

hibits no turbidity upon addition of silver nitrate TS, and dry at 105°C to constant mass: the mass is not more than 25 mg.

(2) Heavy metals—Dissolve 1.0 g of Calcium Hydroxide in 10 mL of dilute hydrochloric acid, evaporate on a water bath to dryness, dissolve the residue in 40 mL of water, and filter. To 20 mL of the filtrate add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: evaporate 5 mL of dilute hydrochloric acid on a water bath to dryness, and add 2 mL of dilute acetic acid, 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 40 ppm).

(3) Magnesium and alkali metals—Dissolve 1.0 g of Calcium Hydroxide in a mixture of 20 mL of water and 10 mL of dilute hydrochloric acid, boil, neutralize with ammonia TS, and precipitate calcium oxalate completely by adding dropwise ammonium oxalate TS. Heat the mixture on a water bath for 1 hour, cool, dilute with water to 100 mL, shake, and filter. To 50 mL of the filtrate add 0.5 mL of sulfuric acid, evaporate to dryness, and ignite at 600°C to constant mass: the mass of the residue does not exceed 24 mg.

(4) Arsenic—Dissolve 0.5 g of Calcium Hydroxide in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution using Apparatus B (not more than 4 ppm).

**Assay** Weigh accurately about 1 g of Calcium Hydroxide, dissolve by adding 10 mL of dilute hydrochloric acid, and add water to make 100 mL. Measure 10 mL of this solution, add 90 mL of water and 1.5 mL of 8 mol/L potassium hydroxide TS, shake, allow to stand for 3 to 5 minutes, and then add 0.1 g of NN indicator. Titrate immediately with 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, until the red-purple color of the solution changes to blue.

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS  
= 3.7046 mg of Ca(OH)<sub>2</sub>

**Containers and storage** Containers—Tight containers.

## Calcium Oxide

### Quick Lime

酸化カルシウム

CaO: 56.08

Calcium Oxide, when incinerated, contains not less than 98.0% of CaO.

**Description** Calcium Oxide occurs as hard, white masses, containing a powder. It is odorless.

It is very slightly soluble in boiling water, and practically insoluble in ethanol (95).

One gram of Calcium Oxide dissolves almost completely in 2500 mL of water.

It slowly absorbs moisture and carbon dioxide from air.

**Identification** (1) Moisten Calcium Oxide with water: heat is generated and a white powder is obtained. Mix the

powder with about 5 times its mass of water: the mixture is alkaline.

(2) Dissolve 1 g of Calcium Oxide in 20 mL of water by adding a few drops of acetic acid (31): the solution responds to the Qualitative Tests for calcium salt.

**Purity** (1) Acid-insoluble substances—Disintegrate 5.0 g of Calcium Oxide with a small amount of water, add 100 mL of water, add dropwise hydrochloric acid with stirring until the solution becomes acidic, and further add 1 mL of hydrochloric acid. Boil the solution for 5 minutes, cool, filter through a glass filter (G4), wash the residue with boiling water until no turbidity is produced when silver nitrate TS is added to the last washing, and dry at 105°C to constant mass: the mass of the residue is not more than 10.0 mg.

(2) Carbonate—Disintegrate 1.0 g of Calcium Oxide with a small amount of water, mix thoroughly with 50 mL of water, allow to stand for a while, remove most of the supernatant milky liquid by decantation, and add an excess of dilute hydrochloric acid to the residue: no vigorous effervescence is produced.

(3) Magnesium and alkali metals—Dissolve 1.0 g of Calcium Oxide in 75 mL of water by adding dropwise hydrochloric acid, and further add 1 mL of hydrochloric acid. Boil for 1 to 2 minutes, neutralize with ammonia TS, add dropwise an excess of hot ammonium oxalate TS, heat the mixture on a water bath for 2 hours, cool, add water to make 200 mL, mix thoroughly, and filter. Evaporate 50 mL of the filtrate with 0.5 mL of sulfuric acid to dryness, and heat the residue strongly at 600°C to constant mass: the mass of the residue is not more than 0.015 g.

**Loss on ignition** Not more than 10.0% (1 g, 900°C, constant mass).

**Assay** Weigh accurately about 0.7 g of Calcium Oxide, previously incinerated at 900°C to constant mass and cooled in a desiccator (silica gel), and dissolve in 50 mL of water and 8 mL of diluted hydrochloric acid (1 in 3) by heating. Cool, and add water to make exactly 250 mL. Pipet 10 mL of the solution, add 50 mL of water, 2 mL of 8 mol/L potassium hydroxide TS and 0.1 g of NN indicator, and titrate with 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, until the red-purple color of the solution changes to blue.

Each mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS  
= 1.1215 mg of CaO

**Containers and storage** Containers—Tight containers.

## Dibasic Calcium Phosphate

リン酸水素カルシウム

CaHPO<sub>4</sub>·2H<sub>2</sub>O: 172.09

Dibasic Calcium Phosphate, when dried, contains not less than 98.0% of CaHPO<sub>4</sub>: 136.06.

**Description** Dibasic Calcium Phosphate occurs as a white, crystalline powder. It is colorless 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 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<sub>4</sub>

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

## Anhydrous Dibasic Calcium Phosphate

無水リン酸水素カルシウム

CaHPO<sub>4</sub>: 136.06

Anhydrous Dibasic Calcium Phosphate, when dried, contains not less than 98.0% of CaHPO<sub>4</sub>.

**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,