## Calcium phosphate (Calcii phosphas)

**Chemical name**. Calcium phosphate (3:2) mixture with calcium phosphate (1:1); CAS Reg. No. 7758-87-4 [$Ca_3(PO_4)_2$]; CAS Reg. No. 7757-93-9 ($CaHPO_4$).

**Other name**. Tribasic calcium phosphate.

**Description**. A white, amorphous powder; odourless or almost odourless.

**Solubility**. Practically insoluble in water and ethanol (~750 g/l) TS; soluble in hydrochloric acid (~70 g/l) TS and nitric acid (~130 g/l) TS.

**Category**. Tablet diluent.

**Storage**. Calcium phosphate should be kept in a well-closed container.

**Additional information**. At relative humidities between 15% and 65%, the equilibrium moisture content at 25 °C is about 2%, but at relative humidities above 75% small additional amounts of moisture are absorbed.

### Requirements

**Definition.** Calcium phosphate is a mixture consisting mainly of $Ca_3(PO_4)_2$ together with $CaHPO_4$.

Calcium phosphate contains not less than **34.0%** and not more than the equivalent of **40.0%** of calcium, Ca, calculated with reference to the ignited substance.

### Identity tests

A. Dissolve 0.05 g in 1 mL of hydrochloric acid (~70 g/l) TS by gentle warming and add 4 mL of water and 0.5 g of sodium acetate R. It yields reaction A described under 2.1 General identification tests as characteristic of calcium.

B. To 0.5 g add 2 mL of nitric acid (~130 g/l) TS and heat gently. This solution yields reaction A described under 2.1 General identification tests as characteristic of orthophosphates.

**Heavy metals**. For the preparation of the test solution use 1.0 g dissolved in 10 mL of hydrochloric acid (~70 g/l) TS. Heat to boiling, cool, dilute to 40 mL with water, and mix. Determine the heavy metals content as described under 2.2.3 Limit test for heavy metals, Method A; not more than 30 µg/g.

**Arsenic**. Use a solution of 3.3 g in 35 mL of hydrochloric acid (~70 g/l) TS, heat to dissolve and proceed as described under 2.2.5 Limit test for arsenic; the arsenic content is not more than 3 µg/g.

**Barium**. Mix 0.5 g with 10 mL of water, heat, and add, drop by drop, hydrochloric acid (~420 g/l) TS until solution is effected. Add an excess of 2 drops of acid, filter, and to the filtrate add 1 mL of potassium sulfate (0.1 g/l) TS; no turbidity appears within 15 minutes.

**Carbonates**. Suspend 5 g in 30 mL of carbon-dioxide-free water R and add slowly 10 mL of hydrochloric acid (~70 g/l) TS; not more than a slight effervescence is observed. (Keep the solution for "Acid-insoluble substances".)

**Chlorides**. Dissolve 0.2 g in a mixture of 2 mL of nitric acid (~130 g/l) TS and 20 mL of water, and proceed as described under 2.2.1 Limit test for chlorides; the chloride content is not more than 1.4 mg/g.

**Fluorides**. Prepare and store all solutions in plastic containers.

Weigh 2.0 g of the test sample into a beaker, add 20 mL of water and 3 mL of hydrochloric acid (~250 g/l) TS. Using a magnetic stirrer and a plastic-coated stirring bar, stir until the sample has dissolved. Then add 50 mL of sodium citrate (250 g/l) TS and dilute to 100 mL with water. Use a fluoride-ion-sensitive electrode and a silver/silver chloride reference electrode system, connected to a potentiometer capable of indicating reproducibly a minimum of ±0.2 mV. Insert the previously rinsed and dried electrodes into the solution, stir for 5 minutes, and read the potential in mV.

Prepare a standard solution of fluoride ion containing 1.1052 mg sodium fluoride R per mL. To 20 mL of this solution add 50 mL of sodium citrate (250 g/l) TS and dilute with sufficient water to produce 100 mL (100 µg F/mL). For the establishment of a standard curve, place 50 mL of sodium citrate (250 g/l) TS in a beaker, add 3 mL of hydrochloric acid (~250 g/l) TS, and dilute to 100 mL with water. Stir as described above for 15 minutes, insert the electrodes, and read the potential in mV. Continue to stir, and at 5-minute intervals add 100 µl, 100 µl, 300 µl, 500 µl, and 500 µl of fluoride ion standard solution (100 µg F/mL), equivalent to the cumulative fluoride ion concentration of 0.1, 0.2, 0.5, 1.0, and 1.5 µg/mL, reading the potential 5 minutes after each addition. Plot the logarithms of the cumulative fluoride ion concentration versus potential.

Determine the concentration of fluoride ion in the solution being examined, reading off from the standard curve the value of mV correlating with the µg of F/mL, and divide by the sample mass taken to obtain the content in the sample; not more than 75 µg/g.

**Sulfates**. Dissolve 0.1 g in 5 mL of hydrochloric acid (~70 g/l) TS, and proceed as described under 2.2.2 Limit test for sulfates; the sulfate content is not more than 8 mg/g.

**Acid-insoluble substances**. Filter the solution prepared for "Carbonates", wash the residue with water, and dry to constant mass at 105 °C; the residue weighs not more than 15 mg (0.3%).

**Loss on ignition**. Ignite 1.0 g at 800 °C for 30 minutes; it loses not more than 80 mg/g.

**Assay**. To about 0.15 g, accurately weighed, add a mixture of 5 mL of hydrochloric acid (~420 g/l) TS and 3 mL of water, use gentle heat to dissolve, and add 125 mL of water. Proceed with the titration as described under 2.5 Complexometric titrations for calcium.

Each mL of disodium edetate (0.05 mol/l) VS is equivalent to 2.004 mg of Ca.