

E60 Sodium Lauryl Sulfate

Revision Stage 2

Definition

Sulfuric acid monododecyl ester sodium salt.

Sodium monododecyl sulfate [151-21-3].

Sodium Lauryl Sulfate is a mixture of sodium alkyl sulfates consisting chiefly of sodium lauryl sulfate [$\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2\text{OSO}_3\text{Na}$]. It contains not less than 85.0% of sodium alkyl sulfates calculated as $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$.

Identification

A: Infrared absorption spectrophotometry – KBr or ATR

Record the infrared absorption spectrum of Sodium Lauryl Sulfate and compare with the spectrum obtained with the Reference Standard: the transmission minima correspond in position and relative size.

B: Place 2.5 g in a silica or platinum crucible, and add 2 mL of 10 N sulfuric acid. Heat on a water bath, then cautiously raise the temperature progressively over an open flame. Ignite, preferably in a muffle furnace, at $600 \pm 25^\circ$. Continue heating until all black particles have disappeared. Cool, add a few drops of 2 N sulfuric acid, and heat and ignite as above. Add a few drops of ammonium carbonate solution, evaporate to dryness, and ignite as above. Cool, dissolve the residue in 50 mL of water, and mix.

To a 2 mL portion of this solution, add 4 mL of *Potassium pyroantimonate solution*. If necessary, rub the inside of the test tube with a glass rod. A white, crystalline precipitate is formed.

C: Prepare a solution (1 in 10) after acidification with hydrochloric acid and boiling for 20 minutes. No precipitate is formed. Add *barium chloride solution*; a white precipitate is produced.

Alkalinity— Dissolve 1.0 g in 100 mL of water, add 0.1 mL of phenol red solution, and titrate with 0.10 N hydrochloric acid: not more than 0.5 mL is required for neutralization.

Sodium chloride—Dissolve about 5 g, accurately weighed, in 50 mL of water. Neutralize the solution with dilute nitric acid, if necessary, add exactly 5.0 mL of 0.1 N Sodium Chloride and titrate, under vigorous stirring (to disperse silver chloride), with 0.1 N Silver Nitrate (indicator: 2 drops of *fluorescein sodium solution*) to the first appearance of turbidity with solution color change from yellow-green to orange through yellow. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N Silver Nitrate = 5.844 mg of NaCl.

Sodium sulfate—

Procedure—Transfer about 1 g of Sodium Lauryl Sulfate, accurately weighed, in 10 mL of water, add 100 mL of alcohol and heat at a temperature just below the boiling point for 2 hours. Filter through a glass or porcelain filter (pore size 4-10 μm) while hot, and wash with 100 mL of boiling alcohol. Dissolve the precipitate by washing with 150 mL of water, collecting the washings in a

45 beaker. Add 10 mL of dilute hydrochloric acid, heat to boiling, add 25 mL of barium chloride
46 solution and allow to stand overnight. Collect the precipitate by filtration through a filter
47 (maximum pore size 16 μm) and wash with water until the last washing shows no opalescence
48 with 0.1 N Silver Nitrate. Dry the precipitate, ignite to constant mass between 500 and 600 degrees
49 by raising the temperature gradually, and weigh as barium sulfate (BaSO_4 : 233.39)

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51 Amount (mg) of sodium sulfate (Na_2SO_4) = amount (mg) of barium sulfate (BaSO_4) times
52 0.6086

53 The combined content of sodium chloride and sodium sulfate is NMT 8.0%.

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55 **Unsulfated alcohols**— Dissolve about 10 g, accurately weighed, in 100 mL of water, and add 100
56 mL of alcohol. Transfer the solution to a separator, and extract with three 50-mL portions of
57 Petroleum Ether. If an emulsion forms, sodium chloride may be added to promote separation of
58 the two layers. Wash the combined Petroleum Ether extracts with three 50-mL portions of water,
59 and dry with anhydrous sodium sulfate. Filter the Petroleum Ether extract into a tared beaker,
60 evaporate on a water bath and dry the residue at 105° for 30 minutes, cool, and weigh. The weight
61 of the residue is not more than 4.0% of the weight of the Sodium Lauryl Sulfate taken.

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63 **Assay**— Dissolve 1.15 g of Sodium Lauryl Sulfate in water, warming if necessary, and dilute to
64 exactly 1000.0 ml with water. Mix 20.0 mL of the solution with 15 ml of methylene chloride and
65 10 mL of Dimidium Bromide-Sulfan Blue Mixed Solution. Titrate with 0.004 M benzethonium
66 chloride, shaking vigorously and allowing the layers to separate before each addition, until the
67 pink color of the methylene chloride layer is completely discharged and a greyish-blue color is
68 obtained.

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70 1 ml of 0.004 M benzethonium chloride is equivalent to 1.154 mg of sodium alkyl sulfates,
71 calculated as $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$.

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73 REAGENTS

74 **Ammonium Carbonate solution**—Dissolve 20 g of ammonium carbonate and 20 mL of ammonia
75 in water to make 100 mL

76 **Potassium pyroantimonate solution**—To 2 g of potassium pyroantimonate add 100 mL of water.
77 Boil the solution for about 5 minutes, cool quickly, and add 10 mL of a solution of potassium
78 hydroxide (3 in 20). Allow to stand for 24 hours, and filter.

79 **Barium Chloride solution**—Dissolve 12 g of barium chloride in water to make 100 mL.

80 **Phenol Red solution (Phenolsulfonphthalein TS)**—Dissolve 100 mg of phenolsulfonphthalein
81 in 100 mL of alcohol, and filter if necessary.

82 **Dilute Nitric Acid (10 percent HNO_3) [7697-37-2]**—Dilute 105 mL of nitric acid with water to
83 1000 mL

84 **Dilute Hydrochloric Acid (10 percent) [7647-01-0]**—Prepare by mixing 226 mL of hydrochloric
85 acid with sufficient water to make 1000 mL.

86 **Fluorescein sodium solution**—Dissolve 0.2 g of fluorescein sodium in water to make 100 mL

87 **Benzethonium chloride (0.004 M) [121-54-0]**—Dissolve 1.792 g of benzethonium
88 chloride previously dried to constant mass at 100-105 °C in water, and dilute to 1000.0 mL with
89 water. Standardize the *Benzethonium chloride* solution using properly validated analytical method.

90 **Dimidium Bromide-Sulfan Blue Mixed Solution (Indicator)**— Dissolve separately 0.5 g of
91 Dimidium bromide and 0.25 g of Sulfan blue in 30 mL of a hot solution of 10% ethanol in water,
92 combine these solutions and dilute to 250 mL with 10% ethanol. Mix 20 mL of this solution with
93 20 mL of a 14.0% V/V solution of Sulfuric acid previously diluted with about 250 mL of water
94 and diluted to 500 mL with water.