filtrate, dissolve the residue in 10 mL of ethanol (95), and to 3 mL of the solution add 1 to 2 drops of dilute iron (III) chloride TS: a grayish green color develops, and it changes to purple-brown later.

(2) To 2.0 g of Powdered Scutellaria Root add 10 mL of methanol, warm on a water bath for 3 minutes, cool, filter, and use the filtrate as the sample solution. Separately, dissolve 1 mg of baicalin 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) (4:2:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly a solution of iron (III) chloride hexahydrate in methanol (1 in 100) on the plate: one spot among the spots from the sample solution and dark green spot from the standard solution show the same in color tone and *Rf* value.

**Purity** Foreign matter—Under a microscope, Powdered Scutellaria Root does not show crystals of calcium oxalate.

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

**Acid-insoluble ash** Not more than 1.0%.

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

**Assay** Weigh accurately about 0.5 g of Powdered Scutellaria Root, add 30 mL of the mobile phase, heat under a reflux condenser on a water bath for 30 minutes, and cool. Transfer the mixture to a glass-stoppered centrifuge tube, centrifuge, and separate the supernatant liquid. Wash the vessel for the reflux extraction with 30 mL of the mobile phase, transfer the washings to the glass-stoppered centrifuge tube, centrifuge after shaking for 5 minutes, and separate the supernatant liquid. To the residue add 30 mL of the mobile phase, shake for 5 minutes, centrifuge, and separate the supernatant liquid. Combine all the extracts, add the mobile phase to make exactly 100 mL, then pipet 2 mL of the extract, 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 Baicalin Reference Standard, and dissolve in methanol to make exactly 20 mL. Pipet 2 mL of the solution, add the mobile phase to make exactly 20 mL, and use this solution as the standard solution. Pipet 10 μL of the sample solution and the standard solution, and perform the test as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas, $A_T$ and $A_S$, of baicalin in each solution.

Amount (mg) of baicalin ($C_{21}H_{18}O_{11}$)
= amount (mg) of Baicalin Reference Standard, calculated on the anhydrous basis

$$\times \frac{A_T}{A_S} \times 5$$

*Operating conditions*—

Detector: An ultraviolet absorption photometer (wavelength: 277 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 for liquid chromatography (5 to 10 μm in particle diameter).

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

Mobile phase: A mixture of diluted phosphoric acid (1 in 146) and acetonitrile (18:7).

Flow rate: Adjust the flow rate so that the retention time of baicalin is about 6 minutes.

Selection of column: Dissolve 1 mg of Baicalin Reference Standard and 2 mg of methyl parahydroxybenzoate in methanol to make 100 mL. Perform the test with 10 μL of this solution under the above operating conditions and calculate the resolution. Use a column giving elution of baicalin and methyl parahydroxybenzoate in this order with the resolution between these peaks being not less than 3.

System repeatability: When repeat the test 6 times with the standard solution under the above operating conditions, the relative standard deviation of the peak area of baicalin is not more than 1.5%.

# Senega

*Senegae Radix*

セネガ

Senega is the root of *Polygala senega* Linné or *Polygala senega* Linné var. *latifolia* Torrey et Gray (*Polygalaceae*).

**Description** Slender, conical root often branched, 3 - 10 cm in length; main root 0.5 - 1.5 cm in diameter; externally light grayish brown to grayish brown; with many longitudinal wrinkles and sometimes with twisted protruding lines; tuberously enlarged crown, with remains of stems and red buds; branched rootlets twisted; a transverse section reveals grayish brown cortex and yellowish white xylem; usually round, and sometimes cuneate to semicircular; cortex on the opposite side is thickened. Odor, characteristic, resembling the aroma of methyl salicylate; taste, sweet at first but leaving an acrid taste.

Under a microscope, a transverse section of the main root reveals a cork layer consisting of several rows of light brown cork cells; secondary cortex composed of parenchyma cells and sieve tubes, traversed by medullary rays, 1 to 3 cells wide; medullary rays on zylem not distinct. Its parenchyma cells contain oil droplets, but starch grains and calcium oxalate crystals are absent.

**Identification** (1) Shake vigorously 0.5 g of pulverized Senega with 10 mL of water: a lasting fine foam is produced.

(2) Shake 0.5 g of pulverized Senega with 30 mL of water for 15 minutes, and filter. Take 1 mL of the filtrate, mix with 50 mL of water, and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum at about 317 nm.

**Purity** (1) Stem—The amount of stems contained in Senega does not exceed 2.0%.

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

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

**Total ash** Not more than 5.0%.

**Acid-insoluble ash** Not more than 2.0%.

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

# Powdered Senega

*Senegae Radix Pulverata*

セネガ末

Powdered Senega is the powder of Senega.

**Description** Powdered Senega occurs as a light brown powder, and has a characteristic odor resembling the aroma of methyl salicylate; taste, sweet at first, but later acrid.

Under a microscope, Powdered Senega reveals fragments of pitted vessels, reticulate vessels and tracheids; fragments of xylem fibers with oblique pits; fragments of xylem parenchyma cells with simple pits; fragments of phloem parenchyma containing oily droplets; fragments of exodermis often composed of cells suberized and divided into daughter cells; oily droplets stained red by sudan III TS. The parenchyma cells of Powdered Senega do not contain starch grains and crystals of calcium oxalate.

**Identification** **(1)** Shake vigorously 0.5 g of Powdered Senega with 10 mL of water: a lasting fine foam is produced.

**(2)** Shake 0.5 g of Powdered Senega with 30 mL of water for 15 minutes, and filter. Take 1 mL of the filtrate, mix with 50 mL of water, and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum at about 317 nm.

**Purity** Foreign matter—Under a microscope, stone cells, starch grains or crystals of calcium oxalate are not observable.

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

**Total ash** Not more than 5.0%.

**Acid-insoluble ash** Not more than 2.0%.

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

# Senega Syrup

*Syrupus Senegae*

セネガシロップ

**Method of preparation**

| | |
|---|---|
| Senega, in medium cutting | 40 g |
| Sucrose | 780 g |
| 10 vol% Ethanol | a sufficient quantity |
| Purified Water | a sufficient quantity |
| | To make 1000 mL |

Add 400 mL of 10 vol% ethanol to Senega, and macerate for one or two days. Filter the extract, wash the residue with a small amount of 10 vol% Ethanol, filter, and combine the filtrate of the extracts and washings until total volume measures about 500 mL. Dissolve Sucrose in the mixture, by warming if necessary, and dilute to 1000 mL with Purified Water. May be prepared with an appropriate quantity of Ethanol and Purified Water in place of 10 vol% Ethanol.

**Description** Senega Syrup is a yellow-brown, viscous liquid. It has a characteristic odor resembling methyl salicylate and a sweet taste.

**Identification** Add 5 mL of water to 1 mL of Senega Syrup, and shake: lasting small bubbles are produced.

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

# Senna Leaf

*Sennae Folium*

センナ

Senna Leaf is the leaflets of *Cassia angustifolia* Vahl or *Cassia acutifolia* Delile (*Leguminosae*).

It contains not less than 1.0% of total sennosides (sennoside A and sennoside B), calculated on the basis of dried material.

**Description** Lanceolate to narrow lanceolate leaflets, 1.5 – 5 cm in length, 0.5 – 1.5 cm in width, light grayish yellow to light grayish yellow-green in color; margin entire, apex acute, base asymmetric, petiole short; under a magnifying glass, vein marked, primary lateral veins running toward the apex along the margin and joining the lateral vein above; lower surface having slight hairs. Odor slight; taste, bitter.

Under a microscope, a transverse section of Senna Leaf reveals epidermis with thick cuticle, with numerous stomata, and with thick-walled, warty unicellular hairs; epidermal cells are often separated into two loculi by a septum which is in parallel with the surface of the leaf, and contain mucilage in the inner loculus; palisade of a single layer under each epidermis; spongy tissue, consisting of 3 to 4 layers, and containing clustered or solitary crystals of calcium oxalate; cells adjacent to vascular bundle, forming crystal cell rows.

**Identification** **(1)** Macerate 0.5 g of pulverized Senna Leaf in 10 mL of diethyl ether for 2 minutes, and filter. Add 5 mL of ammonia TS to the filtrate: a yellow-red color is produced in the water layer. To the residue of maceration add 10 mL of water, and macerate for 2 minutes. Filter, and add 5 mL of ammonia TS: a yellow-red color is produced in the water layer.

**(2)** To 2.0 g of pulverized Senna Leaf add 40 mL of a mixture of tetrahydrofuran and water (7:3), shake for 30 minutes, and centrifuge. Transfer the supernatant liquid to a separator, add 13 g of sodium chloride, and shake for 30 minutes. Separate the water layer with undissolved sodium chloride, and adjust the pH to 1.5 by adding 1 mol/L hydrochloric acid TS. Transfer this solution to another separator, shake with 30 mL of tetrahydrofuran for 10 minutes, separate the tetrahydrofuran layer, and use this solution as the sample solution. Separately, dissolve 1 mg of sennoside A for thin-layer chromatography in 1 mL of a mixture of