Anhydrous Dibasic Calcium Phosphate

Change to read:

▲Portions of this monograph that are national *USP* text, and are not part of the harmonized text, are marked with symbols (◆,◆) to specify this fact.▲ USP 1-Dec-2019

$CaHPO_4$ 136.06
Phosphoric acid, calcium salt (1:1);
Calcium phosphate (1:1) [7757-93-9].

Change to read:

DEFINITION

Anhydrous Dibasic Calcium Phosphate contains ▲NLT 97.5% and NMT 102.5%▲ USP 1-Dec-2019 of anhydrous dibasic calcium phosphate ($CaHPO_4$).

IDENTIFICATION

• **A.**
Sample: 0.1 g of Anhydrous Dibasic 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.

• **B.**
Sample: 0.1 g of Anhydrous Dibasic Calcium Phosphate
Analysis: Dissolve the *Sample* in 5 mL of diluted nitric acid. Warm the solution to 70°, and add 2 mL of 10% ammonium molybdate solution (freshly prepared).
Acceptance criteria: A yellow precipitate of ammonium phosphomolybdate is formed.

ASSAY

Change to read:

• **PROCEDURE**
Buffer: Dissolve 53.5 g of ammonium chloride with sufficient water in a 1000-mL volumetric flask. Add 570 mL of ammonia water, stronger, and dilute with water to volume. The pH of this solution is 10.7.
Sample solution: Transfer 400 mg of Anhydrous Dibasic Calcium Phosphate to a 200-mL volumetric flask. Dissolve in 12 mL of diluted hydrochloric acid with the aid of gentle heat, if necessary, and dilute with water to volume.
Blank: 20 mL of water containing 1.2 mL of diluted hydrochloric acid
Titrimetric system
(See *Titrimetry* ⟨541⟩.)
Mode: Residual titration
Titrant: 0.02 M edetate disodium VS
Back-titrant: 0.02 M zinc sulfate VS
Endpoint detection: Visual
Analysis: To 20.0 mL of the *Sample solution* add 25.0 mL of *Titrant*, 50 mL of water, and 5 mL of *Buffer*. Add 25 mg of eriochrome black T–sodium chloride indicator. Titrate the excess *Titrant* with the *Back-titrant*. Perform a *Blank* determination in the same manner.
Calculate the percentage of anhydrous dibasic calcium phosphate ($CaHPO_4$) in the sample taken:

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

V_B = *Back-titrant* volume consumed by the *Blank* (mL)
V_S = *Back-titrant* volume consumed by the *Sample* (mL)
M = actual molarity of the *Back-titrant* (mM/mL)
F = equivalency factor, 136.06 mg/mM
W = sample weight (mg) in 20.0 mL of the *Sample solution*

Acceptance criteria: ▲97.5%–102.5%▲ USP 1-Dec-2019

IMPURITIES

• **CARBONATE**
Sample: 1.0 g of Anhydrous Dibasic Calcium Phosphate
Analysis: Mix the *Sample* with 5 mL of carbon dioxide-free water, and immediately add 2 mL of hydrochloric acid.
Acceptance criteria: No effervescence occurs.

Change to read:

• **CHLORIDE AND SULFATE** ⟨221⟩**,** *Chloride*
Standard ▲solution:▲ USP 1-Dec-2019 To 0.70 mL of ▲0.01▲ USP 1-Dec-2019 N hydrochloric acid, ▲add 6 mL of diluted nitric acid, and dilute with water to 50 mL.▲ USP 1-Dec-2019
Sample ▲solution:▲ USP 1-Dec-2019 To ▲0.20▲ USP 1-Dec-2019 g of Anhydrous Dibasic Calcium Phosphate, add 20 mL of water and 13 mL of diluted nitric acid, and warm gently, if necessary, to completely dissolve. Dilute with water to 100 mL, and filter if necessary. ▲Use 50 mL of the solution as *Sample solution*.▲ USP 1-Dec-2019
Analysis
Samples: *Standard solution* and *Sample solution*
▲Add 1 mL of silver nitrate TS to the *Sample solution* and the *Standard solution*, respectively. Mix well, and allow to stand for 5 min protecting from direct sunlight. Compare the opalescence developed in both solutions against a black background by viewing downward or transversely.▲ USP 1-Dec-2019
Acceptance criteria: The ▲opalescence▲ USP 1-Dec-2019 of the *Sample* ▲*solution*▲ USP 1-Dec-2019 does not exceed that of the *Standard* ▲*solution*▲ USP 1-Dec-2019 (NMT 0.25%).

Change to read:

• **CHLORIDE AND SULFATE** ⟨221⟩**,** *Sulfate*
Standard ▲solution:▲ USP 1-Dec-2019 To 1.0 mL of 0.010 N sulfuric acid, ▲add 1 mL of diluted hydrochloric acid, and dilute with water to 50 mL.▲ USP 1-Dec-2019
Sample ▲solution:▲ USP 1-Dec-2019 To 0.5 g of Anhydrous Dibasic Calcium Phosphate, add 5 mL of water and 5 mL of diluted hydrochloric acid, and warm gently, if necessary, to completely dissolve. Dilute with water to 100 mL, and filter if necessary. To 20 mL of the ▲solution▲ USP 1-Dec-2019 add 1 mL of diluted hydrochloric acid, and dilute with water to 50 mL.
Analysis
Samples: *Standard solution* and *Sample solution*
▲Add 2 mL of barium chloride TS to the *Sample solution* and the *Standard solution*, respectively. Mix well, and allow to stand for 10 min. Compare the white turbidity produced in both solutions against a black background by viewing downward or transversely.▲ USP 1-Dec-2019
Acceptance criteria: The turbidity of the *Sample* ▲*solution*▲ USP 1-Dec-2019 does not exceed that of the *Standard* ▲*solution*▲ USP 1-Dec-2019 (NMT 0.5%).

Change to read:

• ▲◆▲ USP 1-Dec-2019 **ARSENIC** ⟨211⟩**,** *Method I*
Test preparation: 1.0 g in 25 mL of 3 N hydrochloric acid, diluted with water to 55 mL. Omit the addition of 20 mL of 7 N sulfuric acid specified in *Procedure*.

Acceptance criteria: NMT 3 µg/g▲• USP 1-Dec-2019

- **BARIUM**

Sample: 0.5 g Anhydrous Dibasic Calcium Phosphate
Analysis: Heat the *Sample* to boiling with 10 mL of water, and add 1 mL of hydrochloric acid dropwise, stirring after each addition. Allow to cool, and filter if necessary. To the filtrate add 2 mL of potassium sulfate TS.
Acceptance criteria: No turbidity is produced within 10 min.

- **LIMIT OF ACID-INSOLUBLE SUBSTANCES**

Sample solution: Dissolve 5.0 g in a mixture of 40 mL of water and 10 mL of hydrochloric acid by boiling gently for 5 min.
Analysis: After cooling, collect the insoluble substance on ashless filter paper, and wash with water until the last washing does not give a reaction for chloride (no turbidity results from the addition of silver nitrate TS). Ignite to completely incinerate the residue and the ashless filter paper at 600 ± 50°.
Acceptance criteria: The weight of the residue does not exceed 10 mg (NMT 0.2%).

Change to read:

- ▲• USP 1-Dec-2019**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 milliliter of this solution contains 100 µg of fluoride ion.
Sample solution: Transfer 2.0 g of Anhydrous Dibasic Calcium Phosphate to a beaker containing a plastic-coated stirring bar. Add 20 mL of water and 2.0 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 2.0 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 millivolts. Continue stirring, and at 5-min intervals add 100, 100, 300, 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, and 1.0 µg/mL) versus potential in millivolts.
Rinse and dry the electrodes, insert them into the *Sample solution*, stir for 5 min, and read the potential in millivolts. 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 ppm in the portion of Anhydrous Dibasic Calcium Phosphate taken:

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

V = *Sample solution* volume (mL)
C = concentration of fluoride ion, determined from the *Standard response line*, in the *Sample solution* (µg/mL)
W = weight of Anhydrous Dibasic Calcium Phosphate taken to prepare the *Sample solution* (g)

Acceptance criteria: NMT 50 ppm▲• USP 1-Dec-2019

SPECIFIC TESTS

Change to read:

- **LOSS ON IGNITION** ⟨733⟩

Sample: 1 g of Anhydrous Dibasic Calcium Phosphate
Analysis: Ignite the *Sample* at 800°–825° to constant weight.
Acceptance criteria: ▲6.6%–8.7%▲ USP 1-Dec-2019

ADDITIONAL REQUIREMENTS

Change to read:

- ▲• USP 1-Dec-2019**PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements specified.▲• USP 1-Dec-2019
- **USP REFERENCE STANDARDS** ⟨11⟩

USP Sodium Fluoride RS