Calcium Phosphate, Dibasic

Dicalcium Phosphate

CaHPO$_4$·2H$_2$O

Formula wt, anhydrous 136.06
Formula wt, dihydrate 172.09

CAS: anhydrous [7757-93-9]
CAS: dihydrate [7789-77-7]

DESCRIPTION

Dibasic Calcium Phosphate is anhydrous or contains two molecules of water of hydration. It occurs as a white, odorless, tasteless powder that is stable in air. It is practically insoluble in water, but is readily soluble in dilute hydrochloric and nitric acids. It is insoluble in alcohol.

Functional Use in Foods  Leavening agent; dough conditioner; nutrient; dietary supplement; yeast food.

REQUIREMENTS

Labeling  Indicate whether it is anhydrous or the dihydrate.

Identification
   A. Dissolve about 100 mg by warming with a mixture of 5 mL of 2.7 N hydrochloric acid and 5 mL of water, add 2.5 mL of 6 N ammonium hydroxide, dropwise, with shaking, and then add 5 mL of ammonium oxalate TS. A white precipitate is formed.
   B. To 10 mL of a warm solution (1 in 100) in a slight excess of nitric acid add 10 mL of ammonium molybdate TS. A yellow precipitate of ammonium phosphomolybdate is formed.

Assay  Not less than 97.0% and not more than 105.0% of Dibasic Calcium Phosphate (CaHPO$_4$) or of Dibasic Calcium Phosphate, Dihydrate, (CaHPO$_4$·2H$_2$O).

Arsenic (as As)  Not more than 3 mg/kg.
Fluoride  Not more than 0.005%.
Heavy Metals (as Pb)  Not more than 0.0015%.
Lead  Not more than 25 mg/kg.

Loss on Ignition  CaHPO$_4$ (anhydrous): between 7.0% and 8.5%; CaHPO$_4$·2H$_2$O; (dihydrate): between 24.5% and 26.5%.
Assay Dissolve about 250 mg of Dibasic Calcium Phosphate, accurately weighed, with the aid of gentle heat if necessary, in a mixture of 5 mL of hydrochloric acid and 3 mL of water contained in a 250-mL beaker equipped with a magnetic stirrer, and cautiously add 125 mL of water. With constant stirring, add, in the order named, 0.5 mL of triethanolamine, 300 mg of hydroxy naphthol blue indicator, and from a 50-mL buret, about 23 mL of 0.05 M disodium ethylenediaminetetraacetate. Add sodium hydroxide solution (45 in 100) until the initial red color changes to clear blue, then continue to add it dropwise until the color changes to violet, then add an additional 0.5 mL. The pH is between 12.3 and 12.5. Continue the titration dropwise with the 0.05 M disodium ethylenediaminetetraacetate to the appearance of a clear blue endpoint that persists for not less than 60 s. Each mL of 0.05 M disodium ethylenediaminetetraacetate is equivalent to 6.803 mg of CaHPO$_4$ or to 8.604 mg of CaHPO$_4$.2H$_2$O.

Arsenic A solution of 1 g in 5 mL of 2.7 N hydrochloric acid meets the requirements of the Arsenic Test, Appendix IIIB.

Fluoride (Note: Prepare and store all solutions in plastic containers.)

Buffer Solution Dissolve 73.5 g of sodium citrate in water to make 250 mL of solution.

Standard Solution Dissolve an accurately weighed quantity of USP Sodium Fluoride RS quantitatively in water to obtain a solution containing 1.1052 mg/mL. Transfer 20.0 mL of the resulting solution to a 100-mL volumetric flask containing 50 mL of Buffer Solution, dilute with water to volume, and mix. Each mL of this solution contains 100 µg of fluoride ion.

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.

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 mV. Continue stirring, and at 5-min intervals, add 100 µL, 100 µL, 300 µL, 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 mV.

Procedure Transfer 2.0 g of the specimen under test 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 of test solution. Rinse and dry the electrodes, insert them into the test 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 test solution. Calculate the percentage of fluoride ion in the specimen taken by the formula

\[ C \times 0.005. \]

Heavy Metals Warm 2.66 g with 5 mL of 2.7 N hydrochloric acid until no more dissolves, dilute to 50 mL with water, and filter. A 25-mL portion of the filtrate meets the requirements of the Heavy Metals Test, Appendix IIIB, using 20 µg of lead ion (Pb) in the control (Solution A).

Lead A 10-g sample using a 5-µg/mL Standard Lead Solution meets the requirements of the APDC Extraction Method for Lead, Appendix IIIB.

Loss on Ignition Weigh accurately about 3 g, and ignite, preferably in a muffle furnace, at 800° to 825° to constant weight.

Packaging and Storage Store in well-closed containers.