# Corydalis Tuber

*Corydalis Tuber*

エンゴサク

Corydalis Tuber is the tuber of *Corydalis turtschaninovii* Basser forma *yanhusuo* Y. H. Chou et C. C. Hsu (*Papaveraceae*).

It contains not less than 0.08% of dehydrocorydaline (as dehydrocorydaline nitrate), calculated on the basis of dried material.

**Description** Nearly flattened spherical, 1 - 2 cm in diameter, and with stem scar at one end; externally grayish yellow to grayish brown; hard in texture; fractured surface is yellow and smooth or grayish yellow-green in color and granular. Almost odorless; taste, bitter.

**Identification** To 0.5 g of pulverized Corydalis Tuber add 10 mL of dilute acetic acid, heat on a water bath for 3 minutes with occasional shaking, cool, and filter. To 5 mL of the filtrate add 2 drops of Dragendorff's TS: immediately, an orange-yellow precipitate is produced.

**Loss on drying** Not more than 15.0%.

**Total ash** Not more than 3.0%.

**Component determination** Weigh accurately about 1 g of powdered Corydalis Tuber, add 30 mL of a mixture of methanol and dilute hydrochloric acid (3:1), heat under a reflux condenser on a water bath for 30 minutes, and filter after cooling. To the residue add 15 mL of a mixture of methanol and dilute hydrochloric acid (3:1), and repeat the above procedure. Combine the filtrates so obtained, add a mixture of methanol and dilute hydrochloric acid (3:1) to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of dehydrocorydaline nitrate for component determination, previously dried in a desiccator (silica gel) for not less than 1 hour, dissolve in a mixture of methanol and dilute hydrochloric acid (3:1) to make exactly 200 mL, and use this solution as the standard solution. Perform the test with exactly 5 $\mu$L 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 dehydrocorydaline in each solution.

Amount (mg) of dehydrocorydaline [as dehydrocorydaline nitrate $C_{22}H_{24}N_2O_7$)]
= amount (mg) of dehydrocorydaline nitrate for component determination
$\times \frac{A_T}{A_S} \times \frac{1}{4}$

*Operating conditions—*

Detector: An ultraviolet absorption photometer (wavelength: 340 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 $\mu$m in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 17.91 g of disodium hydrogenphosphate 12-water in 970 mL of water, and adjust to pH 2.2 with phosphoric acid. In this solution add 14.05 g of sodium perchlorate to dissolve, and add water to make exactly 1000 mL. To this solution add 450 mL of acetonitrile, then add 0.20 g of sodium lauryl sulfate to dissolve.

Flow rate: Adjust the flow rate so that the retention time of dehydrocorydaline is about 24 minutes.

*System suitability—*

System performance: Dissolve 1 mg each of dehydrocorydaline nitrate for component determination and berberine chloride in 20 mL of a mixture of water and acetonitrile (20:9). When the procedure is run with 5 $\mu$L of this solution under the above operating conditions, berberine and dehydrocorydaline are eluted in this order with the resolution between these peaks being not less than 1.5.

System repeatability: When the test is repeated 6 times with 5 $\mu$L of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of dehydrocorydaline is not more than 1.5%.

# Creosote

クレオソート

Creosote is a mixture of phenols obtained from wood tar.

**Description** Creosote is a colorless or pale yellow, clear liquid. It has a characteristic odor, and a burning taste.

It is miscible with ethanol (95) and with diethyl ether.

It is slightly soluble in water.

Its saturated solution is neutral.

It is highly refractive.

It gradually changes in color by light or by air.

**Identification** To 10 mL of a saturated solution of Creosote add 1 drop of iron (III) chloride TS: a purple color develops, and the solution becomes rapidly turbid, and changes through blue and muddy green to brown.

**Specific gravity** $d_{20}^{20}$: not less than 1.076.

**Purity** **(1)** Bases and hydrocarbons—Shake 1.0 mL of Creosote with 9 mL of sodium hydroxide TS: the solution is clear and does not darken. On further addition of 50 mL of water, the solution is practically clear.

**(2)** Phenol or coal-tar creosote—Shake Creosote with an equal volume of collodion: no coagulum is produced.

**(3)** Other impurities—To 1.0 mL of Creosote add 2 mL of petroleum benzin and 2 mL of barium hydroxide TS, shake, and allow to stand: no blue or muddy brown color develops in the upper layer of the mixture, and no red color develops in the lower layer.

**Distilling range** 200 - 220°C, not less than 85 vol%.

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

Storage—Light-resistant.