natant liquid and floating materials. Wash the residue with 10 mL of diethyl ether, and remove the diethyl ether by suction. To the residue add 2 mL of pyridine and 0.2 mL of sodium hydrogen carbonate TS, and boil: a light blue to blue color is produced (sulfur).

(3) To 1 g of Sulfur, Salicylic Acid and Thianthol Ointment add 15 mL of ethanol (95), stir well while warming on a water bath, cool, and filter. Use the filtrate as the sample solution. Dissolve 0.01 g each of salicylic acid and thianthol in 5 mL of ethanol (95), and use these solutions as the standard solution (1) and standard solution (2). Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the sample solution and the standard solutions on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, acetone and acetic acid (100) (45:5:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots of each component obtained from the sample solution and the standard solutions (1) and (2) show the same *Rf* value. Spray iron (III) chloride TS upon the plate evenly: the spot from the standard solution (1) and that from the corresponding sample solution reveal a purple color.

Containers and storage Containers—Tight containers.

Sweet Hydrangea Leaf

Hydrangeae Dulcis Folium

アマチャ

Sweet Hydrangea Leaf is the leaf and twig of *Hydrangea macrophylla* Seringe var. *thunbergii* Makino (*Saxifragaceae*).

Description Usually wrinkled and contracted leaf, dark green to dark yellow-green in color. When soaked in water and smoothed out, it is lanceolate to acuminately ovate, about 12 cm in length, about 5 cm in width; margin serrated, base slightly wedged; coarse hair on both surfaces, especially on the veins; lateral veins not reaching the margin but curving upwards and connecting with each other; petiole short and less than one-fifth of the length of lamina. Odor, slight; taste, characteristically sweet.

Identification Mix 0.5 g of pulverized Sweet Hydrangea Leaf with 8 mL of a mixture of diethyl ether and petroleum ether (1:1), shake well, filter, and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of dilute ethanol, and add 1 drop of dilute iron (III) chloride TS: a red-purple color develops, which disappears on the addition of 2 to 3 drops of dilute sulfuric acid.

Purity (1) Stem—The amount of stems contained in Sweet Hydrangea Leaf does not exceed 3.0%.

(2) Foreign matter—The amount of foreign matter other than stems contained in Sweet Hydrangea Leaf does not exceed 1.0%.

Loss on drying Not more than 13.0% (6 hours).

Total ash Not more than 12.0%.

Acid-insoluble ash Not more than 2.5%.

Powdered Sweet Hydrangea Leaf

Hydrangeae Dulcis Folium Pulveratum

アマチャ末

Powdered Sweet Hydrangea Leaf is the powder of Sweet Hydrangea Leaf.

Description Powdered Sweet Hydrangea Leaf occurs as a dark yellow-green powder, and has a faint odor and a characteristic, sweet taste.

Under a microscope, Powdered Sweet Hydrangea Leaf reveals fragments of epidermis with wavy lateral membrane; stomata with two subsidiary cells; unicellular and thin-walled hair with numerous protrusions of the surface, 150 – 300 μm in length; fragments of palisade tissue and spongy tissue; fragments of vascular bundle and mucilage cells containing raphides of calcium oxalate 50 – 70 μm in length.

Identification Mix 0.5 g of Powdered Sweet Hydrangea Leaf with 8 mL of a mixture of diethyl ether and petroleum ether (1:1), shake well, filter, and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of dilute ethanol, and add 1 drop of dilute iron (III) chloride TS: a red-purple color develops, which disappears on the addition of 2 to 3 drops of dilute sulfuric acid.

Purity Foreign matter—Under a microscope, Powdered Sweet Hydrangea Leaf does not show stone cells, a large quantity of fibers or starch grains.

Loss on drying Not more than 12.0% (6 hours).

Total ash Not more than 12.0%.

Acid-insoluble ash Not more than 2.5%.

Swertia Herb

Swertiae Herba

センブリ

Swertia Herb is the whole herb of *Swertia japonica* Makino (*Gentianaceae*) collected during the blooming season.

It contains not less than 2.0% of swertiamarin ($C_{16}H_{22}O_{10}$: 374.34), calculated on the basis of dried material.

Description Herb, 20 cm in length, having flowers, opposite leaves, stems, and, usually, with short, lignified roots; stems square, about 0.2 cm in diameter, often with branches; the leaves and stems dark green to dark purple or yellow-brown in color; the flowers white to whitish, and the roots yellow-brown. When smoothed by immersing in water, leaves, linear or narrow lanceolate, 1 – 4 cm in length, 0.1 – 0.5 cm in width, entire, and sessile; corolla split deeply as five lobes; the lobes narrow, elongated ellipse shape, and under a magnifying glass, with two elliptical nectaries juxtaposed at the base of the inner surface; the margin of lobe resembles eyelashes; the five stamens grow on the tube of the corolla

and stand alternately in a row with corolla-lobes; peduncle distinct. Odor, slight; taste, extremely bitter and persisting.

Identification To 2.0 g of pulverized Swertia Herb add 10 mL of ethanol (95), shake for 5 minutes, filter, and use the filtrate as the sample solution. Separately, dissolve 2 mg of swertiamarin for thin-layer chromatography in 1 mL of ethanol (95), 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 complex fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, 1-propanol and water (6:4:3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (broad spectrum wavelength): one spot among several spots from the sample solution and a red spot from the standard solution show the same color tone and the same Rf value.

Purity Foreign matter—The amount of straw and other foreign matters contained in Swertia Herb does not exceed 1.0%.

Loss on drying Not more than 12.0% (6 hours).

Total ash Not more than 6.5%.

Extract content Dilute ethanol-soluble extract: not less than 20.0%.

Assay Weigh accurately about 1 g of medium powder of Swertia Herb in a glass-stoppered centrifuge tube, add 40 mL of methanol, shake for 15 minutes, centrifuge, and separate the supernatant liquid. To the residue add 40 mL of methanol, and proceed in the same manner. Combine the extracts, and add methanol to make exactly 100 mL. Pipet 5 mL of the solution, add the mobile phase to make exactly 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of Swertiamarin Reference Standard (separately determine the water content), dissolve in methanol to make exactly 20 mL. Pipet 5 mL of the solution, add the mobile phase to make exactly 20 mL, and use this solution as the standard solution. Perform the test with exactly 10 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 swertiamarin in each solution.

Amount (mg) of swertiamarin ($C_{16}H_{22}O_{10}$)
= amount (mg) of Swertiamarin Reference Standard, calculated on the anhydrous basis
$\times \frac{A_T}{A_S} \times 5$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 238 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

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

Mobile phase: A mixture of water and acetonitrile (91:9).

Flow rate : Adjust the flow rate so that the retention time of swertiamarin is about 12 minutes.

System suitability—

System performance: Dissolve 1 mg each of Swertiamarin Reference Standard and theophylline in the mobile phase to make 10 mL. When the procedure is run with 10 μL of this solution under the above operating conditions, theophylline and swertiamarin are eluted in this order with the resolution of these peaks being not less than 10.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of swertiamarin is not more than 1.5%.

Powdered Swertia Herb

Swertiae Herba Pulverata

センブリ末

Powdered Swertia Herb is the powder of Swertia Herb.

It contains not less than 2.0% of swertiamarin ($C_{16}H_{22}O_{10}$: 374.34), calculated on the basis of dried material.

Description Powdered Swertia Herb occurs as a grayish yellow-green to yellow-brown powder. It has a slight odor, and extremely bitter, persistent taste.

Under a microscope, Powdered Swertia Herb reveals xylem tissues with fibers (components of stems and roots); assimilation tissues (components of leaves and calyces); striated epidermis (components of stems and peduncles); tissues of corollas and filaments with spiral vessels; cells of anthers and their inner walls; spherical pollen grains with granular patterns (components of flowers), about 33 μm in diameter; starch grains are simple grain, about 6 μm in diameter, and very few.

Identification To 2.0 g of Powdered Swertia Herb add 10 mL of ethanol (95), shake for 5 minutes, filter, and use the filtrate as the sample solution. Separately, dissolve 2 mg of swertiamarin for thin-layer chromatography in 1 mL of ethanol (95), 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 complex fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, 1-propanol and water (6:4:3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (broad spectrum wavelength): one spot among several spots from the sample solution and a red spot from the standard solution show the same color tone and Rf value.

Purity Foreign matter—Under a microscope, crystals of calcium oxalate, a large quantity of starch grains and groups of stone cells are not observable.

Loss on drying Not more than 12.0% (6 hours).

Total ash Not more than 6.5%.

Acid-insoluble ash Not more than 2.0%.

Extract content Dilute ethanol-soluble extract: not less than 20.0%.

Assay Weigh accurately about 1 g of Powdered Swertia