L = percentage calculated in the test for *Loss on Ignition* ⟨733⟩
D = amount of Na_3PO_4 found (mg)
W = weight of the *Sample* (g)

Acceptance criteria: NLT 97.0% of Na_3PO_4 on the ignited basis. $Na_3PO_4 \cdot 12H_2O$ (dodecahydrate) contains NLT 92.0% of Na_3PO_4 on the ignited basis.

IMPURITIES

- **Loss on Ignition ⟨733⟩**

  Sample: 2 g

  Analysis: Dry the *Sample* at 110° for 5 h, and then ignite at 800° for 30 min.

  Acceptance criteria: The anhydrous form loses NMT 2.0% of its weight, the monohydrate loses 8.0%–11.0% of its weight, and the dodecahydrate loses 45.0%–57.0% of its weight.
- **Arsenic,** *Method I* **⟨211⟩**

  Test preparation: Dissolve a portion equivalent to 1.0 g of anhydrous tribasic sodium phosphate in 35 mL of water.

  Analysis: Proceed as directed in the chapter.

  Acceptance criteria: NMT 3 ppm
- **Heavy Metals,** *Method I* **⟨231⟩**

  Test preparation: Dissolve a portion equivalent to 2.0 g of anhydrous tribasic sodium phosphate in 25 mL of water.

  Analysis: Proceed as directed in the chapter.

  Acceptance criteria: 10 ppm

SPECIFIC TESTS

- **Insoluble Substances**

  Sample solution: Dissolve a portion equivalent to 10.0 g of anhydrous tribasic sodium phosphate in 100 mL of hot water.

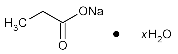
  Analysis: Filter the *Sample solution* through a tared filtering crucible. [Note—Do not use glass.] Wash the insoluble residue with hot water, and dry at 105° for 2 h.

  Acceptance criteria: The weight of the residue so obtained does not exceed 20 mg (0.2%).

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers. No storage requirements specified.
- **Labeling:** Label it to indicate whether it is anhydrous, the monohydrate, or the dodecahydrate.

Sodium Propionate



$C_3H_5NaO_2 \cdot xH_2O$

$C_3H_5NaO_2$ 96.06

Propanoic acid, sodium salt, hydrate;
Sodium propionate hydrate [6700-17-0].
Anhydrous [137-40-6].

DEFINITION

Sodium Propionate, dried at 105° for 2 h, contains NLT 99.0% and NMT 100.5% of sodium propionate ($C_3H_5NaO_2$).

IDENTIFICATION

- **A. Infrared Absorption ⟨197K⟩**

  Analysis: Perform test on an undried sample.

  Acceptance criteria: Meets the requirements
- **B. Identification Tests—General,** *Sodium* **⟨191⟩**

  Sample solution: 1 in 20

  Acceptance criteria: Meets the requirements

ASSAY

- **Procedure**

  Sample: 200 mg of Sodium Propionate, previously dried at 105° for 2 h

  Titrimetric system

  (See *Titrimetry* ⟨541⟩.)

  Mode: Direct titration

  Titrant: 0.1 N perchloric acid VS

  Blank: 50 mL of glacial acetic acid

  Endpoint detection: Visual

  Analysis: Dissolve the *Sample* in 50 mL of glacial acetic acid, and add 1 drop of crystal violet TS. Titrate with 0.1 N perchloric acid VS to a green endpoint. Perform a blank determination, and make any necessary correction.

  Calculate the percentage of sodium propionate ($C_3H_5NaO_2$) in the *Sample* taken:

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

  V_S = volume of the *Titrant* consumed by the *Sample* (mL)
  V_B = volume of the *Titrant* consumed by the *Blank* (mL)
  N = actual normality of the *Titrant* (mEq/mL)
  F = equivalency factor, 96.06 mg/mEq
  W = weight of the *Sample* (mg)

  Acceptance criteria: 99.0%–100.5% on the dried basis

IMPURITIES

- **Heavy Metals,** *Method I* **⟨231⟩**

  Test preparation: Dissolve 2 g of Sodium Propionate in 1 mL of 1 N acetic acid and sufficient water to make 25 mL.

  Acceptance criteria: NMT 10 ppm

SPECIFIC TESTS

- **Water Determination,** *Method I* **⟨921⟩:** NMT 1.0%
- **Alkalinity**

  Sample solution: 2.0 g of Sodium Propionate in 20 mL of water

  Analysis: Add phenolphthalein TS to the *Sample solution*.

  Acceptance criteria: If a pink color is produced, it is discharged by 0.60 mL of 0.10 N sulfuric acid.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers.
- **USP Reference Standards ⟨11⟩**

  USP Sodium Propionate RS

Sodium Starch Glycolate

Starch carboxymethyl ether, sodium salt.

DEFINITION

Sodium Starch Glycolate is the sodium salt of a carboxymethyl ether of starch or of a cross-linked carboxymethyl ether of starch. It may contain NMT 7.0% of Sodium Chloride. The pH and assay requirements for Type A and Type B are set forth in the accompanying table.

Type	pH		% Sodium, Combined as Sodium Starch Glycolate	
	Min.	Max.	Min.	Max.
A	5.5	7.5	2.8	4.2
B	3.0	5.0	2.0	3.4

IDENTIFICATION

- **A. Infrared Absorption** ⟨197K⟩
- **B.** A slightly acidified solution is colored blue to violet by the addition of iodine and potassium iodide TS 1.
- **C. Procedure**

  Potassium pyroantimonate solution: To 2 g of potassium pyroantimonate add 100 mL of water. Boil the solution for 5 min, cool quickly, and add 10 mL of a solution of potassium hydroxide (3 in 20). Allow to stand for 24 h, and filter.

  Analysis: To a 2-mL portion of the *Sample solution* prepared for the test for *Limit of Iron*, add 4 mL of *Potassium pyroantimonate solution*. If necessary, rub the inside of the test tube with a glass rod.

  Acceptance criteria: A white, crystalline precipitate is formed.
- **D.** Sodium Starch Glycolate imparts an intense yellow color to a nonluminous flame.

ASSAY

- **Procedure**

  Sample: 1 g

  Analysis: Transfer the *Sample* to a conical flask, add 20 mL of 80% alcohol, stir for 10 min, and filter. Repeat the extraction until the chloride has been completely extracted, as shown by a test with silver nitrate. Dry the insoluble portion at 105° to constant weight, and transfer an accurately weighed portion (700 mg) of the dried 80% alcohol–insoluble portion to a suitable flask. Add 80 mL of glacial acetic acid, heat the mixture under reflux on a boiling water bath for 2 h, cool to room temperature, and titrate with 0.1 N perchloric acid VS, determining the endpoint potentiometrically.

  Calculate the percentage of sodium combined in the form of sodium starch glycolate:

$$\text{Result} = 100 \times (22.99) \times V \times N/W$$

  V = volume of perchloric acid consumed (mL)
  N = normality of the perchloric acid
  W = weight of the dried alcohol–insoluble residue taken for the *Assay* (mg)

  Acceptance criteria: 2.8%–4.2% for Type A; 2.0%–3.4% for Type B

OTHER COMPONENTS

- **Limit of Sodium Chloride**

  Sample: 500 mg of Sodium Starch Glycolate

  Titrimetric system

  (See *Titrimetry* ⟨541⟩.)

  Mode: Direct titration

  Titrant: 0.1 N silver nitrate VS

  Endpoint detection: Potentiometric

  Indicator electrode: Suitable silver-based

  Reference electrode: Double junction electrode containing a 10% potassium nitrate filling solution in the outer jacket and a standard filling solution in the inner jacket

  Analysis: Transfer the *Sample* to a beaker, and suspend in 100 mL of water. Add 1 mL of nitric acid. Titrate with the *Titrant*. Each mL of 0.1 N silver nitrate is equivalent to 5.844 mg of sodium chloride.

  Acceptance criteria: NMT 7.0%
- **Limit of Sodium Glycolate**

  [Note—Conduct this test without exposure to daylight. Use low-actinic glassware.]

  Solution A: 0.1 mg/mL of 2,7-dihydroxynaphthalene in sulfuric acid; allow to stand until decolorized, and use within 2 days.

  Standard solution: Transfer 310 mg of glycolic acid, previously dried over phosphorus pentoxide in a desiccator at room temperature overnight, to a 500-mL volumetric flask, and dissolve in and dilute with water to volume. Transfer 5.0 mL of this solution to a 100-mL beaker, add 4 mL of 6 N acetic acid, and allow to stand for about 30 min. Add 50 mL of acetone and 1 g of sodium chloride, mix, and pass through fast filter paper moistened with acetone into a 100-mL volumetric flask. Rinse the beaker and the filter paper with acetone. Combine the filtrate and washings, dilute with acetone to volume, and mix. Allow to stand for 24 h without shaking. Use the clear supernatant as the *Standard solution*.

  Sample solution: Transfer 200 mg to a 100-mL beaker. Add 4 mL of 6 N acetic acid and 5 mL of water. Stir until dissolution is complete (about 10 min). Add 50 mL of acetone and 1 g of sodium chloride, and pass through fast filter paper moistened with acetone into a 100-mL volumetric flask. Rinse the beaker and filter paper with acetone. Combine the filtrate and washings, and dilute with acetone to volume. Allow to stand for 24 h without shaking. Use the clear supernatant as the *Sample solution*.

  Analysis: Treat the *Sample solution* and the *Standard solution* as follows. Heat 2.0 mL of the solution on a water bath for 20 min to remove the acetone. Cool to room temperature. Add 20.0 mL of *Solution A* to the solution under test, mix, and heat on a water bath for 20 min. Cool under running water, and quantitatively transfer to a 25-mL volumetric flask. Maintain the flask under running water, and dilute with sulfuric acid to volume. Within 10 min, determine the absorbance of the solution at 540 nm with a suitable spectrophotometer, using water as the blank.

  Acceptance criteria: The absorbance of the *Sample solution* is NMT that of the *Standard solution* (2.0%).

IMPURITIES

Inorganic Impurities

- **Heavy Metals,** *Method II* ⟨ **231**⟩**:** 20 ppm
- **Limit of Iron**

  Standard solution: Dissolve 863.4 mg of ferric ammonium sulfate $[FeNH_4(SO_4)_2 \cdot 12H_2O]$ in water, add 25 mL of 2 N sulfuric acid, dilute with water to 500.0 mL, and mix. Pipet 10 mL of this solution into a 100-mL volumetric flask, dilute with water to volume, and mix. Pipet 5 mL of this solution into a 100-mL volumetric flask, dilute with water to volume, and mix. This solution contains the equivalent of 1.0 µg/mL of iron.

  Sample solution: 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 ± 25°. 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 2 M ammonium carbonate, evaporate to dryness, and ignite as above. Cool, dissolve the residue in 50 mL of water, and mix.

  [Note—Reserve a portion of this solution for *Identification* test *C*.]

  Analysis: Treat the *Sample solution* and the *Standard solution* as follows. Transfer 10 mL of the solution to a suitable beaker, add 2 mL of citric acid solution (1 in 5) and 0.1 mL of thioglycolic acid, and mix. Render the solution alkaline, using litmus paper as an external indicator, by the addition of ammonium hydroxide. Dilute with water to 20 mL, and mix. Allow the solutions to stand for 5 min.

  Acceptance criteria: The color of the solution from the *Sample solution* is a shade of pink no deeper than that of the solution from the *Standard solution* (0.002%).

SPECIFIC TESTS

- **Microbial Enumeration Tests** ⟨**61**⟩ and **Tests for Specified Microorganisms** ⟨**62**⟩**:** It meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*.

- **pH** ⟨791⟩**:** Disperse 1 g in 30 mL of water. The pH of the resulting suspension is either 5.5–7.5 for Type A or 3.0–5.0 for Type B.
- **Loss on Drying** ⟨731⟩**:** Dry a sample at 130° for 90 min: it loses NMT 10.0% of its weight.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers, preferably protected from wide variations in temperature and humidity, which may cause caking.
- **Labeling:** Label it to indicate the botanical source of the starch from which it was derived, the cross-linking agent (if used), the pH range, and whether it is Type A or Type B.
- **USP Reference Standards** ⟨11⟩
  USP Sodium Starch Glycolate Type A RS
  USP Sodium Starch Glycolate Type B RS

Sodium Stearate

Octadecanoic acid, sodium salt.
Sodium stearate [822-16-2].

» Sodium Stearate is a mixture of sodium stearate ($C_{18}H_{35}NaO_2$) and sodium palmitate ($C_{16}H_{31}NaO_2$), which together constitute not less than 90.0 percent of the total content. The content of $C_{18}H_{35}NaO_2$ is not less than 40.0 percent of the total. Sodium stearate contains small amounts of the sodium salts of other fatty acids.

Packaging and storage—Preserve in well-closed, light-resistant containers.

USP Reference standards ⟨11⟩—
USP Palmitic Acid RS
USP Stearic Acid RS

Identification—

A: When heated, it fuses. At a high temperature it decomposes, emitting flammable vapors and the odor of burning fat, finally leaving a residue that, when moistened with water, is alkaline to litmus paper, effervesces with acids, and colors a nonluminous flame intensely yellow.

B: Dissolve 25 g in 300 mL of hot water, add 60 mL of 2 N sulfuric acid, and heat the solution, with frequent stirring, until the separated fatty acid layer is clear. Wash the fatty acids with boiling water until they are free from sulfate, collect in a small beaker, and warm on a steam bath until the water has settled and the fatty acids are clear. Allow the acids to cool, pour off the water layer, then melt the acids, filter into a dry beaker while hot, and dry at 105° for 20 minutes: the solidification temperature of the fatty acids is not less than 54°.

Acid value of fatty acids ⟨401⟩: between 196 and 211, determined on 1 g of the fatty acids obtained in *Identification* test *B*.

Iodine value of fatty acids ⟨401⟩: not more than 4.0, determined on the fatty acids obtained in *Identification* test *B*.

Acidity—Heat 50 mL of alcohol to the same temperature, ±5°, as that attained when the pink endpoint is reached in the titration of the test specimen. Add 3 drops of phenolphthalein TS and sufficient 0.020 N sodium hydroxide to produce a faint pink color. Add 2.00 g of Sodium Stearate, and dissolve with the aid of a small amount of heat: no pink color is produced. Titrate the solution with 0.020 N sodium hydroxide until a pink color is produced: between 1.00 mL and 4.25 mL of 0.020 N sodium hydroxide is required (between 0.28% and 1.2% as stearic acid).

Loss on drying ⟨731⟩—Tare a beaker containing about 1 g of washed sand, previously dried at 105°, add about 500 mg of Sodium Stearate, and again weigh. Add 10 mL of alcohol, evaporate the mixture at about 80° to dryness, and dry at 105° for 4 hours: it loses not more than 5.0% of its weight.

Alcohol-insoluble substances—Reflux 1.0 g with 25 mL of alcohol: it dissolves completely, and the resulting solution is clear or not more than slightly opalescent.

Assay—Proceed with Sodium Stearate as directed in the *Assay* under *Stearic Acid*, using about 100 mg of Sodium Stearate, accurately weighed. Determine the percentage of $C_{18}H_{35}NaO_2$ in the portion of Sodium Stearate taken by the formula:

$$100(A / B)$$

in which *A* is the area due to the methyl stearate peak, and *B* is the sum of the areas of all fatty acid ester peaks in the chromatogram. Similarly, determine the percentage of $C_{16}H_{31}NaO_2$.

Sodium Stearyl Fumarate

DEFINITION

Sodium Stearyl Fumarate contains NLT 99.0% and NMT 101.5% of sodium stearyl fumarate ($C_{22}H_{39}NaO_4$), calculated on the anhydrous basis.

IDENTIFICATION

- **Infrared Absorption** ⟨197K⟩
  Analysis: Perform test on an undried specimen (1 in 300).
  Acceptance criteria: Meets the requirements

ASSAY

- **Procedure**
  Sample: 250 mg
  Blank: 10 mL of chloroform
  Titrimetric system
  (See *Titrimetry* ⟨541⟩.)
  Mode: Direct titration
  Titrant: 0.1 N perchloric acid VS
  Endpoint detection: Visual
  Analysis: Transfer the *Sample* to a 50-mL conical flask, mix with 10 mL of chloroform, and add 20 mL of glacial acetic acid to dissolve. Add quinaldine red TS, and titrate with 0.1 N perchloric acid VS.
  Calculate the percentage of sodium stearyl fumarate ($C_{22}H_{39}NaO_4$) in the *Sample* taken:

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

V_S = volume of *Titrant* consumed by the *Sample* (mL)
V_B = volume of *Titrant* consumed by the *Blank* (mL)
N = actual normality of the *Titrant* (mEq/mL)
F = equivalency factor, 390.5 mg/mEq
W = weight of the *Sample* (mg)

Acceptance criteria: 99.0%–101.5% on the anhydrous basis

OTHER COMPONENTS

- **Limit of Sodium Stearyl Maleate and Stearyl Alcohol**
  Solvent: Chloroform and glacial acetic acid (4:1)
  Standard monostearyl maleate stock solution: 1 mg/mL of USP Monostearyl Maleate RS in *Solvent*
  Standard monostearyl maleate solution: Pipet 5.0 mL of *Standard monostearyl maleate stock solution*, and dilute with chloroform to 50 mL.
  Standard stearyl alcohol stock solution: 1 mg/mL of USP Stearyl Alcohol RS in *Solvent*
  Standard stearyl alcohol solution: Pipet 5.0 mL of *Standard stearyl alcohol stock solution*, and dilute with chloroform to 50 mL.
  Sample solution: Transfer 200 mg of Sodium Stearyl Fumarate to a small, glass-stoppered conical flask. Add 10.0 mL of *Solvent*. Dissolve by placing the flask in an ultrasonic bath for 10 min.
  Chromatographic system
  (See *Chromatography* ⟨621⟩*, Thin-Layer Chromatography*.)