

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 [$\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2\text{OSO}_3\text{Na}$]. 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

$\text{Na}_2\text{S}_2\text{O}_5$

190.11

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

DEFINITION

Sodium Metabisulfite contains an amount of sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) 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 ($\text{Na}_2\text{S}_2\text{O}_5$) 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)

Acceptance criteria: 65.0%–67.4% of SO_2

IMPURITIES

• HEAVY METALS, Method I (231)

Test preparation: 1 g

Analysis: Dissolve the *Test preparation* in 10 mL water. Add 5 mL of hydrochloric acid, evaporate on a steam bath to dryness, and dissolve the residue in 25 mL of water.

Acceptance criteria: NMT 20 ppm

• LIMIT OF CHLORIDE

Standard solution: 0.71 mL of 0.020 N hydrochloric acid in 100 mL of water

Sample solution: 1.0 g in 10 mL of water. [NOTE—Pass through a small chloride-free filter, if necessary.] Add 6 mL of 30% hydrogen peroxide. Add 1 N sodium hydroxide until the solution is slightly alkaline to phenolphthalein, and dilute with water to 100 mL.

Analysis

Samples: *Standard solution* and *Sample solution*
Dilute 2.0 mL of the *Samples* with water to 20 mL. Add 1 mL of nitric acid and 1 mL of silver nitrate TS. Allow to stand for 5 min protected from direct sunlight, and compare the turbidity from the *Samples* (see *Spectrophotometry and Light-Scattering* (851), *Visual Comparison*).

Acceptance criteria: Any turbidity produced by the *Sample solution* does not exceed that of the *Standard solution* (0.05%).

• LIMIT OF THIOSULFATE

Standard solution: Mix 0.10 mL of 0.10 N sodium thiosulfate with 10 mL of 1 N hydrochloric acid.

Sample solution: Mix 2.2 g with 10 mL of 1 N hydrochloric acid.

Analysis

Samples: *Standard solution* and *Sample solution*
Gently boil the *Samples* for 5 min. Cool, then transfer each solution to a small test tube.

Acceptance criteria: Any turbidity produced by the *Sample solution* does not exceed that of the *Standard solution* (0.05%).

• IRON (241)

Test preparation: Dissolve 500 mg of Sodium Metabisulfite in 14 mL of dilute hydrochloric acid (2 in 7), and evaporate on a steam bath to dryness. Dissolve the residue in 7 mL of dilute hydrochloric acid (2 in 7), and again evaporate to dryness. Dissolve the resulting residue in a mixture of 2 mL of hydrochloric acid and 20 mL of water. Add 3 drops of bromine TS, and boil to expel the bromine. Cool, then dilute with water to 47 mL.

Analysis: Proceed as directed in the chapter.

Acceptance criteria: NMT 20 ppm

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-filled, tight containers, and avoid exposure to excessive heat.

Sodium Phosphate, Dibasic—see *Dibasic Sodium Phosphate General Monographs*

Sodium Phosphate, Monobasic—see *Monobasic Sodium Phosphate General Monographs*

Tribasic Sodium Phosphate

Na_3PO_4 (anhydrous)	163.94
Trisodium phosphate, monohydrate	181.96
Phosphoric acid, trisodium salt, dodecahydrate;	
Trisodium phosphate, dodecahydrate	380.13
[10101-89-0].	
Anhydrous [7601-54-9].	

DEFINITION

Tribasic Sodium Phosphate is anhydrous or contains one to twelve molecules of water of hydration. Na_3PO_4 (anhydrous) and $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$ (monohydrate) contain NLT 97.0% of Na_3PO_4 , calculated on the ignited basis. $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ (dodecahydrate) contains NLT 92.0% of Na_3PO_4 , calculated on the ignited basis.

IDENTIFICATION

- **A. IDENTIFICATION TESTS—GENERAL,** *Sodium* (191) and *Phosphate* (191): A solution (1 in 20) meets the requirements.

ASSAY

• PROCEDURE

Sample: 5.5 g of Tribasic Sodium Phosphate, on the anhydrous basis

Blank: 100.0 mL of 1 N hydrochloric acid, accurately measured

Titrimetric system

(See *Titrimetry* (541).)

Mode: Residual titration

Titrant: 1 N sodium hydroxide VS

Endpoint detection: Potentiometric

Analysis: Transfer the *Blank* to a 400-mL beaker, and titrate with the *Titrant* to the endpoint at a pH of 7.0. Record as the volume consumed, and designate as *A*. Transfer the *Sample* to a 400-mL beaker, add 100.0 mL of 1 N hydrochloric acid, and stir until dissolved. Pass a stream of carbon dioxide-free air, in fine bubbles, through the solution for 30 min to expel carbon dioxide, covering the beaker loosely to prevent any loss by spraying. Wash the cover and sides of the beaker with a few mL of water.

Titrate the excess acid potentiometrically with the *Titrant* to the inflection point at a pH of 4. Record the buret reading, and designate as *B*. Protect the solution from carbon dioxide absorbed from the air, and continue the titration with 1 N sodium hydroxide VS to the inflection point at a pH of 8.8. Record the buret reading, and designate as *C*.

Calculate the amount of *Titrant* consumed by the *Sample* to the first inflection point, correcting for the *Blank* ($V_1 = A - B$) and the amount of *Titrant* consumed by the *Sample* between the two inflection points ($V_2 = C - B$). If V_1 is equal to or greater than $2V_2$, calculate the amount of Na_3PO_4 in the portion of *Sample* taken:

$$D = V_2 \times N \times F$$

V_2 = volume of *Titrant* consumed between the two inflection points (mL)

N = actual normality of the *Titrant* (mEq/mL)

F = equivalency factor, 163.9 mg/mEq

If V_1 is less than $2V_2$, calculate the amount of Na_3PO_4 in the portion of *Sample* taken:

$$D = (V_1 - V_2) \times N \times F$$

V_1 = volume of the *Titrant* consumed to the first inflection point, correcting for the *Blank* (mL)

N = actual normality of the *Titrant* (mEq/mL)

F = equivalency factor, 163.9 mg/mEq

Calculate the percentage of Na_3PO_4 on the ignited basis in the portion of Tribasic Sodium Phosphate taken:

$$\text{Result} = [10/(100 - L)] \times (D/W)$$