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## 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Units information and other information was editorially revised in July 2011.

### 1. Scope

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:

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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 SI units are to be regarded as the standard. The values given in parentheses are for information only.  
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 ASTM Standards:<sup>2</sup>

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)

D249 Specification for Asphalt Roll Roofing (Organic Felt) Surfaced with Mineral Granules

<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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<sup>2</sup> 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.

- 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 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  $25 \pm 0.5^\circ\text{C}$  ( $77[77 \pm 1^\circ\text{F}]-1^\circ\text{F}$ ).

6.1.2 *Balance*, accurate to 0.01 g.

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

6.2 *Procedure:*

6.2.1 Stir the sample, and place in the  $25^\circ\text{C}$  ( $77^\circ\text{F}$ )[ $77^\circ\text{F}$ ] water bath until the sample temperature reaches  $25 \pm 0.5^\circ\text{C}$  ( $77[77 \pm 1^\circ\text{F}]-1^\circ\text{F}$ ). Time required for temperature equilibration depends on sample size and configuration.

6.2.2 Condition cup and lid to  $25 \pm 0.5^\circ\text{C}$  ( $77[77 \pm 1^\circ\text{F}]-1^\circ\text{F}$ ). 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 weight-per-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:

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

where:

$A$  = 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  $\text{kg}/\text{m}^3$ , multiply  $D$  by 119.83.

6.3.2 Calculate the specific gravity of the sample as follows:

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

where:

$SG$  = specific gravity,

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

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

#### 6.4 Report:

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

6.4.2 Report the specific gravity of the sample to the nearest hundredth at 25°C (77°F) [77°F].

### 7. Nonvolatile Content

#### 7.1 Apparatus:

7.1.1 *Metal Dish*, flat-bottom, having a diameter of 65 mm (2.5 in.) [2.5 in.] with walls 10 mm (5/8 in.) [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 ±0.01 g.

7.2 *Procedure*—Weigh 10 ± 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 ± 3°C (325 [325 ± 5°F] 5°F) for asphalt products or 105 to 110°C (221 [221 to 230°F] 230°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:

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

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:

$$S_I = (R/S) \times 100 \quad (4)$$

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 ±0.01 g.

9.1.3 *Muffle Furnace*, capable of maintaining a temperature of 593 ± 2.5°C (1100 [1100 ± 10°F] 10°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 ± 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) [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:

$$A_r = (A/S) \times 100 \quad (5)$$

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:

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

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.

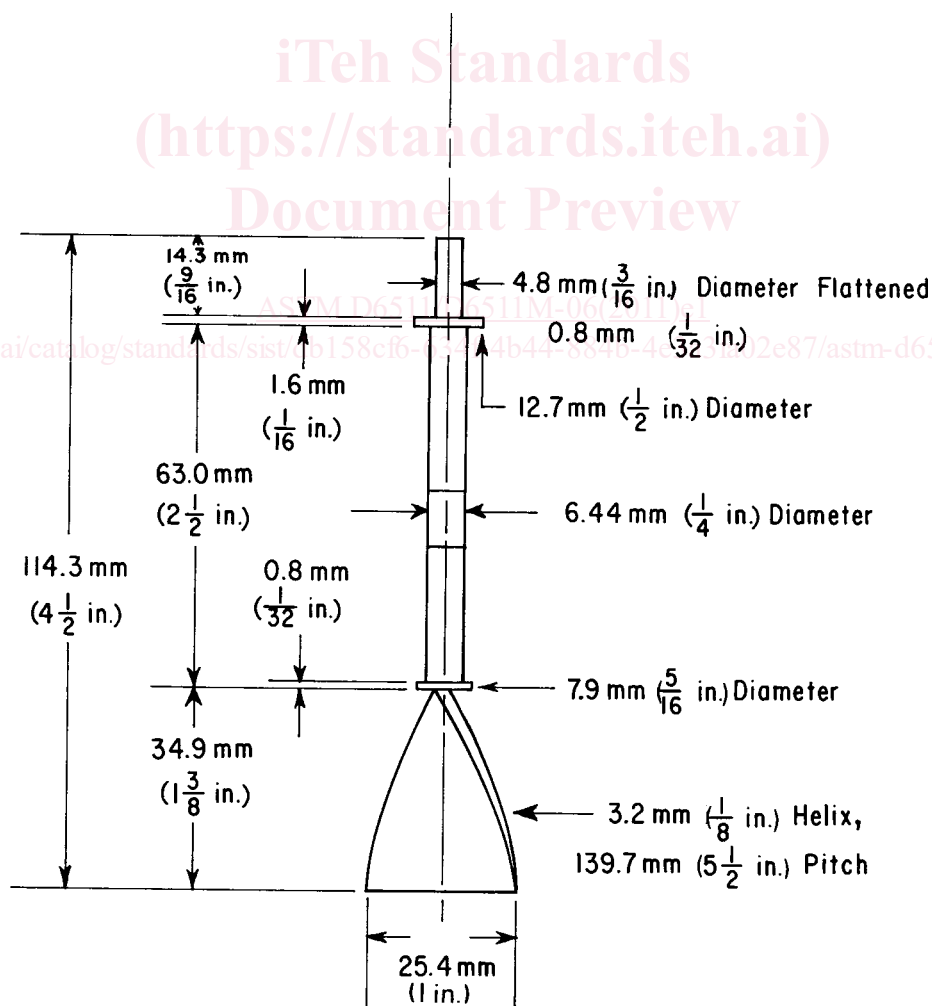
11.1.2.5 *Thermometer*—ASTM stormer viscosity thermometer having a range from 20 to 70°C, and conforming to the requirements for Thermometer 49C as prescribed in Specification E1.

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.); [40 in.], or sufficient distance to produce about 125 revolutions of the rotor. Insert the shaft of the propeller-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.); [in.]. Tighten the set screw on the position collar, if available, while the collar is in contact with



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

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.~~)[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 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  $25 \pm 0.2^\circ\text{C}$  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 (~~1/4 in.~~)[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  $25 \pm 0.2^\circ\text{C}$ . 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  $25 \pm 0.2^\circ\text{C}$ , 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 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~~)[1-pt] container to within 20 mm (~~3/4 in.~~)[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  $25 \pm 0.2^\circ\text{C}$  (~~77~~[77  $\pm$  0.3 $^\circ\text{F}$ ]; 0.3 $^\circ\text{F}$ ), 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~~)[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~~[3 by 5 in.] in.) and shall be 2.4 mm (~~3/32 in.~~)[in.]  $\pm$  10 % thick. The panels shall be 150 by 150 mm (~~6~~[6 by 6 in.] in.); 30 to 28 gage (~~0.32~~[0.32 to 0.40 mm]) nominal thickness; capable of being bent smoothly and uniformly through 180° over a 25 mm (~~1 in.~~)[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~~[6 by 6 in.] in.) steel panels and two pieces of smooth surface asphalt roll roofing, each covered by a 0.8 mm (~~1/32 in.~~)[in.]  $\pm$  10 % thick mask with a 75 by 125-mm (~~3~~[3 by 5 in.] 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~~)[75-mm] dimension, parallel to and no more than 50 mm (~~2 in.~~)[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 (~~1/32 in.~~)[in.]

12.2.2 Expose the test panels horizontally in a well ventilated area for 60 min at  $23 \pm 2^\circ\text{C}$  (~~73.4~~[73.4  $\pm$  3.6 $^\circ\text{F}$ ]; 3.6 $^\circ\text{F}$ ), but not in direct sunlight.

12.2.3 Suspend the panels vertically with the thread closest to the top edge in an oven at  $62 \pm 2^\circ\text{C}$  (~~140~~[140  $\pm$  3.6 $^\circ\text{F}$ ]; 3.6 $^\circ\text{F}$ ).

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)[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°C (32°F)[32°F] for 1 h along with a 25-mm (1-in.)[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  $[\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}]$  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 cm<sup>3</sup>.

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°C (225°F)[225°F] for 15 min, and allow to cool in a desiccator.

14.3.2 Place the crucible in a 100-cm<sup>3</sup> 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 flask 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°C (50[50 to 60°F]60°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.

14.3.5 *Calculation:*

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