
Standard Method of Test for Emulsified Asphalts

AASHTO Designation: T 59-22

Technically Revised: 2022

Technical Subcommittee: 2a, Emulsified Asphalts



**American Association of State Highway and Transportation Officials
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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 $\pm 0.5^\circ\text{C}$ ($\pm 0.9^\circ\text{F}$) (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 $\pm 0.75^\circ\text{C}$ ($\pm 1.35^\circ\text{F}$) (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.

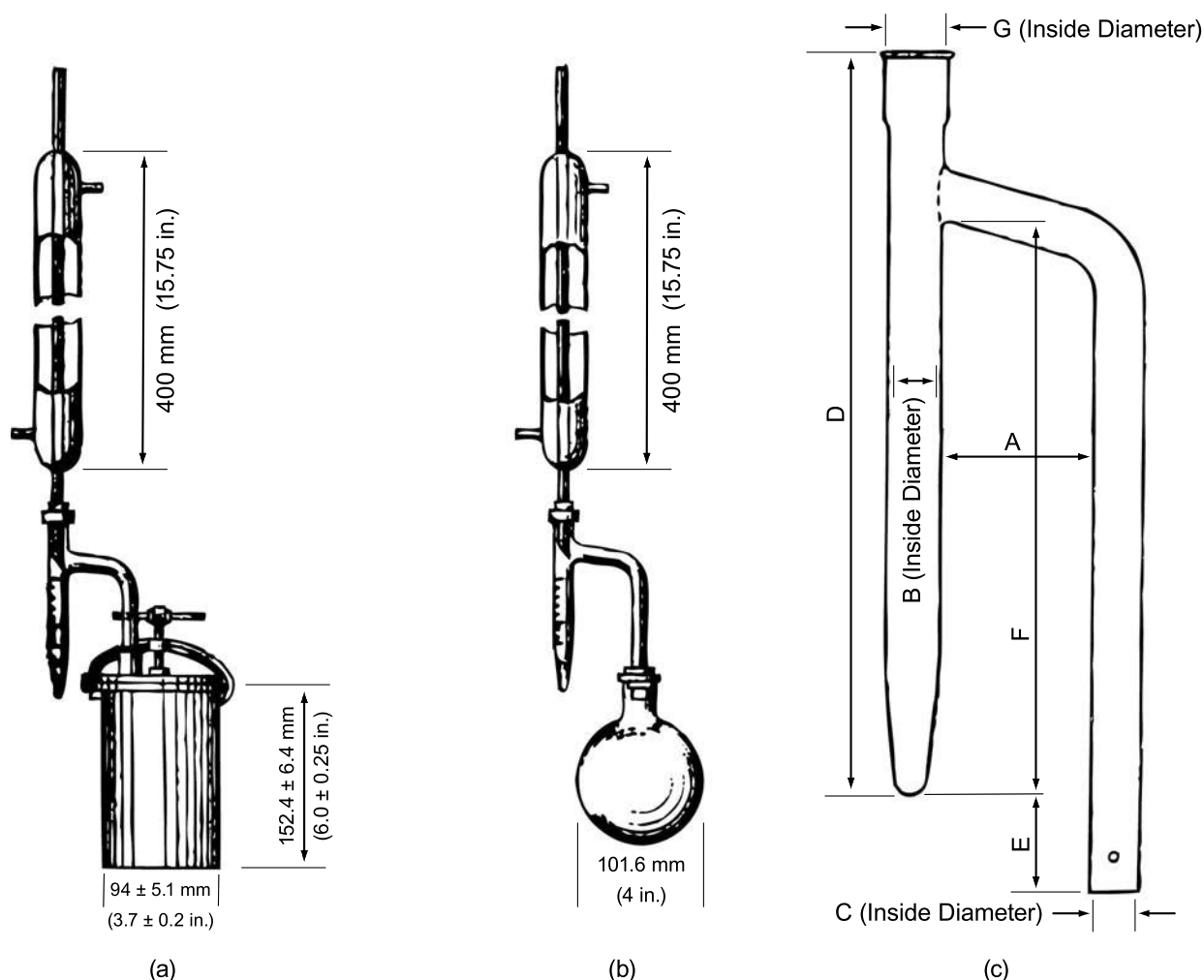
COMPOSITION

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 (15³/₄ 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.



Metric Equivalents

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

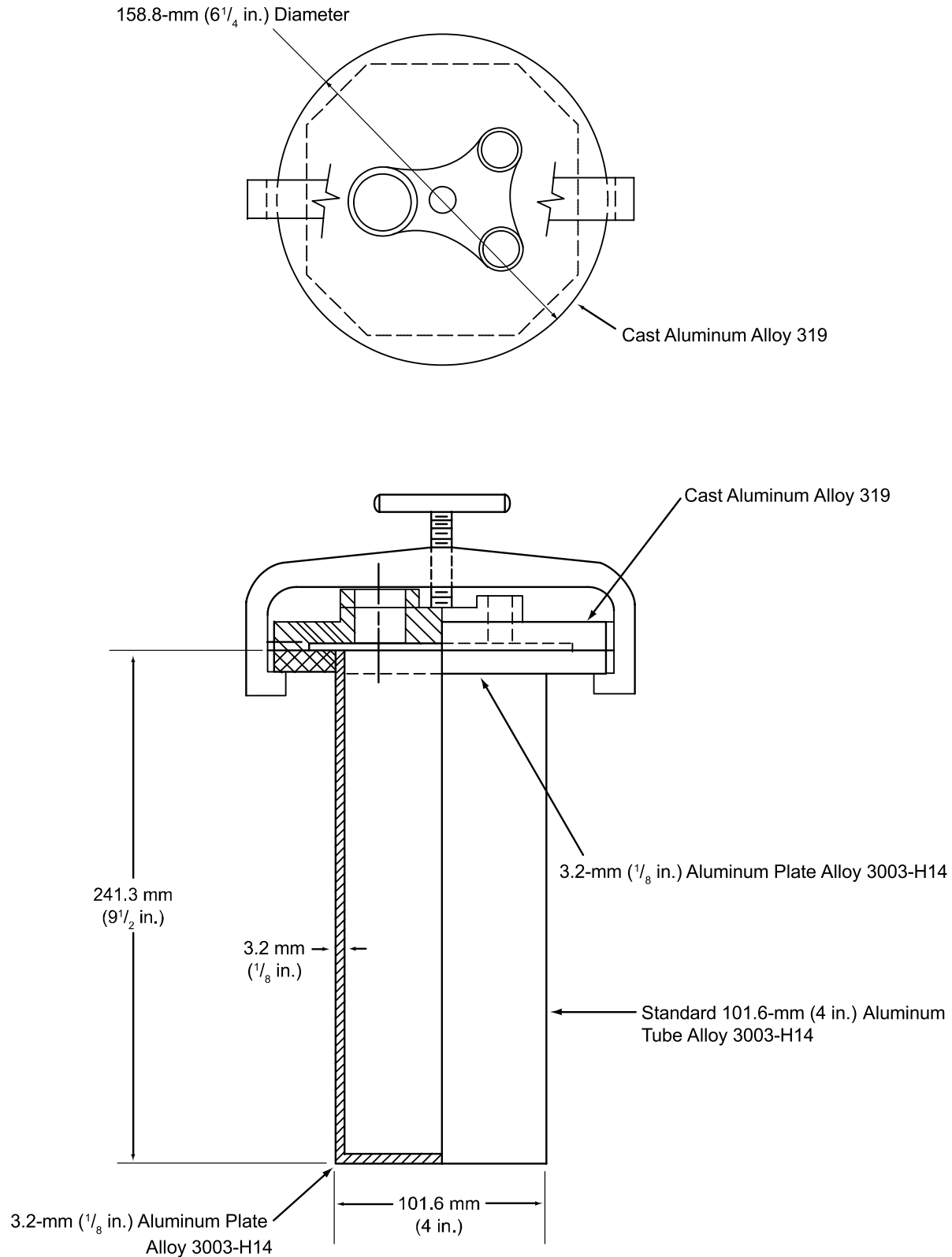
6. **RESIDUE AND OIL DISTILLATE BY DISTILLATION**

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 Pot¹ and Heat Source*—Approximately 240 mm (9½ in.) in height by 95 mm (3¾ in.) in inside diameter (Figure 2) with one 121-mm (4¾ 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.

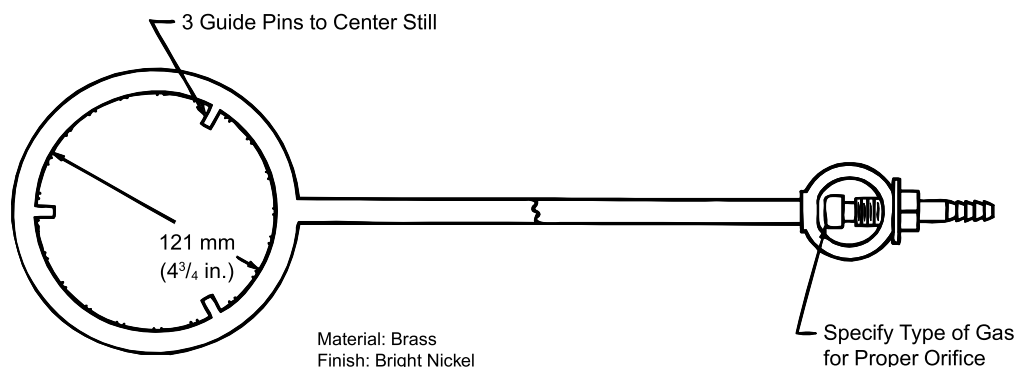


Figure 3—Ring Burner with 121-mm (4¾ in.) Inside Diameter

- 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.

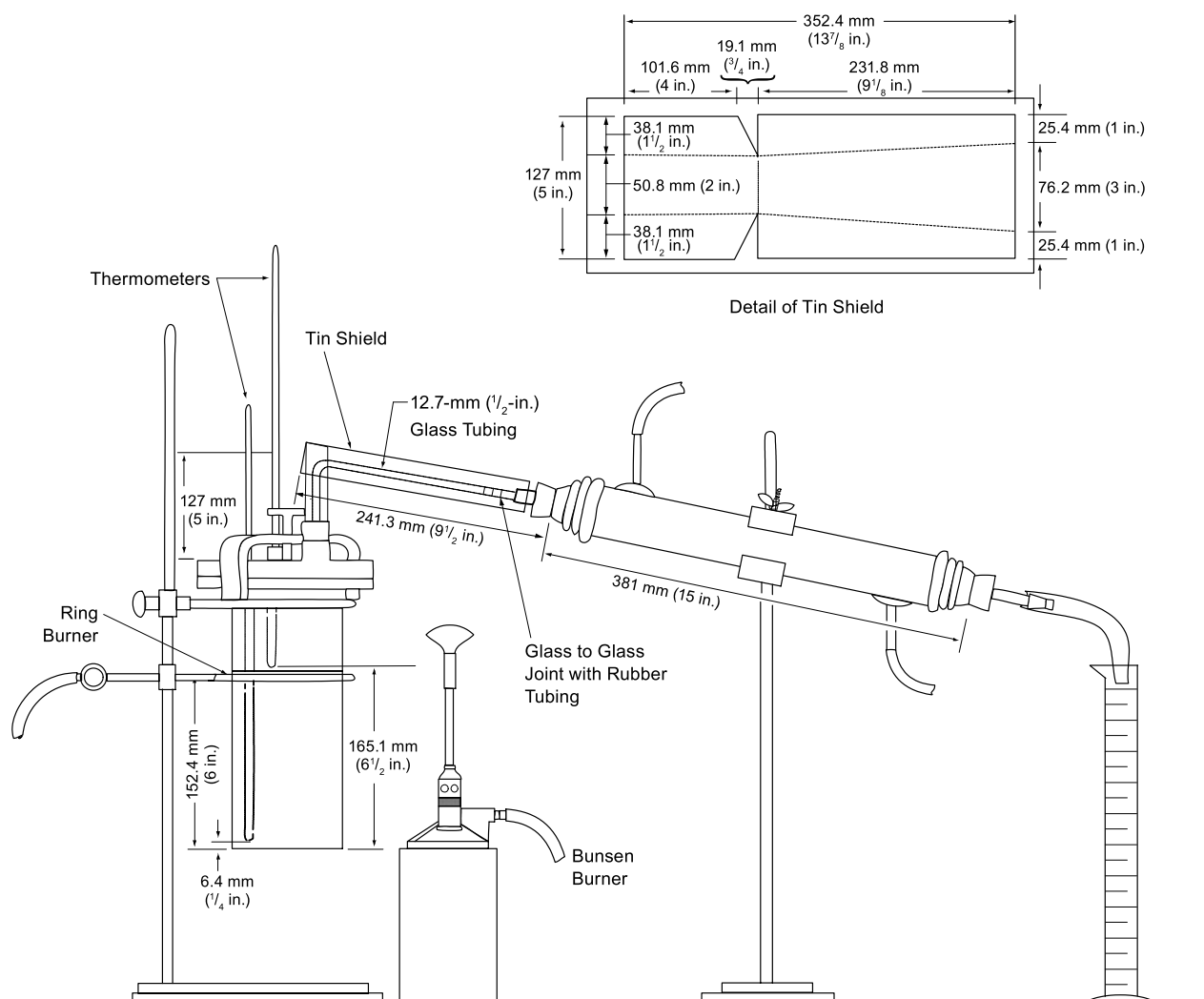
- 6.3.4. *Thermometers*—Two thermometers shall be constructed such that they can be positioned in the manner described in Section 6.4.3. The thermometers shall meet the requirements of M 339M/M 339 with a temperature range of at least -2 to 300°C (30 to 580°F), and an accuracy of ±0.5°C (±0.9°F) (see 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:

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

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:

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

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:*