

# Standard Test Methods for Solvent Bearing Bituminous Compounds<sup>1</sup>

This standard is issued under the fixed designation D6511/D6511M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

 $\epsilon^1$  NOTE—Units information and other information was editorially revised in July 2011.

# 1. Scope

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1.1 These test methods cover procedures for sampling and testing solvent bearing bituminous compounds for use in roofing and waterproofing.

1.2 The test methods appear in the following order:

	Section
Sampling	4
Uniformity	5
Weight per gallon	6
Nonvolatile content	7
Solubility	8
Ash content	9
Water content	10
Consistency	11
Behavior at 60°C [140°F]	12
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Aluminum content	14
Reflectance of aluminum roof coatings	15
Strength of laps of rolled roofing adhered with roof adhesive	16
Adhesion to damp, wet, or underwater surfaces	17
Mineral stabilizers and bitumen	18
Mineral matter	19
Volatile organic content	20

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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  ASIM Siunuurus.	2.1	ASTM	Standards: <sup>2</sup>
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C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials D4 Test Method for Bitumen Content D88 Test Method for Saybolt Viscosity D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation **D140** Practice for Sampling Bituminous Materials D146 Test Methods for Sampling and Testing Bitumen-Saturated Felts and Woven Fabrics for Roofing and Waterproofing D224 Specification for Smooth-Surfaced Asphalt Roll Roofing (Organic Felt) (Withdrawn  $2002)^3$ D249 Specification for Asphalt Roll Roofing (Organic Felt) Surfaced with Mineral Granules (Withdrawn 2002)<sup>3</sup> D562 Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer D1475 Test Method For Density of Liquid Coatings, Inks, and Related Products D2369 Test Method for Volatile Content of Coatings D2824 Specification for Aluminum-Pigmented Asphalt Roof Coatings, Nonfibered, Asbestos Fibered, and Fibered without Asbestos D4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method E1 Specification for ASTM Liquid-in-Glass Thermometers E145 Specification for Gravity-Convection and Forced-Ventilation Ovens E200 Practice for Preparation, Standardization, and Storage <sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or

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<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and are the direct responsibility of Subcommittee D08.05 on Solvent-Bearing Bituminous Compounds for Roofing and Waterproofing.

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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.

 $<sup>^{3}\,\</sup>text{The}$  last approved version of this historical standard is referenced on www.astm.org.

# of Standard and Reagent Solutions for Chemical Analysis

#### 3. Significance and Use

3.1 These tests are useful in sampling and testing solvent bearing bituminous compounds to establish uniformity of shipments.

#### 4. Sampling

4.1 Determine the number of containers sampled to represent a shipment in accordance with Practice D140.

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, 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 72 h.

5.2 *Report*—Make a notation of any separation or settlement of suspended matter that cannot be overcome by moderate agitation.

#### 6. Weight-per-Gallon/Specific Gravity

6.1 Apparatus:

6.1.1 *Weight-per-Gallon Cup*, with lid, stainless steel, calibrated to contain 83.3 g of water at  $25 \pm 0.5$ °C [77  $\pm 1$ °F].

6.1.2 Balance, accurate to 0.01 g.

6.1.3 *Water Bath*, constant temperature, maintained at  $25 \pm 0.5^{\circ}$ C [77  $\pm 1^{\circ}$ F].

# 6.2 Procedure:

6.2.1 Stir the sample, and place in the 25°C [77°F] water bath until the sample temperature reaches  $25 \pm 0.5$ °C [77 ± 1°F]. Time required for temperature equilibration depends on sample size and configuration.

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

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

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

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

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

#### 6.3 Calculations:

6.3.1 Calculate the weight-per-gallon of the sample as follows:

- A = tare weight of weight-per-gallon cup, g,
- B = weight of sample and tare g, and
- D = weight-per-gallon of sample, lb/gal. To convert units to kg/m<sup>3</sup>, multiply D by 119.83.

D = (B - A)/10

6.3.2 Calculate the specific gravity of the sample as follows:

$$SG = D/8.33 \tag{2}$$

(1)

where:

SG = specific gravity,

D = weight-per-gallon of sample calculation from 6.3.1, and

8.33 = weight-per-gallon of water at 25  $\pm$  0.5° C [77  $\pm$  1°F].

6.4 Report:

6.4.1 Report the weight-per-gallon of the sample in poundsper-gallon to the nearest 0.1 lb at 25°C [77°F].

6.4.2 Report the specific gravity of the sample to the nearest hundredth at  $25^{\circ}$ C [77°F].

#### 7. Nonvolatile Content

7.1 Apparatus:

7.1.1 *Metal Dish*, flat-bottom, having a diameter of 65 mm [2.5 in.] with walls 10 mm [<sup>5</sup>/<sub>8</sub> in.] high.

7.1.2 *Oven*, forced draft, conforming to Specification E145, Type III B, for asphalt products, or a standard convection oven for coal tar products.

7.1.3 *Balance*, capable of weighing 50 g to within  $\pm 0.01$  g.

7.2 *Procedure*—Weigh  $10 \pm 1.00$  g in the tared metal dish to the nearest 0.01 g. Dry the dish and its contents in a forced draft oven at  $163 \pm 3^{\circ}$ C [ $325 \pm 5^{\circ}$ F] for asphalt products or 105 to  $110^{\circ}$ C [221 to  $230^{\circ}$ F] in standard convection oven for coal tar products until the residue shows a loss of not more than 0.05 g on successive hourly weighings (approximately 4 h), after cooling in a desiccator.

7.3 *Calculation*—Calculate the percent nonvolatile content  $R_{_{I}}$ , from the mass of the dry residue and the mass of the original sample, as follows:

$$R_I = (R/S) \times 100 \tag{3}$$

where:

R = mass of dry residue, g, and

S = mass of sample, g.

7.4 *Report*—Record the average of two determinations.

# 8. Solubility of Residue in Carbon Disulfide or Trichloroethylene

8.1 Apparatus—See Test Method D4.

8.2 *Procedure*—Determine the matter soluble in carbon disulfide or trichloroethylene on a representative portion of the nonvolatiles (Section 7), in accordance with Test Method D4.

8.3 *Calculation*—Calculate the percent solubility in carbon disulfide or trichloroethylene  $S_I$  from the mass of the residue and the mass of the original sample as follows:

$$S_I = (R/S) \times 100 \tag{4}$$

where:

R = mass of insoluble residue, g, and

S = mass of sample, g.

8.4 *Report*—Percent by weight of the residue that was soluble in the carbon disulfide or trichloroethylene.

# 9. Ash Content

9.1 Apparatus:

9.1.1 Porcelain Crucible, 30-cm<sup>3</sup> capacity, or equivalent.

9.1.2 *Balance*, capable of weighing 50 g to within  $\pm 0.01$  g. 9.1.3 *Muffle Furnace*, capable of maintaining a temperature of 593  $\pm 2.5^{\circ}$ C [1100  $\pm 10^{\circ}$ F].

9.2 *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 7) and weigh  $3 \pm 0.5$  go 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. (Warning—This incineration will produce black smoke. This procedure should be carried out under a fume hood.)

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

$$A_r = (A/S) \times 100 \tag{5}$$

9.4 *Report*—Ash as a percentage of the residue by evaporation.

#### 10. Water Content

10.1 Apparatus—See Test Method D95.

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

10.3 *Calculation*—Calculate the water in the sample, as mass percent, as follows:

Water, 
$$\% = \frac{V(0.99707)}{W \times 100}$$
 (6)

where:

V = volume of water in trap (ml), and

W = mass of sample (gm).

Volatile water-soluble material, if present, may be measured as water.

10.4 Report-Report as mass percent of the sample.

#### 11. Consistency

11.1 Asphalt Roof Coatings:

11.1.1 Summary of Test Method—Consistency is determined using the stormer viscometer and the rate of shear reported in terms of the time required for 100 revolutions of the rotor produced by a specified load (compare with Test Method D562).

11.1.2 Apparatus:

11.1.2.1 Standard Stormer Viscometer.

11.1.2.2 *Water Bath-Test Cup Assembly*, without central baffle or thermometer holder.

11.1.2.3 *Rotor*, propeller-type (see Fig. 1).

11.1.2.4 Slotted Weights, and a suitable hanger.

11.1.2.5 *Thermometer*—ASTM stormer viscosity thermometer having a range from 20 to 70°C, and conforming to the requirements for Thermometer 49C as prescribed in Specification E1.

11.1.2.6 Stop Watch.

11.1.3 Preparation of Apparatus:

11.1.3.1 Place the stormer viscometer on a table or shelf high enough to permit the weight to drop about 1 m [40 in.], or sufficient distance to produce about 125 revolutions of the rotor. Insert the shaft of the propellar-type rotor in the chuck as far as it will go and secure it with the set screw.

11.1.3.2 Raise the water bath-test cup assembly until the bottom of the test cup just touches the bottom of the rotor blade; then, lower the assembly 6 mm [ $\frac{1}{4}$  in.]. Tighten the set screw on the position collar, if available, while the collar is in contact with the bracket and the assembly, and use this setting to position the cup for all test runs. Using the set screws in the rim of the bath holder, adjust the water bath-test cup assembly so that the cup and rotor are visually concentric.

11.1.3.3 For convenience in weight adjustment, attach a slotted metal can cover approximately 50 mm [2 in.] in diameter to the hanger to support the slotted weights required. 11.1.4 *Calibration:* 

11.1.4.1 Use a 94.85 % by weight aqueous glycerin solution a calibration standard. Determine the initial concentration of

as a calibration standard. Determine the initial concentration of reagent grade glycerin by accurately measuring its specific gravity (see Appendix X1), and then add additional water to make up the desired total of 5.15 % by weight water.

11.1.4.2 Determine the weight on the viscometer cord (approximately 100 g) required to produce 100 revolutions of the rotor in the glycerin calibration standard at  $25 \pm 0.2^{\circ}$ C in 18.0  $\pm$  0.2 s, this is the standardizing load. Check the calibration periodically, particularly after prolonged use of the instrument, and recalibrate as necessary.

11.1.5 Procedure:

11.1.5.1 Remove the test cup and fill it with the sample to a level that will extend 6 mm [ $\frac{1}{4}$  in.] above the top of the rotor blade. Agitate the sample in the cup thoroughly to remove any trapped air bubbles. Place the test cup in the water bath, and then move the assembly up into test position against the preset collar.

11.1.5.2 Add 450 g to the standardizing load determined in 11.1.4.2. This will be the test load or driving weight.

11.1.5.3 Adjust the temperature of the sample and the entire test assembly to  $25 \pm 0.2^{\circ}$ C. To expedite reaching equilibrium, raise the driving weight and release the brake to provide agitation. When the water bath test cup, sample, and rotor have all reached  $25 \pm 0.2^{\circ}$ C, remove the thermometer.

11.1.5.4 Raise the driving weight on the cord so that it nearly touches the pulley. With stop watch in hand, release the brake and observe the moving pointer. After 8 to 10 revolutions have been made, time the next 100 revolutions. Take the average of at least three determinations and record as the Stormer consistency in s/100 revolutions. Record the driving weight.

11.1.5.5 Consistencies of up to approximately 100 s/100 revolutions shall be determined directly by timing 100 revolutions. For higher consistencies, when timing 100 revolutions

℣ D6511/D6511M – 06 (2011)<sup>ε1</sup> 14.3 mm (<u>9</u> in.) 4.8 mm ( $\frac{3}{16}$  in.) Diameter Flattened 0.8 mm  $(\frac{1}{32}$  in.) 1.6 mm 12.7 mm ( $\frac{1}{2}$  in.) Diameter  $(\frac{1}{16} \text{ in.})$ 63.0 mm 6.44 mm ( $\frac{1}{4}$  in.) Diameter  $(2\frac{1}{2}$  in.) 114.3 mm 0.8 mm  $(4\frac{1}{2} in.)$  $(\frac{1}{32} in.)$ 7.9 mm ( $\frac{5}{16}$  in.) Diameter 34.9 mm  $(1 \frac{3}{8} in.)$ – 3.2 mm (<u>1</u> in.) Helix, 139.7 mm (5 $\frac{1}{2}$  in.) Pitch 25.4 mm (1 in.)

Note 1—All dimensions are subject to a tolerance of  $\pm 0.1$  mm [ $\pm 0.004$  in.]. Material: nickel-plated brass or stainless steel. FIG. 1 Propeller-Type Rotor for Use with Stormer Viscometer

might unduly prolong the test, calculate the time required for 100 revolutions from direct measurement of the time required for no less than 25 revolutions.

11.1.6 *Report*—Time required for 100 revolutions using 450-g test load (driving weight) in addition to the standardizing load.

#### 11.2 Aluminum-Pigmented Asphalt Roof Coatings:

11.2.1 *Apparatus*—Use the apparatus and equipment specified in Test Method D562, Procedure A.

11.2.2 Procedure:

11.2.2.1 Testing D2824 Type I Coating—Thoroughly mix the sample and fill a 500-ml [1-pt] container to within 20 mm [ $^{3}/_{4}$  in.] of the top. Stir the coating carefully to remove any trapped air bubbles. Maintain the temperature of the sample and the test assembly at 25 ± 0.2 °C [77 ± 0.3 °F], and start the test within 1 h after filling the container. Place the container on the platform of the viscometer so that the paddle-type rotor is immersed just to the mark on its shaft, and turn the rotor through approximately 100 revolutions in 25 to 30 s before starting the test. Then determine the time required for 100 revolutions with a 100-g load in addition to the standardizing load from a running start, that is, permit the rotor to make at least 10 revolutions before timing the next 100.

11.2.2.2 *Testing* **D2824** *Type II or Type III Coating*—Follow the procedure in 11.2.2.1, except use the propeller-type rotor (see Fig. 1) with a 300-g load in addition to the standardizing load.

11.2.3 *Report*—Time required for 100 revolutions using 300-g test load (driving weight) in addition to the standardizing load.

11.3 Viscosity of Asphalt Primer:

11.3.1 Saybolt Furol Viscosity—Test Method D88.

#### **12.** Behavior at 60°C [140°F]

# 12.1 Preparation and Apparatus:

12.1.1 For solvent-based roof cement prepare two test specimens by troweling cement through masks centered over two steel panels. The masks shall have openings 75 by 125 mm [3 by 5 in.] and shall be 2.4 mm [ $\frac{3}{32}$  in.]  $\pm$  10 % thick. The panels shall be 150 by 150 mm [6 by 6 in.]; 30 to 28 gage [0.32 to 0.40 mm] nominal thickness; capable of being bent

smoothly and uniformly through  $180^{\circ}$  over a 25 mm [1 in.] diameter mandrel; and shall be free of oil and scale.

12.1.2 For coating, prepare two test specimens with each base by brushing roof coating on two 150 by 150 mm [6 by 6 in.] steel panels and two pieces of smooth surface asphalt roll roofing, each covered by a 0.8 mm [ $\frac{1}{32}$  in.]  $\pm$  10 % thick mask with a 75 by 125-mm [3 by 5-in.] opening in the center. The steel panels shall conform to requirements of 12.1.1. The smooth surface asphalt roofing roll shall conform to the requirements of Specification D224.

#### 12.2 Procedure:

12.2.1 Immediately after troweling the cement or brushing the coating on the test panels, remove the mask and embed a thread in each coating across the 3-in. [75-mm] dimension, parallel to and no more than 50 mm [2 in.] from one edge of the coating. Measure the distance of the thread from the edge of each test panel to the nearest 0.8 mm [ $\frac{1}{32}$  in.].

12.2.2 Expose the test panels horizontally in a well ventilated area for 60 min at 23  $\pm$  2°C [73.4  $\pm$  3.6°F], but not in direct sunlight.

12.2.3 Suspend the panels vertically with the thread closest to the top edge in an oven at  $62 \pm 2^{\circ}C$  [140  $\pm 3.6^{\circ}F$ ]. After 5h, remove the test panels from the oven and examine for any sign of blistering. Then, measure to the nearest 0.8 mm [ $\frac{1}{32}$  in.] the distance of the thread from the edge of each panel again to determine the extent of any sagging or sliding. Average the two determinations from each type of panel. Report results to the nearest 0.8 mm [ $\frac{1}{32}$  in.].

12.2.4 Observe and estimate to the nearest 5 % the area stained on the reverse side of the prepared roofing panel due to the absorption of the roof coating.

## **13.** Pliability at 0°C [32°F]

13.1 Cool the coated metal panels from the preceding test to room temperature, and then immerse them in a water bath at  $0^{\circ}$ C [32°F] for 1 h along with a 25-mm [1-in.] diameter mandrel. Bend samples over the mandrel while still in water bath. The bending shall be accomplished in approximately 2 s at a uniform rate, with the metal side of the test panel against the mandrel.

13.2 Immediately after bending, maintain the panels in the bent position, dry the panels thoroughly and examine the coating visually for cracking or bond failure. Ignore cracks less than 3 mm [ $\frac{1}{8}$  in.] long unless they extend to the metal.

13.3 Report large cracks and bond failure.

#### 14. Metallic Aluminum

14.1 *Reagents*—Reagent-grade chemicals, or equivalent, prepared in conformance with Practice E200, shall be used in the determination of metallic aluminum.

14.1.1 Water-Use only distilled or deionized water.

14.1.2 *Ferric Sulfate Solution*—Dissolve 330 g of ferric sulfate  $[Fe_2(SO_4)_39H_2O]$  in 750 cm<sup>3</sup> of water and 75 cm<sup>3</sup> of concentrated sulfuric acid (sp gr 1.84). Heat the solution and let stand two days to dissolve the ferric sulfate completely. Then, add water to make up a total volume 1000 cm<sup>3</sup>.

14.1.3 Indicator (Diphenylamine Sulfonate Solution)— Dissolve 0.32 g of the barium salt of diphenylamine sulfonic

acid in 100 cm<sup>3</sup> of water. Then, add 0.5 g of sodium sulfate and filter off the precipitate of barium sulfate.

14.1.4 Phosphoric Acid—CP 85 % orthophosphoric acid.

14.1.5 *Potassium Dichromate Solution*—Prepare a 0.5 N solution by dissolving 24.52 g of potassium dichromate in water and making up to  $1000 \text{ cm}^3$ .

14.1.6 *Sodium Bicarbonate Solution*—Prepare a saturated solution of sodium bicarbonate (approximately 10 %).

#### 14.2 Filtering Apparatus:

14.2.1 *Gooch Crucible*, glazed inside and outside with the exception of the outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top tapering to 36 mm at the bottom and a depth of 28 mm.

14.2.2 Filter Pad, glass-fiber 32 mm in diameter.

14.2.3 *Filter Flask*, heavy-wall with side tube, 500-mL capacity.

14.2.4 *Rubber Tubing*, of adapter for holding Gooch crucible on the filter tube.

Note 1—A photograph of a typical filtering assembly is shown in Fig. 2. The filter pads, No. 934-AH, may be purchased from almost any scientific supply house.

#### 14.3 Procedure:

14.3.1 Place the Gooch crucible plus one thickness of the glass-fiber pad in an oven at about  $107^{\circ}C$  [225°F] for 15 min, and allow to cool in a desiccator.

14.3.2 Place the crucible in a  $100\text{-cm}^3$  beaker and tare weigh to the nearest 1 mg. Then, add about 1 g of the well-mixed sample to the crucible and reweigh as quickly as possible to the nearest 1 mg. Add 50 cm<sup>3</sup> of benzene to the crucible and beaker, filling the crucible about three-fourths full and pouring the remainder into the beaker around the crucible. Cover and let stand 15 min. Then, transfer the crucible to a suction flask and apply suction. Wash the crucible and mat twice with acetone and twice with water.

14.3.3 Transfer the crucible to a 500-cm<sup>3</sup> wide mouth Erlenmeyer flask. Add 50 cm<sup>3</sup> of ferric sulfate solution and stopper the flash immediately with a rubber stopper fitted with a 50-cm<sup>3</sup> separatory funnel and with an outlet tube. Immerse the end of the outlet tube in a flask of sodium bicarbonate solution. Quickly add 50 cm<sup>3</sup> of sodium bicarbonate solution to the Erlenmeyer flask through the separatory funnel, and shake the flask gently. This produces a CO<sub>2</sub> atmosphere, which prevents oxidation of the reduced ferric iron. Keep the separatory funnel stopcock closed and the outlet tube immersed in the sodium bicarbonate solution to retain the inert CO<sub>2</sub> atmosphere. Heat the solution in the Erlenmeyer flask gently until all of the aluminum is dissolved (about 15 min), and then bring the solution to a boil for 10 min. Cool to 10 to  $15^{\circ}C$  [50 to  $60^{\circ}F$ ] during the remainder of the procedure.

14.3.4 Add 15 cm<sup>3</sup> of phosphoric acid through the separatory funnel, followed by 50 cm<sup>3</sup> of water. Shake gently to mix thoroughly. Remove the stopper assembly, add 1 cm<sup>3</sup> of the indicator, and quickly titrate with 0.5 N potassium dichromate solution to a purple end point (see Note 2).

NOTE 2—Reduce the rate of addition of potassium dichromate near the titration end-point in order to be able to recognize the transition from green to intense purple. An excess beyond this point will produce an olive green color.

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FIG. 2 Filtration Apparatus

#### 14.3.5 Calculation:

Metallic aluminum,  $\% = (VN \times 0.009/S) \times 100$  (7)

where:

- V = volume of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution, cm<sup>3</sup>,
- $N = \text{normality of } K_2 Cr_2 O_7 \text{ solution, and}$
- S = sample weight, g.

14.3.6 Report weight percent metallic aluminum.

#### 15. Reflectance

15.1 Prepare two panels at least 100 by 125 mm [4 by 5 in.] by coating with a doctor blade, and allow to dry 48 h at room temperature. The wet film thickness of Type I coatings shall be 0.38 mm [0.015 in.] and Type II coatings shall be 0.51 mm [0.020 in.]. Any smooth, flat substrate, such as uncoated

aluminum or steel may be used for the test panels. Hiding power or opacity charts, suitably supported, also may be used.

15.2 Measure the percent luminous reflectance of each panel (the CIE<sup>4</sup> tristimulus Y value) for CIE Source C (daylight), using an instrument that integrates diffuse and specular reflectance by means of a hollow integrating sphere, calibrated according to the manufacturer's instructions using a Gardner Laboratory Gray-White Reflectance Standard with a CIE tristimulus Y value of 40. Take at least three readings on different areas of each panel and calculate the mean luminous reflectance for each panel. Report the luminous reflectance of the coating sample as the mean of the two panel values to the nearest 0.5 %.

<sup>&</sup>lt;sup>4</sup> Commission Internationale de l'Eclairage.

15.3 Age approximately 300 cm<sup>3</sup> of coating in a tightlyclosed 500 cm<sup>3</sup> [1 pt] container in an oven at  $49 \pm 2^{\circ}$ C [120  $\pm$  3.5°F] for three days. Remove from the oven, cool to room temperature, prepare two panels, and determine the luminous reflectance.

15.4 Report the mean of the two panel values as the luminous reflectance of the sample after high-temperature shelf aging.

# 16. Strength of Lap

# 16.1 Apparatus:

16.2 *Significance*—For those specifications requiring a lap strength test, the following is the procedure to be used.

#### 16.3 Test Specimen and Sample:

16.3.1 Use any roll roofing with a 100-mm [4-in.] wide selvage conforming to the requirements of Specification D249.

16.3.2 Four test specimens are required. For each specimen, cut a 25 by 305 mm [1 by 12 in.] transverse strip of roofing that includes the selvage; then, cut this strip in half so that one piece contains the selvage and the other is plain.

16.3.3 Spread 2.0 g of lap cement evenly over the selvage and set aside for 10 min at 20 to 30°C [68 to 86°F]; then, place the reverse side of the plain half over the cement-coated selvage in the same manner as a field lap would be made.

16.3.4 Place a 4.5-kg [10-lb] mass over the cemented lap. The mass shall be large enough to cover the 25 by 100-mm [1 by 4-in.] lap completely. After 2 min, remove the mass, and age four specimens for 24 h at 20 to  $30^{\circ}$ C [68 to  $86^{\circ}$ F].

16.4 *Procedure*—Test each specimen immediately after the specified aging period in accordance with Test Methods D146, Section 13, except set apart the edges of the clamps 127 mm [5.0 in.]  $\pm 2 \%$ , and use a driven clamp speed of 305 mm [12 in.]/min  $\pm 0.7 \%$ .

#### 17. Adhesion to Damp, Wet, or Underwater Surfaces

#### 17.1 Apparatus:

17.1.1 *Metal Lids*—Slip on covers, supplied with three-fluid oz capacity seamless metal containers, approximately 55 mm in diameter and with a raised rim 1 mm high and 3 mm wide.

17.1.2 *Water Bath*—A bath with a flat bottom surface having a capacity of at least 1 qt, which can be filled with tap water to a minimum depth of 51mm [2 in.] and maintained at  $20 \pm 3^{\circ}$ C [68  $\pm$  5°F].

17.2 Procedure:

17.2.1 Spread 5 g of the cement on the top surface of each of three metal lids and place them in the water bath.

17.2.2 Flame three other metal lids in an open gas flame until each entire lid has an oxidized blue-gray surface and place them in the water bath after they have cooled to room temperature.

17.2.3 After 5 min, place a flamed lid on top of each cement-covered lid, and press only on the rim of the flamed lid for 1 minute or until the flow of the cement stops.

Note 3—Covers that have raised rim [1 mm] on the top surface are intended for this test. When the two lids are pressed together, approximately 4 of the 5 g sample will be contained between the surfaces. If lids with raised rims are not available, smooth lids may be used except that

care should be exercised in pressing the lids together to prevent squeezing out all of the sample.

17.2.4 Remove each set of lids from the water bath and scrape off the excess cement around the edges with a spatula or knife.

17.2.5 Separate the lids without twisting and estimate the percentage of area covered by the cement on each of the flamed lids.

17.3 Report:

17.3.1 Report the average of the three estimates on coverage of the cement on the flamed lids as the adhesion, in percent at  $20^{\circ}$ C [68°F].

Note 4—There may be a need for testing at lower temperature Committee D08 would welcome data supporting suitable modification.

# 18. Mineral Stabilizers, and Bitumen

18.1 Test Method D4, Procedure No. 2—Mineral matter shall be reported as mineral stabilizers. Calculate bitumen as percent of original,  $X_a$ , as follows:

$$X_a = N - (100 B/A) \tag{8}$$

where:

N =nonvolatile matter from 7.4,

- B = net mass of insoluble residue, including corrections, and
- A = mass of the sample taken.

# **19.** Mineral Matter Based on Original Mass of Insoluble Residue

19.1 Using the data obtained in accordance with Test Method D4, Procedure No. 2, calculate the percent mineral matter based on the original mass of the insoluble residue,  $M_i$ , as follows:

$$M_i = (C/B) \times 100 \tag{9}$$

where:

- C = net mass of ignited, reconstituted mineral matter, including any corrections, and
- B = net mass of insoluble residue, including any corrections.

#### 20. Volatile Organic Content (VOC)

20.1 The accepted method is EPA Method 24A, which utilizes Specifications D2369, D4017, D1475 to calculate VOC.

# 21. Precision and Bias<sup>5</sup>

21.1 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 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

<sup>&</sup>lt;sup>5</sup> For definition of terms and significance of the parameters, reference should be made to Practice C670.

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TABLE 1 Precision Criteria				
Material Test	Standard Deviation, %	Acceptable Range of Two Results, %		
Single Operator Precision:				
weight-per-gallon	0.23	0.7		
nonvolatile content	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		

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. 21.2 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.

21.3 No statement is made about either the precision or bias of test methods in Sections 4, 5, 13, and 14, since the result merely states whether there is conformance to the criteria for success specified in these procedures.

21.4 With reference to Sections 9, 11, 12, 15, 16, 17, 18, and 19, precision and bias statements are currently being developed.

# 22. Keywords

22.1 aluminum; asphalt; bituminous; roof coating; solvent bearing

# APPENDIX

## (Nonmandatory Information)

## **X1. SPECIFIC GRAVITY OF AQUEOUS GLYCERIN SOLUTIONS**

X1.1 Aqueous solutions of glycerin are suitable liquids for use as standards in calibrating the Stormer viscometer. By means of an accurate specific gravity determination, the percent glycerin concentration can be determined with  $\pm 0.02$  (see Table X1.1).

Sp Gr, <sup>4</sup> 25/25C	Glycerin, weight %
1.23585	90.00
1.23850	91.00
1.24115	92.00
1.24380	93.00
1.24645	94.00
1.24910	95.00
1.25165	96.00
1.25425	97.00
1.25685	98.00
1.25945	99.00
1.26201	100.00

TABLE X1.1 Specific Gravity of Aqueous Glycerin Solutions

<sup>A</sup> Taken from Handbook of Chemistry and Physics, 30th Ed., Chemical Rubber Publishing Co., 19801 Cranwood Parkway, Cleveland, OH 44128, 1948, p. 1742. See also Sheeley, M.L., Industrial and Engineering Chemistry, IECHA, Vol 24, 1932, p. 1060.

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