Calcium hydrogen phosphate (Calcii hydrogenophosphas)

Calcium hydrogen phosphate, anhydrous

Calcium hydrogen phosphate dihydrate

$CaHPO_4$ (anhydrous)

$CaHPO_4,2H_2O$ (dihydrate)

Relative molecular mass. 136.1 (anhydrous); 172.1 (dihydrate).

Chemical name. Calcium phosphate (1:1); CAS Reg. No. 7757-93-9 (anhydrous).

Calcium phosphate (1:1) dihydrate; CAS Reg. No. 7789-77-7 (dihydrate).

Other name. Dibasic calcium phosphate.

Description. A white or almost white powder; odourless.

Solubility. Practically insoluble in cold water and ethanol (~750 g/l) TS; soluble in dilute acids.

Category. Tablet and capsule diluent.

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

Labelling. The designation on the container of Calcium hydrogen phosphate should state whether it is the dihydrate or the anhydrous form.

Requirements

Calcium hydrogen phosphate contains not less than **30.9%** and not more than the equivalent of **31.7%** of calcium, Ca, calculated with reference to the ignited substance.

Identity tests

A. To 0.2 g add a mixture of 10 mL of hydrochloric acid (~70 g/l) TS and 10 mL of water, and heat to dissolve. To 10 mL of this solution add 2.5 mL of ammonia (~100 g/l) TS (keep the remaining solution for test B); it yields reaction A described under 2.1 General identification tests as characteristic of calcium.

B. Acidify the remaining solution from test A with nitric acid (~130 g/l) TS; it 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, filter if necessary, and add ammonia (~100 g/l) TS until a precipitate is formed. Add just sufficient hydrochloric acid (~70 g/l) TS to dissolve the precipitate and determine the heavy metals content as described under 2.2.3 Limit test for heavy metals, Method A; not more than 40 µg/g.

Arsenic. Use a solution of 1.0 g in 35 mL of hydrochloric acid (~70 g/l) TS and proceed as described under 2.2.5 Limit test for arsenic; the arsenic content is not more than 3 µg/g.

Barium. Dissolve 1.25 g in 10 mL of hydrochloric acid (~70 g/l) TS, filter if necessary, and add ammonia (~100 g/l) TS until a precipitate is formed. Add just sufficient hydrochloric acid (~70 g/l) TS to dissolve the precipitate and dilute with water to 25 mL. Place a 10-mL portion in each of two separate matched tubes. To one portion add 0.5 mL of sulfuric acid (~100 g/l) TS, and to the other 0.5 mL of water; the solutions remain equally clear when viewed after 15 minutes.

Carbonates. To 1 g add 5 mL of carbon-dioxide-free water R and 2 mL of hydrochloric acid (~420 g/l) TS and shake; no effervescence is produced.

Chlorides. Dissolve 0.1 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 2.5 mg/g.

Fluorides. Prepare and store all solutions in plastic containers.

Weigh 2.0 g of the test sample into a beaker and add 20 mL of water and 2.0 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 2 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, 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, and 1.0 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 50 µg/g.

Sulfates. Dissolve 0.10 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 5 mg/g.

Acid-insoluble substances. To 5 g add a mixture of 40 mL of water and 10 mL of hydrochloric acid (~420 g/l) TS, heat until no more dissolves, and dilute to 100 mL with water. Filter any residue, wash with hot water until the washing is free of chlorides, dry the residue at 105 °C for 1 hour, and weigh; not more than 2 mg/g.

Loss on ignition. Ignite 1.0 g to constant mass between 800 and 825 °C. The anhydrous form loses not less than 66 mg/g and not more than 85 mg/g. The dihydrate loses not less than 0.245 g/g and not more than 0.265 g/g.

Assay. To about 0.2 g, accurately weighed, add a mixture of 1 mL of hydrochloric acid (~420 g/l) TS and 5 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.