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Tribasic Calcium Phosphate

$\text{Ca}_5(\text{OH})(\text{PO}_4)_3$ 502.31

Calcium hydroxide phosphate CAS RN®: 12167-74-7.

DEFINITION

Tribasic Calcium Phosphate consists of a variable mixture of calcium phosphates with the approximate composition $(10\text{CaO} \cdot 3\text{P}_2\text{O}_5 \cdot \text{H}_2\text{O})$. It contains NLT 34.0% and NMT 40.0% of calcium (Ca).

IDENTIFICATION

• A.

Sample solution: Dissolve 100 mg in 5 mL of [diluted nitric acid](#).

Analysis: Warm the *Sample solution*, and add 2 mL of [ammonium molybdate TS](#).

Acceptance criteria: A yellow precipitate is formed.

• B.

Sample: 100 mg of Tribasic Calcium Phosphate

Analysis: Dissolve the *Sample* by warming in 10 mL of 2 N [hydrochloric acid](#). Add 2.5 mL of [ammonia TS](#) dropwise, with shaking, and then add 5 mL of [ammonium oxalate TS](#).

Acceptance criteria: A white precipitate is formed.

ASSAY

• PROCEDURE

Sample: 150 mg of Tribasic Calcium Phosphate

Titrimetric system

(See [Titrimetry \(541\)](#).)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

Analysis: Dissolve the *Sample*, with the aid of gentle heat if necessary, in a mixture of [hydrochloric acid](#) and [water](#) (5:3) contained in a 250-mL beaker equipped with a magnetic stirrer, and cautiously add 125 mL of [water](#). With constant stirring, add in the following order: 0.5 mL of [triethanolamine](#), 300 mg of [hydroxy naphthol blue](#), and from the titration 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 *Titrant* to the appearance of a clear blue endpoint that persists for NLT 60 s. Calculate the percentage of calcium (Ca) in the portion of the *Sample* taken:

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

V_s = *Titrant* volume consumed by the *Sample* (mL)

M = actual molarity of the *Titrant* (mmol/mL)

F = equivalency factor, 40.08 mg/mmol

W = *Sample* weight (mg)

Acceptance criteria: 34.0%–40.0%

IMPURITIES

• [CHLORIDE AND SULFATE \(221\)](#), [Chloride](#)

Standard: 1.0 mL of 0.020 N [hydrochloric acid](#)

Sample: 0.500 g of Tribasic Calcium Phosphate

Analysis: Dissolve the *Sample* in 25 mL of 2 N [nitric acid](#), and add 1 mL of silver nitrate TS.

Acceptance criteria: NMT 0.14%

• [CHLORIDE AND SULFATE \(221\)](#), [Sulfate](#)

Standard: 1.0 mL of 0.020 N [sulfuric acid](#)

Sample: 0.500 g of Tribasic Calcium Phosphate

Analysis: Dissolve the *Sample* in the smallest possible amount of 3 N [hydrochloric acid](#). Dilute with [water](#) to 100 mL, and filter, if necessary.

To 25 mL of the filtrate add 1 mL of [barium chloride TS](#).

Acceptance criteria: NMT 0.8%

Change to read:

- **▲ [ARSENIC \(211\), Procedures, Procedure 1](#) ▲** (CN 1-Jun-2023)

Test preparation: Dissolve 1.0 g in just sufficient 3 N [hydrochloric acid](#).

Acceptance criteria: NMT 3 ppm

- **BARIUM**

Sample: 500 mg of Tribasic Calcium Phosphate

Analysis: Mix the *Sample* with 10 mL of [water](#), heat, add [hydrochloric acid](#), dropwise, until solution is effected, and then add 2 drops of the acid in excess. Filter, and add to the filtrate 1 mL of [potassium sulfate TS](#).

Acceptance criteria: No turbidity appears within 15 min.

- **CARBONATE**

Sample: 2 g of Tribasic Calcium Phosphate

Analysis: Mix the *Sample* with 20 mL of [water](#), and add 3 N [hydrochloric acid](#), dropwise, to effect solution.

Acceptance criteria: No effervescence is produced.

- **ACID-INSOLUBLE SUBSTANCES**

Analysis: If an insoluble residue remains in the test for *Carbonate*, boil the solution, filter, wash the residue well with hot water until the last washing is free from chloride, and ignite the residue to constant weight.

Acceptance criteria: NMT 0.2%; the weight of the residue is NMT 4 mg.

- **DIBASIC SALT AND CALCIUM OXIDE**

Sample: 1.5 g of Tribasic Calcium Phosphate

Titrimetric system

(See [Titrimetry \(541\)](#).)

Mode: Residual titration

Titrant: 1 N [hydrochloric acid VS](#)

Back-titrant: 0.1 N [sodium hydroxide VS](#)

Endpoint detection: Potentiometric

Analysis: Dissolve the *Sample* by warming with 25.0 mL of *Titrant*. Cool, and slowly titrate the excess of *Titrant*, while agitating constantly, with the *Back-titrant* to a pH of 4.0.

Acceptance criteria: 13.0–14.3 mL of 1 N [hydrochloric acid](#) is consumed for each g of salt, calculated on the ignited basis.

- **LIMIT OF FLUORIDE**

[NOTE—Prepare and store all solutions in plastic containers.]

Buffer solution: 294 mg/mL of [sodium citrate dihydrate](#) in [water](#)

Standard stock solution: 1.1052 mg/mL of [USP Sodium Fluoride RS](#) in [water](#)

Standard solution: Transfer 20.0 mL of *Standard stock solution* to a 100-mL volumetric flask containing 50.0 mL of *Buffer solution*, dilute with [water](#) to volume, and mix. Each mL of this solution contains 100 µg of fluoride ion.

Sample solution: Transfer 2.0 g of Tribasic Calcium Phosphate to a beaker containing a plastic-coated stirring bar. Add 20 mL of [water](#) and 3.0 mL of [hydrochloric acid](#), and stir until dissolved. Add 50.0 mL of *Buffer solution* and sufficient [water](#) to make 100 mL.

Electrode system: Use a fluoride-specific ion-indicating electrode and a silver–silver chloride reference electrode connected to a pH meter capable of measuring potentials with a minimum reproducibility of ±0.2 mV (see [pH \(791\)](#)).

Analysis

Samples: *Standard solution* and *Sample solution*

Standard response line: Transfer 50.0 mL of *Buffer solution* and 3.0 mL of [hydrochloric acid](#) to a beaker, and add [water](#) to make 100 mL.

Add a plastic-coated stirring bar, insert the electrodes into the solution, stir for 15 min, and read the potential, in mV. Continue stirring, and at 5-min intervals add 100, 100, 300, 500, and 500 µL of *Standard solution*, reading the potential 5 min after each addition. Plot the logarithms of the cumulative fluoride ion concentrations (0.1, 0.2, 0.5, 1.0, and 1.5 µg/mL) versus potential, in mV.

Rinse and dry the electrodes, insert them into the *Sample solution*, stir for 5 min, and read the potential, in mV. From the measured potential and the *Standard response line* determine the concentration, *C* (in µg/mL), of fluoride ion in the *Sample solution*.

Calculate the content of fluoride in the portion of Tribasic Calcium Phosphate taken:

$$\text{Result} = (V \times C)/W$$

V = volume of the *Sample solution* (mL)

C = concentration of fluoride ion in the *Sample solution* determined from the *Standard response line* (µg/mL)

W = weight of Tribasic Calcium Phosphate taken to prepare the *Sample solution* (g)