



Standard Test Methods for Emulsified Bitumens Used as Protective Coatings¹

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This standard has been approved for use by agencies of the Department of Defense.

ASTM Test Methods D 2939 is the method recommended for use by Committee D-8 in place of Methods D 1010 and D 1167.

1. Scope

1.1 These test methods cover procedures for sampling and testing emulsified bitumens used in relatively thick films as protective coatings for metals and built-up roofs.

1.2 The test methods appear in the following order:

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

1.4 *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.*

2. Referenced Documents

2.1 ASTM Standards:

- C 67 Test Methods of Sampling and Testing Brick and Structural Clay Tile³
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials⁴
- D 4 Test Method for Bitumen Content⁵
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester⁶
- D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation⁶
- D 140 Practice for Sampling Bituminous Materials⁷
- D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products⁸
- D 3699 Specification for Kerosine⁹
- D 4798 Test Method for Accelerated Weathering Test Conditions and Procedures for Bituminous Materials (Xenon-Arc Method)⁵
- D 4799 Test Method for Accelerated Weathering Test Conditions and Procedures for Bituminous Materials (Fluorescent and UV Condensation Method)⁵
- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens¹⁰

3. Significance and Use

3.1 These tests are useful in evaluating and characterizing coal tar emulsion and other bituminous emulsions to establish uniformity of shipments.

4. Sampling

4.1 Determine the number of containers sampled to represent a shipment in accordance with Practice D 140.

4.2 Open the original containers and examine them for uniformity of contents. Record the degree of separation, if any,

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² This test is intended to evaluate the relative resistance of coatings which might be exposed to fuel spills.

³ *Annual Book of ASTM Standards*, Vol 04.05.

⁴ *Annual Book of ASTM Standards*, Vol 04.02.

⁵ *Annual Book of ASTM Standards*, Vol 04.04.

⁶ *Annual Book of ASTM Standards*, Vol 05.01.

⁷ *Annual Book of ASTM Standards*, Vol 04.03.

⁸ *Annual Book of ASTM Standards*, Vol 06.01.

⁹ *Annual Book of ASTM Standards*, Vol 05.02.

¹⁰ *Annual Book of ASTM Standards*, Vol 14.02.

into portions of appreciably different consistency, such as thick or thin layers, sedimentation or coagulation, etc. Also note any difficulty encountered in stirring to a uniform condition.

4.3 Take the samples for laboratory examination from the original containers immediately after stirring to a uniform condition. Restir individual or combined samples immediately before taking out portions for tests.

5. Uniformity

5.1 *Procedure*—Examine the contents of a full container of not less than 1 L or 1 qt in volume that has stood undisturbed for 48 h.

5.2 *Report*—Make a notation of any separation of water, coagulation of the emulsified bitumen, or settlement of suspended matter that cannot be overcome by moderate agitation.

6. Resistance to Freezing

6.1 *Procedure*—Expose a representative specimen of the emulsion to a temperature of -18°C (0°F) for 24 h, then warm the specimen in an environment not exceeding 100°F and stir thoroughly.

6.2 *Report*—Make a notation if the specimen was unable to return to a homogeneous consistency when stirred.

7. Weight per Gallon

7.1 *Apparatus*:

7.1.1 *Weight-per-Gallon Cup*,¹¹ with lid, stainless steel, calibrated to contain 83.2 g of water at $25^{\circ} \pm 0.5^{\circ}\text{C}$ ($77^{\circ} \pm 1^{\circ}\text{F}$).

7.1.2 *Balance*, accurate to 0.01 g.

7.1.3 *Water Bath*, constant temperature, maintained at $25^{\circ} \pm 0.5^{\circ}\text{C}$ ($77^{\circ} \pm 1^{\circ}\text{F}$).

7.2 *Procedure*:

7.2.1 Stir the emulsion sample, and place in the 25°C (77°F) water bath for a minimum of 2 h until the sample temperature reaches $25^{\circ} \pm 0.5^{\circ}\text{C}$ ($77^{\circ} \pm 1^{\circ}\text{F}$).

7.2.2 Weigh the weight-per-gallon cup with lid to the nearest 0.01 g and record as tare weight. Condition cup and lid to $25^{\circ} \pm 0.5^{\circ}\text{C}$ ($77^{\circ} \pm 1^{\circ}\text{F}$).

7.2.3 Remove the emulsion sample from the bath, and stir until homogeneous. Avoid trapping air in the sample during stirring.

7.2.4 Carefully fill the weight-per-gallon cup with the emulsion, avoiding the entrapment of air. Jar or vibrate the cup until no further change in volume occurs.

7.2.5 Immediately place the lid on the weight-per-gallon cup and remove, with a clean rag or paper, the excess emulsion oozing through the orifice in the lid.

7.2.6 When the lid is placed on tightly, clean the weight-per-gallon cup carefully, weigh on the balance to the nearest 0.01 g, and record as weight of emulsion and tare.

7.3 *Calculations*:

7.3.1 Calculate the weight per gallon of the emulsion as follows:

$$D = (B - A)/10 \quad (1)$$

where:

A = tare weight of weight-per-gallon cup, g,

B = weight of emulsion and tare, g, and

D = weight per gallon of emulsion, lb/gal.

7.3.2 Calculate the specific gravity of the emulsion as follows:

$$SG = D/8.33 \quad (2)$$

where:

SG = specific gravity,

D = weight per gallon of emulsion, calculation from 7.3.1,

8.33 = weight per gallon of water at $25^{\circ} \pm 0.5^{\circ}\text{C}$ ($77^{\circ} \pm 1^{\circ}\text{F}$).

7.4 *Report*:

7.4.1 Report the weight per gallon of the emulsion in pounds per gallon to the nearest 0.1 lb at 25°C (77°F).

7.4.2 Report the specific gravity of the emulsion to the nearest hundredth at 25°C (77°F).

8. Residue by Evaporation

8.1 *Apparatus*:

8.1.1 *Metal Dish*, flat-bottom, having a diameter of 65 mm (2.5 in.) with walls 10 mm ($\frac{3}{8}$ in.) high.

8.1.2 *Oven*, forced draft, conforming to Specification E 145, Type II B.

8.1.3 *Balance*, capable of weighing 50 g to within ± 0.01 g.

8.2 *Procedure*—Weigh 10 ± 0.25 g in the tared metal dish to the nearest 0.01 g. Dry the dish and its contents in a forced draft oven at $105 \pm 2^{\circ}\text{C}$ ($221 \pm 4^{\circ}\text{F}$) until the residue shows a loss of not more than 0.05 g on successive hourly weighings (approx. 4 h), after cooling in a desiccator.

8.3 *Calculation*—Calculate the percent residue by evaporation, R_1 , from the mass of the dry residue and the mass of the original sample, as follows:

$$R_1 = (R/S) \times 100 \quad (3)$$

where:

R = mass of dry residue, g, and

S = mass of sample, g.

8.4 *Report*—Record the average of two determinations.¹²

9. Volatiles

9.1 *Procedure*—Determine by difference between residue by evaporation (Section 8) and 100 %.

9.2 *Calculation*—Percent volatiles = $100 - R_1$.

9.3 *Report*—Report as percent volatiles.

10. Ash Content

10.1 *Apparatus*:

10.1.1 *Porcelain Crucible*, 30 cm³ capacity, or equivalent.

10.1.2 *Balance*, capable of weighing 50 g to within ± 0.01 g.

¹¹ The sole source of supply of the apparatus known to the committee at this time is Paul N. Gardner Company, Inc., 316 NE First Street, Pompano Beach, FL 33060. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹² In the case of coal tar emulsion: The Specification D 490 tar used to manufacture this emulsion contains some "light ends." In running the residue by evaporation test some of these light ends come off as if they are water.

10.1.3 *Muffle Furnace*, capable of maintaining a temperature of $1100^{\circ}\text{F} \pm 10^{\circ}\text{F}$.

10.2 *Procedure*—Thoroughly mix the dry residue from the determination of residue by evaporation (store the residue in a desiccator at all times prior to this test) (Section 8) and weigh 3 ± 0.5 g to the nearest 0.01 g in a previously ignited and tared crucible. Incinerate the contents inside a muffle furnace at a temperature of 600°C (1110°F) to constant weight.¹³

10.3 *Calculation*—Calculate the ash thus obtained, A_r , as percent of the residue by evaporation as follows:

$$A_r = (A/S) \times 100 \quad (4)$$

where:

A = mass of ash after ignition, g, and

S = mass of sample, g.¹³

10.4 *Report*—Record the ash content A_r .

11. Water Content

11.1 *Procedure*—Determine water content in accordance with Test Method D 95.

11.2 *Report*—Report as mass percent of the emulsion.

12. Flash Point

12.1 *Procedure*—Prepare and test the sample in accordance with Test Methods D 93.

12.2 *Report*—Record and report the flash point if at or below 140°F .

13. Drying Time

13.1 *Apparatus*:

13.1.1 *Metal Panels*—Metal panels 150-mm (6-in.) square, 0.30 to 0.40 mm (28 to 30 gage) thick.

13.1.2 *Brass Mask*¹⁴—150-mm (6-in.) square, and 1.6-mm ($\frac{1}{16}$ -in.) nominal thickness, with a 100-mm (4-in.) square opening in its center.

13.1.3 *Preparation of Metal Panels for Coating*—Prepare the metal panels for the application of the emulsion in accordance with Practice D 609. The panels shall be free of oil and rust or other corrosion and one face of each panel shall be lightly abraded with 00 steel wool or 00 garnet paper to a clean surface and wiped with a clean dry cloth.

13.2 *Preparation of Test Panel*—Thoroughly stir the sample of emulsion. Apply the brass mask to one metal panel so that the sides of the opening are approximately 25 mm (1 in.) from the edges of the panel, and spread the emulsion over the area within the mask opening. Doctor-off the excess level with a flat scraper so that the test film, when prepared, is the same thickness as the mask.

13.3 *Procedure*—Expose two panels prepared in accordance with 13.1.1 through 13.2 in a horizontal position at a temperature of $73.4 \pm 3.6^{\circ}\text{F}$ and approximately 50 % relative humidity. After 24 h (in the case of coal tar emulsion test, after 8 h), test the condition of the surface of the emulsion coat by lightly rubbing with the finger. Consider the emulsion to have

reached a firm set when a light rubbing of the finger does not break, roll, or displace the surface of the coating.

13.4 *Report*—Record whether or not firm set has been attained.

14. Resistance to Heat

14.1 *Apparatus*—A forced draft oven with internal dimensions not less than 300 by 300 by 300 mm (12 by 12 by 12 in.) and capable of maintaining a uniform temperature of $100 \pm 3^{\circ}\text{C}$ ($212 \pm 5^{\circ}\text{F}$).

14.2 *Preparation of Test Panel*—Prepare one panel in accordance with 13.1.2 through 13.2. Allow the test panels to dry for 48 h in a horizontal position at $73.4^{\circ} \pm 3.6^{\circ}\text{F}$.

14.3 *Procedure*—Scratch light reference lines 25 mm (1.0 in.) apart, parallel to the original reference line across the test film, and continue them to the edges of the test panel. Suspend the test panel vertically in the oven with the reference lines horizontal, and maintain at a temperature of $100 \pm 3^{\circ}\text{C}$ ($212 \pm 9^{\circ}\text{F}$) for 2 h. At the end of the test period, examine the coating for blistering, sagging, and slipping.

14.3.1 When coal tar emulsions are tested according to this procedure, maintain the oven at a temperature of $80 \pm 3^{\circ}\text{C}$ ($176 \pm 5^{\circ}\text{F}$).

14.4 *Report*—Record any sagging of the lines within the test film or slipping of the film beyond the lower reference line.

15. Resistance to Water

15.1 *Method A*:

15.1.1 *Apparatus*—Use an oven as described in 8.1.2 and other equipment described in 13.1 and 13.1.3.

15.1.2 *Preparation of Test Panel*—Prepare in accordance with 13.2.

15.1.3 *Procedure*—Dry the coated panels for 24 h in a horizontal position in a forced draft circulation oven at a temperature of $60 \pm 3^{\circ}\text{C}$ ($140 \pm 5^{\circ}\text{F}$). After 24 h completely immerse the test panels in distilled water in a suitably sized glass container at $24 \pm 3^{\circ}\text{C}$ ($75 \pm 5^{\circ}\text{F}$) for 24 h.

15.1.4 *Report*—After taking the test panels out of the water, examine them for development of blistering and reemulsification as inferred from the presence of dispersed bitumen particles in the water. Record the extent of blistering or reemulsification.

15.2 *Method B - For Coal Tar Emulsions*:

15.2.1 *Apparatus*—Prepare apparatus as described in 25.1 through 25.3.3.

15.2.2 *Procedure*—After completion of curing, apply a water resistant cement to the bottom of the metal ring and press onto the coating surface. Apply more of the cement if necessary to the ring/coating joint to prevent leakage. After the cement has cured properly, fill the ring with distilled water undisturbed for 24 h maintained at a temperature of $27 \pm 5^{\circ}\text{C}$ ($80 \pm 10^{\circ}\text{F}$). At the end of the 24 h period, examine the submerged film. Determine adhesion of bond by making intersecting cuts with a knife or needle and lifting the cut film at the point of intersection. Determine reemulsification if the water becomes darkened by rubbing the submerged of the uncut film lightly with a rubber policeman.

15.2.3 *Report*—Assess and report according to the following rating guide:

¹³ This incineration will produce black smoke. This procedure should be carried out under a fume hood.

¹⁴ Other materials such as plexiglass have also been used successfully for these tests.

Rating Number	Description
1	No softening, loss of adhesion, or reemulsification.
2	Slight softening, no loss of adhesion or reemulsification
3	Evidence of softening and loss of adhesion. No reemulsification.
4	Evidence of softening, loss of adhesion and reemulsification.

Report the rating number that describes the condition of the film and/or the description of the film.

16. Flexibility

16.1 *Apparatus*—The apparatus shall consist of 150 by 150 mm (6 by 6 in.) aluminum panel. The panel shall be 0.010 to 0.025 in. thick, clean and free of any deleterious matter. Provide a brass mask 150 by 150 mm (6 by 6 in.), 1.6 mm (1/16 in.) thick, with a 100 by 100-mm (4 by 4-in.) square opening in the center. The thickness of this mask is equivalent to the application of approximately 1.6 L/m² (4 gal/100 ft²) of emulsion.

16.2 *Preparation of Film*—Prepare two test panels as follows: Clamp the brass mask on the metal or aluminum panel and proceed as outlined in 13.2. Place the panels and apply the emulsion in a horizontal position. After allowing the film to set at standard conditions for 2 h, remove the mask.

16.3 *Procedure*—Dry the two test panels for 48 h in a horizontal position at 73.4° ± 3.6°F and approximately 50 % relative humidity. Then dry the panels suspended vertically in an oven at 60 ± 5°C (140 ± 10°F) for 5 h. After drying remove the panels from the oven and cool at room temperature for 1 h.

16.3.1 For most emulsions, immerse the panels in water at a temperature of 0 ± 0.5°C (32 ± 1°F) for 1 h. At the completion of this period, take the panels from the water and immediately bend each panel smoothly and snugly around a 51 mm (2 in.) diameter mandrel that has been preconditioned to 32 ± 1°F.

16.3.2 For coal tar emulsions, immerse the panels in water at a temperature of 23 ± 1.8°C (73.4 ± 3.6°F) for 1 h. At the completion of this period, take the panels from the water and immediately bend each panel smoothly and snugly around a 12.7 mm (1/2 in.) diameter mandrel that has been preconditioned to 23 ± 1.8°C (73.4 ± 3.6°F).

NOTE 1—This bending must take place at a uniform speed through an arc of 180° in approximately 2 s. Make the bend with the uncoated surface of the panel next to the mandrel.

16.4 Report:

16.4.1 Immediately after bending examine the coating for cracking. Use the following crack rating table for assessment of the cracking condition.

Crack Rating No.	Description
1	Perfect, no cracks hairline or otherwise, no loss of adhesion.
2	Hairline cracks present, no loss of adhesion
3	Slight cracking present, no loss of adhesion, hairline cracks may or not be present.
4	Moderate cracking and/or loss of adhesion. Slight cracking, hairline cracking, may or may not be present.
5	Severe cracking typically with loss of adhesion. Other forms of cracking may be present.

16.4.2 Report the crack rating number and optionally the corresponding description.

17. Brush-Application Behavior

17.1 Apparatus:

17.1.1 *Black Iron Panel*, cold-rolled, 1.6 mm thick (16 gage), free of rust and oil, 300 by 300 mm (12 by 12 in.).

17.2 *Procedure*—The temperature of the materials used and the surroundings shall be 73.4° ± 3.6°F. Mount the metal panel in a vertical position, and with a 75-mm (3-in.) paint brush predampened with material to be tested, apply a reasonably uniform layer of the emulsion to one side of the plate at the rate of 1.0 L/m² (0.26 gal/ft²) ± 10 %. Check the quantity applied by weighing the brush and container during application.

17.3 *Report*—Record the location and extent of sag or slip of the emulsion on the plate and any runoff during drying.

18. Spray-Application Behavior

18.1 Apparatus:

18.1.1 *Black Iron Panel*, as specified in 17.1.1.

18.2 *Procedure*—The temperature of the materials used and the surroundings shall be 73.4° ± 3.6°F. Mount the metal panel at an incline of 50 % (6 in./ft). By means of any suitable spray equipment, spray a reasonably uniform coat of the emulsion on one side of the plate at the rate of 1.0 L/m² (0.26 gal/ft²) ± 10 %. Check this coverage by weighing the plate immediately before and after application of the emulsion.

18.3 *Report*—Record the location and extent of sag or slip of the emulsion coat on the plate, and any runoff from the plate during drying.

19. Wet Flow

19.1 *Preparation of Metal Panels*—Prepare the metal panels as outlined in 13.1 through 13.2.

19.2 *Procedure*—Immediately after application of the film, as outlined in 13.2, carefully remove the mask and place the test panel in a vertical position with a reference line drawn horizontally at the lower edge of the test film. Expose the test panel in a room at a temperature of 73.4° ± 3.6°F and 50 ± 2 % relative humidity.

19.3 *Report*—At the end of 30 min, measure the amount of flow beyond the reference line.

20. Direct Flame Test

20.1 *Test Procedure*—Use the coated side of the panels from Section 14 when at room temperature. Mount the panels in a vertical position and subject them to the blue flame of a bunsen burner applied at the center of the coated area for exactly 10 s. Remove the flame and after 10 s record any continued combustion of the coating.

20.2 *Report*—Again apply the flame of the bunsen burner, this time continuously to the center of the coated area, and make observations for slipping, run-down, or loss or charred material from the test panel during the direct-flame test. Do not record slight bleeding of oily material at the area contacted by the flame as run-down.

21. Solubility of Residue in Trichloroethylene

21.1 *Procedure*—Determine the matter soluble in trichloroethylene on a representative portion of the residue by evaporation (Section 8), in accordance with Test Method D 4.

21.2 *Report*—Percent by weight of the residue that was

solubility in the trichloroethylene.

22. Wet Film Continuity

22.1 *Procedure*—Using a spatula, spread the wet emulsion to a thin film on a sheet of standard 66.7-g/m² (18-lb) copy machine paper. As the emulsion is drawn out to a smear, it shall show a uniformly homogeneous consistency.

22.2 *Report*—Make a notation of any inconsistency.

23. Resistance to Volatilization

23.1 *Procedure*—Weigh a representative specimen from the residue by evaporation determination (Section 8) and then expose it for 30 min in a 270 ± 2°C (518 ± 4°F) gravity convection oven. Weigh the specimen again after cooling in a desiccator and calculate the percent weight loss.

23.2 *Calculation*:

$$V_r = (R_v / R_d) \times 100 \quad (5)$$

where:

V_r = % loss in weight,

R_v = mass of dry residue in g after volatilization, and

R_d = mass of dry residue in g obtained from Section 8.

23.3 *Report*—Percent loss in weight.

24. Solubility of Residue in Carbon Disulfide

24.1 *Procedure*—Determine the matter soluble in carbon disulfide on a representative portion of the residue by evaporation (Section 8), in accordance with Test Method D 4.

24.2 *Report*—Percent by weight of the residue that was solubility in the carbon disulfide.

25. Resistance to Kerosine

25.1 *Apparatus*:

25.1.1 *Tiles*—Unglazed ceramic tiles, white, nonvitreous, dust-pressed body with an absorption range of 10 to 18 % (determined in accordance with Test Methods C 67) approximately 150 by 150 mm (6 by 6 in.) by 9.5 to 13 mm (3/8 to 1/2 in.) in thickness, or of sufficient size to accommodate the opening of the mask.

25.1.2 *Brass Masks*—150-mm (6-in.) square, and 1.6 mm (1/16 in.) in thickness for the first mask (first coat), and 3.2 mm (1/8 in.) in thickness for the second mask (second coat) with a 100-mm (4-in.) square opening in its center.

25.1.3 *Metal Ring*—Approximately 50 mm (2 in.) in diameter and 5 mm (1 in.) in height.

25.2 *Preparation of Tiles for Coating*—The tiles shall be free of oil and other foreign matter. Immerse the tiles in distilled water for at least 10 min and remove the excess water immediately before application of the emulsion by wiping with a clean dry cloth.

25.3 *Preparation of Film*:

25.3.1 Thoroughly stir the sample of emulsion. Apply the 1.6 mm (1/16 in.) brass mask to one tile so that the sides of the opening are approximately 25 mm (1 in.) from the edges of the tile, and spread the emulsion over the area within the mask opening. Doctor-off the excess with a flat scraper so that the test film when prepared is the same thickness as the mask.

25.3.2 Apply the material in two coats using the 3.2 mm (1/8 in.) brass mask for the second coat. Leave the mask for the first

coat in place during the drying period, but remove it before the second mask is positioned for the final coating application.

25.3.3 Cure each coat for 96 h at 73.4° ± 3.6°F and 50 ± 10 % relative humidity.

25.4 *Procedure*—After completion of curing, place the metal ring on the tile. Apply a solvent resistant cement¹⁵ to the ring/emulsion joint to prevent kerosine leakage. After the cement has cured properly fill the ring with kerosine. The kerosine shall comply with Specification D 3699. Expose the coating to kerosine for 24 h before evaluation. Remove the kerosine from the metal ring, blot dry, and immediately examine the film for softness and loss of adhesion. Immediately after this examination, break the tile in half exposing that part of the tile whose film was subject to the kerosine.

25.5 *Report*—Record any evidence of leakage of kerosine, loss of adhesion, or discoloration of the tile.

26. Resistance to Impact

26.1 *Preparation of Test Panel*—Prepare the specimens used in this test using the same procedure as outlined in 13.1.1 through 13.1.3, except coat one metal plate that is 150 mm by 155 mm by 3 mm (6 by 6 by 1/8 in.) in size. Apply the emulsion with a doctor blade using the brass mask described in 25.1.2 (one coat only) at a thickness of 1.16 mm (1/16 in.) to the prepared surface, and allow to dry as described in 25.3.2.

26.2 *Procedure*—Place each specimen, coating side up, on a solid horizontal base, and subject it to the impact of a 0.9-kg (2-lb) steel ball dropped from a height of 2.4 m (8 ft). Conduct the test at 73.4° ± 3.6°F.

26.3 *Report*—Examine the coating immediately for evidence of chipping, cracking, or loss of adhesion to the metal, and record same.

27. Resistance to Impact After Accelerated Weathering

27.1 *Preparation of Test Panel*—Prepare a specimen used in this test method in accordance with Section 26, then subject it to 1000 h accelerated weathering in a fluorescent UV/condensation device in accordance with Test Method D 4799, cycle B, using UVA-340 lamps, or 1000 h of xenon arc exposure in accordance to Test Method D 4798, Cycle B with an irradiance level of 0.35 W/m²/nm band at 340 nm and water spray temperature shall be 21 ± 3°C (70 ± 5°F).¹⁶

27.2 *Procedure*—At the completion of the accelerated weathering period, subject the specimen to the impact test as described in 26.2.

27.3 *Report*—Examine the coating immediately for evidence of chipping, cracking, or loss of adhesion to the metal, and record same.

28. Aggregate (Sand) Content in Bituminous Emulsions

28.1 *Apparatus*:

28.1.1 Clean No. 20 and No. 80 mesh sieves. The 3 in. diameter sieves are normally used.

¹⁵ Permatex High Temperature Form-A-Gasket #1372 type material has been found suitable.

¹⁶ Test Methods D 4798 and D 4799 are two different test methods and may result in two different results. A special task group is currently studying accelerated weathering concerns as they relate to bituminous materials. As their findings become available they will be incorporated into this test method.

28.1.2 *Balance*, analytical capable of weighing to 0.01 g with a minimum capacity of 200 g.

28.1.3 *Beaker*, 600 mL, clean, dry, and tared.

28.1.4 *Crucibles*, of suitable type and capacity to receive the sieved sand.

28.1.5 *Mixer*, kitchen type, low speed.

28.1.6 *Oven*, forced draft type, set at 155°C.

28.1.7 Muffle furnace of size and capacity to maintain 800°C.

28.1.8 *Vacuum Filtration Assembly: Buckner funnel*, large side arm flask, 1/8 in. rubber gasket insert for funnel, and vacuum pump. The gasket is sized to provide an airtight seal of the bottom rim of the sieve to the surface of the Buckner funnel.

28.2 *Reagent*—Sodium tripolyphosphate, purified.

28.3 *Sample Preparation*—Prior to start of testing, mix each sample for a minimum of 30 s or until homogenous with mixer. After mixing, start testing the sample immediately. Delay in testing will cause inconsistent results due to settling of sand additive.

28.4 *Test Procedure*:

28.4.1 Immediately after mixing sample, pour out 100 g into suitable sized tared beaker 600 mL size or larger. Record as wet weight to the nearest 0.1 g.

28.4.2 Add about 200 g of water and 1 ± 0.2 g of sodium tripolyphosphate. Pour through the No. 20 sieve into a beaker.

28.4.3 Place the No. 80 sieve into the Buckner funnel on top of the rubber insert. Slowly, without stirring, pour the contents of the No. 20 filtered beaker through the No. 80 sieve with the vacuum turned on. While pouring through the sieve, continuously rinse the residue with a wash bottle. Continually dilute the No. 20 filter material with water so that only thin watered down material is poured through the No. 80 sieve. Rinse the retained material.

28.4.4 Swirl and rinse the sieve residue through the sieve under vacuum, there should be very little emulsion retained on the No. 80 sieve when finished.

28.4.5 Transfer the contents of the two sieves into a previously tared 600 mL beaker. Use as much water as necessary to transfer all of the contents out of the sieves. Excess water can be decanted carefully out of the beaker through the No. 80 sieve with vacuum until about 150 mL of liquid remain. If any sand is accidentally lost then start over.

28.4.6 Place beaker with sand and water in forced draft oven to dry to constant weight. Record as dry weight to the nearest 0.1 g.

28.4.7 Transfer contents to a tared crucible and record as before ash weight to nearest 0.01g.

28.4.8 Place crucible and contents in muffle furnace and ash out for 1 h at 800°C.

28.4.9 At end of 1 h, allow contents to cool and weigh. Record as ashed weight to nearest 0.01 g.

28.5 *Calculations*:

28.5.1 Calculate percent aggregate in emulsion as follows:

$$\text{Percent Aggregate} = (B \times (D \div C) \div A) \times 100 \quad (6)$$

where:

A = wet weight,

B = dry weight,

C = before ash weight, and

D = after ash weight.

28.6 *Report*—Percent aggregate in emulsion by weight.

28.7 *Precision and Bias*:

28.7.1 *Precision*—The research necessary to report precision has not been conducted.

28.7.2 *Bias*—The research necessary to report bias has not been conducted.

28.8 *Keywords*:

28.8.1 aggregate content, bituminous emulsion, sand

29. Precision and Bias¹⁷

29.1 Criteria for judging the acceptability of test results obtained by the weight-per-gallon, residue-by-evaporation, and ash content test methods are provided in Table 1. The figures in

TABLE 1 Precision Criteria

Material Test	Standard Deviation, %	Acceptable Range of Two Results, %
Single Operator Precision:		
weight per gallon	0.23	0.7
residue by evaporation	0.58	1.6
ash content	1.34	3.8
Multilaboratory Precision:		
weight per gallon	0.55	1.5
residue by evaporation	0.94	2.7
ash content	2.08	5.9

column two of Table 1 are standard deviations or coefficients of variation that have been found to be appropriate for the tests described in column one. The figures given in column three are the limits that should not be exceeded by the difference between the results of two properly conducted tests. Single-operator precision is to be evaluated based on results of two properly conducted tests by the same operator on samples of the same material. Multilaboratory precision is to be evaluated based on two properly conducted tests from two different laboratories on samples of the same material.

29.2 During the interlaboratory study to determine precision of the weight per gallon, residue by evaporation, and ash content tests, no other test methods were evaluated. Therefore, no statement about bias with reference to these three tests can be made.


29.3 No statement is made about either the precision or bias of test methods in Sections 5, 6, 12, 14, 15, 16, 17, 18, 20, 22, 25, 26, and 27, since the result merely states whether there is conformance to the criteria for success specified in these procedures.

29.4 With reference to Sections 9, 11, 13, 19, 21, 23, and 24, precision and bias statements are currently being developed.

30. Keywords

30.1 asphalt; bitumen; coal tar; coating; emulsified

¹⁷ For definition of terms and significance of the parameters, reference should be made to Practice C 670.

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