



# Standard Test Methods for Chemical Analysis of Soaps Containing Synthetic Detergents<sup>1</sup>

This standard is issued under the fixed designation D 820; 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 procedures for the chemical analysis of soaps containing synthetic detergents.

1.2 The analytical procedures appear in the following order:

	Sections
Moisture and Other Matter Volatile at 105°C (Oven Method)	5 and 6
Free Alkali or Free Acid	7 and 8
Anhydrous, Salt-Free, Soda Soap	9-12
Alcohol-Soluble Matter	13 and 14
Matter Insoluble in Water	15
Total Alkalinity of Matter Insoluble in Alcohol (Alkaline Salts)	16 and 17
Sodium Silicate	18-20
Phosphates	21-28
Phosphate (Colorimetric Method Using Molybdenum Blue)	29-34
Unsaponified and Unsaponifiable Matter	35-39
Free Fatty Matter	40
Chlorides in Alcohol-Soluble Matter	41-43
Rosin (McNicol Method)	44-47
Synthetic Detergent (by Difference)	48
Neutral, Inorganic Salts	49

1.3 *This standard does not purport to address all of the safety problems, 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 216 Method for Distillation of Natural Gasoline<sup>2</sup>

D 459 Terminology Relating to Soaps and Other Detergents<sup>3</sup>

D 875 Method for Calculation of Olefins and Aromatics in Petroleum Distillates from Bromine Number and Acid Absorption<sup>4</sup>

D 1193 Specification for Reagent Water<sup>5</sup>

D 1768 Test Method for Sodium Alkylbenzene Sulfonate in

Synthetic Detergents by Ultraviolet Absorption<sup>3</sup>

D 2357 Qualitative Classification of Surfactants by Infrared Absorption<sup>3</sup>

D 2358 Test Method for Separation of Active Ingredient from Surfactant and Syndet Compositions<sup>3</sup>

D 3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.2 The term *synthetic detergent* in these test methods is defined in accordance with Terminology D 459, as follows:

3.3 *synthetic detergent*—a detergent produced by chemical synthesis and comprising an organic composition other than soap.

3.4 For definitions of other terms used in these test methods, refer to Terminology D 459.

## 4. Purity of Reagents and Materials

4.1 *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>6</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.

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

### MOISTURE AND OTHER MATTER VOLATILE AT 105°C (OVEN METHOD)

## 5. Apparatus

5.1 *Dish*—A porcelain or glass dish about 6 to 8 cm in diameter and about 2 to 4 cm in depth will be required.

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

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<sup>2</sup> Discontinued, see 1987 *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.04.

<sup>4</sup> Discontinued, see 1984 *Annual Book of ASTM Standards*, Vol 05.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>6</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.

## 6. Procedure

6.1 Weigh  $5 \pm 0.01$  g of the sample in the dish, and dry to constant weight in an air oven at a temperature of  $105 \pm 2^\circ\text{C}$ . Constant weight is attained when heating for successive 1-h periods shows a loss (or gain) of not more than 0.1 %.

NOTE 1—Because of its established use in the trade, the term *weight* is employed in these methods in place of the technically correct term *mass*.

### FREE ALKALI OR FREE ACID

## 7. Reagents

7.1 *Ethyl Alcohol, Neutral* (95 %)—Freshly boiled, reagent grade, ethyl alcohol, 95 % or higher, neutral to phenolphthalein, and containing only volatile denaturants, 95 mL,<sup>7</sup> plus 5 mL of water.

7.2 *Phenolphthalein Indicator Solution* (10 g/litre)—Dissolve 1 g of phenolphthalein in 50 mL of neutral ethyl alcohol (95 %) and mix with 50 mL of water (see 7.1).

## 8. Procedure

8.1 Weigh 5 to  $10 \pm 0.01$  g of the sample into a 300-mL Erlenmeyer flask. Add 200 mL of neutral ethyl alcohol (95 %). Equip the flask with an air-cooled reflux condenser, and digest the sample on a steam bath until the soap is dissolved (see Note 2). Remove the condenser, add 0.5 mL of the phenolphthalein indicator solution, and titrate immediately with standard acid or alkali. Calculate as NaOH, if alkaline, or as oleic acid, if acid.

NOTE 2—In the analysis of soaps known to contain little or no alkaline salts, it is unnecessary to filter the hot alcoholic soap solution. However, the filtration should be carried out in all cases where alkaline salts such as silicates, phosphates, borates, and similar salts are present, since these are known to affect the free alkali determination. Free alkali figures in soap or surfactant mixtures containing borax are unreliable, due to solubility of borax in hot alcohol.

### ANHYDROUS, SALT-FREE, SODA SOAP

## 9. Apparatus

9.1 *Extraction Cylinder*, 250-mL, graduated, glass-stoppered, about 39 mm (1½ in.) in diameter and about 35.5 cm (14 in.) in length.

9.2 *Stokes Flask*, 100-mL, round-bottom (with the bottom blown out), sealed onto a 150-mL Erlenmeyer flask. A diagram of the Stokes flask is shown in Fig. 1.

9.3 *Siphon*, consisting of a two-hole rubber stopper fitted with small-diameter glass tubing as shown in Fig. 2.

## 10. Reagents

10.1 *Ethyl Alcohol, Neutral* (95 %)—Freshly boiled, reagent grade, ethyl alcohol, 95 % or higher, neutral to phenolphthalein, and containing only volatile denaturants, 95 mL,<sup>7</sup> plus 5 mL of water.

10.2 *Methyl Orange Indicator Solution* (1 g/litre)—Dissolve 0.1 g of methyl orange in water and dilute to 100 mL.

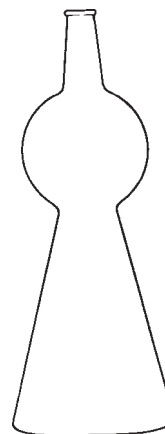


FIG. 1 Stokes Flask

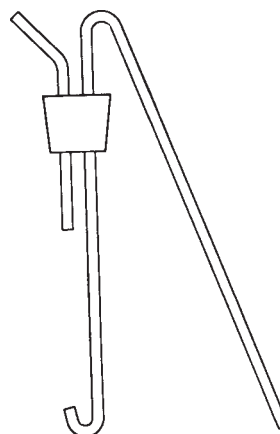


FIG. 2 Siphon

10.3 *Petroleum Ether*—The solvent used shall be of the pentane type, containing a minimum amount of isopentane, isohexane, and hexane, and boiling in the range 35 to  $60^\circ\text{C}$ .<sup>8</sup>

#### Distillation test:<sup>A</sup>

Initial boiling point	35 to $38^\circ\text{C}$
Dry flask end point	$52$ to $60^\circ\text{C}$
Distilling under $54^\circ\text{C}$ , min	95 %
Distilling under $40^\circ\text{C}$ , max	60 %
Specific gravity at $15.5/15.5^\circ\text{C}$ ( $60/60^\circ\text{F}$ )	0.630 to 0.660
Color	water-white
Doctor test	sweet
Evaporation residue, 100 mL, max	0.0011 g
Copper-strip corrosion test <sup>B</sup>	noncorrosive
Unsaturated compounds <sup>C</sup>	trace only permitted
Residue in distilling flask	neutral to methyl orange
Blotter-strip odor test <sup>D</sup>	odorless within 12 min
Aromatic compounds <sup>E</sup>	no nitrobenzene odor
Saponification value	less than 1.0 mg KOH/100 mL

<sup>A</sup> The distillation test shall be made in accordance with Method D 216. As a check on the evaporation residue, 250 mL of the petroleum ether and 0.25 g of stearin or other hard fat (previously brought to constant weight by heating) when dried as in the actual determination (10.4) shall not show an increase in weight exceeding 0.003 g.

<sup>B</sup> The copper-strip corrosion test shall be made by inserting a small polished copper strip into the petroleum ether in the distilling flask. There should be no appreciable darkening of the copper.

<sup>7</sup> Fischer Scientific A962, or its equivalent, is suitable for this purpose.

<sup>8</sup> J. T. Baker Analyzed Reagent 9268, or its equivalent, is suitable for this purpose.



<sup>C</sup> Unsaturated compounds shall be determined by the method for determining olefins described in Method D 875.

<sup>D</sup> Odor test: Immerse 1 in. of a strip of white unglazed blotting paper, approximately 1 by 4 by 0.166 in. in size, in the petroleum ether for 30 s, remove the strip, and allow to dry at room temperature in still air for 12 min.

<sup>E</sup> Aromatic compounds: Add 5 drops of petroleum ether to 40 drops of sulfuric acid ( $\text{H}_2\text{SO}_4$ , sp gr 1.84) and 10 drops of nitric acid ( $\text{HNO}_3$ , sp gr 1.42) in a test tube, warm for 10 min, allow to cool for 30 min, transfer to a shallow dish, and dilute with water.

10.4 *Phenolphthalein Indicator Solution* (10 g/litre)—Dissolve 1 g of phenolphthalein in 50 mL of neutral ethyl alcohol (95 %) and then mix with 50 mL of water (see 10.1).

10.5 *Sodium Hydroxide, Standard Solution* (0.1 N)—Prepare and standardize a 0.1 N sodium hydroxide (NaOH) solution.

10.6 *Sodium Sulfate* ( $\text{Na}_2\text{SO}_4$ ), anhydrous.

10.7 *Sulfuric Acid, Standard* (0.5 N)—Prepare and standardize a 0.5 N sulfuric acid ( $\text{H}_2\text{SO}_4$ ) solution.

10.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ).

10.9 *Sulfur Acid* (1+1)—Gradually pour 10 g of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ) (sp gr 1.84) onto 10 g of cracked ice made from distilled water, gently swirling the mixing vessel; or gradually pour the acid down the sides of the mixing vessel into an equal weight of water, swirling gently, while submersing the vessel in an ice bath.

## 11. Procedure

11.1 Weigh  $2 \pm 0.001$  g of the sample into a tared 250-mL beaker. Add 25 mL of water and 25 mL of neutral ethyl alcohol (95 %), and warm on the steam bath until solution is complete. Cool, add 5 drops of methyl orange indicator solution, and titrate with 0.5 N  $\text{H}_2\text{SO}_4$  to a pink color. Add 5 mL of  $\text{H}_2\text{SO}_4$  (1+1) in excess.

11.2 Transfer the contents of the beaker to a 250-mL extraction cylinder or a Stokes flask, equipped with a siphon. Wash the beaker alternately with equal parts of hot water and hot ethyl alcohol (95 %), adding the washings to the extraction cylinder or Stokes flask. Keep the total volume for extraction under 160 mL in the extraction cylinder, or within the constricted portion of the Stokes flask. Wash the beaker with a small amount of petroleum ether to remove any traces of fatty acids and fatty matter and add to the extraction cylinder or Stokes flask. Cool the cylinder or flask under tap water to a temperature not to exceed 25°C. Add 50 mL of petroleum ether and allow to stand for  $\frac{1}{2}$  h without shaking. Remove the greater part of the fatty acids by drawing off the petroleum ether layer as closely as possible, by means of a glass siphon, into a 500-mL separatory funnel. Repeat the extractions five more times with petroleum ether, using 50-mL portions, and shaking the cylinder thoroughly each time.

NOTE 3—If an emulsion appears at this point, it may be broken by the addition of 10 g of anhydrous  $\text{Na}_2\text{SO}_4$ .

11.3 Combine the petroleum ether extracts and wash with small portions of distilled water until the water washings are no longer acid to methyl orange indicator solution. Dry the combined, washed, petroleum ether extracts with anhydrous  $\text{Na}_2\text{SO}_4$ , and filter through paper into the original tared 250-mL beaker. Wash the separatory funnel with two small portions of petroleum ether, filtering and adding the washings to the beaker.

11.4 Evaporate the petroleum ether extract on the steam bath until about 1 mL remains. Then swirl manually until the last trace of solvent evaporates and the odor of petroleum ether is no longer perceptible. Cool in a desiccator and weigh as total fatty matter, which is defined as fatty and rosin acids plus unsaponified and unsaponifiable fatty matter.

11.5 Dissolve the total fatty matter in 50 mL of neutral ethyl alcohol (95 % v) with warming. Add phenolphthalein indicator and titrate with 0.1 NaOH solution to a pink end point.

## 12. Calculations

12.1 Calculate the percentage of anhydrous, salt-free, soda soap as follows:

$$A = G - F \quad (1)$$

$$G = [(VN \times 0.022) + E]/W \times 100$$

where:

- A = weight percent of anhydrous, salt-free, soda soap,
- G = weight percent of soda soap plus unsaponified and unsaponifiable fatty matter,
- F = weight percent of unsaponified and unsaponifiable fatty matter (Section 39),
- V = millilitres of NaOH solution used in titration (11.5),
- N = normality of NaOH solution,
- E = grams of extract (11.4), and
- W = grams of sample (11.1), and
- 0.022 = net gain in milliequivalent weight from the conversion of the fatty acid to the sodium salt by replacement of a proton with a sodium ion.

## ALCOHOL-SOLUBLE MATTER

### 13. Reagents

13.1 *Ethyl Alcohol, Neutral* (95 %)—Freshly boiled, reagent grade, ethyl alcohol, 95 % or higher, neutral to phenolphthalein, and containing only volatile denaturants, 95 mL<sup>7</sup> plus 5 mL of water.

13.2 *Ethyl Alcohol, Neutral* (absolute)—Freshly boiled absolute ethyl alcohol, neutral to phenolphthalein.<sup>7</sup>

### 14. Procedure

14.1 Weigh  $2 \pm 0.001$  g of the sample into 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 a glass wool pad, with suction into a tared 300-mL Erlenmeyer flask, retaining as much of the residue as possible in the beaker. Repeat this extraction three times with 25-mL 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 if sufficient). Reprecipitate the alcohol-insoluble matter by slowly adding, while stirring vigorously, 50 mL of neutral ethyl alcohol (absolute).

NOTE 4—Solution and reprecipitation of alcohol-insoluble matter is necessary for complete separation from alcohol-soluble matter.

14.2 Heat the solution to boiling on the steam bath, filter, and transfer the precipitate quantitatively to the Gooch crucible, washing several times with neutral ethyl alcohol (95 %). Evaporate the combined filtrate and washings in the Erlenmeyer flask on the steam bath, and then dry to constant weight at  $105 \pm 2^\circ\text{C}$ . Calculate total alcohol-soluble matter.

14.3 Reserve the flask and contents for the determination of free fatty matter (Section 37). Reserve the Gooch crucible and contents, without drying, for the determination of matter insoluble in water (Section 15) and total alkalinity of matter insoluble in alcohol (alkaline salts) (Section 17).

## MATTER INSOLUBLE IN WATER

### 15. Procedure

15.1 Wash the alcohol-insoluble matter retained in the Gooch crucible (Section 14) thoroughly with hot water until the washings are no longer alkaline to phenolphthalein. Reserve the filtrate for determination of total alkalinity of matter insoluble in alcohol (Section 17). Dry the crucible and residue to constant weight at  $105 \pm 2^\circ\text{C}$  and calculate the percentage of matter insoluble in water.

## TOTAL ALKALINITY OF MATTER INSOLUBLE IN ALCOHOL (ALKALINE SALTS)

### 16. Reagents

16.1 *Hydrochloric Acid*—Prepare and standardize a 1 N hydrochloric acid solution.

16.2 *Methyl Orange Indicator Solution* (1 g/litre)—Dissolve 0.1 g of methyl orange in water and dilute to 100 mL.

### 17. Procedure

17.1 Titrate the water solution obtained in the determination of matter insoluble in water (Section 15) with the standard 1 N hydrochloric acid solution of 16.1, using methyl orange as indicator.

17.2 Calculate the alkalinity as sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) as follows:

$$\text{Na}_2\text{CO}_3, \% \text{ weight} = 106 \text{ VN}/20 \text{ W} \quad (2)$$

where:

V = millilitres of standard acid used,

N = normality of standard acid, and

W = weight of sample (14.1), g

## SODIUM SILICATE

### 18. Reagents

18.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

18.2 *Hydrofluoric Acid* (sp gr 1.15)—Prepare a solution of hydrofluoric acid (HF) having a specific gravity of 1.15.

18.3 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ).

### 19. Procedure

19.1 When the material contains no mineral matter that is insoluble in water, ignite a portion of the sample containing not

to exceed 0.2 g of silica ( $\text{SiO}_2$ ) in a platinum dish (Note 5) at a low temperature. When charred, extract the soluble salts with water, return the paper and charred residue to the dish, and complete the ignition. Unite the residue in the dish and the water extract, carefully acidify with HCl, and finally add the equivalent of from 5 to 10 mL of HCl in excess. The dish or casserole containing the solution should be covered with a watch glass while adding acid, so as to avoid loss by spray.

NOTE 5—When phosphates are present, a platinum dish should not be used.

19.2 When the material contains mineral matter insoluble in water, or a determination of highest accuracy is not necessary, take a portion of the solution after titrating the matter insoluble in alcohol (Section 17) containing not more than 0.2 g of  $\text{SiO}_2$  and add 5 to 10 mL of HCl.

19.3 Evaporate the acidified solution obtained in accordance with 19.1 or 19.2 (washing off and removing the cover glass if used) to dryness on a steam bath or hot plate at a temperature not exceeding  $120^\circ\text{C}$ . Cool, moisten with HCl and let stand 5 to 10 min, breaking up all lumps with a stirring rod. Add about 25 mL of hot water. Heat a few minutes and filter through a small ashless paper. Wash thoroughly with hot water.

19.4 Evaporate the filtrate to dryness and proceed as described in 19.3, filtering on a second paper. Carefully ignite the two papers and contents in a weighed platinum crucible, first at a low temperature until the paper is consumed, then over a blast lamp. Cool in a desiccator, weigh, and repeat until constant weight is obtained.

19.5 If extreme accuracy is desired, moisten the weighed contents of the crucible with water, add 10 mL of HF and 4 drops of concentrated  $\text{H}_2\text{SO}_4$ , and evaporate to dryness over a low flame. Ignite at the temperature of the blast lamp for about 2 min, cool in a desiccator, and weigh.

### 20. Calculation

20.1 Calculate the sodium silicate having the ratio 1  $\text{Na}_2\text{O}$ :3.25  $\text{SiO}_2$  as follows:

$$\text{Sodium silicate, \% w} = (A - B) \times 1.308 \quad (3)$$

where:

A = grams of ignited residue before treatment with HF (19.4), and

B = grams of ignited residue after treatment with HF (19.5).

## PHOSPHATES

### 21. Application

21.1 This test method is applicable to any species of alkali metal phosphates free of interfering ions. This test method can be used for the analysis of soap and synthetic detergent builders if the sample is properly prepared (see Section 26). This test method does not apply when the level of phosphate present is equivalent to or less than 2 %  $\text{P}_2\text{O}_5$ .

### 22. Summary of Test Method

22.1 All of the phosphate present is converted, by acid hydrolysis, to the ortho form and titrated between pH 4.3 and 8.8 with NaOH solution.



## 23. Interferences

23.1 Heavy metals such as iron, aluminum, calcium, magnesium, etc., that will precipitate, either as insoluble phosphates or hydroxides, before the upper end point is reached, will interfere. Interference also occurs if borates, sulfites, carbonates, or other buffering materials are present. The last two compounds and some of the borate will be expelled during the acid hydrolysis boil. Borate is removed by conversion to methyl borate and subsequent volatilization. Ammonia or other weak bases also will interfere. The most common interference is from silicic acid. Experiment and experience in analysis of spray-dried synthetics have shown that unless the ratio of the percentage of  $\text{SiO}_2$  to the percentage of  $\text{P}_2\text{O}_5$  approaches or exceeds 0.2, the interference by silicates will be so slight that it may be neglected. Larger amounts must be dehydrated as directed, but need not be removed by filtration during preparation of the sample.

## 24. Apparatus

24.1 *Electrometric Titration Apparatus*, equipped with glass and calomel electrodes. Any standard pH meter, capable of performing titrations accurate to  $\pm 0.1$  pH and accurately standardized at pH 4.0 and 8.0 is suitable.

24.2 *Gas Burners*, suitable for heating the sample to approximately  $550^\circ\text{C}$ .

24.3 *Muffle Furnace*, with suitable pyrometer and controls for maintaining temperatures up to  $550^\circ\text{C}$ .

24.4 *Motor Stirrer*, air or electric.

## 25. Reagents

25.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

25.2 *Mixed Indicator* (optional)—Prepare the following solutions:

25.2.1 *Methyl Orange Solution* (0.5 g/litre)—Dissolve 0.05 g of methyl orange in water and dilute to 100 mL.

25.2.2 *Phenolphthalein, Alcohol Solution* (5.0 g/litre)—Dissolve 0.50 g of phenolphthalein in alcohol (50 %) and dilute to 100 mL with alcohol.

25.2.3 *Thymol Blue Solution* (0.4 g/litre)—Dissolve 0.04 g of thymol blue in water and dilute to 100 mL.

25.2.4 *Methylene Blue Solution* (1.0 g/litre)—Dissolve 0.10 g of methylene blue in water and dilute to 100 mL.

25.2.5 *Alcohol* (90 %)—Alcohol (90 %) prepared from alcohol conforming to Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue. Mix the solutions in the following proportions:

Methyl orange solution	32 mL
Phenolphthalein solution	32 mL
Thymol blue solution	8 mL
Methylene blue solution	4 mL
Alcohol	24 mL

The individual components are stable indefinitely. The mixed indicator should be prepared at least weekly. In practice, 3 mL of this mixed indicator are used in a final volume of approximately 250 mL of solution to be titrated. The lower end point is taken as the first change from gray to a definite green; the upper end point is the change from pink to a bright purple.

25.3 *Sodium Hydroxide, Standard Solution* (0.5 or 1.0 N)—Prepare a 0.5 or 1.0 N carbonate-free solution of sodium hydroxide (NaOH) and standardize accurately.

25.4 *Sodium Hydroxide Solution* (1+1)—Dissolve sodium hydroxide (NaOH) in an equal weight of water. When using, decant the solution from the settled carbonate. A more dilute solution may be used. NaOH solutions must be protected from carbon dioxide ( $\text{CO}_2$ ) contamination.

## 26. Preparation of Sample Solutions

26.1 Commercial sodium or potassium phosphates need no special preparation except solution in water. Weigh a portion of the well-mixed sample to the nearest 0.001 g, transfer directly to a 400-mL beaker, and dissolve in about 100 mL of water. Neutralize to litmus paper with HCl (sp gr 1.19) and add 10 mL excess. The optimum size of sample is given by the formula:

$$\text{Grams of sample} = (N \times 280) / P \quad (4)$$

where:

$N$  = normality of the NaOH solution to be used in the titration, and

$P$  = percentage of  $\text{P}_2\text{O}_5$  expected in the sample.

26.2 Soap products may be analyzed by using the filtrate from the silicon dioxide ( $\text{SiO}_2$ ) determination. Use care not to exceed the sample weight prescribed in 26.1. Alternatively the sample may be prepared as described in 26.3.

26.3 Built synthetic detergent samples are treated by ashing. Weigh a sample of suitable size (but not to exceed 10 g) to the nearest 0.001 g. When the expected percentage of  $\text{P}_2\text{O}_5$  in the sample is known, the equation in 26.1 may be used to calculate a suitable sample weight. Place the sample in a 400-mL high-silica glass beaker, or a new, well-glazed porcelain or silica evaporating dish or large crucible and ignite gently over a low gas burner until most of the volatile combustible matter is burned off. Use care so that the sample is not heated to above  $550^\circ\text{C}$  with the gas burner. Transfer to a muffle, operated at not over  $550^\circ\text{C}$ , for 10 to 15 min. The ignited residue need not be free from carbon and usually is of a grayish color. Cool and add cautiously 10 mL of HCl. Evaporate to dryness on open steam. If the ratio of the percentage of  $\text{SiO}_2$  to the percentage of  $\text{P}_2\text{O}_5$  approaches or exceeds 0.2 or is unknown, dehydrate the silicates completely by cooling the sample and repeating the HCl addition and evaporation two additional times. After the third evaporation, continue to heat the residue for an additional 15 to 20 min after dryness is attained to ensure complete dehydration of  $\text{SiO}_2$ . After the sample appears dry, last traces of water or HCl may be removed in a 110 to  $120^\circ\text{C}$  oven, if desired, in this step. Cool the sample and transfer into a 400-mL beaker using distilled water and proceed as in 26.3.1 or 26.3.2.

26.3.1 If the sample contains perborate or borate, evaporate to dryness on a steam bath, add about 200 mL of methanol, 10 mL of HCl and two or three hollow glass beads. Partially cover the beaker with a watch glass and boil down to a volume of about 20 mL. (The boiling time must be at least 30 min.) Evaporate down to less than 10 mL on a steam bath under a stream of nitrogen or clean, dry air. Proceed as described in Section 27.

26.3.2 If the sample is known to be free from perborate and borate, add distilled water to make a total of about 90 mL. Add 10 mL of HCl and proceed as in Section 27.

## 27. Procedure

27.1 Each solution in a 400-mL beaker, prepared as described in 26.1, should have a volume of about 100 mL and contain an excess of at least 10 mL of HCl (sp gr 1.19). Cover with a watch glass and boil gently for a minimum of 30 min. Up to 60 min may be necessary for phosphates of the glass type. All phosphate must be in the ortho form. Cool to room temperature (20 to 30°C).

27.2 Dilute to 200 mL, place on an electrometric titration stand (Note 6), and neutralize to a pH of 4.3. Most of the neutralization may be made with NaOH solution (1+1), but final adjustment should be made with the standard NaOH solution (0.5 or 1.0 *N*) to be used in the titration. Cool again, if necessary, to maintain the temperature below 30°C. Titrate carefully to the upper end point (pH 8.8) recording the titration between end points (*T*).

NOTE 6—The mixed indicator may be used for this titration but with some small sacrifice of accuracy. If the samples have been prepared by the ignition method, they must be filtered and the paper washed thoroughly, after the acid hydrolysis, as particles of carbon obscure the visual end point. The color changes can be checked by comparison with pH meter readings to acquire familiarity with the exact shade required. For greatest accuracy, titration with a pH meter is recommended.

## 28. Calculation

28.1 Calculate the percentage of total  $P_2O_5$  as follows:

$$\text{Total } P_2O_5, \% \text{ weight} = (TN \times 7.098)/W \quad (5)$$

where:

*T* = millilitres of NaOH solution required for titration of the sample,

*N* = normality of the NaOH solution, and

*W* = grams of sample in the sample solution.

## PHOSPHORUS (COLORIMETRIC METHOD USING MOLYBDENUM BLUE)

## 29. Summary of Test Method

29.1 The sample is dissolved in water and the phosphates hydrolyzed to orthophosphate with mineral acid. A diluted aliquot of the hydrolyzed sample is reacted with a single-solution molybdate reagent to form the molybdenum blue complex with the ortho-phosphate present. After extraction into isobutanol to remove any interference from product colorants or turbidity, the molybdenum blue complex is measured colorimetrically.

## 30. Scope

30.1 This procedure must be used to determine the level of phosphate present in detergents when the level present is equal to or less than that equivalent to 2 %  $P_2O_5$ . Results are reported as percent weight P.

## 31. Reagents

31.1 The single-solution molybdate reagent is prepared from solutions of sulfuric acid, ammonium molybdate, potassium antimonyltartrate, and ascorbic acid as follows:

31.1.1 *Solution A, Sulfuric Acid*—Cautiously add, with cooling, 140 mL of concentrated sulfuric acid ( $H_2SO_4$ , sp gr 1.82) to 900 mL of water. Cool to room temperature.

31.1.2 *Solution B, Ammonium Molybdate*—Dissolve 15.0 g of ammonium molybdate in 500 mL of water. Store in the dark.

31.1.3 *Solution C, Ascorbic Acid*—Dissolve 13.5 g of ascorbic acid in 250 mL of water. Store at 4°C. Discard after 1 week.

31.1.4 *Solution D, Potassium Antimonyltartrate*—Dissolve 0.35 g of potassium antimonyltartrate ( $C_4H_4KO_7Sb$ ) in 500 mL of distilled water. Store at 4°C in the dark.

31.2 Mix thoroughly 125 mL of Solution A, 50 mL of Solution B, 50 mL of Solution C, and 25 mL of Solution D. The resulting single-solution molybdate reagent should be yellow in color. Allow to reach room temperature prior to use. A greenish or blue color indicates phosphate contamination, in which case the solution should be discarded. The single-solution molybdate reagent must be prepared fresh daily. The individual solutions are stable as stated above. *Add solutions in the order specified* “A” 1st, “B” 2nd, “C” 3rd, and “D” 4th.

31.3 *Potassium dihydrogen phosphate (anhydrous)*, reagent grade.

## 32. Calibration

32.1 A phosphate stock solution for calibration may be prepared by dissolving in a 1000-mL volumetric flask 0.4394 ± 0.0002 g of potassium dihydrogen phosphate (anhydrous) ( $KH_2PO_4$ ) which has been previously dried at 105°C for 1 h. This stock solution contains ortho-phosphate equivalent to 100 µg P/mL.

32.2 Make a dilute stock solution containing 1.00 µg P/mL by pipeting 10 mL of the original stock solution into a 1-L volumetric flask and diluting to volume with water.

32.3 Transfer by pipet 10, 20, 30, 40, and 50-mL aliquots of the dilute stock solution to separate 250-mL separatory funnels containing about 50 mL of water. Add additional water to each separatory funnel to bring the total volume to 100 mL. Use as a blank 100 mL of water added to an additional separatory funnel.

32.4 To each calibration point and the blank add 20 mL of mixed reagent from a dispensing flask or graduated cylinder; stopper and shake the separatory funnel vigorously to mix well. Let stand for 10 min but no longer than 15 min. (The formation of the molybdenum blue complex is not instantaneous; however color formation is essentially complete in the first 2 to 3 min.) Add 40 mL of isobutanol using a graduated cylinder and shake for 60 ± 10 s. Let stand 5 min but no longer than 10 min to allow the layers to separate. Drain off and discard the aqueous layer; drain the isobutanol layer into a 50-mL volumetric flask, washing down the walls of the separatory funnel with 5 mL of ethanol. Dilute the extract to volume with ethanol and mix well. Determine the absorbance of the extract at 690 nm in a 1-cm spectrophotometric cell versus distilled water as a reference. Absorbance measurement should be made within 1 h of color development.

32.5 Plot a calibration curve of absorbance values determined versus micrograms of P present where the water blank represents 0 and the 10, 20, 30, 40, and 50-mL aliquots of dilute stock solution represent 10, 20, 30, 40, and 50 µg of P, respectively. The curve should be linear with an intercept of + 0.01 – 0.03 absorbance units.

### 33. Procedure for Detergent Samples

33.1 Using the following table, weigh out accurately to  $\pm 0.001$  g a sample of well mixed, riffled, detergent material into a 50-mL beaker:

Estimated % Weight P	Sample Weight, g	Volumetric, mL	Aliquot, mL
0 to 1	10.0	100	5
1 to 2	10.0	1000	10

Transfer to a 1-L volumetric flask, adding water to a total volume of approximately 500 mL. Carefully, and with gentle mixing, add 50 mL of concentrated sulfuric acid to the sample solution. If *carbonates* are present, take care during acid addition so that evolution of CO<sub>2</sub> does not cause the sample to foam out of the neck of the flask. After acid addition, rinse down the neck of the flask with water, mix gently, and place on a hot steam bath for 1 h. Remove, cool, and dilute to volume with water. Mix well. Dilute the hydrolyzed sample solution by pipeting an aliquot into a volumetric flask. Use an aliquot and flask size as specified in the table above as a guide. Other combinations of aliquots and flask sizes may be used as desired. The diluted sample solution should contain about 20 to 40 µg P/10 mL (2 to 4 µg/mL). Mix well. Pipet 10 mL of the diluted sample solution into a 250-mL separatory funnel containing 90 mL of water. Add 2 to 3 drops of phenolphthalein indicator solution and 3 to 4 drops of 50 % NaOH solution. Add 1 N H<sub>2</sub>SO<sub>4</sub> dropwise until the sample is just colorless. Proceed as described in 32.4, starting with “Add 20 mL of the mixed reagent from a dispensing flask or .....”

### 34. Calculations

34.1 Calculate the percent weight P present in the sample from the sample weight, dilution and aliquot volumes, and micrograms of phosphorus found in the final aliquot as determined from the calibration curve of phosphorus as follows:

$$A = (B \times V)/(W \times C \times 100) \quad (6)$$

where:

- A = weight percent of phosphorus,
- B = micrograms of phosphorus, (found using calibration curve),
- V = volume, mL, (see 33.1),
- W = sample weight, g, and
- C = aliquot, mL.

34.2 Round off and report data to two decimals; report any value less than 0.005 % w P as 0.00 % w; report 0.007 % w P

as 0.01 % w P, etc. Duplicate runs which agree within 8.5 % relative are acceptable for averaging (95 % confidence level).<sup>9</sup>

34.3 To convert from % w P to % w P<sub>2</sub>O<sub>5</sub>, multiply the % w P by 2.29.

34.4 *Repeatability (Single Analyst)*—The coefficient of variation of results (each the average of duplicate determinations), obtained by the same analyst on different days, was estimated to be 2.9 % relative at 14 degrees of freedom. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 5.7 % relative.

34.5 *Reproducibility (Multilaboratory)*—The coefficient of variation of results, (each the average of duplicate determinations), obtained by analysts in different laboratories, has been estimated to be 4.3 % relative at 12 degrees of freedom. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 13.1 % relative.

## UNSAPONIFIED AND UNSAPONIFIABLE FATTY MATTER

NOTE 7—In the case of superfatted soaps, free fatty acids, which are the superfatting agents in highest percentage, plus this unsaponified and unsaponifiable matter, constitute the major portion of the superfatting agents used.

### 35. Summary of Test Method

35.1 Unsaponified and unsaponifiable fatty matter plus free rosin and free fatty acids in the alcohol-soluble matter are extracted with petroleum ether from a 50/50 volume percent solution of ethanol and water. The acids are then removed with NaOH wash (38.3). If it is desired to include these acids in a calculation for free fatty matter, proceed to Section 40.

### 36. Apparatus

36.1 *Extraction Cylinder or Stokes Flask, and Siphon*—See Section 9.

### 37. Reagents

37.1 *Ethyl Alcohol, Neutral (95 %)*—Freshly boiled ethyl alcohol, 95 % or higher and neutral to phenolphthalein, (see 7.1).

37.2 *Petroleum Ether*—See 10.3.

37.3 *Phenolphthalein Indicator*—Prepare a 1 % solution in neutral ethyl alcohol (95 %) (see 36.1).

37.4 *Sodium Hydroxide, Standard Solution (0.2 N)*—Prepare and standardize a 0.2 N sodium hydroxide (NaOH) solution.

37.5 *Sodium Sulfate, (Na<sub>2</sub>SO<sub>4</sub>), anhydrous.*

### 38. Procedure

38.1 Dissolve the alcohol-soluble matter obtained in accordance with Section 14 in a mixture of 25 mL of water and 25 mL of neutral ethyl alcohol (95 %), warming if necessary.

<sup>9</sup> Data supporting the precision statements are available at ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Request RR: D12-1006.



Transfer the solutions to a 250-mL extraction cylinder or a Stokes flask, equipped with siphons. Wash the Erlenmeyer flask alternately with equal parts of hot water and hot neutral ethyl alcohol (95 %), adding the washings to the extraction cylinder or Stokes flask. Keep the total volume for extraction under 160 mL in the extraction cylinder, or within the constricted portion of the Stokes flask. Wash the Erlenmeyer flask with a small amount of petroleum ether to remove any traces of fatty matter and add to the extraction cylinder or Stokes flask.

38.2 Cool the cylinder or flask under tap water to a temperature not to exceed 25°C. Add 50 mL of petroleum ether and shake. Draw off the petroleum ether layer as closely as possible, by means of a glass siphon, into a separatory funnel of 500-mL capacity. Repeat the extractions six more times with petroleum ether, using 50-mL portions, shaking the cylinder thoroughly each time.

NOTE 8—If an emulsion appears at this point, it may be broken by adding 10 g of anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ).

38.3 Combine the petroleum ether extracts and wash four times with 10-mL portions of 0.2 *N* NaOH solution adding the washings to the alcoholic solution which shall be reserved for the determination of chlorides (Section 40). Finally, wash the petroleum ether extract with small portions of water until the water washings are no longer alkaline to phenolphthalein. Transfer the washed petroleum ether extract to a tared 300-mL Erlenmeyer flask, washing the separatory funnel with two small portions of petroleum ether.

38.4 Evaporate on a steam bath until about 1 mL remains. Swirl manually until all solvent is evaporated and the odor of petroleum ether is no longer perceptible. Cool in a desiccator and weigh.

### 39. Calculation

39.1 Calculate the percentage of unsaponified and unsaponifiable fatty matter as follows:

$$F = E \times 100/W \quad (7)$$

where:

F = weight percent of unsaponified and unsaponifiable fatty matter,

E = weight of dried petroleum ether extract (38.4), and

W = weight of sample in grams (14.1).

### 40. Free Fatty Matter

40.1 The free fatty matter is calculated as the percentage of unsaponified and unsaponifiable fatty matter (see 39.1) plus the weight percentage of free rosin and free fatty acids (see Section 8).

## CHLORIDES IN ALCOHOL-SOLUBLE MATTER

### 41. Reagents

41.1 *Calcium Carbonate* ( $\text{CaCO}_3$ ) chloride-free.

41.2 *Magnesium Nitrate Solution* (200 g/L)—Dissolve 200 g of chloride-free magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) in 1 L of water.

41.3 *Potassium Chromate Indicator Solution*—Dissolve 5 g of chloride-free potassium chromate ( $\text{K}_2\text{CrO}_4$ ) in water and add 0.1 *N*  $\text{AgNO}_3$  solution until a slight red precipitate is produced. Filter the solution, and dilute the filtrate to 100 mL.

41.4 *Silver Nitrate, Standard Solution* (0.1 *N*)—Prepare and standardize a 0.1 *N* silver nitrate ( $\text{AgNO}_3$ ) solution.

### 42. Procedure

42.1 To the alcoholic solution remaining after the determination of fatty matter (Section 38), add 15 mL of  $\text{Mg}(\text{NO}_3)_2$  solution. Heat on the steam bath until the precipitate is coagulated, filter, and wash with water into a 500-mL beaker.

42.2 Add 1 mL of  $\text{K}_2\text{CrO}_4$  indicator for every 100 mL of solution. Titrate the solution containing the sample with 0.1 *N*  $\text{AgNO}_3$  solution until the red color formed by each drop begins to disappear more slowly upon stirring, showing that most of the chloride has been precipitated.

42.3 Prepare a blank by adding to another 500-mL beaker the same volume of distilled water,  $\text{Mg}(\text{NO}_3)_2$  solution, and  $\text{K}_2\text{CrO}_4$  indicator present in the beaker containing the sample. Add enough calcium carbonate ( $\text{CaCO}_3$ ) to the blank, so that the turbidity in both solutions appears to be the same when they are swirled. Using the blank for comparison, continue the titration of the solution containing the sample until a faint but distinct change of color occurs. At the end point the color should not be dark, just distinctly different (reddish yellow) from that of the blank containing no silver chromate.

42.4 Add to the blank enough 0.1 *N*  $\text{AgNO}_3$  solution so that the colors of the two solutions exactly match; this is the “blank” titration required to produce the end point.

### 43. Calculation

43.1 Calculate the percentage of chlorides (as NaCl) in alcohol-soluble matter as follows:

$$C = [(V_1 - V_2)N \times 0.0585]/W \times 100 \quad (8)$$

where:

C = weight percent of chlorides (as NaCl) in alcohol-soluble matter,

$V_1$  = millilitres of  $\text{AgNO}_3$  solution required for titration of the sample (42.3),

$V_2$  = millilitres of  $\text{AgNO}_3$  solution required for titration of the blank (42.4),

*N* = normality of the  $\text{AgNO}_3$  solution, and

W = weight of sample, g.

## ROSIN (McNICOLL METHOD)<sup>10</sup>

### 44. Apparatus

44.1 The apparatus required consists of a glass flask connected, preferably by a ground-glass joint, to a reflux condenser.

<sup>10</sup> Cox and Evers, “Report of British Standards Committee,” *Analyst*, Vol 62, No. 741, pp 865–870 (1937); also McNicoll, D., “The Estimation of Rosin Acids in Fatty Mixtures,” *Journal, Soc. Chemical Industry*, Vol 40, p. 124 T (1921).



44.1.1 *Esterification Flask*—A 150-mL flask of either the round-bottom or Erlenmeyer type shall be used.

44.1.2 *Reflux Condenser*—Any suitable water-cooled, glass reflux condenser may be used.

## 45. Reagents

45.1 *Naphthalene-β-Sulfonic Acid Solution*—Dissolve 40 g of Eastman grade or equivalent reagent in 1 L of absolute methyl alcohol.

45.2 *Phenolphthalein Indicator Solution*—Prepare a 1 % solution in neutral ethyl alcohol (95 %) (see 10.1).

45.3 *Potassium Hydroxide, Standard Alcoholic Solution* (0.2 N)—Accurately standardize a 0.2 N solution of potassium hydroxide (KOH) in neutral ethyl alcohol (95 %) (see 10.1). Due to volatility of alcohol, this solution should be restandardized frequently.

## 46. Procedure

46.1 *Preparation of Total Fatty Matter*—Prepare total fatty matter for the rosin determination in accordance with the extraction procedure described in 11.1-11.4, using enough sample to yield approximately 5 g of total fatty matter.

46.2 *Esterification and Titration*—Weigh  $2 \pm 0.001$  g of the total fatty matter into the esterification flask. Add 25 mL of naphthalene-β-sulfonic acid solution. Add a few glass beads to ensure smooth boiling, attach the reflux condenser, and boil for 30 min; also, run a blank test using 25 mL of the reagent. At the end of the boiling period cool the contents of the flask, add 0.5 mL of phenolphthalein indicator, and titrate immediately with 0.2 N alcoholic KOH solution.

## 47. Calculations

47.1 Calculate the results as follows (Note 9):

$$R = [(V_1 - V_2)N \times 0.346/W_1] \times 100 \quad (9)$$

$$R_1 = R - 1.0$$

$$B = E/W_2 \times 100$$

$$R_2 = (R_1 \times B)/100$$

$$R_s = R_2 \times 1.064$$

where:

$R$  = weight percent of rosin in total fatty matter,

$R_1$  = corrected weight percent of rosin in total fatty matter (Note 10),

$R_2$  = weight percent of rosin on basis of original sample,

$R_s$  = weight percent of rosin-soda soap on basis of original sample,

$V_1$  = millilitres of KOH solution required for titration of sample,

$V_2$  = millilitres of KOH solution required for titration of blank,

$N$  = normality of KOH solution,

$W_1$  = grams of sample (46.2),

$B$  = weight percent of total fatty matter,

$E$  = grams of extract (11.3), and

$W_2$  = grams of sample used in preparation of total fatty matter (46.1).

NOTE 9—In all cases where the rosin content is found to be less than 5 %, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test, as follows:

tively by the Liebermann-Storch test, as follows:

Transfer 1 to 2 mL of the sample of fatty acids plus fatty matter to a test tube, add 5 to 10 mL of acetic anhydride, and warm on a steam bath. After cooling, pour 1 to 2 mL into a white porcelain dish and allow a drop or two of sulfuric acid ( $H_2SO_4$ , sp gr 1.53) to run down the side of the vessel. (The  $H_2SO_4$  (sp gr 1.53) is prepared by diluting 34.7 mL of  $H_2SO_4$  (sp gr 1.84) with 35.7 mL of water.) If rosin is present, a fugitive violet coloration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. The test should be checked with a sample of fatty acids plus fatty matter to which a small amount of rosin has been added.

NOTE 10—Cooperative studies have shown that the McNicoll method gives results approximately 1 % higher than the amount of rosin present. Consequently, the committee recommends deducting 1 % from the percentage of rosin found in the fatty acids plus fatty matter.

47.2 If true fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap.

## SYNTHETIC DETERGENT (BY DIFFERENCE)

### 48. Calculation

48.1 Calculate the percentage of anhydrous, salt-free, synthetic detergent as follows:

$$D = K - (A + F + C) \quad (10)$$

where:

$D$  = weight percent of anhydrous, salt-free synthetic detergent,

$K$  = weight percent of alcohol-soluble matter (Section 14),

$A$  = weight percent of anhydrous, salt-free, soda soap (Section 12),

$F$  = weight percent of free fatty matter (Section 40), and

$C$  = weight percent of chlorides (as NaCl) in alcohol-soluble matter (Section 43).

48.2 In many cases, actual identification and a more accurate determination of the percentage of synthetic detergent than that provided by 48.1 will be required. A complete listing of analytical methods for the isolation of anionic, cationic, and nonionic surfactants is beyond the scope of this standard; see Test Methods D 1768 and D 3049, Test Method D 2358, and Classification D 2357 for this information. Other applicable standards may be found by consulting the annual index.

## NEUTRAL INORGANIC SALTS

### 49. Calculation

49.1 Calculate the percentage of neutral inorganic salts as follows:

$$S = (100 + C) - (M + K + I + S_a) \quad (11)$$

where:

$S$  = weight percent of neutral, inorganic salts,

$C$  = weight percent of chlorides (as NaCl) in alcohol-soluble matter (Section 43),

$M$  = weight percent of moisture and other matter volatile at 105°C (Section 6),

$K$  = weight percent of alcohol-soluble matter (Section 14),

$I$  = weight percent of matter insoluble in water (Section 15), and  $S_a$  = total alkalinity of matter insoluble in alcohol (alkaline salts) (Section 17).

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