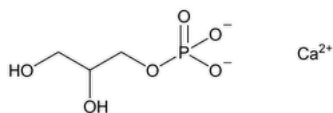


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Calcium Glycerophosphate



$C_3H_7CaO_6P$ 210.14

1,2,3-Propanetriol, mono(dihydrogen phosphate) calcium salt (1:1);

Calcium glycerophosphate CAS RN®: 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 trituate.

Analysis: Dissolve the *Sample* in 300 mL of water, add 6 mL of 10 M sodium hydroxide and 15 mg of calconcarboxylic acid trituate. 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

Change to read:

• ▲ [LEAD \(251\), Procedures, Procedure 1](#) ▲ (CN 1-JUN-2023) : NMT 4 ppm

Change to read:

- [▲ Iron \(241\), Procedures, Procedure 1 ▲](#) (CN 1-JUN-2023)

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

- **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 solution (17 mg/mL), and allow to stand for 5 min protected from light. When viewed against a dark background, the *Sample solution* is not more turbid than the *Standard solution*.

Acceptance criteria: NMT 0.04%

- **LIMIT OF SULFATE**

Standard solution: 36.2 µg/mL of potassium sulfate in water

Sample solution: Use the *Sample solution* prepared as directed in the test for *Appearance of Solution*.

Analysis: To 15 mL of the *Standard solution* add 0.5 mL of 5 M acetic acid and 1 mL of barium chloride solution (250 mg/mL). Repeat, using 15 mL of the *Sample solution*. Allow the solutions to stand for 5 min protected from light. When viewed against a dark background, the *Sample solution* is not more turbid than the *Standard solution*.

Acceptance criteria: NMT 0.2%

- **LIMIT OF ARSENIC**

Standard stock solution: In a 250-mL volumetric flask dissolve 330 mg of arsenic trioxide in 5 mL of 2 N sodium hydroxide, and dilute with water to volume. Transfer 1 mL of this solution to a 100-mL volumetric flask, and dilute with water to volume.

Standard solution: Transfer 1 mL of *Standard stock solution* into a 10-mL volumetric flask, and dilute with water volume.

Tin(II) chloride solution: Heat 20 g of tin with 85 mL of hydrochloric acid until no more hydrogen is released. Allow to cool. Dilute 1 volume of this solution with 10 volumes of dilute hydrochloric acid (200 mg/mL of hydrochloric acid in water).

Sample solution: Transfer 330 mg of Calcium Glycerophosphate to a 25.0-mL volumetric flask, and dilute with water to volume.

Apparatus: Prepare a 100-mL side-arm conical flask containing a magnetic stirring bar. Attach to the conical flask a ground-glass stopper through which passes a glass tube 20-cm long, with an internal diameter of 5 mm. The lower end of the tube is inside the conical flask and has been drawn to a tip with an internal diameter of 1 mm. An orifice 3 mm in diameter is 15 mm from the tip and at least 3 mm below the lower surface of the stopper. The upper end of the tube has a flat ground surface at a right angle to the axis of the tube. A second glass tube of the same internal diameter and 30 mm long, with a similar flat ground surface, is placed in contact with the ground surface of the first tube and is held in position by a clamp and springs. Into the lower tube, insert 55 mg of loosely packed lead acetate cotton. Place a disk of mercuric bromide paper between the flat surfaces of the tubes.

Analysis: Before placing the tube assembly into the flask, transfer the *Sample solution* to the flask, and add 15.0 mL of hydrochloric acid, 0.1 mL of *Tin(II) chloride solution*, and 5 mL of potassium iodide TS. Allow to stand for 15 min, and add 5 g of activated zinc. Assemble the apparatus immediately, and immerse the flask in a water bath at a temperature such that a uniform evolution of gas is maintained. After not less than 2 h, examine the stain produced on the mercuric bromide paper. Perform the same procedure using 1.0 mL of the *Standard solution*. The stain produced on the mercuric bromide paper by the *Sample solution* is not more intense than that produced by the *Standard solution*.

Acceptance criteria: NMT 3 ppm

- **LIMIT OF PHOSPHATES**

Sulfomolybdic solution: Dissolve with heating 2.5 g of ammonium molybdate in 20 mL of water. Dilute 28 mL of sulfuric acid with 50 mL of water, and cool. Mix the two solutions, and dilute with water to 100 mL.

Tin(II) chloride solution: Prepare as directed in the test for *Limit of Arsenic*.

Standard stock solution: 14.3 µg/mL of monobasic potassium phosphate in water.

Standard solution: Transfer 1.0 mL of *Standard stock solution* to a 100-mL volumetric flask, and dilute with water to volume.

Sample solution: Transfer 2.5 mL of the *Sample solution* from the test for *Appearance of Solution* to a 100-mL volumetric flask, and dilute with water to volume.

Analysis: To 100 mL of the *Sample solution* add 4 mL of *Sulfomolybdic solution*, mix, and add 0.1 mL of *Tin(II) chloride solution*. To 100 mL of the *Standard solution*, add 4 mL of *Sulfomolybdic solution*, mix, and add 0.1 mL of *Tin(II) chloride solution*. Allow the preparations to stand for 10 min, then examine 20 mL of each preparation. Any color produced by the *Sample solution* is not more intense than that produced by the *Standard solution*.

Acceptance criteria: NMT 0.04%

- **CITRIC ACID**

Mercury(II) sulfate solution: 1 g of mercuric oxide in 20 mL of water and 4 mL of sulfuric acid

Analysis: Mix 5 g of Calcium Glycerophosphate with 20 mL of carbon dioxide-free water, and filter. To the filtrate add 0.15 mL of sulfuric acid, and filter. To the filtrate add 5 mL of *Mercury(II) sulfate solution*, and heat to boiling. Add 0.5 mL of 0.2 M potassium permanganate, and heat to boiling.