Designation: D2939 – 03

Standard Test Methods for Emulsified Bitumens Used as Protective Coatings[1]

This standard is issued under the fixed designation D2939; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.
Test Methods D 2939 are the methods recommended for use by Committee D08 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, built-up roofs, and bituminous pavements.

1.2 The test methods appear in the following order:

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:*[3]

C67 Test Methods for Sampling and Testing Brick and Structural Clay Tile

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D4 Test Method for Bitumen Content

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

D140 Practice for Sampling Bituminous Materials

D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products

D3699 Specification for Kerosine

D4798 Practice for Accelerated Weathering Test Conditions and Procedures for Bituminous Materials (Xenon-Arc Method)

D4799 Practice for Accelerated Weathering Test Conditions and Procedures for Bituminous Materials (Fluorescent UV, Water Spray, and Condensation Method)

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

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 D140.

[1] These test methods are under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and are the direct responsibility of Subcommittee D08.09 on Bituminous Emulsions.

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

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4.2 Open the original containers and examine them for uniformity of contents. Record the degree of separation, if any, into portions of appreciably different consistency, such as thick or thin layers, sedimentation or coagulation, and so forth. 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,*[4] with lid, stainless steel, calibrated to contain 83.2 g of water at 25 ± 0.5°C (77 ± 1°F).

7.1.2 *Balance*, accurate to 0.01 g.

7.1.3 *Water Bath*, constant temperature, maintained at 25 ± 0.5°C (77 ± 1°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 ± 0.5°C (77 ± 1°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 ± 0.5°C (77 ± 1°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 ± 0.5°C (77 ± 1°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 (⅝ in.) high.

8.1.2 *Oven*, forced draft, conforming to Specification E145, 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 ± 2°C (221 ± 4°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.[5]

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 cm^3 capacity, or equivalent.

[4] 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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] In the case of coal tar emulsion: The Specification D490 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.

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

10.1.3 *Muffle Furnace*, capable of maintaining a temperature of 1100 ± 10°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.[6]

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.

10.4 *Report*—Record the ash content A_r.

11. Water Content

11.1 *Procedure*—Determine water content in accordance with Test Method D95.

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 D93.

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*[7]—150-mm (6-in.) square, and 1.6-mm (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 D609. 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 ± 3.6°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 ± 3°C (212 ± 5°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 ± 3.6°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 ± 3°C (212 ± 9°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 ± 3°C (176 ± 5°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 ± 3°C (140 ± 5°F). After 24 h completely immerse the test panels in distilled water in a suitably sized glass container at 24 ± 3°C (75 ± 5°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 ± 5°C (80 ± 10°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.

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

[7] Other materials such as plexiglass have also been used successfully for these tests.