. Development of a procedure to quantitatively determine the comparative coating will be considered.

26. IDENTIFICATION TEST FOR CATIONIC SLOW-SETTING (CSS) EMULSIFIED ASPHALTS

26.1. *Scope*:

26.1.1. This test method is intended to identify a CSS grade of emulsified asphalts if the results of the particle charge test are inconclusive (Section 8.4.6).

26.2. *Summary of Test Method*:

26.2.1. A specified amount of washed and dried silica sand is hand-mixed with a specified amount of emulsified asphalt. The mixing time should extend until the aggregate is completely coated. The amount of CSS-grade emulsified asphalt in the mix should yield a total mix asphalt content of about 5.0 percent. The sample is spread out and allowed to air-cure for 24 h. The retained coating, after boiling, is then determined.

26.3. *Significance and Use*:

26.3.1. The conditions of the test are designed to identify the ability of a CSS grade of emulsified asphalt from M 208 to properly mix, coat, and adhere to a specified silica sand.

26.3.2. This test method is intended to confirm the adhesive properties that exist between a cationic emulsified asphalt and silica sand and the ability of the sample to remain coated after being submerged in boiling water.

26.4. *Apparatus and Material*:

26.4.1. *Heat Source*—Hot plate or Bunsen burner. The use of a Bunsen burner will necessitate the placement of an interfacial material between the flame and beaker.

26.4.2. *Screen*—No. 20 mesh folded into a circular configuration with the edges folded downward; or a strip of 0.850-mm (No. 20) mesh screen, 25.4 mm by 355.6 mm (1 in. by 14 in.) in length, and a circular screen of the circumference of the 1000-mL beaker. Bend the strip into a circle and insert it in the bottom of the beaker. Place the circular screen on top of the strip.

26.4.3. *Beaker*—1000-mL capacity.

26.4.4. *Paper Napkin.*

26.4.5. *20–30 Standard Ottawa Silica Sand*—As described in ASTM C778 and washed to remove impurities prior to testing.[4]

26.4.6. *Spatula*—A steel spatula or its equivalent, having a blade of 203.2 mm (8 in.) in length.

26.4.7. *Mixing Container*—400-mL capacity, glass or stainless-steel beaker or bowl.

26.4.8. *Beaker Tongs.*

26.4.9. *Eye Protection*—Suitable safety glasses with a side shield or any other appropriate eye protection.

26.4.10. *Hand Protection*—Heat-resistant gloves.

26.4.11. *Balance*—Conforming to the requirements of M 231, Class G 2.

26.4.12. *Distilled Water.*

26.5. *Procedure*:

26.5.1. Weigh 150 g of dry silica sand into the mixing container.

26.5.2. Add 12 g of the emulsified asphalt and immediately begin to mix vigorously for 20 to 60 s or until silica sand is completely coated.

Note 37—Water may be added to facilitate mixing; 1.0 percent is generally sufficient.

26.5.3. The sample should be spread onto a paper napkin in such a manner as to facilitate curing. Allow the sample to air-cure for 24 h. Remove the sample from the paper napkin and break it into several pieces; observe whether the sample is completely cured. If the sample is not completely cured, allow additional curing time.

26.5.4. Boil about 500 mL of distilled water in a 1000-mL beaker containing 0.850-mm (No. 20) mesh screen shelf. Place the coated silica sand in the boiling water on top of the screen.

26.5.5. Boil for 10 min and then decant the water.

26.5.6. Spread the sample on a level surface and observe the coating of the mix.

26.6. *Report*:

26.6.1. From the visual examination of the amount of coated and uncoated areas in the mixture, record the retained coating, after boiling, of the total sand surface area. If the coating is more than 50 percent, the emulsified asphalt should be considered as having a passing rating for classification of CSS emulsified asphalt.

26.7. *Precision and Bias*:

26.7.1. This test method, which requires a subjective evaluation of test results and reports only two possible conditions, does not lend itself readily to a statistical round-robin exercise. At present, there is no precision and bias statement for this test method.

COATING TESTS ON EMULSIFIED ASPHALTS

27. FIELD COATING TEST

27.1. *Scope*:

27.1.1. This is a quick field method to ascertain the compatibility of emulsified asphalt with the job aggregate.

27.2. *Significance and Use*:

27.2.1. This test method is used at the project site to determine (1) the ability of the emulsified asphalt to coat the job aggregate; (2) the ability of the emulsified asphalt to withstand mixing; and (3) the water resistance of the aggregate coated with emulsified asphalt.

27.3. *Summary of Test Method*:

27.3.1. A measured amount of the job aggregate is hand-mixed with a measured amount of the emulsified asphalt supplied to the job. The ability of the emulsified asphalt to remain as a coating during a 5-min mixing cycle is observed. The resistance offered by the coating to washing off is determined by repeated filling of a container of coated aggregate with water and emptying.

27.4. *Apparatus*:

27.4.1. *Metal Containers*—$^1/_2$-L (1-pt) capacity (friction-top pint cans).

27.4.2. *Metal Porcelain Saucepan*—$2^1/_2$ to 3-L ($2^1/_2$ to 3-qt), equipped with a handle.

27.4.3. *Dispensing Graduate*—50-mL capacity, preferably plastic.

27.4.4. *Serving Spoon*—Long-handled.

27.4.5. *Sieve*—A 19-mm ($^3/_4$-in.) sieve, made of wire cloth and conforming to ASTM E11.

27.4.6. *Timer*—Graduated in 0.1 s and accurate within 0.1 percent when tested over a 15-min interval.

27.4.7. *Newspapers*.

27.5. *Procedure*:

27.5.1. Remove the rim from the $^1/_2$-L (1-pt) can.

27.5.2. Fill the can level with the job aggregate, discarding any sizes above 19 mm ($^3/_4$ in.).

27.5.3. Measure out 50 mL of emulsified asphalt.

27.5.4. Transfer the aggregate and the emulsified asphalt into the saucepan.

27.5.5. Hand-mix vigorously for 5 min with the spoon.

27.5.6. Observe (1) whether the stone is fully coated with the emulsified asphalt and rate the coating as "good," "fair," or "poor," using the ratings as defined in Section 17.7.1.1; and (2) the presence, if any, of free water, which denotes breakdown of the emulsified asphalt.

27.5.7. Refill the $^{1}/_{2}$-L (1-pt) can with the coated stone.

27.5.8. Set the can of coated stone upright in the saucepan.

27.5.9. Fill the can with water and pour if off. Repeat this step five times.

27.5.10. Transfer the contents of the can onto newspapers. Repeat the observations made in Section 29.5.6 and record the ratings.

27.6. *Report*:

27.6.1. Report the observations made in Sections 29.5.6 and 29.5.10 as the results from this test.

27.7. *Precision and Bias*:

27.7.1. The usual methods of analysis for precision cannot be applied to this test method because it is not fully quantitative.

28. EMULSIFIED ASPHALT/JOB AGGREGATE COATING TEST

28.1. *Scope*:

28.1.1. This test method may be used to identify the ability of SS grades of emulsified asphalt to mix with and coat a dense and fine-graded job aggregate. It is a laboratory test method for screening emulsified asphalt candidates for mixing with and coating job aggregates and is not to be construed as a mix design test method.

28.2. *Significance and Use*:

28.2.1. The conditions of the test are designed to identify the ability of emulsified asphalt, SS grades (CSS from M 208 and SS from M 140), to mix with and coat dense-graded and fine-graded job aggregate.

28.3. *Summary of Test Method*:

28.3.1. A specified amount of dry job aggregate is hand-mixed with a specified amount of water in order to "pre-wet" the aggregate. The wetted aggregate is then hand-mixed with a specified amount of emulsified asphalt of known asphalt content, until the maximum coating of the job aggregate is obtained. (Mix time is usually 15 to 120 s.) The ability of the emulsified asphalt to mix with the job aggregate is determined by using various amounts of water and emulsified asphalt until a maximum coating of the job aggregate is obtained. This coating is rated as "good," "fair," or "poor," using the ratings as defined in Section 17.7.1.1.

28.4. *Apparatus*:

28.4.1. *Containers*—A 1000-mL glass beaker, a 1.0-L (l-qt) friction-top metal can, or 1000-mL stainless steel beaker or bowl.

28.4.2. *Mixing Tool*—A steel spatula or its equivalent, having a blade approximately 203.2 mm (8 in.) in length.

28.4.3. *Balance*—Conforming to the requirements of M 231, Class G 2.

28.5. *Procedure*:

28.5.1. Add 300 g of dry job aggregate into the container. Add and record the amount of water necessary to "pre-wet" the aggregate (based on the dry mass of the aggregate). Immediately begin to mix vigorously for 1 min or until all aggregate surfaces subjectively appear to be wetted (as a guide, use 2 to 8 percent water for dense-graded aggregate and 4 to 12 percent water for fine-graded aggregate). The natural moisture in a job aggregate may be used in the test if the moisture content is predetermined. Additional water may then be added, if necessary, to obtain the desired level of water to be used for "pre-wetting" the aggregate.

28.5.2. Add and record the amount of emulsified asphalt (of known asphalt residue content) necessary to coat the aggregate. Immediately begin to mix vigorously, scraping the sides and bottom of the container, for 15 to 120 s or until the maximum coating has been attained (as a guide, based on the dry mass of the aggregate, use 3 to 7 percent asphalt residue for dense-graded aggregate and 4 to 8 percent residue for fine-graded aggregate). For example, 8 percent emulsified asphalt in 60 percent solids would be equivalent to 4.8 percent asphalt residue in the mix.

28.5.3. If the mix appears to be too dry and insufficiently coated, repeat Section 30.5.1 or 30.5.2, using an increased amount of water or emulsified asphalt, or both. If the mix appears to be too wet from excessive water or emulsified asphalt, or both, repeat Section 30.5.1 or 30.5.2, using less water or emulsified asphalt, or both.

28.5.4. For each job aggregate mix, observe and record the amount of water used to "pre-wet" the aggregate and asphalt residue from the emulsified asphalt, and note the one mix that provides the best aggregate coating.

28.5.5. Rate the best coating as "good," "fair," or "poor," using the ratings as defined in Section 17.7.1.1.

28.6. *Report*:

28.6.1. Report the amount of water used to "pre-wet" the aggregate and residual asphalt needed for the best aggregate coating obtainable.

28.6.2. Report the maximum coating achieved as "good," "fair," or "poor," in accordance with Section 17.7.1.1.

28.7. *Precision and Bias*:

28.7.1. The usual methods of analysis for precision cannot be applied to this test method because it is not fully quantitative.

DENSITY OF EMULSIFIED ASPHALTS

29. TEST TO DETERMINE MASS PER LITER (GALLON)

29.1. *Scope*:

29.1.1. This test method is used to determine the mass per liter (gallon) of emulsified asphalt used in highway construction. This density is computed by determining the mass of emulsified asphalt contained in a standard measure of known volume.

Note 38—The calculation of Imperial Gallon Weight may be made by using proper conversion factors.

29.2. *Significance and Use*:

29.2.1. This test method provides a controlled laboratory test for the determination of quantitative volumes of emulsified asphalt for use in bills-of-lading, invoicing, and application rates.

29.3. *Apparatus*:

29.3.1. *Mass-per-Gallon Cup*—Stainless steel measure of known standard volume (83.2 mL).

29.3.2. *Balance*—Conforming to the requirements of M 231, Class G 2.

29.3.3. *Water Bath*—Capable of maintaining a constant temperature of 25 ± 0.5°C (77 ± 1°F). The water bath shall be capable of operation at the temperatures required, as corrected, if necessary, by calibration. The thermometer for measuring the temperature shall meet the requirements of M 339M/M 339 with a temperature range of at least 20 to 30°C (68 to 86°F), and an accuracy of ±0.13°C (±0.23°F) (see Note 39).

Note 39—Thermometer types suitable for use include ASTM E1 mercury thermometers; ASTM E879 thermistor thermometer; ASTM E1137/E1137M Pt-100 RTD platinum resistance thermometer, Special order; or IEC 60751: 2008 Pt-100 RTD platinum resistance thermometer, Special order.

29.3.4. *Timer*—Graduated in 0.1 s and accurate within 0.1 percent when tested over a 15-min interval.

29.3.5. *Sieve*—A 0.850-mm (No. 20) sieve, made of wire cloth and conforming to ASTM E11.

29.4. *Procedure*:

29.4.1. Stir the emulsified asphalt sample and place it in a constant-temperature water bath maintained at 25 ± 0.5°C (77 ± 1°F) for approximately 1 h.

29.4.2. Place the measure and its cap on the balance and zero the balance.

29.4.3. Remove the emulsified asphalt sample from the bath and stir it, using care to avoid trapping air in the sample. If necessary, strain it through an 850-mm (No. 20) sieve to remove any skin or film that might be present in the emulsified asphalt.

29.4.4. Bring the measure to approximately 25°C (77°F) and pour the emulsified asphalt into the measure, filling it completely.

29.4.5. Start placing the cap onto the measure, and remove, with a clean dry rag or paper, the excess emulsified asphalt oozing through the orifice in the cap.

29.4.6. When the cap is tightly placed on the measure, clean the measure carefully, and determine the mass on the tared balance to the nearest 0.01 g. Record this mass.

29.5. *Calculation*:

29.5.1. Calculate the mass per liter of emulsified asphalt as follows:

$$D_e = M_e \times 11.98 \qquad (10)$$

where:

D_e = density of the emulsified asphalt, g/L; and

M_e = mass of the emulsified asphalt in the measure, g.

29.5.2. When desired, calculate the mass per gallon of emulsified asphalt as follows:

$$D_e = M_e/10 \qquad (11)$$

where:

D_e = density of the emulsified asphalt, lb/gal; and

M_e = mass of the emulsified asphalt in the measure, g.

29.6. *Report*:

29.6.1. Report the density of emulsified asphalts in grams per liter (pounds per gallon) to the nearest 5 g (0.01 lb) at 25°C (77°F).

29.7. *Precision and Bias*:

29.7.1. The following criteria should be used for judging the acceptability of results (95 percent probability):

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

Density	Repeatability
Grams per liter at 25°C (77°F)	0.019
Pounds per gallon at 25°C (77°F)	0.019

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

Density	Reproducibility
Grams per liter at 25°C (77°F)	0.034
Pounds per gallon at 25°C (77°F)	0.034

Note 40—Mass per liter (gallon) at 25°C (77°F) may be translated to mass per liter (gallon) at 15.6°C (60°F) by using a multiplier of 1.00475.

30. KEYWORDS

30.1. Asphalt residue; cationic; coating; demulsibility; distillation; emulsified asphalt; evaporation; medium-setting; miscibility; particle charge; rapid-setting; settlement; slow-setting; storage stability; viscosity; water content.

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[1] Available from Koehler Instrument Company, Inc., 1595 Sycamore Avenue, Bohemia, NY 11716; and Humboldt Manufacturing Co., 3801 North 25th Avenue, Schiller Park, IL 60176.

[2] Available from Humboldt Manufacturing Co., Catalog No. H-1876, 3801 North 25th Avenue, Schiller Park, IL 60176.

[3] Limestone from Vulcan Materials Midwest Division, 6857 North U.S. Highway 421, Monon, IN 47959, has been found suitable as a reference aggregate.

[4] Fine aggregate is available from U.S. Silica Co., 701 Boyce Memorial Drive, Ottawa, IL 61350.