It is practically insoluble in water, in ethanol (95) and in diethyl ether.

It dissolves in dilute hydrochloric acid and in dilute nitric acid.

**Identification** (1) Dissolve 0.1 g of Dibasic Calcium Phosphate in 10 mL of diluted hydrochloric acid (1 in 6) by warming, add 2.5 mL of ammonia TS dropwise with shaking, and add 5 mL of ammonium oxalate TS: a white precipitate is produced.

(2) Dissolve 0.1 g of Dibasic Calcium Phosphate in 5 mL of dilute nitric acid, and add 2 mL of hexaammonium heptamolybdate TS after warming for 1 to 2 minutes at 70°C: a yellow precipitate is produced.

**Purity** (1) Acid-insoluble substance—Dissolve 5.0 g of Dibasic Calcium Phosphate in 40 mL of water and 10 mL of hydrochloric acid, and boil for 5 minutes. After cooling, collect the insoluble substance using filter paper for assay. Wash with water until no more turbidity of the washing is produced when silver nitrate is added. Ignite to incinerate the residue and filter paper: the mass is not more than 2.5 mg (not more than 0.05%).

(2) Chloride—Dissolve 0.20 g of Dibasic Calcium Phosphate in 20 mL of water and 13 mL of dilute nitric acid, add water to make 100 mL, and filter, if necessary. Perform the test using a 50-mL portion of this solution as the test solution. Prepare the control solution with 0.70 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.248%).

(3) Sulfate—Dissolve by warming 1.0 g of Dibasic Calcium Phosphate in 5 mL of water and 5 mL of dilute hydrochloric acid, add water to make 100 mL, and filter, if necessary. Take 30 mL of the filtrate, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 1.0 mL of 0.005 mol/L sulfuric acid VS (not more than 0.160%).

(4) Carbonate—Mix 1.0 g of Dibasic Calcium Phosphate with 5 mL of water, and add immediately 2 mL of hydrochloric acid: no effervescence occurs.

(5) Heavy metals—Dissolve 0.65 g of Dibasic Calcium Phosphate in a mixture of 5 mL of water and 5 mL of dilute hydrochloric acid by warming, cool, and add ammonia TS until precipitates begin to form in the solution. Dissolve the precipitates by adding a small amount of dilute hydrochloric acid dropwise, add 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, add 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 31 ppm).

(6) Barium—Heat 0.5 g of Dibasic Calcium Phosphate with 10 mL of water, add 1 mL of hydrochloric acid dropwise with stirring, and filter, if necessary. Add 2 mL of potassium sulfate TS to the filtrate, and allow to stand for 10 minutes: no turbidity forms.

(7) Arsenic—Dissolve 1.0 g of Dibasic Calcium Phosphate in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

**Loss on drying** 19.5 - 22.0% (1 g, 200°C, 3 hours).

**Assay** Weigh accurately about 0.4 g of Dibasic Calcium Phosphate, previously dried, dissolve in 12 mL of dilute hydrochloric acid, and add water to make exactly 200 mL. Pipet 20 mL of this solution, add exactly 25 mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess disodium dihydrogen ethylenediamine tetraacetate with 0.02 mol/L zinc acetate VS (indicator: 0.025 g of eriochrome black T-sodium chloride indicator). Perform a blank determination.

Each mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 2.7211 mg of $CaHPO_4$

**Containers and storage** Containers—Well-closed containers.

# Anhydrous Dibasic Calcium Phosphate

無水リン酸水素カルシウム

$CaHPO_4$: 136.06

Anhydorous Dibasic Calcium Posphate, when dried, contains not less than 98.0% of $CaHPO_4$.

**Description** Anhydrous Dibasic Calcium Phosphate occurs as white, crystalline powder or granules. It is odorless and tasteless.

It is practically insoluble in water, in ethanol (95) and in diethyl ether.

It dissolves in dilute hydrochloric acid and in dilute nitric acid.

**Identification** (1) Dissolve 0.1 g of Anhydrous Dibasic Calcium Phosphate in 10 mL of diluted hydrochloric acid (1 in 6) by warming, add 2.5 mL of ammonia TS dropwise with shaking, and add 5 mL of ammonium oxalate TS: a white precipitate is produced.

(2) Dissolve 0.1 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of dilute nitric acid, and add 2 mL of hexaammonium heptamolybdate TS after warming for 1 to 2 minutes at 70°C: a yellow precipitate is produced.

**Purity** (1) Acid-insoluble substances—Dissolve 5.0 g of Anhydrous Dibasic Calcium Phosphate in 40 mL of water and 10 mL of hydrochloric acid, and boil for 5 minutes, After cooling, collect the insoluble substance using filter paper for assay. Wash with water until no more turbidity of the washing is produced when silver nitrate is added. Ignite to incinerate the residue with the filter paper: the mass is not more than 2.5 mg (not more than 0.05%).

(2) Chloride—Dissolve 0.20 g of Anhydrous Dibasic Calcium Phosphate in 20 mL of water and 13 mL of dilute nitric acid, add water to make 100 mL, and filter, if necessary. Perform the test using a 50-mL portion of this solution as the test solution. Prepare the control solution with 0.70 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.248%).

(3) Sulfate—Dissolve by warming 0.80 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of water and 5 mL of dilute hydrochloric acid, add water to make 100 mL, and filter,

if necessary. Take 30 mL of the filtrate, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 1.0 mL of 0.005 mol/L sulfuric acid VS (not more than 0.200%).

**(4)** Carbonate—Mix 1.0 g of Anhydrous Dibasic Calcium Phosphate with 5 mL of water, and add immediately 2 mL of hydrochloric acid: no effervescence occurs.

**(5)** Heavy metals—Dissolve 0.65 g of Anhydrous Dibasic Calcium Phosphate in a mixture of 5 mL of water and 5 mL if dilute hydrochloric acid by warming, cool, and add ammonia TS until precipitates begin to form in the solution. Dissolve the precipitates by adding a small amount of dilute hydrochloric acid dropwise, add 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, add 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 31 ppm).

**(6)** Barium—Heat 0.5 g of Anhydrous Dibasic Calcium Phosphate with 10 mL of water, add 1 mL of hydrochloric acid dropwise with stirring, and filter, if necessary. Add 2 mL of potassium sulfate TS to the filtrate, and allow to stand for 10 minutes: no turbidity forms.

**(7)** Arsenic—Dissolve 0.5 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

**Loss on drying** Not more than 1.0% (1 g, 200°C, 3 hours).

**Assay** Weigh accurately about 0.4 g of Anhydrous Dibasic Calcium Phosphate, previously dried, dissolve in 12 mL of dilute hydrochloric acid, and add water to make exactly 200 mL. Pipet 20 mL of this solution, add exactly 25 mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess disodium dihydrogen ethylenediamine tetraacetate with 0.02 mol/L zinc acetate VS (indicator: 0.025 g of eriochrome black T-sodium chloride indicator). Perform a blank determination.

Each mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 2.7211 mg of $CaHPO_4$

**Containers and storage** Containers—Well-closed containers.

# Monobasic Calcium Phosphate

リン酸二水素カルシウム

$Ca(H_2PO_4)_2.H_2O$: 252.07

Monobasic Calcium Phosphate, when dried, contains not less than 90.0% of $Ca(H_2PO_4)_2.H_2O$.

**Description** Monobasic Calcium Phosphate occurs as white crystals or crystalline powder. It is odorless and has an acid taste.

It is sparingly soluble in water, and practically insoluble in ethanol (95) and in diethyl ether.

It dissolves in dilute hydrochloric acid and in dilute nitric acid.

It is slightly deliquescent.

**Identification** **(1)** Dissolve 0.1 g of Monobasic Calcium Phosphate in 10 mL of diluted hydrochloric acid (1 in 6) by warming, add 2.5 mL of ammonia TS dropwise with shaking, and add 5 mL of ammonium oxalate TS: a white precipitate is produced.

**(2)** Dissolve 0.1 g of Monobasic Calcium Phosphate in 5 mL of dilute nitric acid, and add 2 mL of hexaammonium heptamolybdate TS after warming for 1 to 2 minutes at 70° C: a yellow precipitate is produced.

**Purity** **(1)** Clarity and color of solution—Dissolve 1.0 g of Monobasic Calcium Phosphate in 19 mL of water and 2 mL of diluted hydrochloric acid (3 in 4), and heat on a water bath for 5 minutes with occasional shaking: the solution is clear and colorless.

**(2)** Dibasic phosphate and acid—Triturate 1.0 g of Monobasic Calcium Phosphate with 3 mL of water, and add 100 mL of water and 1 drop of methyl orange TS: a red color develops. Then add 1.0 mL of 1 mol/L sodium hydroxide VS: the color changes to yellow.

**(3)** Chloride—Dissolve 1.0 g of Monobasic Calcium Phosphate in 20 mL of water and 12 mL of dilute nitric acid, add water to make exactly 100 mL, and filter, if necessary. Perform the test using 50 mL of this solution as the test solution. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.018%).

**(4)** Sulfate—Dissolve 1.0 g of Monobasic Calcium Phosphate in 20 mL of water and 1 mL of hydrochloric acid, add water to make 100 mL, and filter, if necessary. Perform the test using 50 mL of this solution as the test solution. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS (not more than 0.048%).

**(5)** Heavy metals—Dissolve 0.65 g of Monobasic Calcium Phosphate in a mixture of 5 mL of water and 5 mL of dilute hydrochloric acid by warming, cool, and add ammonia TS until precipitates begin to form in the solution. Dissolve the precipitates by adding a small amount of dilute hydrochloric acid dropwise, add 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, add 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 31 ppm).

**(6)** Arsenic—Dissolve 1.0 g of Monobasic Calcium Phosphate in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

**Loss on drying** Not more than 3.0% (1 g, silica gel, 24 hours).

**Assay** Weigh accurately about 0.4 g of Monobasic Calcium Phosphate, previously dried, dissolve in 3 mL of dilute hydrochloric acid, and add water to make exactly 100 mL. Pipet 20 mL of this solution, add exactly 25 mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess disodium dihydrogen ethylenediamine tetraacetate with 0.02 mol/L zinc