

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 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 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<sub>4</sub>

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

## Monobasic Calcium Phosphate

リン酸二水素カルシウム

Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O: 252.07

Monobasic Calcium Phosphate, when dried, contains not less than 90.0% of Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O.

**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

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  
= 5.041 mg of  $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$

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

## Calcium Stearate

ステアリン酸カルシウム

Calcium Stearate mainly consists of calcium salts of stearic acid ( $\text{C}_{18}\text{H}_{36}\text{O}_2$ ) and palmitic acid ( $\text{C}_{16}\text{H}_{32}\text{O}_2$ ).

Calcium Stearate, when dried, contains not less than 6.4% and not more than 7.1% of calcium (Ca: 40.08).

**Description** Calcium Stearate occurs as a white, light, bulky powder. It feels smooth when touched, and is adhesive to the skin. It is odorless or has a faint, characteristic odor.

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

**Identification** (1) Shake vigorously 3 g of Calcium Stearate with 20 mL of diluted hydrochloric acid (1 in 2) and 30 mL of diethyl ether for 3 minutes, and allow to stand: the separated aqueous layer responds to the Qualitative Tests (1), (2) and (4) for calcium salt.

(2) Wash the diethyl ether layer obtained in (1) with 20 mL and 10 mL of dilute hydrochloric acid and 20 mL of water successively, and evaporate the diethyl ether on a water bath: the residue melts at a temperature not below 54°C (Method 2).

**Purity** (1) Heavy metals—Heat gently 1.0 g of Calcium Stearate with caution at the beginning, and heat further, gradually raising the temperature, to incineration. After cooling, add 2 mL of hydrochloric acid, evaporate on a water bath to dryness, warm the residue with 20 mL of water and 2 mL of dilute acetic acid for 2 minutes, cool, filter, and wash the residue with 15 mL of water. Combine the filtrate and the washings, add water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution by evaporating 2 mL of hydrochloric acid on a water bath to dryness and by adding 2 mL of dilute acetic acid, 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 20 ppm).

(2) Arsenic—To 1.0 g of Calcium Stearate add 5 mL of diluted hydrochloric acid (1 in 2) and 20 mL of chloroform, shake vigorously for 3 minutes, allow to stand, and separate the water layer. Perform the test using Apparatus B with this water layer as the test solution (not more than 2 ppm).

**Loss on drying** Not more than 4.0% (1 g, 105°C, 3 hours).

**Assay** Weigh accurately about 0.5 g of Calcium Stearate, previously dried, heat gently with caution at first, and then ignite gradually to ash. Cool, add 10 mL of dilute hydrochloric acid to the residue, warm for 10 minutes on a water bath, and transfer the contents to a flask with the aid of 10-mL, 10-mL, and 5-mL portions of hot water. Add sodium hydroxide TS until the solution becomes slightly turbid, and then add 25 mL of 0.05 mol/L disodium dihydrogen ethylenediamine

tetraacetate VS, 10 mL of ammonia-ammonium chloride buffer solution, pH 10.7, 4 drops of eriochrome black T TS and 5 drops of methyl yellow TS, and titrate rapidly the excess disodium dihydrogen ethylenediamine tetraacetate with 0.05 mol/L magnesium chloride VS, until the green color of the solution disappears and a red color develops. Perform a blank determination.

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS  
= 2.0039 mg of Ca

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

## Calumba

### *Calumbae Radix*

コロンボ

Calumba is the cross-sectioned root of *Jateorhiza columba* Miers (*Menispermaceae*).

**Description** Disk-like slices, 0.5–2 cm in thickness, 3–8 cm in diameter; mostly with concave center and slightly waved; side surface grayish brown in color, with irregular wrinkles; cut surface light yellow and powdery, with pale and dark radiating stripes; cortex rather yellowish; cambium and its neighborhood light grayish brown, warty protrusions in the center; hard in texture, but brittle. Odor characteristic; taste, bitter.

**Identification** To 3 g of pulverized Calumba add 30 mL of water, allow to stand for 5 minutes with occasional shaking, and filter. To 2 mL of the filtrate add gently 1 mL of sulfuric acid, and, after cooling, add carefully chlorine TS to make two layers: a light red to red color develops at the zone of contact.

**Total ash** Not more than 7.5%.

## Powdered Calumba

### *Calumbae Radix Pulverata*

コロンボ末

Powdered Calumba is the powder of Calumba.

**Description** Powdered Calumba occurs as a grayish yellow powder, and has a characteristic odor and a bitter taste.

Under a microscope, Powdered Calumba reveals numerous starch grains, fragments of parenchyma cells containing them; fragments of cork cells, stone cells, fibers, substitute fibers, vessels, tracheids, and also solitary crystals of calcium oxalate; starch grains consisting of solitary grains or 2- to 3-compound grains; hilum, unevenly scattered, usually 25–50  $\mu\text{m}$ , but up to 90  $\mu\text{m}$  in diameter.

**Identification** To 3 g of Powdered Calumba add 30 mL of water, allow to stand for 5 minutes with occasional shaking.