(3) Heavy metals—Proceed with 1.0 g of Purified Gelatin according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Arsenic—Place 15.0 g of Purified Gelatin in a flask, add 60 mL of diluted hydrochloric acid (1 in 5), and heat until solution is effected. Add 15 mL of bromine TS, heat until the excess of bromine is expelled, neutralize with ammonia TS, add 1.5 g of disodium hydrogenphosphate 12-water, and allow to cool. To this solution add 30 mL of magnesia TS, allow to stand for 1 hour, and collect the precipitates. Wash the precipitates with five 10-mL portions of diluted ammonia TS (1 in 4), and dissolve in diluted hydrochloric acid (1 in 4) to make exactly 50 mL. Perform the test with 5 mL of this solution using Apparatus B: the solution has no more color than the following standard solution.

Standard solution: Proceed with 15 mL of Standard Arsenic Solution, instead of Purified Gelatin, in the same manner (not more than 1 ppm).

(5) Mercury—Place 2.0 g of Purified Gelatin in a decomposition flask, add 20 mL of diluted sulfuric acid (1 in 2) and 100 mL of a solution of potassium permanganate (3 in 50), heat gently under a reflux condenser, and boil for 2 hours. If the solution becomes clear during boiling, reduce the temperature of the solution to about 60°C, add further 5 mL of a solution of potassium permanganate (3 in 50), boil again, and repeat the above-mentioned procedure until the precipitate of manganese dioxide remains for about 20 minutes. Cool, add a solution of hydroxylammonium chloride (1 in 5) until the precipitate of manganese dioxide disappears, add water to make exactly 150 mL, and use the solution as the sample solution. Perform the test as directed under the Atomic Absorption Spectrophotometry (Cold vapor type) using the sample solution. Place the sample solution in a sample water bottle of the atomic absorption spectrophotometer, add 10 mL of tin (II) chloride-sulfuric acid TS, connect the bottle immediately to the atomic absorption spectrophotometer, and circulate air. Determine the absorbance $A_T$ of the sample solution at 253.7 nm when the indication of the recorder has risen rapidly and become constant. On the other hand, place 2.0 mL of Standard Mercury Solution in a decomposition flask, add 20 mL of diluted sulfuric acid (1 in 2) and 100 mL of a solution of potassium permanganate (3 in 50), and proceed in the same manner as for the sample solution. Determine the absorbance AS of the standard solution: $A_T$ is not more than $A_S$ (not more than 0.1 ppm).

**Loss on drying** Not more than 15.0%. Take about 1 g of Purified Gelatin, accurately weighed, in a tared 200-mL beaker containing 10 g of sea sand (No. 1), previously dried at 110°C for 3 hours. Add 20 mL of water, allow to stand for 30 minutes with occasional shaking, evaporate on a water bath to dryness with occasional shaking, and dry the residue at 110°C for 3 hours.

**Residue on ignition** Not more than 2.0% (0.5 g).

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

# Gentian

*Gentianae Radix*

ゲンチアナ

Gentian is the root and rhizome of *Gentiana lutea* Linné (*Gentianaceae*).

**Description** Nearly cylindrical pieces, 10 - 50 cm in length, 2 - 4 cm in daiameter; externally dark brown; the rhizome short, with fine, transverse wrinkles, and sometimes with buds and remains of leaves at the upper edge. The root longitudinally and deeply wrinkled, and more or less twisted; fractured surface yellow-brown and not fibrous, and a cambium and its neighborhood tinged dark brown. Odor, characteristic; taste, sweet at first, later persistently bitter.

Under a microscope, a transverse section of the root reveals several layers of collenchyma adjoined internally to 4 to 6 layers of thin-walled cork; secondary cortex of the parenchyma with irregularly distributed phloem; xylem consisting chiefly of parenchyma, with individual or clustered vessels and tracheids, and exhibiting some sieve tubes of xylem; parenchyma of the xylem and the cortex containing oil droplets, minute needle crystals of calcium oxalate and very rarely starch grains 10 - 20 μm in diameter.

**Identification** (1) Place 0.1 g of pulverized Gentian, previously dried in a desiccator (silica gel) for 48 hours, on a slide glass, put a glass ring 10 mm in both inside diameter and in height on it, then cover with another slide, and heat gently and gradually: pale yellow crystals are sublimed on the upper slide. The crystals are insoluble in water and in ethanol (95), and soluble in potassium hydroxide TS.

(2) To 0.5 g of pulverized Gentian add 10 mL of methanol, shake for 5 minutes, filter, and use the filtrate as the sample solution. Separately, dissolve 1 mg of gentiopicroside for thin-layer chromatography in 1 mL of methanol, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, ethanol (99.5) and water (8:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the sample solution and a dark purple spot from the standard solution show the same color tone and the same *R*f value.

**Total ash** Not more than 6.0%.

**Acid-insoluble ash** Not more than 3.0%.

# Powdered Gentian

*Gentianae Radix Pulverata*

ゲンチアナ末

Powdered Gentian is the powder of Gentian.