



Standard Method for ANALYSIS OF CALCIUM AND BARIUM PETROLEUM SULFONATES BY LIQUID CHROMATOGRAPHY¹

This Standard is issued under the fixed designation D 2894; 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 reappraisal.

1. Scope

1.1 This method covers the determination of mineral oil, calcium or barium sulfonate, average molecular weight, water, and specific gravity of crude and refined natural and synthetic calcium and barium sulfonates.

NOTE 1—A method for analysis of the same type of materials is described in ASTM Method D 1216, Analysis of Calcium and Barium Petroleum Sulfonates.²

NOTE 2—The values stated in U.S. customary units are to be regarded as the standard.

2. Summary of Methods

2.1 The sample is dissolved in ethyl ether and converted to sulfonic acid, using dilute hydrochloric acid. The sulfonic acid and mineral oil, after extraction, are converted to sodium sulfonate and the isolated sodium sulfonate and mineral oil dissolved in chloroform. An aliquot of the chloroform solution is adsorbed on silica gel. The oil is eluted with chloroform and the sulfonate with ethyl alcohol, and both are determined gravimetrically. Average molecular weight is calculated from the average equivalent weight of the isolated sodium sulfonate, which is determined by ashing a portion of the isolated sodium sulfonate. Water is determined by ASTM Method D 95, Test for Water in Petroleum Products and Bituminous Materials By Distillation.² Specific gravity is determined by pycnometer.

3. Apparatus

3.1 *Chromatographic Column*, made of glass and consisting of a reservoir and a separator section, and fitted with TFE-fluorocarbon stopcock with a 2-mm bore, as shown in Fig. 1 of ASTM Method D 2548, Analysis of Oil-Soluble Sodium Petroleum Sulfonates by Liquid Chromatography.² 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. (259 to 635 mm) Hg vacuum.

3.5 *Muffle Furnace*, capable of operating at 1500 to 1800 F (800 to 1000 C).

3.6 *Water Bath*, capable of being maintained at 77 ± 0.3 F (25 ± 0.2 C).

3.7 *Distillation Apparatus*, as described in Method D 95.

3.8 *Dish*, platinum, 100-ml capacity.

3.9 *Pycnometer*, as shown in Fig. 2 of Method D 1216. To calibrate, weigh to the nearest 1 mg, with cap in place, then fill with distilled water at 60 to 68 F (15 to 20 C) and place in a water bath at 77 ± 0.3 F (25 ± 0.2 C). After 30 min, adjust the water meniscus at the top of the neck so that it is exactly level. To obtain a flat meniscus, add a minute amount of wetting agent to the water surface. Remove the pycnometer from the bath and dry the outside. Replace the cap and weigh to the nearest 1 mg. Record the weight of water contained as W_c .

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

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

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² *Annual Book of ASTM Standards*, Part 18.

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."