Designation: D1568 − 05 (Reapproved 2014)

Standard Test Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates[1]

This standard is issued under the fixed designation D1568; 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 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:

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.* For a specific hazards statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1172 Guide for pH of Aqueous Solutions of Soaps and Detergents

D1193 Specification for Reagent Water

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.

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

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[1] These test methods are under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergentsand is the direct responsibility of D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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[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.

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 ½ to ¾ 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. Hazards

8.1 All reagents and chemicals should be handled with care. Before using any chemical, read and follow all safety precautions and instructions on the manufacturer's label or MSDS (Material Safety Data Sheet).

9. Preparation of Sample

9.1 *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.

9.2 *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.

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

METHODS OF CHEMICAL ANALYSIS

10. Purity of Reagents

10.1 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.[3] 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.

10.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

MOISTURE BY THE DISTILLATION METHOD

(Preferred Method)

11. Apparatus

11.1 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.

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[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.

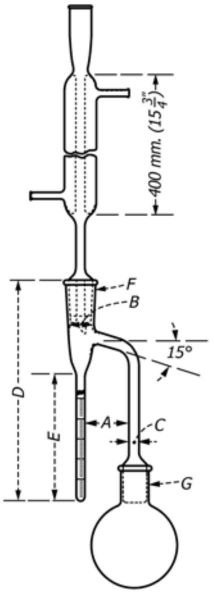

FIG. 1 Assembly of Distillation Apparatus

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

11.1.2 *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.

11.1.3 *Condenser*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately 15¾ in. (400 mm) in length, with an inner tube ⅜ to ½ in. (9.5 to 12.7 mm) in outside diameter, and not less than ¼ 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 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.

11.1.4 *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:

11.1.4.1 *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.

11.1.4.2 *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.

11.1.4.3 *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.

12. Solvent

12.1 *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.

13. Procedure

13.1 Transfer to the 1000-mL flask, equipped with the size of the trap specified in 11.1.4, an amount of sample according to the percentage of moisture expected, as follows:

Moisture Expected, %	Weight of Sample to be Used, g[A]
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

[A] 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[4] wool on the surface of the toluene. The glass wool should be thoroughly dried in the oven and held in the desiccator before use.

13.2 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.

13.3 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.

13.4 Wash down condenser with 1 mL of absolute ethanol (CH_3CH_2OH). Adjust the temperature of the distillate to 20°C and read the volume of water.

14. Calculations

14.1 Calculate the percentage of moisture as follows:

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

where:

V = volume of water, mL at 20°C, and

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[4] Borosilicate glass has been found satisfactory for this purpose.

W = weight of the sample, g.

WATER BY THE KARL FISCHER TITRATION METHOD
(Alternative Method)

15. Application

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

16. Apparatus

16.1 *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 suitable drying agent such as anhydrous calcium sulfate (Drierite)[5] to protect the Karl Fischer reagent from moisture in the air. Calcium chloride also is satisfactory. Magnesium perchlorate (Dehydrite) must not be used, due to the possible danger of explosion in contact with organic vapors.[6]

NOTE 4—Instead of the described and recommended buret there are a number of other practical arrangements of titration apparatus.

17. Reagents

17.1 *Karl Fischer Reagent*—Purchase as separate stable solutions of iodine in methanol and sulfur dioxide in pyridine with directions for mixing prior to use.[7] Alternatively, stable single-solution, pyridine-free reagent solutions are available from laboratory supply houses.[8]

17.2 *Methyl Alcohol, Anhydrous*—The water content of the methyl alcohol (CH_3OH) shall not be more than 0.1 weight percent. Run a blank on each new container of methyl alcohol to establish the water content.

17.3 *Sodium Tartrate Dihydrate* ($Na_2C_4 \cdot H_4O_6 \cdot 2H_2O$)—The water content of the sodium tartrate should be 15.66 ± 0.05 %. This value can be checked by heating a sample at 150°C for 3 h.

18. Procedure

18.1 Weigh to the nearest 0.1 mg a portion of the sample, containing from 50 to 200 mg of water, into a *dry*, 125-mL Erlenmeyer flask and stopper immediately.

18.2 Add 50 mL of methyl alcohol and immediately cover the flask aperture with a rubber dam.[9] Allow to stand for a few minutes with occasional swirling.

18.3 Pierce the rubber dam, insert the tip of the buret, and titrate with the Karl Fischer reagent. Approach of the end point is shown by a decrease in speed of discharge of the brown color of the reagent and a slight change in color of the sample from yellow to reddish yellow. Continue to titrate in 0.1 to 0.2-mL increments until the red-brown color of iodine persists.

18.4 *Blank*—Run a blank determination on 50 mL of methyl alcohol, using the procedure described in 18.2 and 18.3.

18.5 Repeat the procedure described in 18.1 – 18.3 using 0.3 to 0.7 g of sodium tartrate dihydrate in place of the sample.

19. Calculations

19.1 Calculate the water equivalent of the Karl Fischer reagent as follows:

$$C = (A \times 0.1566)/B \tag{2}$$

where:

C = grams of water equivalent to 1 mL of Karl Fischer reagent,
A = grams of sodium tartrate used (18.5), and
B = millilitres of Karl Fischer reagent required for titration of the sodium tartrate.

19.2 Calculate the percentage of water in the sample as follows:

$$\text{Water, \%} = [(A - B) \times C/D] \times 100 \tag{3}$$

where:

A = millilitres of Karl Fischer reagent required to titrate the sample,
B = millilitres of Karl Fischer reagent required to titrate the blank,
C = grams of water equivalent to 1 mL of Karl Fischer reagent, and
D = grams of sample used.

20. Precision

20.1 Based on experimental results, the intralaboratory precision (95 % limits) for simultaneous duplicate determinations by one analyst is ± 0.22 % at a moisture level of 70 %.

20.2 The over-all precision (intralaboratory) is statistically estimated (95 % limits) to be ± 0.17 % at a moisture level of 70 %.

TOTAL MATTER INSOLUBLE IN ALCOHOL

21. Reagent

21.1 *Ethyl Alcohol, Neutral (95 %)*—Freshly boiled ethyl alcohol (95 % or higher) and neutral to phenolphthalein indicator, conforming to either Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue.

22. Procedure

22.1 Transfer 2 g of the sample, weighed to nearest 0.001 g, to a 250-mL beaker. Add 100 mL of neutral ethyl alcohol (95 %), cover the beaker, and heat on the steam bath with frequent stirring and maceration of the sample until completely disintegrated. Let settle and filter the supernatant liquid through a tared Gooch crucible, with suction, into a 300-mL Erlenmeyer flask, retaining as much of the residue as possible in the beaker. Repeat this extraction three times with 25-mL

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[5] Drierite, which is a special indicating type of anhydrous calcium sulfate, is obtainable from the W. A. Hammond Co., Yellow Springs, Ohio.

[6] A suitable buret can be obtained from the Precision Scientific Co., 3737 Cortland St., Chicago, Ill. 60647.

[7] Instructions for laboratory preparation of the Karl Fischer Reagent are given in Section 2(1.1) of ASTM Method D890, Test for Water in Liquid Naval Stores, *1983 Annual Book of ASTM Standards,* Vol 06.03.

[8] Examples of brands available are Hydranal® of Riedel-de Faën AG, and Aquastar® of EM Science, Inc.

[9] Davol "Sani Tab" caps have been found suitable for this purpose.

portions of hot neutral ethyl alcohol (95 %), each time retaining as much of the residue as possible in the beaker. Finally, evaporate any remaining alcohol and dissolve the residue in the smallest possible quantity of hot water (5 mL is sufficient). Reprecipitate the alcohol-insoluble matter by slowly adding, while stirring vigorously, 50 mL of neutral ethyl alcohol.

NOTE 5—Solution and reprecipitation of alcohol-insoluble matter is necessary for complete separation from alcohol-soluble matter.

22.2 Transfer the precipitate quantitatively to the tared Gooch crucible, washing well with 95 % alcohol. Dry 2 h or longer at 105 ± 2°C. Cool in a desiccator and weigh. Repeat heating until constant weight is attained.

23. Calculation

23.1 Calculate the percentage of total matter insoluble in alcohol as follows:

$$T = (M/W) \times 100 \tag{4}$$

where:

T = percentage of total matter insoluble in alcohol,
M = grams of insoluble matter, and
W = grams of sample used.

NEUTRAL OIL

24. Reagents

24.1 *Acetone.*

24.2 *Ethyl Alcohol, Neutral (95 %)*—Freshly boiled ethyl alcohol, 95 % or higher and neutral to phenolphthalein indicator, conforming to either Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue..

24.3 *Petroleum Ether,* having a distillation range between 30 and 60°C, or *n*-pentane having a distillation range between 33 and 41°C.

25. Procedure

25.1 Accurately weigh a sample equivalent to 3 to 5 g of active ingredient and transfer to a 250-mL extraction cylinder. Add 50 mL of neutral ethyl alcohol and 50 mL of water. Shake to disperse.

25.2 Add 50 mL of petroleum ether (or *n*-pentane), stopper the cylinder, and shake up and down vigorously for 1 min (Note 6). Remove the stopper and wash it and the internal walls of the cylinder with a small volume of petroleum ether. Allow to stand until the emulsion breaks and both layers are perfectly clear.

NOTE 6—Use of ethyl ether yields higher values because of increased solubility of sulfones, etc.

25.3 Siphon off the ether layer into a dry, tared 250-mL wide-mouth Erlenmeyer flask. Care should be taken not to siphon off any of the bottom alcohol-water layer with the ether extract. The end of the siphon tube should be near the top of the ether layer at the start of the siphoning operation and away from the cylinder wall. During the siphoning, push the tube down gradually as far as possible without contamination to within ⅛ to ¼ in. (3 to 6 mm) of the boundary between the ether and water layers. If the operation of siphoning off the ether extract is carefully carried out, no water will be siphoned off with the ether layer. After the siphoning step, slide the siphon up and away from the liquid in the cylinder. Gently blow the ether remaining in the siphon into the Erlenmeyer flask. Rinse the exit end of the siphon with a small volume of ether.

25.4 Remove the ether by distillation in a controlled temperature water bath held at 60 to 70°C.

NOTE 7—The use of a regulated, steam-heated water bath is recommended. An electrically-heated sand bath may be used in place of the water bath, provided certain safety measures are carried out. The switch control should be some distance away from the hot plate and place of work. A sand bath of such size as to fit on top of the hot plate should be provided. The temperature of the sand bath should be automatically maintained between 60 and 70°C by a suitable regulator.[10]

25.5 Repeat the procedure described in 25.2 – 25.4 four additional times, making a total of five extractions.

NOTE 8—The same siphon must be used throughout the determination.

NOTE 9—The ether should be evaporated to a small volume after each extraction, permitting the total volume of ether from the next extraction to be held conveniently in the 250-mL flask. Do not attempt to evaporate more than 125 mL of ether at one time, as losses due to foaming may occur.

25.6 Evaporate as much as possible of the petroleum ether from the combined extracts on the water bath. Remove the flask from the water bath when the ether no longer distills from the flask.

25.7 Place the flask on a sheet of asbestos ⅛ in. (3 mm) thick on a boiling water bath and remove the last traces of ether with the aid of a stream of clean, dry air. Do not leave on the water bath for any time longer than absolutely necessary to volatilize the petroleum ether. This should take no more than 5 min.

NOTE 10—If the operation described in 25.7 requires more than 5 min, either too great a volume of ether remained in the flask in the procedure described in 25.6, or the stream of air is of insufficient volume, or both conditions are abnormal. Take the proper steps to correct either or both sources of error.

25.8 Add 2 mL of acetone and repeat the procedure described in 25.7. Wipe the flask with a clean cloth, cool in a desiccator, and weigh.

26. Calculation

26.1 Calculate the results as follows:

$$\text{Neutral oil, (unsulfonated material) \%} = (A/B) \times 100 \tag{5}$$

where:

A = grams of residue, and
B = grams of sample used.

27. Precision

27.1 Based on experimental results, the intralaboratory precision of this method is statistically estimated (95 % limits) to be ± 0.03 %.

27.2 For simultaneous duplicate determinations by one analyst, the estimated precision is ± 0.01 %.

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[10] The Foxboro regulator has been found satisfactory for this purpose.

CHLORIDES CALCULATED AS SODIUM CHLORIDE (NaCl)

28. Apparatus

28.1 *Stirrer Motor and Small Glass Rod Stirrer.*

28.2 *Potentiometer.*[11]

28.3 *Calomel Reference Electrode,* saturated.

28.4 *Silver Wire Electrode,* 1 mm in diameter by 120 mm in length.

29. Reagents and Materials

29.1 *Acetone.*

29.2 *Ethyl Alcohol*—Freshly boiled ethyl alcohol conforming to Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue.

29.3 *Methyl Orange Indicator Solution.*

29.4 *Nitric Acid (1+1)*—Mix 1 volume of concentrated nitric acid (HNO_3, sp gr 1.42) containing 0.3 % sodium nitrite ($NaNO_2$) with 1 volume of water.

29.5 *Nitric Acid (1+4)*—Mix 1 volume of HNO_3 (sp gr 1.42) with 4 volumes of water.

29.6 *Silver Nitrate, Standard Solution (0.2 N)*—Prepare and standardize a 0.2 *N* silver nitrate ($AgNO_3$) solution as follows: Weigh 17 g of $AgNO_3$ to the nearest 0.001 g. Dissolve in water and transfer to a 500-mL volumetric flask. Dilute to the mark. Standardize as follows: Dry about 10 g of NaCl at 110°C to constant weight. Weigh about 2.00 g of the dried NaCl to the nearest 0.001 g. Dissolve in a solvent consisting of 60 % water and 40 % alcohol. Transfer to a 100-mL volumetric flask and dilute to the mark with solvent. Pipet 100 mL of the NaCl solution to a beaker and titrate with the $AgNO_3$ solution as described in Section 30.

29.6.1 Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality} = (A \times 10)/(B \times 58.45) \tag{6}$$

where:

A = grams of NaCl used, and
B = millilitres of $AgNO_3$ solution required for titration of the NaCl.

30. Procedure

30.1 Chlorides may be determined on the original sample, the alcohol-insoluble portion, or on the alcohol-soluble matter, and should be reported on these bases, the total chlorides calculated as NaCl being reported for the analysis of the original sample.

30.2 Weigh to ± 0.001 g a portion of the sample approximately equal to 30 g divided by the percentage of NaCl expected, but the sample should not exceed 10 g.

30.3 Dissolve in 250 mL of hot water, add 2 drops of methyl orange indicator solution, and acidify to the acid color by adding HNO_3 (1+4). Warm slightly and stir to effect maximum solution. Add 50 mL of acetone.

30.4 Clean the silver electrode in the HNO_3 (1+1) containing $NaNO_2$. Set up the titration cell with the silver electrode connected to the top terminal and the saturated calomel cell connected to the bottom terminal. Set the pH meter on + mV. Start the stirring and titrate the solution potentiometrically as follows:

30.4.1 Add 0.5 mL of $AgNO_3$ solution and measure the emf. If appreciable chloride is present, the emf should be in the range of 100 mV.

30.4.2 Add $AgNO_3$ solution slowly in 2 to 3-mL portions until the emf reaches 200 mV. Stir well.

30.4.3 Add $AgNO_3$ solution in 0.1-mL portions, allowing sufficient time after each addition for the solution to reach equilibrium (60 to 80 s). Measure the emf (stirrer off) at each 0.1-mL point.

30.4.4 Calculate the end point by the rate of change method (Note 11). The end point is usually in the range of 260 to 270 mV.

NOTE 11—*Example*—The method for determining the maximum rate of change is as follows:

mL	emf	ΔE†	ΔE'
21.20	210		
21.30	220	10	10
21.40	240	20	17[A]
21.50	277	37	
21.60	302	25	12

[A] Maximum rate of change.

$$\text{End point} = 21.4 + ([17/(17+21)] \times 0.1) \tag{7}$$

$$= 21.46 \text{ mL}$$

30.5 Run a blank and subtract the value obtained from the value calculated in 30.4.4.

31. Calculations

31.1 Calculate as sodium chloride (NaCl) the percentage of chlorides present, as follows:

$$A = [(S - B)N \times 5.85]/C \tag{8}$$

where:

A = percentage of chlorides present, calculated as NaCl,
S = millilitres of $AgNO_3$ solution required for titration of the sample,
B = millilitres of $AgNO_3$ solution required for titration of the blank,
N = normality of the $AgNO_3$ solution, and
C = grams of sample used.

pH

32. Procedure

32.1 Determine the pH in accordance with Test Method D1172, except measure 10 min after adjusting the solution of the sample to volume.

33. Keywords

33.1 alkylbenzene sulfonates; chemical analysis; sampling

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[11] The Beckman Model G pH meter has been found satisfactory for this purpose.

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