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3 **Sodium Lauryl Sulfate**
4 **(Stage 4)**

5 Sulfuric acid monododecyl ester sodium salt.
6

7 Sodium monododecyl sulfate [151-21-3].
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9 » Sodium Lauryl Sulfate is a mixture of sodium alkyl sulfates consisting chiefly of sodium
10 lauryl sulfate [$\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2\text{OSO}_3\text{Na}$]. The combined content of sodium chloride and
11 sodium sulfate is not more than 8.0 percent. It contains not less than 85.0% of sodium alkyl
12 sulfates calculated as $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$.
13

14 **Identification—**

15 **A:** Place 2.5 g in a silica or platinum crucible, and add 2 mL of 10 N sulfuric acid. Heat on
16 a water bath, then cautiously raise the temperature progressively over an open flame. Ignite,
17 preferably in a muffle furnace, at $600 \pm 25^\circ$. Continue heating until all black particles have
18 disappeared. Cool, add a few drops of 2 N sulfuric acid, and heat and ignite as above. Add a
19 few drops of 2 M ammonium carbonate, evaporate to dryness, and ignite as above. Cool,
20 dissolve the residue in 50 mL of water, and mix.
21

22 To a 2 mL portion of this solution, add 4 mL of *Potassium pyroantimonate solution*. If
23 necessary, rub the inside of the test tube with a glass rod. A white, crystalline precipitate is
24 formed.
25

26 **B:** Prepare a solution (1 in 10) after acidification with hydrochloric acid and boiling for 20
27 minutes. No precipitate is formed. Add *barium chloride solution*; a white precipitate is
28 produced.
29

30 **C:** Dissolve 0.1 g in 10 mL of water and shake. A copious foam is formed.
31

32 **D:** To 0.1 mL of the solution prepared for identification C, add 0.1 mL of a 1 g/L solution
33 of methylene blue and 2 mL of dilute sulfuric acid. Add 2 mL of methylene chloride and shake.
34 An intense blue color develops in the methylene chloride layer.
35

36 **Alkalinity—**Dissolve 1.0 g in 100 mL of water, add phenol red solution, and titrate with 0.10
37 N hydrochloric acid: not more than 0.5 mL is required for neutralization.
38

39 **Sodium chloride**—Dissolve about 5 g, accurately weighed, in about 50 mL of water.
40 Neutralize the solution with dilute nitric acid, if necessary, add exactly 5.0 mL of 0.1 N Sodium
41 Chloride and titrate with 0.1 N Silver Nitrate (indicator: 2 drops of *fluorescein sodium solution*)
42 to the first appearance of turbidity. Perform a blank determination, and make any necessary
43 correction. Each mL of 0.1 N Silver Nitrate = 5.844 mg of NaCl.

44

45 **Sodium sulfate**—

46 *Procedure*—Transfer about 1 g of Sodium Lauryl Sulfate, accurately weighed, in 10 mL of
47 water, add 100 mL of alcohol and heat at a temperature just below the boiling point for 2 hours.
48 Filter through a glass filter while hot, and wash with 100 mL of boiling alcohol. Dissolve the
49 precipitate by washing with 150 mL of water, collecting the washings in a beaker. Add 10 mL
50 of dilute hydrochloric acid, heat to boiling, add 25 mL of *barium chloride solution* and allow to
51 stand overnight. Collect the precipitate and wash with water until the last washing shows no
52 opalescence with 0.1 N Silver Nitrate. Dry the precipitate, ignite to constant mass between 500
53 and 600 degrees by raising the temperature gradually, and weigh as barium sulfate (BaSO_4 :
54 233.39)

55

56 Amount (mg) of sodium sulfate (Na_2SO_4) = amount (mg) of barium sulfate (BaSO_4) times
57 0.6086

58

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

67

68 **Assay**—Dissolve 1.5 g of Sodium Lauryl Sulfate in water, warming if necessary, and dilute to
69 exactly 1000.0 ml with water. To 10.0 ml of the solution add 25 mL *Methylene blue solution*
70 (0.003 % w/v), 15 ml of methylene chloride and 20 mL of water. Titrate with 0.004 M
71 benzethonium chloride, shaking vigorously and allowing the layers to separate before each
72 addition, until almost the same blue color of the two layers is obtained.

73

74 1 ml of 0.004 M benzethonium chloride is equivalent to 1.154 mg of sodium alkyl sulphates,
75 calculated as $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$.

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77

78 **REAGENTS:**

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

82

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

84 *Dilute Sulfuric Acid*—Cautiously add 57 mL of sulfuric acid to about 100 mL of water, cool to
85 room temperature, and dilute with water to 1000 mL.

86

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

89

90 **Dilute Nitric Acid** (10 percent HNO₃) [7697-37-2]—Dilute 105 mL of nitric acid with
91 water to 1000 mL.

92

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

95

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

97

98 **Methylene blue solution**—Dissolve 0.003g of methylene blue, 5.0g of anhydrous sodium
99 sulfate and 1.2g of sulfuric acid in 100 mL of water. Anhydrous sodium sulfate is the emulsion
100 breaker.

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