Designation: D5348 – 95 (Reapproved 2012)

Standard Test Method for Determination of the Moisture Content of Sulfonated and Sulfated Oils by Distillation with Xylene[1]

This standard is issued under the fixed designation D5348; 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.

1. Scope

1.1 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 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; or alkali hydroxides, carbonates or acetates: or alcohol, glycerin, diethylene glycol, acetone, or other water-miscible volatile compounds. This test method was derived from Test Methods D500, Sections 4 through 9.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils

3. Significance and Use

3.1 This test method is intended to determine the moisture content of fats, oils, and fatliquors used in the softening and stuffing of leather. The moisture content is measured for the purpose of quality assurance.

4. Apparatus

4.1 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.

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

4.1.2 *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.

4.1.3 *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.

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

5. Purity of Reagents

5.1 *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.[3]

6. Reagents

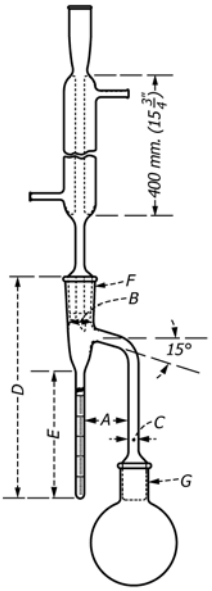
6.1 *Oleic Acid,* heated previous to use for 5 to 10 min over a free flame at a temperature of 130 to 135°C.

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 40-1957).

Current edition approved April 1, 2012. Published April 2012. Originally approved in 1993. Last previous edition approved in 2006 as D5348 – 95(2006). DOI: 10.1520/D5348-95R12.

[2] 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.

[3] *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.



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

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, 8.3, and 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 ($K_2Cr_2O_7$) and 990 mL of sulfuric acid (H_2SO_4, 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 jellying 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:

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

where:

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

10. Precision and Bias

10.1 Although this test method is widely used, precision and bias information is not available at this time.

11. Keywords

11.1 distillation; leather; moisture; sulfonated and sulfated oils; xylene

TABLE 1 Specific Gravity of Water[A]

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

[A] This table is taken from *Smithsonian Tables*, compiled from various authors.