



Standard Test Methods for Chemical Analysis of Soaps Containing Synthetic Detergents¹

This standard is issued under the fixed designation D820; 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 and 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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:*²

¹ 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 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.

D216 Method of Test for Distillation of Natural Gasoline (Withdrawn 1988)³

D459 Terminology Relating to Soaps and Other Detergents

D875 Method for Calculating of Olefins and Aromatics in Petroleum Distillates from Bromine Number and Acid Absorption (Withdrawn 1984)³

D1193 Specification for Reagent Water

D1768 Test Method for Sodium Alkylbenzene Sulfonate in Synthetic Detergents by Ultraviolet Absorption

D2357 for Qualitative Classification of Surfactants by Infra-red Absorption

D2358 Test Method for Separation of Active Ingredient from Surfactant and Syndet Compositions

D3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration

3. Terminology

3.1 *Definitions:*

3.2 The term *synthetic detergent* in these test methods is defined in accordance with Terminology D459, 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 D459.

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.⁴ Other grades may be used, provided it is first ascertained that the reagent is of

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ *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.

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 D1193.

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.

6. Procedure

6.1 Weigh 5 ± 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,⁵ 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 ± 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,⁵ 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.

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°C.⁶

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

^A The distillation test shall be made in accordance with Method D216. 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.

^B 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.

^C Unsaturated compounds shall be determined by the method for determining olefins described in Method D875.

⁶ J. T. Baker Analyzed Reagent 9268, or its equivalent, is suitable for this purpose.

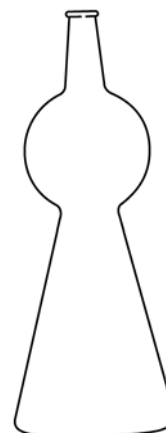


FIG. 1 Stokes Flask

⁵ Fischer Scientific A962, or its equivalent, is suitable for this purpose.

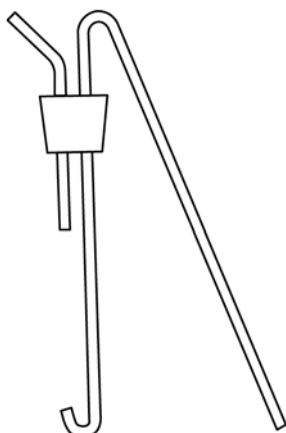


FIG. 2 Siphon

^D 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.

^E Aromatic compounds: Add 5 drops of petroleum ether to 40 drops of sulfuric acid (H₂SO₄, sp gr 1.84) and 10 drops of nitric acid (HNO₃, 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* (Na₂SO₄), anhydrous.

10.7 *Sulfuric Acid, Standard* (0.5 N)—Prepare and standardize a 0.5 N sulfuric acid (H₂SO₄) solution.

10.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

10.9 *Sulfur Acid* (1+1)—Gradually pour 10 g of concentrated sulfuric acid (H₂SO₄) (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 ± 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 H₂SO₄ to a pink color. Add 5 mL of H₂SO₄ (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 ½ 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 Na₂SO₄.

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 Na₂SO₄, 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 mL⁵ plus 5 mL of water.

13.2 *Ethyl Alcohol, Neutral* (absolute) —Freshly boiled absolute ethyl alcohol, neutral to phenolphthalein.⁵