purple color at the outer portion of cortex, and light brown inner portion making irregular wave; xylem yellowish in color; the center of the crown is often cracked, and the surrounding part red-purple. Odor, slight; taste, slightly sweet.

**Identification** (1) Heat 0.5 g of pulverized Lithospermum Root in a test tube: red vapor evolves, which condenses on the wall of the upper part of the tube into red-brown oil drops.

- (2) Shake 0.5 g of pieces or powder of Lithospermum Root with 1 mL of ethanol (95), and to the red solution thereby obtained add 1 drop of sodium hydroxide TS: the red color changes to blue-purple. To this solution add 1 to 2 drops of dilute hydrochloric acid: the color turns red again.
- (3) To 0.5 g of pulverized Lithospermum Root add 5 mL of ethanol (95), shake for 30 minutes, filter, and evaporate the filtrate at a temperature not higher than 40°C under reduced pressure. Add 1 mL of ethanol (95) to the residue, and use this solution as the sample solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot  $5 \mu$ L of the sample solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and ethanol (95) (3:1) to a distance of about 10 cm, and air-dry the plate: a red-purple spot appears around the Rf 0.75.

Total ash Not more than 11.0%.

Acid-insoluble ash Not more than 3.5%.

## Longgu

Fossilia Ossis Mastodi

リュウコツ

Longgu is the ossified bone of large mammal, and is mainly composed of calcium carbonate.

**Description** Irregular masses or fragments, occasionally cylindrical masses; externally light grayish white, sometimes with grayish black or yellow-brown spots here and there; the outer part consists of a layer 2 – 10 mm in thickness, and is minute in texture, surrounding the light brown, porous portion; heavy and hard, but somewhat fragile in texture; when crushed, it changes into pieces and powder. Odorless, tasteless, and strongly adhesive to the tongue on licking.

- **Identification** (1) Dissolve 0.5 g of pulverized Longgu in 10 mL of dilute hydrochloric acid: it evolves a gas, and forms a slightly brownish and turbid solution. Pass the gas evolved through calcium hydroxide TS: a white precipitate is produced.
- (2) The turbid solution, obtained in (1), has a characteristic odor. Filter this solution, and neutralize with ammonia TS: the solution responds to the Qualitative test for calcium salt.
- (3) Dissolve 0.1 g of pulverized Longgu in 5 mL of nitric acid by warming, and add hexaammonium heptamolybdate TS: a yellow precipitate is produced.
- **Purity** (1) Heavy metals—To 2.0 g of pulverized Longgu add 5 mL of water, shake to mix, add carefully 6 mL of hydrochloric acid, and evaporate on a water bath to dryness.

Dissolve the residue in 50 mL of water, and filter. To 25 mL of the filtrate add 2 mL of dilute acetic acid, 1 drop of ammonia TS and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: Evaporate 3 mL of hydrochloric acid on a water bath to dryness, add 2 mL of dilute acetic acid and 2.0 mL of Standard Lead Solution, and add water to make 50 mL (not more than 20 ppm).

(2) Arsenic—Prepare the test solution with 0.20 g of pulverized Longgu according to Method 2, and perform the test using Apparatus B (not more than 10 ppm).

## **Macrogol 400**

## Polyethylene Glycol 400

マクロゴール 400

Macrogol 400 is a polymer of ethylene oxide and water, represented by the formula  $HOCH_2$  ( $CH_2OCH_2$ ) $_nCH_2OH$ , in which the value of n ranges from 7 to 9.

**Description** Macrogol 400 occurs as a clear, colorless and viscous liquid. It has no odor or a slight, characteristic odor.

It is miscible with water, with methanol, with ethanol (95) and with pyridine.

It is soluble in diethyl ether.

It is slightly hygroscopic.

Congealing point: 4 - 8°C

Specific gravity  $d_{20}^{20}$ : 1.110 – 1.140

**Identification** Dissolve 0.05 g of Macrogol 400 in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake, and filter, if necessary. To the filtrate add 1 mL of a solution of phosphomolybdic acid *n*-hydrate (1 in 10): a yellow-green precipitate is formed.

**pH** Dissolve 1.0 g of Macrogol 400 in 20 mL of water: the pH of this solution is between 4.0 and 7.0.

- **Purity** (1) Acid—Dissolve 5.0 g of Macrogol 400 in 20 mL of neutralized ethanol, and add 2 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide VS: the solution is red in color.
- (2) Ethylene glycol and diethylene glycol—Dissolve 4.0 g of Macrogol 400 in water to make exactly 10 mL, and use this solution as the sample solution. Weigh accurately about 0.05 g each of ethylene glycol and diethylene glycol, dissolve in water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with  $2\,\mu\text{L}$  each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions. Determine the peak heights,  $H_{\text{Ta}}$  and  $H_{\text{Sa}}$ , of ethylene glycol of each solution, and the peak heights,  $H_{\text{Tb}}$  and  $H_{\text{Sb}}$ , of diethylene glycol, and calculate the amount of ethylene glycol and diethylene glycol: the sum of the contents of ethylene glycol and diethylene glycol is not more than 0.25%.