lowing conditions. Measure the peak area A_T of oxygen. Separately, introduce 1.0 mL of oxygen into the gas mixer, add carrier gas to make exactly 100 mL, mix thoroughly, and use this as the standard gas mixture. Proceed with 1.0 mL of this mixture in the same manner under Nitrogen, and measure the peak area A_S of oxygen.

$$\text{Amount (vol\%) of } N_2 = 100 - \frac{A_T}{A_S}$$

Operating conditions—

Detector: A thermal-conductivity detector.

Column: A column about 3 mm in inside diameter and about 3 m in length, packed with zeolite for gas chromatography (250 to 350 μm in particle diameter).

Column temperature: A constant temperature of about 50°C.

Carrier gas: Hydrogen or helium

Flow rate: Adjust the flow rate so that the retention time of oxygen is about 3 minutes.

Selection of column: Introduce 1.0 mL of oxygen into the gas mixer, add Nitrogen to make 100 mL, and mix thoroughly. Proceed with 1.0 mL of this mixture under the above operating conditions. Use a column giving well-resolved peaks of oxygen and Nitrogen in this order.

System repeatability: Repeat the test 5 times according to the above conditions with the standard gas mixture. Relative standard deviation of peak area of oxygen is not more than 2.0%.

Containers and storage Containers—Metal cylinders.

Storage—Not exceeding 40°C.

Nuphar Rhizome

Nupharis Rhizoma

センコツ

Nuphar Rhizome is the longitudinally split rhizome of *Nuphar japonicum* De Candolle.

Description Usually, longitudinally split irregular column, twisted, bent or somewhat pressed, 20 - 30 cm in length, about 2 cm in width; the outer surface, dark brown, and the cross section, white to grayish white in color; one side shows nearly round to blunt triangular scars of petiole about 1 cm in diameter, and the other side numerous scars of roots less than 0.3 cm in diameter; light, spongy in texture, and easily broken; fractured surface flat and powdery. Under a magnifying glass, a transverse section reveals a black outer portion, and porous tissue with scattered vascular bundles in the inner portion. Odor, slight; taste, slightly bitter and unpleasant.

Identification Boil 1 g of pulverized Nuphar Rhizome with 20 mL of methanol under a reflux condenser on a water bath for 15 minutes, cool, and filter. Evaporate the filtrate to dryness, warm the residue with 5 mL of dilute acetic acid on a water bath for 1 minute, cool, and filter. Spot 1 drop of the filtrate on a piece of filter paper, air-dry the paper, spray Dragendorff's TS for spraying on it, and allow it to stand: a yellow-red color appears.

Purity (1) Petiole—The amount of its petioles contained in Nuphar Rhizome does not exceed 3.0%.

(2) Foreign matter—The amount of foreign matter other than petiole contained in Nuphar Rhizome does not exceed 1.0%.

Loss on drying Not more than 15.0% (6 hours).

Total ash Not more than 10.0%.

Acid-insoluble ash Not more than 1.0%.

Nux Vomica

Strychni Semen

ホミカ

Nux Vomica is the seed of *Strychnos nux-vomica* Linné (*Loganiaceae*).

When dried, it contains not less than 1.07% of strychnine ($C_{21}H_{22}N_2O_2$: 334.41).

Description Disk, often slightly bent, 1 - 3 cm in diameter, 0.3 - 0.5 cm in thickness; externally light grayish yellow-green to light grayish brown, covered densely with lustrous appressed hairs radiating from the center to the circumference; on both sides, the margin and the central part bulged a little; the dot-like micropyle situated at one point on the margin, and from which usually a raised line runs to the center on one side; extremely hard in texture; when cracked upon soaking in water, the seed coat thin, the interior consisting of two horny, light grayish yellow endosperms, and leaving a central narrow cavity at the center; a white embryo, about 0.7 cm in length, situated at one end between the inner surfaces of the endosperms. Odorless; taste, very bitter and persisting.

Identification (1) To 3 g of pulverized Nux Vomica add 3 mL of ammonia TS and 20 mL of chloroform, macerate for 30 minutes with occasional shaking, and filter. Remove most of the chloroform from the filtrate by warming on a water bath, add 5 mL of diluted sulfuric acid (1 in 10), and warm on a water bath while shaking well until the odor of chloroform is no longer perceptible. After cooling, filter through a pledget of absorbent cotton, and add 2 mL of nitric acid to 1 mL of the filtrate: a red color develops.

(2) To the remaining filtrate obtained in (1) add 1 mL of potassium dichromate TS, and allow to stand for 1 hour: a yellow-red precipitate is produced. Collect the precipitate by filtration, and wash with 1 mL of water. Transfer a part of the precipitate to a small test tube, add 1 mL of water, dissolve by warming, cool, and add 5 drops of sulfuric acid dropwise carefully along the wall of the test tube: the layer of sulfuric acid shows a purple color which turns immediately red to red-brown.

Total ash Not more than 3.0%.

Assay Weigh accurately about 1.0 g of pulverized Nux Vomica, previously dried at 60°C for 8 hours, place in a glass-stoppered centrifuge tube, and moisten with 1 mL of ammonia solution (28). To this solution add 20 mL of diethyl ether, stopper the centrifuge tube tightly, shake for