» Sodium Formaldehyde Sulfoxylate contains an amount of CH_3NaO_3S equivalent to not less than 45.5 percent and not more than 54.5 percent of SO_2, calculated on the dried basis. It may contain a suitable stabilizer, such as sodium carbonate.

Packaging and storage—Preserve in well-closed, light-resistant containers, and store at controlled room temperature.

Clarity and color of solution—Dissolve 1 g in 20 mL of water, and transfer 10 mL to a 20- × 150-mm test tube. Compare with water in a similar test tube: the liquids are equally clear and, when viewed transversely by transmitted light, exhibit no apparent difference in color.

Identification—

A: Dissolve about 4 g in 10 mL of water in a test tube, and add 1 mL of silver-ammonia-nitrate TS: metallic silver is produced, either as a finely divided, gray precipitate or as a bright metallic mirror on the inner surface of the tube.

B: Dissolve about 40 mg of salicylic acid in 5 mL of sulfuric acid, add about 50 mg of Sodium Formaldehyde Sulfoxylate, and warm very gently: a permanent, deep red color appears.

Alkalinity—Dissolve 1.0 g in 50 mL of water, add phenolphthalein TS, and titrate with 0.10 N sulfuric acid: not more than 3.5 mL is required for neutralization.

pH 〈791〉: between 9.5 and 10.5, in a solution (1 in 50).

Loss on drying 〈731〉—Dry it at 105° for 3 hours: it loses not more than 27.0% of its weight.

Sulfide—Dissolve 6 g in 14 mL of water in a test tube, and wet a strip of lead acetate test paper with the clear solution: no discoloration is evident within 5 minutes.

Iron—Transfer 1.0 g to a suitable crucible, and carefully ignite, initially at a low temperature until thoroughly charred, and finally, preferably in a muffle furnace, at 500° to 600° until the carbon is all burned off. Cool, dissolve the residue in 2 mL of hydrochloric acid, and dilute with water to 50 mL. Add about 50 mg of ammonium persulfate and 5 mL of ammonium thiocyanate TS, mix, and transfer to a color-comparison tube. Treat in the same manner 5.0 mL of a solution of ferric ammonium sulfate, prepared by dissolving 43.2 mg of ferric ammonium sulfate in 10 mL of 2 N sulfuric acid and adding water to make 1000 mL, each mL representing 5 µg of Fe. The color of the test solution is not deeper than that of the solution containing the standard iron solution (0.0025%).

Sodium sulfite—Transfer 4.0 mL of the solution prepared for the *Assay* to a conical flask containing 100 mL of water. Add 2 mL of formaldehyde TS, and titrate with the same 0.1 N iodine VS that is used for the *Assay*, adding 3 mL of starch TS as the endpoint is approached. Calculate the percentage of Na_2SO_3 in the Sodium Formaldehyde Sulfoxylate taken by the formula:

$$(1.25)(63.02)(V_2 - V_1)(N / W)$$

in which 63.02 is the equivalent weight of sodium sulfite; V_1 and V_2 are the volumes, in mL, of 0.1 N iodine VS consumed in this titration and in the titration performed in the *Assay*, respectively; N is the exact normality of the iodine solution; and W is the weight, in g, of Sodium Formaldehyde Sulfoxylate taken for the *Assay:* not more than 5.0% of Na_2SO_3, calculated on the dried basis, is found.

Assay—Transfer about 1 g of Sodium Formaldehyde Sulfoxylate, accurately weighed, to a 50-mL volumetric flask, dissolve in about 25 mL of water, dilute with water to volume, and mix. Reserve a portion of this solution for the test for *Sodium sulfite*. Transfer 4.0 mL of this solution to a conical flask containing 100 mL of water, and titrate with 0.1 N iodine VS, adding 3 mL of starch TS as the endpoint is approached. Each mL of 0.1 N iodine is equivalent to 1.602 mg of SO_2.

Sodium Hydroxide

$NaOH$ 40.00

Sodium hydroxide [1310-73-2].

DEFINITION

Sodium Hydroxide contains NLT 95.0% and NMT 100.5% of total alkali, calculated as sodium hydroxide (NaOH), including NMT 3.0% of sodium carbonate (Na_2CO_3).

[**Caution—**Exercise great care in handling sodium hydroxide, because it rapidly destroys tissues.]

IDENTIFICATION

- **A. Identification Tests—General,** *Sodium* **〈191〉:** A solution (1 in 25) meets the requirements.

ASSAY

- **Procedure**

Sample solution: 1.5 g of Sodium Hydroxide in 40 mL of carbon dioxide-free water. Cool the solution to room temperature.

Blank: 40.0 mL of carbon dioxide-free water

Titrimetric system

(See *Titrimetry* 〈541〉.)

Mode: Direct titration

Titrant: 1 N sulfuric acid

Endpoint detection: Visual

Analysis: To the *Sample*, add phenolphthalein TS. Titrate with 1 N sulfuric acid VS. At the discharge of the pink color of the indicator, record the volume of *Titrant* (V_{S1}). Add methyl orange TS, and continue the titration until a persistent pink color is produced. Record the volume of *Titrant* (V_{S2}). Perform a blank determination, and make any necessary corrections.

Calculate the percentage of total alkali, calculated as sodium hydroxide (NaOH), in the *Sample* taken:

$$\text{Result} = \{[(V_{S1} - V_B) \times N \times F_1]/W\} \times 100$$

V_{S1} = volume of *Titrant* consumed by the *Sample* to the first endpoint (mL)
V_B = volume of *Titrant* consumed by the *Blank* (mL)
N = actual normality of the *Titrant* (mEq/mL)
F_1 = equivalency factor, 40.00 (mg/mEq)
W = weight of the *Sample* (mg)

Calculate the percentage of sodium carbonate (Na_2CO_3) in the *Sample* taken:

$$\text{Result} = \{[(V_{S2} - V_{S1}) \times N \times F_2]/W\} \times 100$$

V_{S2} = volume of *Titrant* consumed by the *Sample* to the second endpoint (mL)
V_{S1} = volume of *Titrant* consumed by the *Sample* to the first endpoint (mL)
N = actual normality of the *Titrant* (mEq/mL)
F_2 = equivalency factor, 106.0 (mg/mEq)
W = weight of the *Sample* (mg)

Acceptance criteria: 95.0%–100.5% of total alkali; NMT 3.0% of sodium carbonate (Na_2CO_3)

IMPURITIES

- **Potassium**

Sample solution: 1 in 20

Analysis: Acidify 5 mL of the *Sample solution* with 6 N acetic acid, then add 5 drops of sodium cobaltinitrite TS.

Acceptance criteria: No precipitate is formed.

- **Heavy Metals 〈231〉**

Test preparation: Dissolve 0.67 g in a mixture of 5 mL of water and 7 mL of 3 N hydrochloric acid. Heat to boiling, cool, and dilute with water to 25 mL.

Analysis: Proceed as directed in the chapter.
Acceptance criteria: NMT 30 ppm

SPECIFIC TESTS

- **Insoluble Substances and Organic Matter:** A solution (1 in 20) is complete, clear, and colorless to slightly colored.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers.

Sodium Lauryl Sulfate

Sulfuric acid monododecyl ester sodium salt;
Sodium monododecyl sulfate [151-21-3].

DEFINITION

Sodium Lauryl Sulfate is a mixture of sodium alkyl sulfates consisting chiefly of sodium lauryl sulfate [$CH_3(CH_2)_{10}CH_2OSO_3Na$]. The combined content of sodium chloride and sodium sulfate is NMT 8.0%.

IDENTIFICATION

- **A. Identification Tests—General,** *Sodium* ⟨**191**⟩**:** Ignite 500 mg at 800° until the carbon is consumed. The residue dissolved in 10 mL of water meets the requirements.
- **B. Identification Tests—General,** *Sulfate* ⟨**191**⟩**:** A solution (1 in 10), after acidification with hydrochloric acid and gentle boiling for 20 min, meets the requirements.

IMPURITIES

Inorganic Impurities

- **Heavy Metals,** *Method II* ⟨**231**⟩**:** 20 ppm
- **Sodium Chloride**
  Sample solution: 100 mg/mL in water
  Analysis: Neutralize 50 mL of *Sample solution* with 0.8 N nitric acid, using litmus paper as the indicator. Add 2 mL of potassium chromate TS, and titrate with 0.1 N silver nitrate VS. Each mL of 0.1 N silver nitrate is equivalent to 5.844 mg of NaCl.

SPECIFIC TESTS

- **Sodium Sulfate**
  Lead nitrate solution: 33.1 g/L of lead nitrate in water
  Analysis: Transfer 1 g of Sodium Lauryl Sulfate, weighed, to a 250-mL beaker. Add 35 mL of water, and warm to dissolve. To the warm solution add 2.0 mL of 1 N nitric acid, mix, and add 50 mL of alcohol. Heat the solution to boiling, and slowly add 10 mL of *Lead nitrate solution*, with stirring. Cover the beaker, simmer for 5 min, and allow to settle. If the supernatant is hazy, allow to stand for 10 min, heat to boiling, and allow to settle. When the solution is almost to the boiling point, decant as much liquid as possible through 9-cm filter paper (Whatman No. 41 or equivalent). Wash four times by decantation, each time using 50 mL of 50% alcohol, and bring the mixture to a boil. Transfer the filter paper to the original beaker, and immediately add 30 mL of water, 20.0 mL of 0.05 M edetate disodium VS, and 1 mL of ammonia–ammonium chloride buffer TS. Warm to dissolve the precipitate, add 0.2 mL of eriochrome black TS, and titrate with 0.05 M zinc sulfate VS. Each mL of 0.05 M edetate disodium is equivalent to 7.102 mg of Na_2SO_4.
  Acceptance criteria: NMT 8.0% of the combined content of sodium chloride and sodium sulfate
- **Alkalinity**
  Sample solution: Dissolve 1.0 g in 100 mL of water, add phenol red TS, and titrate with 0.10 N hydrochloric acid.
  Acceptance criteria: NMT 0.60 mL for neutralization
- **Total Alcohols:** Transfer 5 g to an 800-mL Kjeldahl flask, and add 150 mL of water, 50 mL of hydrochloric acid, and a few boiling chips. Attach a reflux condenser to the Kjeldahl flask, heat carefully to avoid excessive frothing, and boil for 4 h. Cool the flask, rinse the condenser with ether, collecting the ether in the flask, and transfer the contents to a 500-mL separator, rinsing the flask twice with ether and adding the washings to the separator. Extract the solution with two 75-mL portions of ether, evaporate the combined ether extracts in a tared beaker on a steam bath, dry the residue at 105° for 30 min, cool, and weigh.
  Acceptance criteria: The residue represents the total alcohols and is NLT 59.0% of the weight of Sodium Lauryl Sulfate taken.
- **Unsulfated Alcohols**
  Sample solution: Dissolve 10 g in 100 mL of water, and add 100 mL of alcohol.
  Analysis: Transfer the solution to a separator, and extract with three 50-mL portions of solvent hexane. If an emulsion forms, sodium chloride may be added to promote separation of the two layers. Wash the combined solvent hexane extracts with three 50-mL portions of water, and dry with anhydrous sodium sulfate. Filter the solvent hexane extract into a tared beaker, evaporate on a steam bath until the odor of solvent hexane no longer is perceptible, dry the residue at 105° for 30 min, cool, and weigh.
  Acceptance criteria: The weight of the residue is NMT 4.0% of the weight of Sodium Lauryl Sulfate taken.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.

Sodium Metabisulfite

$Na_2S_2O_5$ 190.11
Disulfurous acid, disodium salt;
Disodium pyrosulfite [7681-57-4].

DEFINITION

Sodium Metabisulfite contains an amount of sodium metabisulfite ($Na_2S_2O_5$) equivalent to NLT 65.0% and NMT 67.4% of SO_2.

IDENTIFICATION

- **A. Identification Tests—General,** *Sodium* ⟨**191**⟩ and *Sulfite* ⟨**191**⟩**:** A solution (1 in 20) meets the requirements.

ASSAY

- **Procedure**
  Sample: 200 mg of Sodium Metabisulfite
  Blank: 50.0 mL of 0.1 N iodine VS, accurately measured
  Titrimetric system
  (See *Titrimetry* ⟨541⟩.)
  Mode: Residual titration
  Titrant: 0.1 N iodine VS
  Back-titrant: 0.1 N sodium thiosulfate VS
  Endpoint detection: Visual
  Analysis: Add the *Sample* to 50.0 mL of 0.1 N iodine VS in a glass-stoppered conical flask, and swirl to dissolve. Allow to stand for 5 min, protected from light. Add 1 mL of hydrochloric acid, and titrate the excess iodine with *Back-titrant*, adding 3 mL of starch TS as the endpoint is approached. Perform a blank determination.
  Calculate the percentage of sodium metabisulfite ($Na_2S_2O_5$) in the portion of Sodium Metabisulfite taken:

$$\text{Result} = \{[(V_B - V_S) \times N \times F]/W\} \times 100$$

V_B = *Back-titrant* volume consumed by the *Blank* (mL)
V_S = *Back-titrant* volume consumed by the *Sample* (mL)
N = *Back-titrant* normality (mEq/mL)
F = equivalency factor, 32.03 mg/mEq
W = *Sample* weight (mg)