tion.

Amount (mg) of berberine [as berberine chloride ($C_{20}H_{18}ClNO_4$)]
= amount (mg) of Berberine Chloride Reference Standards, calculated on the anhydrous basis
$\times \frac{A_T}{A_S}$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 345 nm).

Column: A stainless steel column 4 to 6 mm in inside diameter and 15 to 25 cm in length, packed with octadecylsilanized silica gel (5 to 10 mm in particle diameter).

Column temperature: A constant temperature of about 40°C

Mobile phase: Dissolve 3.4 g of potassium dihydrogenphosphate and 1.7 g of sodium lauryl sulfate in 1000 mL of a mixture of water and acetonitrile (1:1).

Flow rate: Adjust the flow rate so that the retention time of berberine is about 10 minutes.

Selection of column: Dissolve 0.001 g each of Berberine Chloride Reference Standard and palmatine chloride in 10 mL of methanol. Perform the test with 20 mL of this solution under the above operating conditions. Use a column giving elution of palmatine and berberine in this order, and clearly dividing each peak.

System repeatability: Repeat the test five times with the standard solution under the above operating conditions the relative deviation of the peak area of berberine is not more than 1.5%.

Powdered Phellodendron Bark

Phellodendri Cortex Pulveratus

オウバク末

Powdered Phellodendron Bark is the powder of Phellodendron Bark.

It contains not less than 1.2% of berberine chloride [as berberine chloride ($C_{20}H_{18}ClNO_4$: 371.81)], calculated on the basis of dried material.

Description Powdered Phellodendron Bark occurs as a bright yellow to yellow powder. It has a slight odor and an extremely bitter taste, is mucilaginous, and colors the saliva yellow on chewing.

Under a microscope, Powdered Phellodendron Bark reveals fragments of yellow, thick-walled fiber bundles or fibers, and fibers often accompanied by crystal cell rows; fewer groups of stone cells together with idioblasts; fragments of parenchyma cells containing starch grains and oil droplets; fragments of medullary ray and phloem; mucilage cells and mucilage masses. Numerous solitary crystals of calcium oxalate, 7 - 20 μm in diameter; starch grains, simple grains and 2- to 4-compound grains, simple grain, 2 - 6 μm in diameter; oil droplets, stained red with sudan III TS.

Identification **(1)** To 1 g of Powdered Phellodendron Bark add 10 mL of diethyl ether, allow to stand for 10 minutes with occasional shaking, and filter to remove the diethyl ether. Collect the powder on the filter paper, add 10 mL of ethanol (95), allow to stand for 10 minutes with occasional shaking, and filter. To 2 to 3 drops of the filtrate add 1 mL of hydrochloric acid, add 1 to 2 drops of hydrogen peroxide TS, and shake: a red-purple color develops.

(2) Use the filtrate obtained in (1) as the sample solution. Separately, dissolve 1 mg of berberine chloride 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 5 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water and acetic acid (100) (7:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the sample solution and a spot with yellow to yellow-green fluorescence from the standard solution show the same in color tone and Rf value.

(3) Stir up Powdered Phellodendron Bark with water: the solution becomes gelatinous owing to mucilage.

Purity Curcuma—Place Powdered Phellodendron Bark on filter paper, drop diethyl ether on it, and allow to stand. Take the powder off the filter paper, and drip 1 drop of potassium hydroxide TS: no red-purple color develops. Under a microscope, Powdered Phellodendron Bark does not contain gelatinized starch or secretory cells containing yellow-red resin.

Loss on drying Not more than 9.0% (60°C, 8 hours).

Total ash Not more than 7.5%.

Acid-insoluble ash Not more than 0.5%.

Assay Weigh accurately about 0.5 g of Powdered Phellodendron Bark, add 30 mL of a mixture of methanol and dilute hydrochloric acid (100:1), heat under a reflux condenser on a water bath for 30 minutes, cool, and filter. Repeat the above procedure twice with the residue, using 30-mL and 20-mL portions of a mixture of methanol and dilute hydrochloric acid (100:1). To obtained residue add 10 mL of methanol, shake well, and filter. Combine the whole filtrates, add methanol to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of Berberine Chloride Reference Standard (separately determine the water content), dissolve in methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of berberine in each solution.

Amount (mg) of berberine ([as berberine chloride) ($C_{20}H_{18}ClNO_4$)]
= amount (mg) of berberine chloride for component determination Berberine Chloride Reference Standard, calculated on the anhydrous basis
$\times \frac{A_T}{A_S}$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 345 nm).

Column: A stainless steel column 4 to 6 mm in inside di-

ameter and 15 to 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 3.4 g of potassium dihydrogenphosphate and 1.7 g of sodium lauryl sulfate in 1000 mL of a mixture of water and acetonitrile (1:1).

Flow rate: Adjust the flow rate so that the retention time of berberine is about 10 minutes.

Selection of column: Dissolve 1 mg each of Berberine Chloride Reference Standard and palmatine chloride in 10 mL of methanol. Proceed with 20 μL of this solution under the above operating conditions. Use a column giving elution of palmatine and berberine in this order, and clearly dividing each peak.

System repeatability: When repeat the test 5 times with the standard solution under the above operating conditions, the relative standard deviation of the peak area of berberine is not more than 1.5%.

Compound Phellodendron Powder for Cataplasm

パップ用複方オウバク散

Method of preparation

Powdered Phellodendron Bark	660 g
Powdered Gardenia Fruit	325 g
d- or *dl*-Camphor	10 g
dl- or *l*-Menthol	5 g
To make	1000 g

Prepare as directed under Powders, with the above ingredients.

Description Compound Phellodendron Powder for Cataplasm occurs as a yellow-brown powder, having a characteristic odor.

Identification Shake thoroughly 0.2 g of Compound Phellodendron Powder for Cataplasm with 5 mL of methanol, filter, and use the filtrate as the sample solution. Dissolve 0.01 g of berberine chloride in 10 mL of methanol, and use the solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water and acetic acid (100) (7:2:1) to a distance of about 10 cm, air-dry the plate, and examine under ultraviolet light (main wavelength: 365 nm): the spots from the sample solution and the standard solution reveal a yellow color, and show the same *Rf* value (berberine).

Containers and storage Containers—Tight containers.

Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder

オウバク・タンナルビン・ビスマス散

Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder contains not less than 12.9% and not more than 16.3% of bismuth (Bi: 208.98).

Method of preparation

Powdered Phellodendron Bark	300 g
Albumin Tannate	300 g
Bismuth Subnitrate	200 g
Scopolia Extract	10 g
Starch, Lactose or their mixture a sufficient quantity	
To make	1000 g

Prepare as directed under Powders, with the above ingredients. Scopolia Extract Powder may be used in place of Scopolia Extract.

Description Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder is brownish yellow in color, and has a bitter taste.

Identification **(1)** Shake thoroughly 0.1 g of Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder with 5 mL of methanol, filter, and use the filtrate as the sample solution. Dissolve 0.01 g of berberine chloride in 10 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 5 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water and acetic acid (100) (7:2:1) to a distance of about 10 cm, air-dry the plate, and examine under ultraviolet light (main wavelength: 365 nm): the spot of berberine chloride reveals a yellow color, and the spots from the sample solution and the standard solution show the same *Rf* value (berberine).

(2) To 0.3 g of Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder add 20 mL of ethanol (95), heat in a water bath for 3 minutes with shaking, cool, and filter. To 10 mL of the filtrate add 1 drop of iron (III) chloride TS: a blue-green color is produced. Allow to stand: a bluish black precipitate is produced (albumin tannate).

(3) To 0.3 g of Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder add 10 mL of diluted pyridine (1 in 5), warm in a water bath for 3 minutes with shaking, cool, and filter. Add 1 mL of ninhydrin-ascorbic acid TS to the filtrate, and heat in a water bath: a blue color is produced (albumin tannate).

(4) To 0.5 g of Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder add 5 mL of dilute hydrochloric acid and 10 mL of water, warm, shake thoroughly, and filter. The filtrate responds to the Qualitative Tests for bismuth salt.

Assay Weigh accurately about 0.7 g of Phellodendron, Albumin Tannate and Bismuth Subnitrate Powder, shake well with 10 mL of water and 20 mL of diluted nitric acid (1 in 3), add water to make exactly 100 mL, and filter. Discard the