



Designation: D820 – 93 (Reapproved 2023)

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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. Referenced Documents

2.1 *ASTM Standards*:²

D216 Method of Test for Distillation of Natural Gasoline (Withdrawn 1988)³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

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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 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 cm to 8 cm in diameter and about 2 cm to 4 cm in depth will be required.

6. Procedure

6.1 Weigh 5 g $\pm$ 0.01 g of the sample in the dish, and dry to constant weight in an air oven at a temperature of 105 °C $\pm$ 2 °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 g to 10 g $\pm$ 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,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 °C to 60 °C.⁶

Distillation test:^A

Initial boiling point	35 °C to 38 °C
Dry flask end point	52 °C 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

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

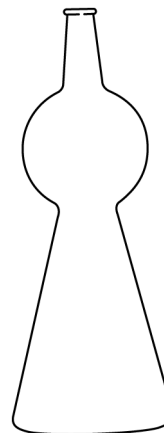


FIG. 1 Stokes Flask

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.

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