



Standard Method for ANALYSIS OF OIL-SOLUBLE SODIUM PETROLEUM SULFONATES BY LIQUID CHROMATOGRAPHY¹

This Standard is issued under the fixed designation D 2548; 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.

1. Scope

1.1 This method covers the determination of oil and sulfonate in crude and refined natural and synthetic sodium sulfonates. Resins, if present, are recovered with the oil phase, and carboxylates are recovered as sulfonates.

NOTE 1—A method for the analysis of the same type of materials is described in ASTM Method D 855, Analysis of Oil-Soluble Sodium Petroleum Sulfonates.² This method also covers the determination of properties other than oil and sulfonate content.

2. Summary of Method

2.1 The sample is dissolved in chloroform and adsorbed on silica gel. The oil is eluted with chloroform and the sulfonate with ethyl alcohol, and both are determined gravimetrically.

3. Apparatus

3.1 *Chromatographic Column*, made of glass and consisting of a reservoir and a separator section, and fitted with a TFE-fluorocarbon stopcock with a 2-mm bore, as shown in Fig. 1. A column with a detachable reservoir connected by a standard taper joint may be used.

3.2 *Steam Bath*.

3.3 *Vacuum Desiccator*, shielded.

3.4 *Vacuum Oven*, capable of being maintained at 212 F (100 C) and connected to 22 to 25 in. (559 to 625 mm) Hg vacuum.

4. 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.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.

4.2 *Chloroform*.

4.3 *Ethyl Alcohol (95 percent)*—Either pure grain or denatured ethyl alcohol conforming to Formula 3A of the U.S. Bureau of Internal Revenue.

4.4 *Silica Gel*, 60 to 200 mesh.⁴

5. Preparation of Column

5.1 Pour 80 to 100 ml of chloroform into the column, and push a wad of cotton to the bottom with a rod (Note 2). Compress the cotton enough to hold back the silica gel but not enough to impede the flow of solvent.

NOTE 2—A coarse fritted disk made of borosilicate glass may be used in place of the cotton wad.

5.2 Pour 15 ± 1 g of silica gel into the column containing the chloroform. The column must be free of air bubbles to avoid channeling. Start the flow of chloroform by opening the stopcock. When the liquid level is within $\frac{1}{2}$ in. (13 mm) of the surface of the gel, close the stopcock.

NOTE 3—Never allow the liquid level to fall below the surface of the silica gel.

¹ This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

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In 1969, the scope and procedure were changed to restrict the method to determination of sodium sulfonates.

² *Annual Book of ASTM Standards*, Part 23.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."

⁴ Silica gel, Grade 62, obtainable from the Davison Chemical Corp., Baltimore, Md., has been found satisfactory for this purpose.