



Designation: D 1568 – 97

## Standard Test Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates<sup>1</sup>

This standard is issued under the fixed designation D 1568; 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 sampling and chemical analysis of paste, powder, or liquid detergent alkylbenzene sulfonates.

1.2 The procedures for sampling and analysis appear in the following order:

	Sections
Sampling:	
Powders and Flakes Packed in Cans or Cartons	3
Powders and Flakes in Bulk	4
Liquids	6
Pastes	7
Preparation of Sample	8
Moisture:	
Distillation Method	10-13
Karl Fischer Method	14-19
Total Matter Insoluble in Alcohol	20-22
Neutral Oil	23-26
Chlorides Calculated as Sodium Chloride (NaCl)	27-30
pH	31

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:

D 1172 Test Method for pH of Aqueous Solutions of Soaps and Detergents<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

### SAMPLING

### 3. General Requirements

3.1 The seller shall have the option of being represented at the time of sampling and when he so requests shall be furnished with a duplicate sample.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and is the direct responsibility of D12.12 on Analysis of Soaps and Synthetic Detergents.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.04.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

### 4. Powders and Flakes Packed in Cans or Cartons

4.1 One can or carton shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb (22.7 kg). In the case of smaller containers, a can or carton shall be taken at random from each lot of containers totaling not more than 5000 lb (2268 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn will not exceed 20 lb (9.1 kg). The individual cans or cartons shall be tightly wrapped at once in paraffined paper and sealed by rubbing the edges with a heated iron. The inspector shall accurately weigh each wrapped can or carton and record its weight and the date of weighing on the wrapper. The wrapped cans or cartons shall be placed in an airtight container which should be nearly filled, and which shall then be sealed, marked, and sent to the laboratory for test. Samples shall be kept cool until tested.

### 5. Powders and Flakes in Bulk

5.1 A grab sample of not less than 0.5 lb (227 g) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 100 lb (45.4 kg). In the case of smaller containers, a grab sample of not less than 0.5 lb (227 g) shall be taken at random from each lot of containers totaling not more than 10,000 lb (4536 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three grab samples of 0.5 lb (227 g) each taken at random from separate containers. In the case of very large lots the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb (9.1 kg). The inspector shall rapidly mix the gross sample and place it in an airtight container which shall be filled, sealed, marked, accurately weighed, with its weight and the date of weighing recorded on the package, and be sent to the laboratory for test. Samples shall be kept cool until tested.

## 6. Liquids

6.1 A sample of not less than 0.5 pt (236.6 mL) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 10 gal (37.9 L). In the case of smaller containers, a sample of not less than 0.5 pt (236.6 mL) shall be taken at random from each lot of containers totaling not more than 1000 gal (3785.4 L) or fraction thereof. The gross sample shall in all cases consist of not less than three samples of 0.5 pt (236.6 mL) each taken at random from separate containers. Before drawing the sample from the container selected, the contents of the container shall be thoroughly agitated. The inspector shall thoroughly mix the gross sample, place it in clean, dry cans or bottles, which shall be completely filled and securely stoppered with clean corks or caps, then sealed, marked, and sent to the laboratory for test.

## 7. Pastes

7.1 *Pastes Packed in Cans or Cartons of 5 lb (2.27 kg) or Less*—One can or carton shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb (22.7 kg). In the case of smaller containers, a can or carton shall be taken at random from each lot of containers totaling not more than 5000 lb (2268 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb (9.1 kg). The samples shall be wrapped, sealed, marked, and sent to the laboratory for test.

7.2 *Pastes Packed in Bulk*—A “trier” sample (Note 1) of not less than 0.5 lb (227 g) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb (22.7 kg). In the case of smaller containers, a “trier” sample of not less than 0.5 lb (227 g) shall be taken at random from each lot of containers totaling not more than 5000 lb (2268 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three 0.5-lb (227-g) samples, each taken at random from separate containers. With very large lots where the sample drawn as above will amount to more than 10 lb (4.5 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 10 lb (4.5 kg). The inspector shall promptly place the gross sample in a clean, dry, airtight and watertight container, which shall be filled, sealed, marked, and sent to the laboratory for test.

NOTE 1—A trier sample is obtained by inserting a “trier” into the material. A trier is a halfround steel cylinder  $\frac{1}{2}$  to  $\frac{3}{4}$  in. (12.7 to 19 mm) in diameter, 6 to 36 in. (152 to 914 mm) in length, pointed on one end and having a grip handle on the other end. After insertion, the trier is turned two or three times, and upon removal a core of the material being sampled is obtained.

## 8. Preparation of Sample

8.1 *Powders and Flakes*—Minimizing exposure to air, rapidly disintegrate and mix the sample of powdered, flake, or chip product. If desired, quarter down to about 1 lb (453.6 g).

Weigh at once all portions for analysis, preserving the remainder in an airtight container in a cool place.

8.2 *Liquids*—No preparation of the sample of liquid, other than a thorough mixing, is necessary unless it is received during very cold weather, when it should be allowed to stand at least 1 h after it has warmed to room temperature (20 to 30°C) before it is tested, particularly for its lathering qualities.

NOTE 2—If pastes or liquids are known to be acidic, and decomposition of sample can result from heating, the samples shall be adequately labeled for precautionary treatment and warmed to room temperature or other maximum temperature as agreed upon for mixing and sampling.

8.3 *Pastes*—Store preferably in glass. If crystals separate, melt on water bath (Note 1).

## METHODS OF CHEMICAL ANALYSIS

### 9. Purity of Reagents

9.1 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>4</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.

9.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

### MOISTURE BY THE DISTILLATION METHOD

(Preferred Method)

### 10. Apparatus

10.1 The apparatus required shall consist 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.

10.1.1 *Flask*—A 1000-mL flask of either the short-neck, round-bottom type or the Erlenmeyer type.

10.1.2 *Heat Source*—Either an oil bath (stearic acid, paraffin wax, etc.), or an electric heater provided with a sliding rheostat or other means of heat control.

10.1.3 *Condenser*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately  $15\frac{3}{4}$  in. (400 mm) in length, with an inner tube  $\frac{3}{8}$  to  $\frac{1}{2}$  in. (9.5 to 12.7 mm) in outside diameter, and not less than  $\frac{1}{4}$  in. (6.35 mm) in inside diameter, shall be used. The end of the condenser to be inserted in the trap may be ground off at an angle of 30° from the

<sup>4</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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

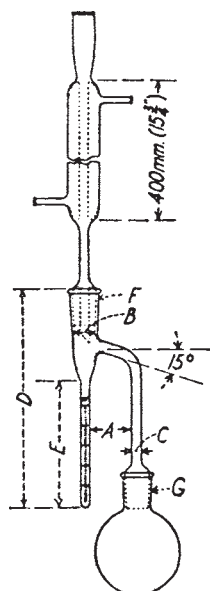


FIG. 1 Assembly of Distillation Apparatus

vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be about ¼ in. (7 mm) 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 of this paragraph may be used.

10.1.4 *Trap*—For greatest accuracy several trap sizes are allowable, depending upon the percentage of moisture expected:

Moisture Expected, percent	Size of Trap, mL
0 to 5, incl	5
Over 5 to 17, incl	10
Over 17 to 30, incl	10
Over 30 to 50, incl	25
Over 50 to 70, incl	25
Over 70 to 85, incl	25

Traps made of well-annealed glass, constructed essentially as shown in Fig. 1, and graduated to contain one of the following specified volumes at 20°C shall be used:

10.1.4.1 *5-mL Trap*—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.

10.1.4.2 *10-mL Trap*—Subdivided from 0 to 1 mL in 0.1-mL divisions and from 1 to 10 mL in 0.2-mL divisions.

10.1.4.3 *25-mL Trap*—Subdivided from 0 to 1 mL in 0.1-mL divisions and from 1 to 25 mL in 0.2-mL divisions.

NOTE 3—The condenser and trap should be thoroughly cleaned before use.

### 11. Solvent

11.1 *Xylene* (or Toluene)—Saturate xylene (or toluene) with water by shaking with a small quantity of water and distill. Use the distillate for the determination.

### 12. Procedure

12.1 Transfer to the 1000-mL flask, equipped with the size of the trap specified in 10.1.4, an amount of sample according to the percentage of moisture expected, as follows:

Moisture Expected, %	Weight of Sample to be Used, g <sup>A</sup>
0 to 5, incl	50 ± 5
Over 5 to 17, incl	50 ± 5
Over 17 to 30, incl	40 ± 4
Over 30 to 50, incl	30 ± 3
Over 50 to 70, incl	30 ± 3
Over 70 to 85, incl	25 ± 2

<sup>A</sup> Weighed to the nearest 0.25 g.

Add immediately about 100 mL of xylene or toluene. Place a small, thin sheet of long-fiber, chemical-resistant glass<sup>5</sup> wool on the surface of the toluene. The glass wool should be thoroughly dried in the oven and held in the desiccator before use.

12.2 Connect the flask and receiver to the condenser and pour sufficient xylene or toluene down the condenser tube to cause a slight overflow through the side tube. Wrap the flask and tube leading to the receiver with an insulating cloth, so that refluxing will be under better control.

12.3 Heat the oil bath with a gas burner or other source of heat, or apply heat directly to the flask with an electric heater and distill slowly. The rate at the start should be approximately 100 drops/min. When the greater part of the water has distilled over, increase the distillation rate to 200 drops/min until no more water is collected. Purge the reflux condenser during the distillation with 5-mL portions of xylene (or toluene) to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the xylene (or toluene) by using a spiral copper or nichrome wire. Move the wire up and down in the condenser occasionally, thus causing the water to settle at the bottom of the receiver. Reflux for at least 2 h, and shut off the heat at the end of this period.

12.4 Wash down condenser with 1 mL of absolute ethanol (CH<sub>3</sub>CH<sub>2</sub>OH). Adjust the temperature of the distillate to 20°C and read the volume of water.

### 13. Calculations

13.1 Calculate the percentage of moisture as follows:

$$\text{Moisture, \%} = [(V \times 0.998)/W] \times 100 \quad (1)$$

where:

V = volume of water, mL at 20°C, and

W = weight of the sample, g.

### WATER BY THE KARL FISCHER TITRATION METHOD (Alternative Method)

### 14. Application

14.1 This method is intended to be used where the amount of moisture is low.

### 15. Apparatus

15.1 *Automatic Buret* (Machlett Type), 50-mL capacity, 1000-mL reservoir of amber glass with a straight tip. The buret should also be equipped with a drying tube containing a

<sup>5</sup> Borosilicate glass has been found satisfactory for this purpose.