Atropine Injection, add exactly 10 mL of the internal standard solution, and use this solution as the sample solution. Separately, weigh accurately about 0.4 g of oxycodone hydrochloride for assay and about 0.1 g of hydrocotarnine hydrochloride for assay previously dried at 105°C for 3 hours, and dissolve in water to make exactly 50 mL. Pipet 2 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Calculate the ratios, Q_{Ta} and Q_{Sb}, of the peak area of oxycodone hydrochloride and hydrocotarnine hydrochloride to that of the internal standard from the sample solution, and the ratios, Q_{Sa} and Q_{Sb}, of the peak area of oxycodone hydrochloride and hydrocotarnine hydrochloride to that of the internal standard from the standard solution.

Amount (mg) of oxycodone hydrochloride ($C_{18}H_{21}NO_4.HCl.3H_2O$)
= amount (mg) of oxycodone hydrochloride for assay, calculated on the anhydrous basis
$\times \frac{Q_{Ta}}{Q_{Sa}} \times 1.1536 \times \frac{1}{25}$

Amount (mg) of hydrocotarnine hydrochloride ($C_{12}H_{15}NO_3.HCl.H_2O$)
= amount (mg) of hydrocotarnine hydrochloride for assay
$\times \frac{Q_{Tb}}{Q_{Sb}} \times 1.0699 \times \frac{1}{25}$

Internal standard solution—Dissolve 0.02 g of phenacetin in 10 mL of ethanol (95), and add water to make 100 mL.
Operating conditions—
Detector: An ultraviolet absorption photometer (wavelength: 285 nm).
Column: A stainless steel column about 4 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized polyvinyl alcohol gel polymer for liquid chromatography (5 μm in particle diameter).
Column temperature: A constant temperature of about 25°C.
Mobile phase: To 500 mL of 0.05 mol/L disodium hydrogenphosphate TS add 0.05 mol/L sodium dihydrogenphosphate TS, and adjust the pH to 8.0. To 300 mL of this solution add 200 mL of acetonitrile, and mix.
Flow rate: Adjust the flow rate so that the retention time of oxycodone hydrochloride is about 8 minutes.
Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and use a column giving elution of the internal standard, oxycodone hydrochloride and hydrocotarine hydrochloride in this order, and complete separation of their peaks.

(2) Atropine sulfate—Pipet 2 mL of Compound Oxycodone and Atropine Injection, and add exactly 2 mL of the internal standard solution. To this solution add 10 mL of diluted dilute hydrochloric acid (1 in 10) and 2 mL of ammonia TS, immediately add 20 mL of dichloromethane, shake vigorously, filter the dichloromethane layer through filter paper on which 5 g of anhydrous sodium sulfate is placed, and evaporate the filtrate to dryness under reduced pressure. To the residue add 0.5 mL of 1,2-dichloromethane and 0.5 mL of bis-trimethylsilylacetamide, stopper tightly, warm in a water bath at 60°C for 15 minutes, and use this solution as the sample solution. Separately, weigh accurately about 0.03 g of Atropine Sulfate Reference Standard (separately determine its loss on drying in the same manner as directed under Atropine Sulfate), and dissolve in water to make exactly 100 mL. Pipet 2 mL of this solution, and add exactly 2 mL of the internal standard solution. Proceed with this solution in the same manner as directed as for the sample solution, and use so obtained solution as the standard solution. Perform the test with 2 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of atropine to that of the internal standards.

Amount (mg) of atropine sulfate [$(C_{17}H_{23}NO_3)_2.H_2SO_4.H_2O$]
= amount (mg) of Atropine Sulfate Reference Standard, calculated on the dried basis
$\times \frac{Q_T}{Q_S} \times \frac{1}{50} \times 1.027$

Internal standard solution—A solution of homatropine hydrobromide (1 in 4000).
Operating conditions—
Detector: A hydrogen flame-ionization detector.
Column: A glass column about 3 mm in inside diameter and about 1.5 m in length, packed with 180- to 250-μm siliceous earth for gas chromatography coated with 1 to 3% of 50% phenyl-methylsilicone polymer.
Column temperature: A constant temperature of about 210°C.
Carrier gas: Nitrogen or helium.
Flow rate: Adjust the flow rate so that the retention time of atropine is about 5 minutes.
Selection of column: Proceed with 2 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of the internal standard and atropine in this order with the resolution between these peaks being not less than 3.

Containers and storage Containers—Hermetic containers, and colored containers may be used.
Storage—Light-resistant.

Oyster Shell

Ostreae Testa

ボレイ

Oyster Shell is the shell of *Ostrea gigas* Thunberg (*Ostreidae*).

Description Irregularly curved, foliaceous or lamellated broken pieces. The unbroken oyster shell forms a bivalve 6 - 10 cm in length and 2 - 5 cm in width. The upper valve is flat and the lower one is somewhat hollow. Both the upper and lower edges of the valve are irregularly curved and bite with each other. The surface of the valve is externally light greenish gray-brown and internally milky in color. Odorless and tasteless.

Identification **(1)** Dissolve 1 g of sample pieces of Oyster

Shell in 10 mL of dilute hydrochloric acid by heating: it evolves a gas, and forms a very slightly red, turbid solution in which a transparent, thin suspended matter remains. Pass the evolved gas through calcium hydroxide TS: a white precipitate is produced.

(2) The solution obtained in (1) has a slight, characteristic odor. Filter this solution and neutralize with ammonia TS: the solution responds to the Qualitative Tests for calcium salt.

(3) Ignite 1 g of pulverized Oyster Shell: it turns blackish brown in color at first, and evolves a characteristic odor. Ignite it further: it becomes almost white.

Purity Barium—Dissolve 1 g of pulverized Oyster Shell in 10 mL of dilute hydrochloric acid: the solution does not respond to the Qualitative Tests (1) for barium salt.

Powdered Oyster Shell

Ostreae Testa Pulverata

ボレイ末

Powdered Oyster Shell is the powder of Oyster Shell.

Description Powdered Oyster Shell occurs as a grayish white powder. It is odorless and tasteless.

Identification **(1)** Dissolve 1 g of Powdered Oyster Shell in 10 mL of dilute hydrochloric acid by heating: it evolves a gas, and forms a very slightly red, turbid solution. Pass the gas evolved through calcium hydroxide TS: a white precipitate is produced.

(2) The solution obtained in (1) has a slight, characteristic odor. Filter this solution, and neutralize with ammonia TS: the solution responds to the Qualitative Tests for calcium salt.

(3) Ignite 1 g of Powdered Oyster Shell: it turns blackish brown in color at first evolving a characteristic odor, and becomes almost white by further ignition.

Purity **(1)** Water-soluble substances—Shake 3.0 g of Powdered Oyster Shell with 50 mL of freshly boiled and cooled water for 5 minutes, filter, and evaporate 25 mL of the filtrate to dryness. Dry the residue at 105°C for 1 hour, cool, and weigh: the mass of the residue does not exceed 15 mg.

(2) Acid-insoluble substances—To 5.0 g of Powdered Oyster Shell add 100 mL of water, and add hydrochloric acid in small portions with stirring until the solution becomes acid. Boil the acidic mixture with additional 1 mL of hydrochloric acid. After cooling, collect the insoluble substance by filtration, and wash it with hot water until the last washing no longer gives any reaction in Qualitative test (2) for chloride. Ignite the residue and weigh: the mass of the residue does not exceed 25 mg.

(3) Barium—Dissolve 1 g of Powdered Oyster Shell in 10 mL of dilute hydrochloric acid: the solution does not responds to the Qualitative Tests (1) for barium salt.

Loss on drying Not more than 4.0% (1 g, 180°C, 4 hours).

Containers and storage Containers—Tight containers.

Panax Rhizome

Panacis Japonici Rhizoma

チクセツニンジン

Panax Rhizome is the rhizome of *Panax japonicus* C. A. Meyer (*Araliaceae*), usually after being treated with hot water.

Description Irregularly cylidrical rhizome with distinct nodes, 3 - 20 cm in length, 1 - 1.5 cm in diameter, internode 1 - 2 cm; externally light yellow-brown, with fine longitudinal wrinkles; stem scars, hollowed at the center, protruding on the upper surface, and root scars protruding as knobs on internodes; easily broken; fractured surface approximately flat, and light yellow-brown in color; horny in texture; odor, slight; taste, slightly bitter.

Identification Shake 0.5 g of pulverized Panax Rhizome with 10 mL of methanol for 10 minutes, filter, and use the filtrate as the sample solution. Separately, dissolve 2 mg of chikusetsusaponin IV 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 ethyl acetate, water and formic acid (5:1:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid on the plate, and heat the plate at 110°C for 5 minutes: one of several spots obtained from the sample solution shows the same color and the same *Rf* value with the purple-red spot from the standard solution.

Total ash Not more than 5.0%.

Extract content Dilute ethanol-soluble extract: not less than 30.0%.

Powdered Panax Rhizome

Panacis Japonici Rhizoma Pulveratum

チクセツニンジン末

Powdered Panax Rhizome is the powder of Panax Rhizome.

Description Powdered Panax Rhizome occurs as a light grayish yellow-brown powder, and has a slight odor and a slightly bitter taste.

Under a microscope, Powdered Panax Rhizome reveals mainly starch grains or gelatinized starch masses, and fragments of parenchyma cells containing them; also fragments of cork tissue, somewhat thick-walled collenchyma, phloem tissue, and reticulate vessels; rarely fragments of scalariform vessels with a simple perforation, fibers and fiber bundles, rosette aggregates of calcium oxalate, and parenchyma cells containing them; yellow to orange-yellow resin; starch grains consisting of simple grains or 2- to 4-compound grains, simple grains, 3 - 18 μm in diameter; rosette aggregates of calci-