



## Standard Test Methods and Practices for Emulsified Asphalts<sup>1</sup>

This standard is issued under the fixed designation D 244; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the 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:

Test	Sections
Composition:	
Water Content	4-10
Residue and Oil Distillate by Distillation	11-15
Residue by Evaporation	16-22
Particle Charge of Cationic Emulsified Asphalts	23-28
Consistency:	
Viscosity (Saybolt Furol)	29-33
Stability:	
Demulsibility	34-39
Settlement	40-45
Cement Mixing	46-52
Sieve Test	53-58
Coating	59-62
Miscibility with Water	63-65
Freezing	66-68
Coating Ability and Water Resistance	69-76
Storage Stability of Asphalt Emulsion	77-83
Examination of Residue	84-91
Identification Test for Rapid Setting Cationic Emulsified Asphalt	92-99
Identification of Cationic Slow Set Emulsions	100-105
Field Coating Test on Emulsified Asphalts	106-111
Emulsified Asphalt/Job Aggregate Coating Test	112-117
Weight per Gallon of Emulsified Asphalt	118-124
Residue by Low-Temperature Vacuum Distillation	126-131

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 and health practices and determine the applicability of regulatory limitations prior to use.*

<sup>1</sup> These test methods 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 Tests.

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### 2. Referenced Documents

#### 2.1 ASTM Standards:

- C 150 Specification for Portland Cement<sup>2</sup>
- C 778 Specification for Standard Sand<sup>2</sup>
- D 5 Test Method for Penetration of Bituminous Materials<sup>3</sup>
- D 70 Test Method for Specific Gravity and Density of Semi-Solid Bituminous Materials<sup>3</sup>
- D 86 Test Method for Distillation of Petroleum Products<sup>4</sup>
- D 88 Test Method for Saybolt Viscosity<sup>5</sup>
- D 113 Test Method for Ductility of Bituminous Materials<sup>3</sup>
- D 128 Test Methods for Analysis of Lubricating Grease<sup>4</sup>
- D 139 Test Method for Float Test for Bituminous Materials<sup>3</sup>
- D 140 Practice for Sampling Bituminous Materials<sup>3</sup>
- D 977 Specification for Emulsified Asphalt<sup>3</sup>
- D 2042 Test Method for Solubility of Asphalt Materials in Trichloroethylene<sup>3</sup>
- D 2397 Specification for Cationic Emulsified Asphalt<sup>3</sup>
- D 3289 Test Method for Specific Gravity or Density of Semi-Solid and Solid Bituminous Materials by Nickel Crucible<sup>3</sup>
- E 1 Specification for ASTM Thermometers<sup>6</sup>
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>7</sup>
- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens<sup>7</sup>

### 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.

<sup>2</sup> Annual Book of ASTM Standards, Vol 04.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 04.03.

<sup>4</sup> Annual Book of ASTM Standards, Vol 04.04.

<sup>5</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>7</sup> Annual Book of ASTM Standards, Vol 14.02.

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 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 divisions from 0 to 2 mL, and in 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 D 86.

7. Sample

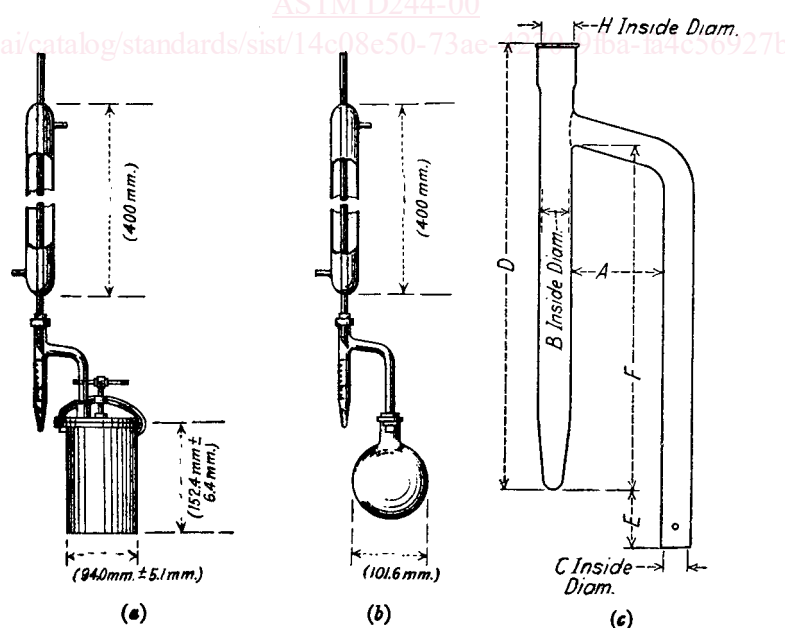
7.1 Obtain a representative sample of the material for test using standard procedures as specified in Practice D 140.

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  $100 \pm 0.1$  g of sample in the still. When the material contains more than 25 % water, use a  $50 \pm 0.1$ -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



A = 45 to 55 mm  
 B = 14 to 16 mm  
 C = 12 to 16 mm  
 D = 235 to 255 mm

E = 25 to 38 mm  
 F = 186 to 194 mm  
 H = 18 to 19 mm

FIG. 1 Apparatus for Determining Water Content

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 D 244."

## 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:

Water Content, weight % 30 to 50	Reproducibility, weight % 2.0
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## RESIDUE AND OIL DISTILLATE BY DISTILLATION

### 11. Scope

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

### 12. Significance and Use

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

### 13. Apparatus

13.1 *Aluminum-Alloy Still*,<sup>8</sup> (see Fig. 2), approximately 241.3 mm (9½ in.) in height by 95.3 mm (3¾ in.) in inside diameter with one 121-mm (4¾-in.) inside diameter ring burner,<sup>9</sup> having holes on the inner periphery and having three spacers, to ensure centering of burner around the still (see Fig. 3).

NOTE 2—Residue by distillation results obtained with iron stills in accordance with Method D 244 – 66 are acceptable. Similarly results obtained with a 127-mm (5-in.) ring burner as in subsequent issues of Test Methods D 244 are acceptable.

13.2 *Connection Apparatus*, consisting of a 12.5 ± 0.5 mm glass or metal connecting tube. The metal connecting tube may be secured to the lid by a threaded compressing fitting. Other condensers of 12.5 ± 0.5 mm glass or metal tubing may be used providing that the wetted length is 400 to 550 mm (15.5 to 21').

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

13.4 *Thermometer*—Two ASTM Low-Distillation Thermometers, graduated either in Fahrenheit or Celsius degrees as specified, having a range from –2 to +300°C, (30 to 580°F) respectively, and conforming to the requirements for Thermometer 7C or 7F as prescribed in Specification E 1, or any other thermometric device of equal accuracy or response.

NOTE 3—For details of the assembly of apparatus for the distillation test, see Fig. 4.

13.5 *Balance*, capable of weighing 3500 g to within ±0.1 g.

### 14. Procedure

14.1 Weigh 200 ± 0.1 g of a representative sample of the emulsion in the previously weighed aluminum-alloy still (including lid, clamp, thermometers and gasket, if gasket is used).

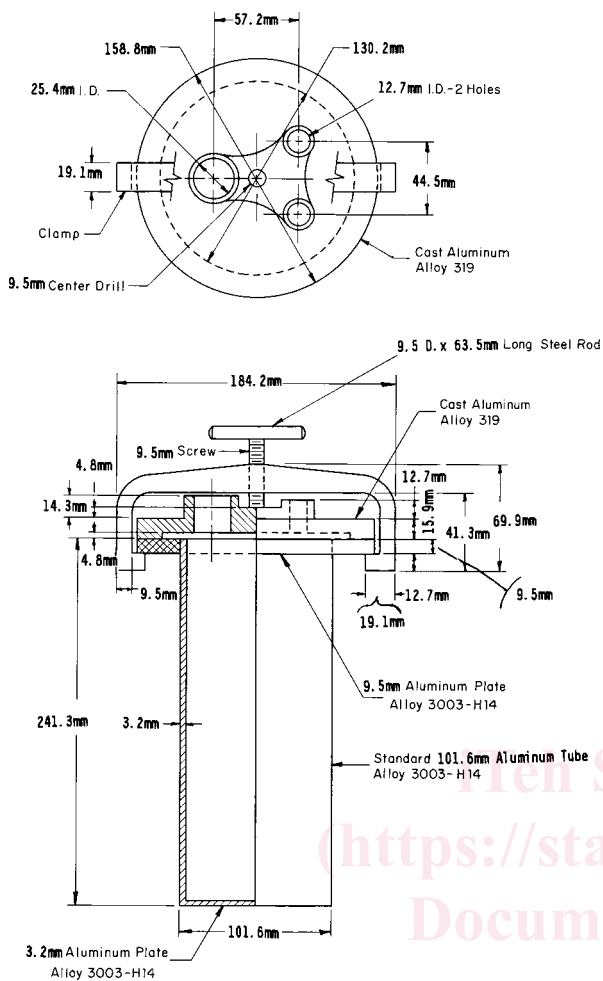
14.2 Use a gasket of oiled paper between the still and its cover, or grind the joint to a tight fit. Securely clamp the cover on the still. Other gasket materials may be used providing they withstand the maximum temperature reached during distillation.

14.3 Insert a thermometer, or other thermometric device, 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 6.4 mm from the bottom of the still and the bulb of the other is approximately 165.1 mm from the bottom of the still.

14.4 Place the ring burner around the still about 152.4 mm (6 in.) from the bottom of the still. Apply heat by lighting this burner and adjusting to low flame. Also apply just enough heat from a bunsen burner to the connecting tube to prevent condensation of water in this tube.

<sup>8</sup> Available from P & H Electronics, 442 Columbia St., Lafayette, IN 47901, Koehler Instruments, Inc., 168-56 Douglas Ave., Jamaica, NY 11433, and Humboldt Mfg. Co., 7300 W. Agatite Ave., Chicago, IL 60656.

<sup>9</sup> Available from Humboldt Manufacturing Co., Catalog No. H-1876, 7302 W. Agatite Ave., Chicago, IL 60656.



Metric Equivalents

in.	mm
1/8	3.2
3/16	4.8
3/8	9.5
1/2	12.7
9/16	14.3
5/8	15.9
3/4	19.1
1	25.4
1 1/8	41.3
1 3/4	44.5
2 1/4	57.2
2 1/2	63.5
4	101.6
5 1/8	130.2
6 1/4	158.8
7 1/4	184.2
9 1/2	241.3

NOTE 1—The still cover may be machined from Rolled Aluminum Plate Alloy 3003-H14.

FIG. 2 Aluminum-Alloy Still

14.5 Move the ring burner approximately level with the bottom when the temperature can be read on the lower thermometer, approximately 215°C. Increase the temperature to 260 ± 5°C, maintaining it at this temperature for 15 min. Complete the total distillation in 60 ± 15 min from the first application of heat.

NOTE 4—The location of the burner at the start of the test is flexible. It may be raised to decrease chance of foam-over or lowered to middle of still for emulsion containing no solvent. A sudden change in temperature reading of upper thermometer indicates foam on bulb. Remove heat until foaming ceases.

14.6 Immediately at the expiration of the heating period, again weigh the still and accessories as described in 14.1. Calculate and report the percentage residue by distillation. Record the volume of oil distillate to the nearest 1/2 mL. Calculate and report the oil distillate as a volume percentage on the total emulsion. Save this oil distillate if identification is desired.

NOTE 5—The aluminum-alloy still at room temperature (9.1) weighs 1.5 g more than at 260°C. Correct for this error by adding 1.5 g to gross weight obtained in 14.6 prior to calculating the percentage of residue by distillation.

14.7 Remove the cover from the still, stir, and immediately pour suitable portions of the residue into an 8-oz tin or suitable molds and containers for making the required tests. Handle or condition molds and containers for desired examination of the residue as described in Sections 86-91, and proceed as required by the appropriate ASTM test method from the points that follow the pouring stage. If there is foreign matter in the residue, the material shall be poured through a 300-µm sieve prior to pouring into the test molds and containers.

15. Precision and Bias

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

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

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

15.1.2 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, weight %	Reproducibility, weight %
50 to 70	2.0

15.2 The precision for penetration of residue from distillation by this method is the same as that shown in Section 79.

RESIDUE BY EVAPORATION

16. Scope

16.1 This test method covers procedures for a relatively rapid determination of the percentage of emulsion residue.

17. Significance and Use

17.1 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 sections. However, properties of the residue from evaporation procedure may differ from those of the distillation residue (see Note 7).

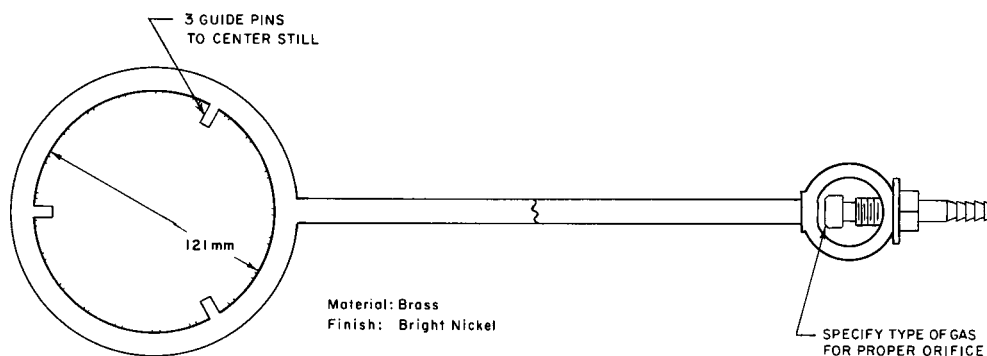


FIG. 3 Ring Burner with 4¾-in. (121-mm) Inside Diameter

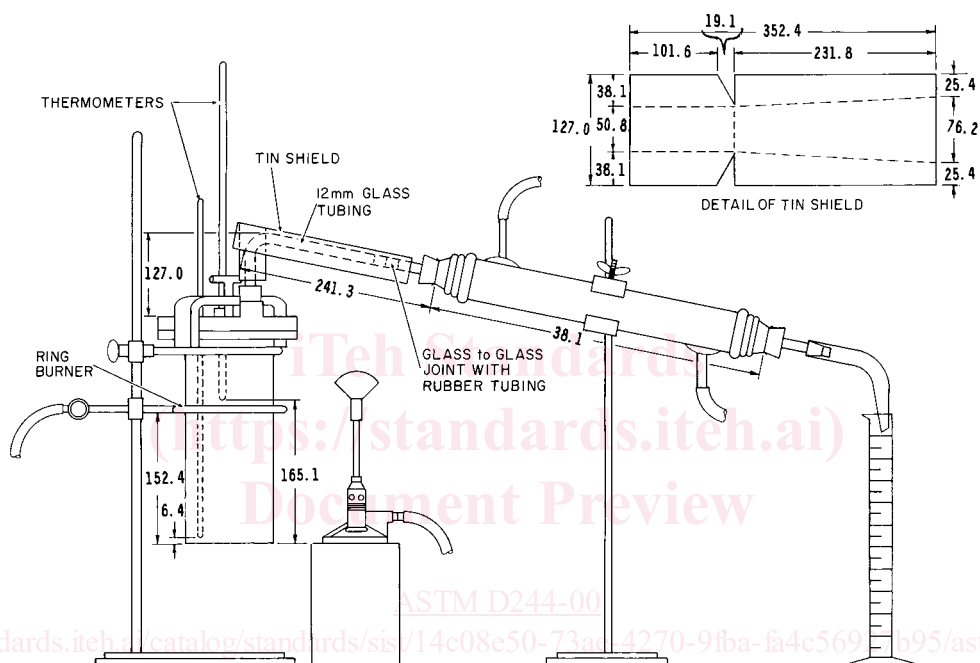


FIG. 4 Apparatus Assembly for Distillation Test of Emulsified Asphalts

		Metric Equivalents														
in.	¼	¾	1	1½	2	3	4	5	6	6½	9⅛	9½	13⅞	15		
mm	6.4	19.1	25.4	38.1	50.8	76.2	101.6	127.0	152.4	165.1	231.8	241.3	352.4	381.0		

**18. Apparatus**

18.1 *Beakers*, low form, 1000-mL capacity, made of borosilicate glass or aluminum.

18.2 *Glass Rods*, with flame-polished ends, 6.4 mm in diameter and 177.8 mm in length.

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

18.4 *Oven*, conforming to Specification E 145, Type 1B.

18.5 *Sieve*—A 300-µm sieve conforming to Specification E 11.

**19. Procedure A**

19.1 Use procedure A when determination of the percentage of residue only is required.

19.2 Weigh 50 ± 0.1 g of thoroughly mixed, emulsified asphalt into each of three beakers, each beaker having previously been weighed with a glass rod. Place the beakers containing the rods and sample in the oven, the temperature of

which has been adjusted to 163 ± 3.0°C, for 2 h. At the end of this period remove each beaker and stir the residue thoroughly. Replace in the oven for 1 h, then remove the beakers from the oven, allow to cool to room temperature, and weigh, with the rods.

NOTE 6—Care must be taken to prevent loss of asphalt from the beaker through foaming or spattering, or both. Also, the placing of beakers and emulsion samples in a cold or warm oven and bringing the oven and sample up to a temperature of 163°C together is permissible. If preferred, preliminary evaporation of water may be accomplished by careful heating on a hot plate, followed by oven treatment at 163°C for 1 h.

**20. Procedure B**

20.1 Use procedure B when tests on the residue from the emulsion are required.

20.2 Proceed in accordance with 22.2 using four 50 ± 0.1-g samples. After the calculation for percentage of residue,



replace the beakers in the oven until the asphalt residue is sufficiently fluid to pass through a 300- $\mu\text{m}$  sieve (usually requiring 15 to 30 min). Pour the residue through the 300- $\mu\text{m}$  sieve into suitable containers and molds for making such tests as desired, as described in Sections 86-90.

**NOTE 7**—As the test method for residue by evaporation described in Sections 18-20 tends to give an asphaltic residue lower in penetration and ductility than the distillation test method described in Sections 13-15, material may be accepted but shall not be rejected as failing to meet specifications containing requirements for determination of residue by distillation, on data obtained by evaporation. If residue from evaporation fails to meet the requirements for properties specified for residue from distillation, tests shall be rerun using the distillation test method.

## 21. Calculation and Report

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

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

where:

$A$  = weight of beaker, rod, and residue, g, and

$B$  = tare weight of beaker and rod, g.

21.2 Report the percentage of residue by evaporation as the average of the three results.

## 22. Precision and Bias

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

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

Residue by Evaporation, weight % 50 to 70	Repeatability, weight % 0.4
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22.1.2 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, weight % 50 to 70	Reproducibility, weight % 0.8
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## PARTICLE CHARGE OF CATIONIC EMULSIFIED ASPHALTS

### 23. Scope

23.1 This test method is used to identify cationic emulsions. Positively charged particles are classified as cationic.

### 24. Significance and Use

24.1 Cationic emulsions are identified by the migration of the particles to a negatively charged electrode (cathode) by means of a direct current.

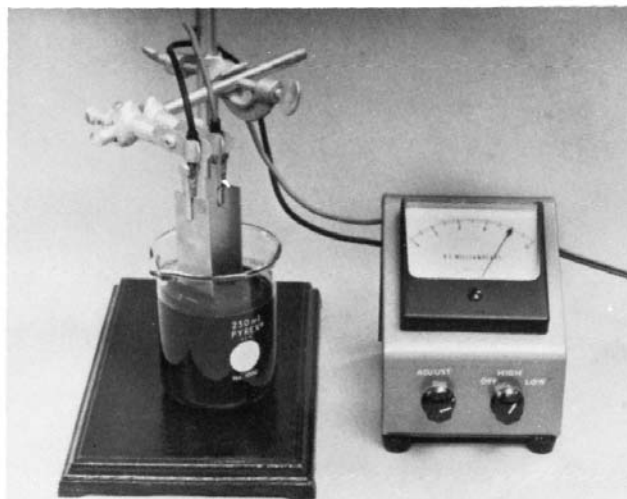
### 25. Apparatus

25.1 *Current Source*, of 12-V dc, a milliammeter, and a variable resistor (see Fig. 5 and Fig. 6).

25.2 *Electrodes*—Two stainless steel plates, 25.4 mm by 101.6 mm insulated from each other and rigidly held parallel 12.7 mm apart (see Fig. 7).

25.3 *Insulator*—Polytetrafluoroethylene resin square rod, virgin electrical grade, 12.7 mm thick (see Fig. 7).

25.4 *Beaker*, 250 mL.



**FIG. 5 Particle Charge Tester**

25.5 *Glass Rod*, 101.6 mm long and 6.35 mm thick or other suitable material or device that is capable of insulating and suspending the electrode assembly in emulsion.

25.6 *Water Bath*, capable of maintaining the required testing temperature within the limits specified in this test method.

25.7 *Thermometer*, ASTM No. 19C or 19F conforming to the requirements of Specification E 1.

## 26. Procedure

26.1 Heat the emulsion to be tested to  $50 \pm 3^\circ\text{C}$ , in a  $71 \pm 3^\circ\text{C}$  water bath. Stir the emulsion thoroughly to ensure uniformity of temperature.

26.2 Pour the emulsion to be tested into the 250-mL beaker to a height that will allow the electrodes to be suspended 25.4 mm in the emulsion. 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 electrode assembly in emulsion may be used if desired.

26.3 Connect the electrodes, that have been properly cleaned (Note 8), to the dc source.

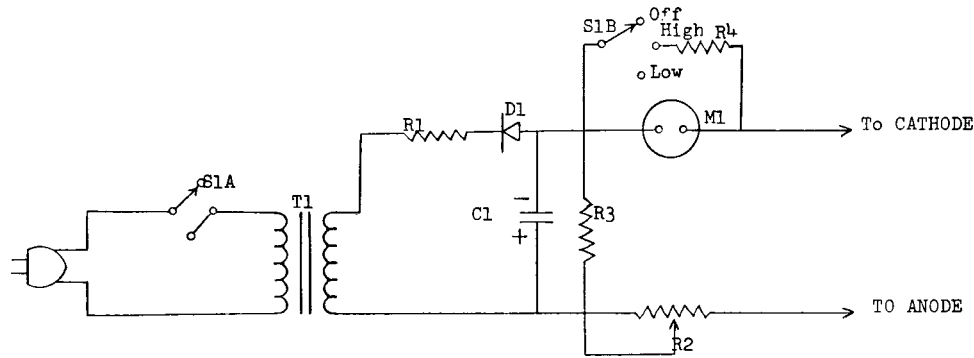
**NOTE 8**—New electrodes and electrodes to be re-used should be cleaned in the following sequence:

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.

26.4 Adjust the current to at least 8 mA with the variable resistor and start timing with a suitable timing device. (The 8 mA is a minimum current value. Higher current levels may be specified. The current used shall be reported.)

26.5 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.

26.6 Observe the asphalt deposit on the electrodes. A cationic emulsion will deposit a discernible amount of asphalt



C1—500  $\mu$ F 25-V capacitor  
 D1—silicon diode  
 R1—47  $\Omega$ , 1-W resistor  
 R2—5000  $\Omega$  potentiometer  
 R3—6800  $\Omega$ , 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  
 M1—0 to 10-mA milliammeter

FIG. 6 Particle Charge Tester Circuit Diagram

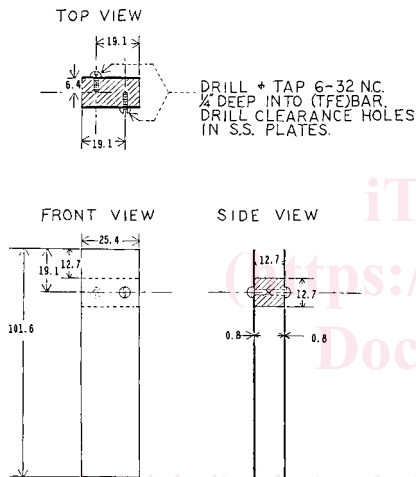


FIG. 7 Insulator

electrode) will be relatively clean. Any evidence of a deposit of asphalt on the cathode which is clearly discernible when compared to the anode is to be considered passing.

NOTE 9—If the emulsion does not produce conclusive results and it is a cationic slow-setting grade, then proceed to Section 100, "Identification of Cationic Slow-Set Emulsion."

27. Report

27.1 Report the following information:

27.1.1 Level of current used, and

27.1.2 Whether the tested emulsion passes or fails as defined in 26.6.

28. Precision and Bias

28.1 This test method, which requires 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.

on the cathode (negative electrode) while the anode (positive

CONSISTENCY

VISCOSITY

29. Scope

29.1 This test method utilizes the Saybolt Furol viscometer to measure the consistency of an asphalt emulsion. It is applicable to all the emulsions specified in Specifications D 977 and D 2397.

30. Significance and Use

30.1 Viscosity has significance in the use of asphalt emulsions because it is a property which affects their utility. When used in application types of construction, the material must be thin enough to be uniformly applied through the spray bar of distributor, yet thick enough so that it will not flow from the crown or grade of the road. For mixing grade emulsions, the viscosity may affect mixability and resulting thickness of film

on the aggregate. The viscosity of many emulsions is affected by shear. Therefore, strict adherence to test procedure is necessary to achieve precision.

31. Apparatus

31.1 *Viscometer*—A Saybolt Furol viscometer conforming to the requirements specified in Test Method D 88.

31.2 *Sieve*—A 850- $\mu$ m sieve or a 20-mesh strainer of wire cloth, framed or unframed.

31.3 *Thermometers*—ASTM No. 17C or 17F for tests at 25°C and ASTM No. 19F or 19C for tests at 50°C, conforming to the requirements of Specification E 1.

31.4 *Water Bath*, capable of maintaining the required testing temperature within the limits specified in Table 2 of Test Method D 88.

### 32. Procedure

32.1 *Tests at 25°C*—Stir the sample thoroughly without incorporating bubbles and pour it into a 118-mL bottle. Place the bottle in the water bath at 25°C for 30 min and mix the sample in the bottle by inverting it several times slowly enough to prevent bubble formation. Pour the sample into the viscometer through the 850- $\mu$ m sieve or 20-mesh strainer, allowing a small portion to flow through the outlet tube to waste. Place the cork in position, fill the viscometer and, without again stirring the sample, determine the viscosity as prescribed in Test Method D 88.

32.2 *Tests at 50°C*—Clean and dry the viscometer and insert the cork. Heat the emulsion sample to  $122 \pm 5^\circ\text{F}$  ( $50 \pm 3^\circ\text{C}$ ) in a  $160 \pm 5^\circ\text{F}$  ( $71 \pm 3^\circ\text{C}$ ) water bath or oven. Stir the sample thoroughly without incorporating bubbles, and then pour approximately 100 mL into a 400-mL glass beaker. Immerse the bottom of the beaker containing the emulsion approximately 2 in. (50.8 mm) below the level of a  $160 \pm 5^\circ\text{F}$  ( $71 \pm 3^\circ\text{C}$ ) water bath. Hold the beaker upright and stir the emulsion with a wide circular motion at a rate of 60 revolutions per minute with the thermometer to obtain uniform temperature distribution. Avoid incorporation of bubbles. Heat the emulsion in the water bath to  $124.5 \pm 0.5^\circ\text{F}$  ( $51.4 \pm 0.3^\circ\text{C}$ ). Immediately pour the emulsion through the 850- $\mu$ m (No. 20) sieve or 20-mesh strainer into the viscometer until it is above the overflow rim. Stir the emulsion in the viscometer at 60 revolutions per minute with the thermometer until the test temperature is attained,

avoiding bubble formation. Adjust the bath temperature until the emulsion temperature remains constant for 1 min at  $50 \pm 0.05^\circ\text{C}$ . Withdraw the thermometer. Quickly remove the excess emulsion from the gallery with a suction pipet. Determine the viscosity as described in Test Method D 88. Report the results to the nearest full second.

NOTE 10—While the Saybolt Furol viscometer is not used for petroleum products and lubricants when the time of flow is less than 25 s, this instrument is satisfactory for testing emulsified asphalt when the time of flow is not less than 20 s.

### 33. Precision and Bias

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

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

Test Temperature, °C	Viscosity, s	Repeatability, % of the mean
25	20 to 100	5
50	75 to 400	9.6

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

Test Temperature, °C	Viscosity, s	Reproducibility, % of the mean
25	20 to 100	15
50	75 to 400	21

## DEMULSIBILITY

### 34. Scope

34.1 This test method, applicable to anionic and cationic emulsions of the RS and MS type, measures the chemical breaking of the emulsion.

### 35. Significance and Use

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

### 36. Apparatus and Reagents

36.1 *Wire Cloth*—Three pieces of 1.40-mm wire cloth approximately 125 mm square, unframed, having wire diameters and openings that conform to Specification E 11.

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

36.3 *Rods*—Three metal rods with rounded ends, approximately (7.9 mm) in diameter.

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

36.5 *Calcium Chloride Solution* (1.11 g/L)—Dissolve 1.11 g of calcium chloride ( $\text{CaCl}_2$ ) in water and dilute to 1 L. The

1.11 g/L calcium chloride solution shall be standardized to be a  $0.02 \text{ N} \pm 0.001$  normal solution of calcium chloride in water.

36.6 *Calcium Chloride Solution* (5.55 g/L)—Dissolve 5.55 g of  $\text{CaCl}_2$  in water and dilute to 1 L. The 5.55 g/L calcium chloride solution shall be standardized to be a  $0.1 \text{ N} \pm 0.001$  normal solution of calcium chloride in water.

36.7 *Dioctyl Sodium Sulfosuccinate Solution* (0.8%)—Dissolve 8.00 g of dioctyl sodium sulfosuccinate in 992 g of water.

36.8 *Balance*, capable of weighing 500 g to within  $\pm 0.1$  g.

### 37. Procedure

37.1 Determine the percentage of residue by distillation as described in Section 14.

37.2 Record the weight of each assembly of beaker, rod, and wire cloth.

37.3 Weigh  $100 \pm 0.1$  g of the emulsified asphalt into each of three 600-mL beakers in the weighed assemblies. Bring the weighed sample of emulsion and the proper reagent to a temperature of ( $25 \pm 0.5^\circ\text{C}$ ). Over a period of approximately 2 min, add to each beaker, from a buret, 35 mL of  $\text{CaCl}_2$  solution (1.11 g/L) (Note 10) for rapid setting emulsions, or 50 mL of  $\text{CaCl}_2$  solution (5.55 g/L) for mixing-type emulsions. While adding the  $\text{CaCl}_2$  solution, 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 emulsion. Continue kneading any lumps for an additional 2 min after the addition of the CaCl<sub>2</sub> solution.

NOTE 11—When testing cationic emulsions, use 35 mL of dioctyl sodium sulfosuccinate solution (0.8 %) instead of 35 mL of CaCl<sub>2</sub> solution (1.11 g/L).

37.4 Decant the mixture of any unbroken emulsion and reagent onto the wire cloth. Rinse the beaker containing the sample and metal rod with distilled water. Knead and break up all lumps, and continue washing the beaker, rod, and wire cloth until the wash water drains clear. Place the wire cloth enclosing the asphalt in the beaker with the metal rod. Place the assembly in a 163°C drying oven and dry to constant weight.

### 38. Calculation

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

$$\text{Demulsibility, \%} = (A/B) \times 100 \quad (3)$$

where:

A = average weight of demulsibility residue from the three tests of each sample of emulsified asphalt, and

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

### 39. Precision and Bias

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

NOTE 12—Precision does not apply when using dioctyl sodium sulfosuccinate solution in the testing of cationic emulsions for demulsibility.

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

Demulsibility, weight % 30 to 100	Repeatability, % of the mean 5
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39.1.2 The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

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

### 40. Scope

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

### 41. Significance and Use

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

### 42. Apparatus

42.1 *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.

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

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

### 43. Procedure

43.1 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. After standing for this period, remove approximately the top 55 mL of emulsion by means of a pipet or siphon without disturbing the balance. Mix each portion thoroughly. Weigh 50 g of each sample into separate weighed 1000-mL low-form glass beakers, and determine the asphaltic residue by evaporation in accordance with Section 19.

43.2 After removal of the top sample, siphon off approximately the next 390 mL from each of the cylinders. Thoroughly mix the emulsion remaining in the cylinders and weigh 50 g into separate weighed 1000-mL low-form beakers. Determine the asphaltic residue of these samples in accordance with Section 19.

NOTE 13—If the emulsion contains appreciable amounts of oil distillate as determined by distillation (see 14.6), the settlement value may be calculated from the difference in the percentage of water content between the top and bottom samples as determined by the procedure described in Section 8.

### 44. Calculation and Report

44.1 Calculate the settlement for each cylinder as follows:

$$\text{Settlement, \% (5 days)} = B - A \quad (4)$$

where:

A = the percentage of residue from the top samples, and  
B = the percentage of residue from the bottom samples.

NOTE 14—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 emulsion, 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 greater than the percent residue of the emulsion, the result is considered suspect and the test shall be repeated.

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

### 45. Precision and Bias

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

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

Settlement, weight % 0 to 1.0 above 1.0	Repeatability 0.4 weight % 5 % of the mean
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45.1.2 The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Settlement, weight % 0 to 1.0 above 1.0	Reproducibility 0.8 weight % 10 % of the mean
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## CEMENT MIXING

### 46. Scope

46.1 This test method is a mixing test used to identify or classify a slow setting, SS or CSS, type of emulsion.

### 47. Significance and Use

47.1 The result of this test indicates the ability of a slow setting emulsified asphalt to mix with a finely divided, high surface area material (high early strength, Type III, portland cement) without breaking the emulsion.

### 48. Apparatus

48.1 *Sieves*—A 180- $\mu\text{m}$  sieve and a 3-in. (76.2-mm) diameter 1.4-mm sieve, made of wire cloth conforming to Specification E 11.

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

48.3 *Stirring Rod*—A steel rod with rounded ends, approximately 13 mm in diameter.

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

48.5 *Balance*, capable of weighing 500 g to within  $\pm 0.1$  g.

### 49. Cement

49.1 High-early-strength portland cement conforming to the requirements for Type III portland cement in Specification C 150 and having a minimum specific surface area of 1900  $\text{cm}^2/\text{g}$ , as measured by the Wagner Turbidimeter.

### 50. Procedure

50.1 Dilute the emulsion with distilled water to a residue of 55 %, as determined by distillation or by evaporation for 3 h at 163°C.

50.2 Sieve a portion of the cement through the 180- $\mu\text{m}$  sieve. Weigh  $50 \pm 0.1$  g of the cement passing the No. 80 (180- $\mu\text{m}$ ) sieve into the iron dish or saucepan.

50.3 Bring the ingredients and apparatus to a temperature of approximately 25°C before mixing. Add 100 mL of the diluted emulsion to the cement and stir the mixture at once with the steel rod, using a circular motion at a rate of 60 revolutions per minute. At the end of the 1-min mixing period, add 150 mL of distilled water, and continue the stirring for 3 min.

50.4 Pour the mixture through a weighed 1.4-mm sieve. Use repeated washings to completely remove material from the mixing bowl. Pour these over the sieve, and rinse the sieve using distilled water held at a height of approximately 152 mm until the water is clear. Place the sieve in a weighed shallow pan, heat at 163°C in an oven, and weigh. Repeat the heating and weighing until successive weights differ by no more than 0.1 g.

### 51. Report

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

### 52. Precision and Bias

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

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

Cement Mixing, weight % 0 to 2	Repeatability, weight % 0.2
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52.1.2 The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Cement Mixing, weight % 0 to 2	Reproducibility, weight % 0.4
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## SIEVE TEST

### 53. Scope

53.1 This test method measures the degree to which an emulsified asphalt may contain particles of asphalt or other discreet solids retained on a 850  $\mu\text{m}$  mesh sieve.

### 54. Significance and Use

54.1 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 are other factors affecting particle formation.

### 55. Apparatus and Reagents

55.1 *Sieve*—A sieve having a 76.2-mm frame conforming to 76.2 mm of Specification E 11, and having a 850- $\mu\text{m}$  wire sieve cloth.

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

55.3 *Sodium Oleate Solution (2 %)*—Dissolve 2 g of pure sodium oleate in distilled water and dilute to 100 mL.

NOTE 15—Replace sodium oleate solution with distilled water in testing cationic emulsions.

55.4 *Balances*, capable of weighing 2000 g to within  $\pm 1$  g, and 500 g to within  $\pm 0.1$  g.

### 56. Procedure

56.1 The temperature at which the sieve test should be performed is related to the emulsion viscosity. For those materials whose viscosity is 100 s or less at 25°C, perform the test at room temperature. For those materials whose viscosity is more than 100 s at 25°C and those whose viscosity is specified at 50°C, use a test temperature of  $50 \pm 3^\circ\text{C}$ . If heating is necessary the emulsion, in a closed container, may be placed in an oven or water bath, followed by stirring to achieve homogeneity.

56.2 Record the weight of the sieve and pan and wet the wire cloth with the 2 % sodium oleate solution. Weigh 1000 g of the emulsified asphalt into a suitable container and pour it through the sieve. Wash the container and the residue on the sieve with the sodium oleate solution until the washings run clear. Place the pan under the sieve and heat for 2 h in a 105°C drying oven. Cool in a desiccator, and weigh the sieve, pan and residue.