Calcium Carbonate Tablets—*see Calcium Carbonate Tablets General Monographs*

Calcium Citrate—*see Calcium Citrate General Monographs*

Calcium Citrate Tablets

DEFINITION

Calcium Citrate Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of calcium (Ca).

IDENTIFICATION

- **A.** The *Sample solution* from the test for *Strength* produces line emissions or absorptions at the characteristic wavelengths for calcium.
- **B. IDENTIFICATION TESTS—GENERAL,** *Calcium* ⟨**191**⟩ and *Citrate* ⟨**191**⟩

Analysis: Grind a Tablet to a fine powder in a mortar. Transfer the powder to a centrifuge tube, add 2–5 mL of water, sonicate for 1 min, shake, and centrifuge.

Acceptance criteria: The supernatant meets the requirements of the tests.

STRENGTH

[NOTE—A standard stock solution is commercially available at different calcium concentrations. Necessary volumetric adjustment can be made in the *Standard solution*. Concentrations of the *Standard solution* and the *Sample solution* may be modified to fit the linear or working range of the instrument.]

- **CONTENT OF CALCIUM,** *Procedure 1*

Standard stock solution: Weigh about 1.001 g of calcium carbonate, previously dried at 300° for 3 h and cooled in a desiccator for 2 h, and dissolve in 25 mL of 1 N hydrochloric acid. Boil to expel carbon dioxide, and dilute with water to 100 mL to obtain a solution having a known concentration of about 4000 µg/mL of calcium.

Standard solution: To a 200-mL volumetric flask add 100 mL of water and 4 mL of nitric acid, and mix thoroughly. Pipet 25.0 mL of the *Standard stock solution* into the volumetric flask, and dilute with water to volume to obtain a solution having a known concentration of about 500 µg/mL of calcium.

Sample solution: Weigh and finely powder NLT 20 Tablets. Transfer a weighed portion of the powdered Tablets, equivalent to about 0.1 g of calcium, to a 50-mL flask. Add 4 mL of nitric acid, and heat the solution to boil gently, during which fuming evolves. Boil the solution for an additional 30 min with constant swirling, during which no fuming should be observed. Cool the solution to room temperature, quantitatively transfer all of the solution to a 200-mL volumetric flask, dilute with water to volume, mix, and filter.

Instrumental conditions

(See *Plasma Spectrochemistry* ⟨730⟩.)

Mode: ICP–AES

Analytical wavelength: 317.93 nm. [NOTE—The operating conditions may be developed and optimized based on the manufacturer's recommendation. A typical setting includes radio frequency (RF) power of about 1300 watts, argon torch flow of about 15 L/min, argon auxiliary flow of about 0.2 L/min, and a nebulizer flow rate of about 0.8 L/min.]

Analysis: Determine the emission of the *Standard solution*, the *Sample solution,* and a 2% nitric acid solution as the blank at the wavelength indicated above.

Calculate the percentage of the labeled amount of calcium (Ca) in the portion of Tablets taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of calcium from the *Sample solution*

r_S = peak response of calcium from the *Standard solution*

C_S = concentration of calcium in the *Standard solution* (µg/mL)

C_U = nominal concentration of calcium in the *Sample solution* (µg/mL)

Acceptance criteria: 90.0%–110.0%

- **CONTENT OF CALCIUM,** *Procedure 2*

Lanthanum chloride solution: 267 mg/mL of lanthanum chloride heptahydrate in 0.125 N hydrochloric acid

Calcium standard solution: Dissolve 1.001 g of calcium carbonate, previously dried at 300° for 3 h and cooled in a desiccator for 2 h, in 25 mL of 1 N hydrochloric acid. Boil to expel carbon dioxide, and dilute with water to 1000 mL to obtain a concentration of 400 µg/mL of calcium.

Standard stock solution: 100 µg/mL of calcium from *Calcium standard solution* in 0.125 N hydrochloric acid

Standard solutions: Into separate 100-mL volumetric flasks pipet 1.0, 1.5, 2.0, 2.5, and 3.0 mL of the *Standard stock solution*. To each flask add 1.0 mL of *Lanthanum chloride solution*, and dilute with water to volume to obtain concentrations of 1.0, 1.5, 2.0, 2.5, and 3.0 µg/mL of calcium.

Sample solution: [NOTE—Finely powder NLT 20 Tablets.] Transfer an equivalent to 5 Tablets from powdered Tablets to a porcelain crucible. Heat the crucible in a muffle furnace maintained at 550° for 6–12 h, and cool. Add 60 mL of hydrochloric acid, and boil gently on a hot plate or steam bath for 30 min, intermittently rinsing the inner surface of the crucible with 6 N hydrochloric acid. Cool, and quantitatively transfer the contents of the crucible to a 100-mL volumetric flask. Rinse the crucible with small portions of 6 N hydrochloric acid, and add the rinsings to the flask. Dilute with water to volume, and filter, discarding the first 5 mL of the filtrate. Dilute this solution quantitatively, with 0.125 N hydrochloric acid to obtain a concentration of 2 µg/mL of calcium, adding 1 mL of *Lanthanum chloride solution* per 100 mL of the final volume.

Instrumental conditions

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

Mode: Atomic absorption spectrophotometry

Analytical wavelength: Calcium emission line at 422.7 nm

Lamp: Calcium hollow-cathode

Flame: Nitrous oxide–acetylene

Blank: 0.125 N hydrochloric acid containing 1 mL of *Lanthanum chloride solution* per 100 mL

Analysis

Samples: *Standard solutions* and the *Sample solution*

Determine the absorbances of the solutions, using the *Blank*. From a linear regression equation, calculated using the absorbance of the *Standard solutions* versus concentrations, determine the concentration, *C*, in µg/mL of calcium in the *Sample solution*.

Calculate the percentage of the labeled amount of calcium (Ca) in the portion of Tablets taken:

$$\text{Result} = (C/C_U) \times 100$$

C = determined concentration of calcium in the *Sample solution*
C_U = nominal concentration of calcium in the *Sample solution*

Acceptance criteria: 90.0%–110.0%

CONTAMINANTS

- **MICROBIAL ENUMERATION TESTS ⟨2021⟩:** The total aerobic microbial count does not exceed 1000 cfu/g. The total combined yeasts and molds count does not exceed 100 cfu/g.
- **ABSENCE OF SPECIFIED MICROORGANISMS ⟨2022⟩:** Meet the requirements of the test for absence of *Escherichia coli*

PERFORMANCE TESTS

- **DISINTEGRATION AND DISSOLUTION OF DIETARY SUPPLEMENTS ⟨2040⟩:** Meet the requirements for *Disintegration*, 15 min
- **WEIGHT VARIATION OF DIETARY SUPPLEMENTS ⟨2091⟩:** Meet the requirements

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **LABELING:** The label states the quantity of calcium in terms of mg/Tablet.

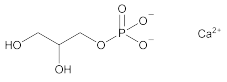
Calcium Glubionate Syrup—*see Calcium Glubionate Syrup General Monographs*

Calcium Gluceptate—*see Calcium Gluceptate General Monographs*

Calcium Gluconate—*see Calcium Gluconate General Monographs*

Calcium Gluconate Tablets—*see Calcium Gluconate Tablets General Monographs*

Calcium Glycerophosphate



$C_3H_7CaO_6P$ 210.14

1,2,3-Propanetriol, mono(dihydrogen phosphate) calcium salt (1:1);
Calcium glycerophosphate [27214-00-2].

DEFINITION

Calcium Glycerophosphate is a mixture, in variable proportions, of calcium (*RS*)-2,3-dihydroxypropyl phosphate and calcium 2-hydroxy-1-(hydroxymethyl)ethyl phosphate, which may be hydrated. Calcium Glycerophosphate contains NLT 18.6% and NMT 19.4% of calcium (Ca), calculated on the dried basis.

IDENTIFICATION

- **A.**
  Analysis: Ignite 0.1 g in a crucible. Take up the residue with 5 mL of nitric acid, heat on a water bath for 1 min, and filter. Mix 1 mL of the filtrate with 2 mL of ammonium molybdate TS.
  Acceptance criteria: A yellow color develops.
- **B.**
  Analysis: Dissolve 20 mg of the substance being examined in 5 mL of 5 M acetic acid, and add 0.5 mL of potassium ferrocyanide solution (53 mg/mL). The resulting solution remains clear. To the clear solution, add 50 mg of ammonium chloride.
  Acceptance criteria: A white crystalline precipitate is produced.

ASSAY

- **PROCEDURE**
  Sample: 200 mg
  Titrimetric system
  (See *Titrimetry* ⟨541⟩.)
  Mode: Direct titration
  Titrant: 0.1 M edetate disodium VS
  Endpoint detection: Colorimetric
  Blank: 300 mL of water. Add 6 mL of 10 M sodium hydroxide and 15 mg of calconcarboxylic acid triturate.
  Analysis: Dissolve the *Sample* in 300 mL of water, add 6 mL of 10 M sodium hydroxide and 15 mg of calconcarboxylic acid triturate. Titrate with *Titrant* until the solution is a distinct blue color.
  Calculate the percentage of calcium (Ca) in the portion of Calcium Glycerophosphate taken:

$$\text{Result} = [(V - B) \times M \times F \times 100]/W$$

V = *Sample* titrant volume (mL)
B = *Blank* titrant volume (mL)
M = titrant molarity (mM/mL)
F = equivalency factor, 40.08 mg/mM
W = weight of the *Sample* (mg)

Acceptance criteria: 18.6%–19.4% on the dried basis

IMPURITIES

- **LEAD ⟨251⟩:** NMT 4 ppm
- **IRON ⟨241⟩**
  Standard solution: Dilute 1 volume of *Standard Iron Solution*, prepared as directed in the chapter, with water to 10 volumes (1 µg/mL).
  Analysis: Dissolve 0.20 g in 10 mL of water. Add 2 mL of a 20% (w/v) solution of citric acid, 0.1 mL of thioglycolic acid, and mix. Make alkaline with 10 M ammonia, dilute with water to 20 mL, and allow to stand for 5 min. Any pink color produced is not more intense than that obtained by treating 4 mL of the *Standard solution* in the same manner.
  Acceptance criteria: NMT 20 ppm
- **HEAVY METALS,** *Method I* **⟨231⟩:** NMT 20 ppm
- **LIMIT OF CHLORIDE**
  Standard solution: 8.24 µg/mL of sodium chloride in water
  Sample solution: Dissolve 125 mg in a 10-mL mixture of 5 M acetic acid and water (2:8), and dilute with water to 15 mL.
  Analysis: To the *Sample solution* add 1 mL of 2 M nitric acid, then add 1 mL of a silver nitrate solution (17 mg/mL), and allow to stand for 5 min protected from light. To 10 mL of the *Standard solution* add 5 mL of water, 1 mL of 2 M nitric acid, and 1 mL of silver nitrate