Scutellaria Root

Scutellariae Radix

オウゴン

Scutellaria Root is the root of *Scutellaria baicalensis* Georgi (*Labiatae*), from which the periderm has been removed.

It contains not less than 10.0% of baicalin, calculated on the basis of dried material.

Description Cone-shaped, semitubular or flattened root, 5 – 20 cm in length, 0.5 – 3 cm in diameter; externally yellow-brown, with coarse and marked longitudinal wrinkles, and with scattered scars of lateral root and remains of brown periderm; scars of stem or remains of stem at the crown; xylem rotted in old roots, often forming a hollow; hard in texture and easily broken; fractured surface fibrous and yellow in color. Almost odorless; taste, slightly bitter.

Identification **(1)** Boil gently 0.5 g of pulverized Scutellaria Root with 20 mL of diethyl ether under a reflux condenser on a water bath for 5 minutes, cool, and filter. Evaporate the 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.

(2) To 2.0 g of pulverized 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 a dark green spot from the standard solution show the same color tone and the same *R*f value.

Total ash Not more than 6.0%.

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

Assay Weigh accurately about 0.5 g of pulverized 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 of the pulverized Scutellaria Root and the mobile phase into a glass-stoppered centrifuge tube, centrifuge after shaking for 5 minutes, 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, 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 the test is repeated 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%.

Powdered Scutellaria Root

Scutellariae Radix Pulverata

オウゴン末

Powdered Scutellaria Root is the powder of Scutellaria Root.

It contains not less than 10.0% of baicalin, calculated on the basis of dried material.

Description Powdered Scutellaria Root occurs as a yellow-brown powder. It is almost odorless, and has a slight, bitter taste.

Under a microscope, Powdered Scutellaria Root reveals fragments of parenchyma cells containing small amount of starch grains, fragments of reticulate vessels, tracheids and elongated stone cells; also a few fragments of spiral vessels and xylem fibers are observed.

Identification **(1)** Boil gently 0.5 g of Powdered Scutellaria Root with 20 mL of diethyl ether under a reflux condenser on a water bath for 5 minutes, cool, and filter. Evaporate the

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%.