



# Standard Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils<sup>1</sup>

This standard is issued under the fixed designation D500; 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.

## 1. Scope

1.1 These test methods cover the chemical analysis of sulfonated and sulfated oils. The analytical procedures appear in the following order:

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1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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## 2. Referenced Documents

- 2.1 *ASTM Standards*:<sup>2</sup>  
D1193 Specification for Reagent Water

## 3. Purity of Reagents

3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>3</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

## MOISTURE

### *Method A. Water by Distillation with Volatile Solvent*

## 4. Scope

4.1 This test method covers the determination of water existing in a sample of sulfonated or sulfated oil, or both, by distilling the sample with a volatile solvent. The method is applicable only to sulfonated and sulfated oils that do not contain the following: mineral acids, free sulfonic acids, or free sulfuric acid esters; or alkali hydroxides, carbonates or acetates; or alcohol, glycerin, diethylene glycol, acetone, or other water-miscible volatile compounds.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

## 5. Apparatus

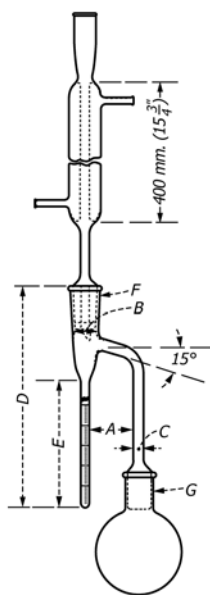
5.1 The apparatus required consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and flask shall be interchangeable ground joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask. A suitable assembly of the apparatus is illustrated in Fig. 1.

5.1.1 *Flask*, 500-mL, of either the short-neck, round-bottom type or the Erlenmeyer type.

5.1.2 *Heat Source*—The source of heat may be either an oil bath (stearic acid, paraffin wax, etc.), or an electric heater provided with a sliding rheostat or other means of heat control.

5.1.3 *Condenser*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately 400 mm (15¾ in.) in length with an inner tube 9.5 to 12.7 mm (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be about 7 mm (¼ in.) above the surface of the liquid in the trap after the distillation conditions have been established. Fig. 1 shows a conventional sealed-in type of condenser, but any other condenser fulfilling the detailed requirements above may be used.

5.1.4 *Trap*—A trap made of well-annealed glass constructed in accordance with Fig. 1 and graduated as shown to contain 5 mL at 20°C. It shall be subdivided into 0.1-mL divisions, with



A = 45 to 55 mm  
 B = 22 to 24 mm in inside diameter  
 C = 9 to 11 mm in inside diameter  
 D = 235 to 240 mm  
 E = 146 to 156 mm  
 F and G are interchangeable joints, standard taper 24/40.

FIG. 1 Apparatus for Water Determination by Distillation with Volatile Solvent, Method A

each 1-mL line numbered (5 mL at top). The error in any indicated capacity may not be greater than 0.05 mL.

## 6. Reagents

6.1 *Oleic Acid*, heated previous to use for 5 to 10 min over a free flame at a temperature of 130 to 135°C.

6.2 *Xylene*.

## 7. Calibration

7.1 To calibrate the apparatus add approximately 1 g of water to a mixture of 80 g of xylene and 10 g of oleic acid. Conduct the distillation as described in 8.2 – 8.4. When all the water has distilled, cool the apparatus, add another g of water, and repeat the distillation. Continue the calibration up to the capacity of the receiving tube.

## 8. Procedure

8.1 Clean the condenser and the receiving tube thoroughly with soap and warm water before using. Rinse well, then treat with hot cleaning solution (a mixture of 10 mL of saturated potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) and 990 mL of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, sp gr 1.84)), and finally thoroughly wash and dry.

8.2 Take enough of the sample to be tested for analysis to yield about 4 mL of water. Introduce the approximate quantity into a weighing bottle and make the weighings from the bottle into the flask, taking care that after removal of the sample no drops of oil are left on the outside of the weighing bottle. Add 80 g of xylene and oleic acid equivalent to about two and one-half times the weight of the bone-dry sample to prevent foaming and jelling of the contents of the flask. Introduce glass beads to prevent bumping and mix the contents of the flask thoroughly by swirling, taking care to avoid any loss of material. Fill the trap with xylene and immediately connect the flask with the distillation apparatus. Insert a loose cotton plug in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

8.3 Heat the flask and regulate the heating so that the condenser tube immediately below the water jacket is just barely hot. In this way a minimum of water will condense farther up the condenser where it may be difficult to volatilize any moisture condensed on the walls.

8.4 Continue the distillation at the specified rate until practically no water is visible on any part of the apparatus except within the graduations of the trap. This operation usually requires less than 1 h. Increase the rate of distillation in order to remove all traces of condensed water in the condenser tube, and continue the distillation until the water level in the trap remains unchanged after a 10-min interval. Dislodge any droplets adhering to the side of the receiver with a thin copper wire twisted into a loop. Immerse the receiving tube in warm water at about 40°C for 15 min or until the xylene layer becomes clear, then read and record the temperature and the exact volume of the water in the trap.

## 9. Calibration

9.1 The volume of condensed water measured in the trap may be converted into its equivalent weight in grams by means of Table 1. Calculate the percentage of water as follows:

**TABLE 1 Specific Gravity of Water<sup>A</sup>**

Temperature, °C	Specific Gravity
4	1.00000
35	0.99406
36	0.99371
37	0.99336
38	0.99299
39	0.99262
40	0.99224
41	0.99186
42	0.99147
43	0.99107
44	0.99066
45	0.99025

<sup>A</sup> This table is taken from *Smithsonian Tables*, compiled from various authors.

$$\text{Water, \%} = (A/B) \times 100 \quad (1)$$

where:

*A* = weight of water, g, and  
*B* = weight of sample, g.

*Method B. Moisture and Volatile Matter by Hot-Plate Method*

## 10. Scope

10.1 This test method covers the determination of the percentage of water and other compounds volatile at about 100°C existing in a sample of sulfonated or sulfated oil, or both, by rapid evaporation. The test method is applicable only to sulfonated and sulfated oils that do not contain the following: mineral acids, free sulfonic acids or free sulfuric acid esters, ammonia, acetic acid or similar volatile acids, alkali hydroxides, carbonates, acetates or similar salts that may react with oleic acid at elevated temperatures liberating volatile acids, or glycerin, diethylene glycol, xylene, or other compounds of similar volatility.

## 11. Apparatus

11.1 The apparatus required consists of a glass-stoppered weighing flask, a glass beaker, and a suitable thermometer.

11.1.1 *Weighing Flasks*—Any suitable glass-stoppered weighing flask of 10 to 15-mL capacity.

11.1.2 *Beaker*—A Griffin low-form glass beaker with an approximate capacity of 150 mL and a diameter of about 5 cm.

11.1.3 *Heat Source*—The source of heat may be either an electric hot plate with or without asbestos paper or board cover, or an open flame under a suitable asbestos board and a wire gauze (to spread the heat).

11.1.4 *Thermometer*, graduated from 90 to 150°C, about 3 in. in length, and substantially constructed.

## 12. Reagents

12.1 *Desiccating Agent*—Any suitable desiccating agent may be used.

NOTE 1—Recent investigations seem to indicate that calcium chloride is unreliable as a laboratory desiccating agent.

12.2 *Oleic Acid*.

## 13. Procedure

13.1 Weigh approximately 5 g of oleic acid into the beaker and insert the thermometer. Heat the oleic acid gradually, while stirring with the thermometer, until the temperature reaches 130°C. Place the beaker in an oven at 105 to 110°C for 15 min, cool in a desiccator, and weigh. Repeat the heating over the hot plate and in the oven until two successive weighings differ by less than 1.5 mg.

13.2 Place about 6 g of the sample in the weighing flask and determine the weight accurately. Transfer the sample to the beaker (containing the oleic acid and the thermometer) and weigh the flask again. Heat the mixture exactly as in the taring of the beaker as described in 13.1. The loss in weight is equivalent to the moisture in the sample.

## 14. Calculation

14.1 Calculate the percentage of moisture and volatile matter in the sample as follows:

$$\text{Moisture and volatile matter, \%} = (A/B) \times 100 \quad (2)$$

where:

*A* = loss of weight, g, and  
*B* = weight of sample, g.

## ORGANICALLY COMBINED SULFURIC ANHYDRIDE

### *Method A. Titration Test (For Sulfated Oils)*

## 15. Scope

15.1 This test method covers the determination of the organically combined sulfuric anhydride existing in a sample of sulfated oil by boiling the sample with sulfuric acid and determining the acidity of the reaction mixture. This method is applicable only to oils that split off their combined SO<sub>3</sub> upon boiling with mineral acids and that do not contain compounds that cannot be accurately titrated in water solution with methyl orange as the indicator.

## 16. Apparatus

16.1 The apparatus required consists of a glass flask provided with a glass stopper and an air condenser. The connection between the flask and the condenser shall be a ground joint. Perforated glass beads shall be used to prevent bumping.

16.1.1 *Flask*—An Erlenmeyer flask (Fig. 2) made of a borosilicate glass, having a capacity of approximately 300 mL and provided with a glass stopper.

16.1.2 *Condenser*—The condenser required consists of a glass tube, 915 mm (36 in.) in length, and 8 mm (5/16 in.) in outside diameter. The lower end of the tube shall be flared and ground to fit the mouth of the Erlenmeyer flask.

16.1.3 *Glass Beads*—Perforated glass beads, made of chemically-resistant glass, approximately 4 mm (5/32 in.) in diameter. Before using, the glass beads shall be boiled thoroughly in several portions of water or until the wash water reacts neutral to methyl orange indicator.

## 17. Reagents

17.1 *Ethyl Ether*.