15 mL of hydrochloric acid and 10 mL of water. Heat on a steam bath, with occasional mixing, to dissolve the dibasic calcium phosphate, but not longer than 30 min. Cool, add water to volume, and mix. If the solution is not clear, filter, discarding the first 10 mL of the filtrate.

Blank: Water

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Indicator: Hydroxy naphthol blue

Endpoint detection: Visual

Analysis: Transfer 25.0 mL of the *Sample solution* to a 250-mL beaker equipped with a magnetic stirrer. With constant stirring, add, in the order named, 0.5 mL of triethanolamine, 300 mg of *Indicator*, and from a 50-mL buret, about 23 mL of *Titrant*. Add sodium hydroxide solution (45 in 100) until the initial red color changes to clear blue. Continue to add it dropwise until the color changes to violet, and add an additional 0.5 mL. The pH is 12.3–12.5. Continue the titration dropwise with the *Titrant* to the appearance of a clear blue endpoint that persists for NLT 60 s. Perform a blank determination.

Calculate the percentage of the labeled amount of dibasic calcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$) in the portion of Tablets taken:

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

V_S = *Titrant* volume consumed by the *Sample solution* (mL)
V_B = *Titrant* volume consumed by the *Blank* (mL)
M = actual molarity of the *Titrant* (mM/mL)
F = equivalency factor, 172.08 mg/mM
W = nominal weight of dibasic calcium phosphate dihydrate in the *Sample solution* taken for *Analysis* (mg)

Acceptance criteria: 92.5%–107.5%

PERFORMANCE TESTS

- **DISSOLUTION** ⟨**711**⟩

Medium: 0.1 N hydrochloric acid; 900 mL

Apparatus 2: 75 rpm

Time: 45 min

Standard solution: Solution having a known concentration of calcium in *Medium*

Sample solution: Filtered portion of the solution under test, suitably diluted with *Medium* if necessary

Instrumental conditions

(See *Spectrophotometry and Light-Scattering* ⟨851⟩.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 422.7 nm

Lamp: Calcium hollow-cathode

Flame: Air-acetylene

Analysis

Samples: *Standard solution* and *Sample solution*

Determine the concentration of calcium (Ca) in the *Sample solution* in comparison with a *Standard solution*.

Calculate the percentage of the labeled amount of dibasic calcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$) dissolved:

$$\text{Result} = (A_U/A_S) \times (C_S \times D \times V/L) \times (M_r/A_r) \times 100$$

A_U = absorbance of the *Sample solution*
A_S = absorbance of the *Standard solution*
C_S = concentration of calcium in the *Standard solution* (mg/mL)
D = dilution factor for the *Sample solution*
V = volume of *Medium*, 900 mL
L = label claim (mg/Tablet)
M_r = molecular weight of dibasic calcium phosphate, 172.08
A_r = atomic weight of calcium, 40.08

Tolerances: NLT 75% (Q) of the labeled amount of dibasic calcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$) is dissolved.

- **UNIFORMITY OF DOSAGE UNITS** ⟨**905**⟩**:** Meet the requirements

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **LABELING:** The quantity of dibasic calcium phosphate stated in the labeling is in terms of dibasic calcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$).

Calcium Polycarbophil

Calcium polycarbophil [9003-97-8].

» Calcium Polycarbophil is the calcium salt of polyacrylic acid cross-linked with divinyl glycol.

Packaging and storage—Preserve in tight containers.

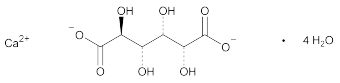
Identification—When tested as directed in the test for *Absorbing power*, it absorbs about 35 times its original weight.

Loss on drying ⟨731⟩**—**Dry it in vacuum at 130 ° for 4 hours: it loses not more than 10.0% of its weight.

Absorbing power—Transfer about 250 mg, accurately weighed, to a tared 50-mL centrifuge tube fitted with a tight closure. Add 35 mL of 0.1 N hydrochloric acid to the tube, seal the tube, and shake by mechanical means for 30 minutes. Centrifuge at 2000 rpm for 20 minutes, and decant and discard the supernatant. [NOTE—Exercise care to avoid any loss of particles.] Add 35 mL of 0.1 N hydrochloric acid, and shake for 30 minutes. Centrifuge, decanting and discarding the supernatant. Repeat the foregoing steps, using water instead of acid. Add 35 mL of a sodium bicarbonate solution (15 in 1000), and shake, venting as necessary to release any carbon dioxide liberated. Shake for 1 hour, centrifuge, and decant the supernatant. Add 35 mL of sodium bicarbonate solution (15 in 1000), and shake for 1 hour. Allow the tube and contents to stand overnight or until the contents have settled, and centrifuge. Withdraw the supernatant, and weigh the tube and contents. Calculate the weight of sodium bicarbonate solution absorbed: not less than 35.0 g of the sodium bicarbonate solution is absorbed by 1.0 g of Calcium Polycarbophil, calculated on the dried basis.

Content of calcium—Transfer about 2 g of Calcium Polycarbophil, accurately weighed, to a tared crucible. Cover, leaving the lid slightly ajar, and place in a muffle furnace. Heat to 600° over 2 hours, increase the temperature to 1000 ° over 1 hour, and maintain at 1000 ° for 1 hour. Allow to cool slowly. Dissolve the residue in dilute hydrochloric acid (1 in 5), quantitatively transfer with the aid of dilute hydrochloric acid (1 in 5) to a 100-mL volumetric flask, and dilute with dilute hydrochloric acid (1 in 5) to volume. Pipet 15 mL of this solution into a 250-mL beaker, and add, while stirring with a magnetic stirrer, 100 mL of water, 20.0 mL of 0.05 M edetate disodium VS, and 300 mg of hydroxy naphthol blue. Adjust with 1 N sodium hydroxide solution to a pH of 9.0 to 9.5. Adjust with about 10 mL of 2 N sodium hydroxide to a pH of 12.4. Titrate with 0.05 M edetate disodium VS to a persistent blue endpoint. Each mL of 0.05 M edetate disodium is equivalent to 2.004 mg of calcium (Ca). The content of Ca found is not less than 18.0% and not more than 22.0%, calculated on the dried basis.

Calcium Saccharate



$C_6H_8CaO_8 \cdot 4H_2O$ 320.26
D-Glucaric acid, calcium salt (1:1) tetrahydrate;
Calcium D-glucarate (1:1), tetrahydrate [5793-89-5].

DEFINITION
Calcium Saccharate is the calcium salt of D-saccharic acid. It contains NLT 98.5% and NMT 102.0% of $C_6H_8CaO_8 \cdot 4H_2O$.

IDENTIFICATION
- **A. IDENTIFICATION TESTS—GENERAL,** *Calcium* ⟨**191**⟩
  Sample: 0.2 g
  Analysis: Dissolve the *Sample* in 10 mL of water by the addition of 2 mL of hydrochloric acid.
  Acceptance criteria: Meets the requirements
- **B. INFRARED ABSORPTION** ⟨**197M**⟩**:** Meets the requirements

ASSAY
- **PROCEDURE**
  Sample: 600 mg
  Blank: 150.0 mL of water
  Titrimetric system
  (See *Titrimetry* ⟨541⟩.)
  Mode: Direct titration
  Titrant: 0.05 M edetate disodium VS
  Endpoint detection: Visual
  Analysis: Dissolve the *Sample* in 150 mL of water with the aid of a sufficient volume of hydrochloric acid. While stirring, preferably with a magnetic stirrer, add 30 mL of *Titrant* from a 50-mL buret. Add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue. Continue the titration to a blue endpoint. Per form a *Blank* determination and make any necessary corrections.
  Calculate the percentage of calcium saccharate ($C_6H_8CaO_8 \cdot 4H_2O$) in the portion of Calcium Saccharate taken:

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

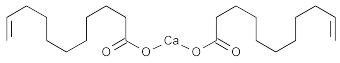
V_S = *Titrant* volume consumed by the *Sample* (mL)
V_B = *Titrant* volume consumed by the *Blank* (mL)
N = actual normality of the *Titrant* (mEq/mL)
F = equivalency factor, 320.2 mg/mEq
W = *Sample* weight (mg)
  Acceptance criteria: 98.5%–102.0%

IMPURITIES
- **CHLORIDE AND SULFATE,** *Chloride* ⟨**221**⟩
  Standard: 0.50 mL of 0.020 N hydrochloric acid
  Sample: 0.50 g dissolved in 10 mL of water by the addition of 2 mL of nitric acid
  Acceptance criteria: NMT 0.07%
- **CHLORIDE AND SULFATE,** *Sulfate* ⟨**221**⟩
  Standard: 0.6 mL of 0.020 N sulfuric acid
  Sample: 0.50 g dissolved in 10 mL of water by the addition of 2 mL of hydrochloric acid
  Acceptance criteria: NMT 0.12%
- **HEAVY METALS,** *Method II* ⟨**231**⟩**:** NMT 20 ppm
- **SUCROSE AND REDUCING SUGARS**
  Sample: 0.5 g
  Analysis: Dissolve the *Sample* in 10 mL of water with the addition of 2 mL of hydrochloric acid, and boil the solution for about 2 min. Cool, add 15 mL of sodium carbonate TS, allow to stand for 5 min, and filter. Add 5 mL of the clear filtrate to about 2 mL of alkaline cupric tartrate TS, and boil for 1 min.
  Acceptance criteria: No red precipitate is formed immediately.

SPECIFIC TESTS
- **OPTICAL ROTATION,** *Specific Rotation* ⟨**781S**⟩
  Sample solution: 60 mg/mL in 4.8 N hydrochloric acid that has been allowed to stand for 1 h
  Acceptance criteria: +18.5° to +22.5°

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Calcium Saccharate RS

Calcium Undecylenate



$C_{22}H_{38}O_4Ca$ 406.61
10-Undecenoic acid, calcium (2+) salt.
Calcium 10-undecenoate.

» Calcium Undecylenate contains not less than 98.0 percent and not more than 102.0 per cent of $C_{22}H_{38}O_4Ca$ (calcium undecylenate), calculated on the dried basis.

Packaging and storage—Preserve in well-closed containers.

Identification—

A: A filtered solution (1 in 20) in 3 N hydrochloric acid responds to the tests for *Calcium* ⟨191⟩.

B: Transfer 40 mL of water and 10 g of calcium undecylenate to a 250-mL separator. Cautiously and slowly add 10 mL of hydrochloric acid, while swirling. Insert the stopper, and shake. [NOTE—The separator will become quite warm, and pressure must be carefully and frequently relieved through the stopcock. If a curdy, white material remains after 5 minutes of shaking, add additional hydrochloric acid, 1 mL at a time, and shake until a clear oily phase is formed.] Allow the phases to separate, drain, and discard the bottom aqueous layer. Drain and discard the middle oily layer, if present. Filter the top layer of undecylenic acid through a pledget of cotton into a 10-mL graduated cylinder, noting the volume obtained. T ransfer the filtrate to a 250-mL flask, and add an equal volume of aniline. Reflux for 1 hour, swirling the flask occasionally. Allow to cool, and pour 60 mL of alcohol through the condenser into the flask. Remove the flask from the condenser, add 1 g of charcoal, and stir. Filter the slurr y into a 250-mL beaker. Add water dropwise until a few cr ystals form or the solution becomes slightly cloudy. [NOTE—If too much water is added, an oil will form. Add alcohol dropwise until the oil dissolves.] Allow the mixture to stand or refrigerate until cr ystals are formed. Collect the cr ystals on a filter paper inserted in a 45-mm porous glass filter funnel. Wash the crystals with 75 mL of 25% alcohol: the cr ystals have a clean, white, glossy appearance. If not, recr ystallize by dissolving the cr ystals in about 50 mL of alcohol. Add about 1 g of charcoal, stir, filter into a 150-mL beaker, and continue as directed above, beginning with "Add water dropwise." Dr y the crystals in vacuum at 50° for 2 hours: the cr ystals so obtained melt between 66° and 67.5°, the procedure for *Class Ia* being used (see *Melting Range or Temperature* ⟨741⟩). [NOTE—If the melting point is low, additional dr ying or recrystallization may be necessary.]

Loss on drying ⟨731⟩**—**Dry it at 105° for 2 hours: it loses between 2.0% and 5.7% of its weight.

Particle size ⟨786⟩**—**Test in accordance with this procedure, except to use not more than 25 g, and except that a single No. 100 sieve is used and is to be shaken for not less than 30 minutes or until sifting is practically complete: not less than 99.0% of it passes through a No. 100 sieve.