(emetine and cephaeline), calculated on the basis of dried material.

Description Slender, curved, cylindrical root, 3 - 15 cm in length, 0.3 - 0.9 cm in diameter; mostly twisted, and sometimes branched; outer surface gray, dark grayish brown, reddish brown in color and irregularly annulated; when root fractured, cortex easily separable from the xylem; the cortex on the fractured surface is grayish brown, and the xylem is light brown in color: thickness of cortex up to about two-thirds of radius in thickened portion. Scales in rhizome opposite. Odor, slight; powder irritates the mucous membrane of the nose; taste, slightly bitter and unpleasant.

Under a microscope, the transverse section of Ipecac reveals a cork layer, consisting of brown thin-walled cork cells; in the cortex, sclerenchyma cells are absent; in the xylem, vessels and tracheids arranged alternately; parenchyma cells filled with starch grains and sometimes with raphides of calcium oxalate.

Identification To 0.5 g of pulverized Ipecac add 2.5 mL of hydrochloric acid, allow to stand for 1 hour with occasional shaking, and filter. Collect the filtrate into an evaporating dish, and add a small pieces of chlorinated lime: circumference of it turns red.

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

Total ash Not more than 5.0%.

Acid-insoluble ash Not more than 2.0%.

Component determination Weigh accurately about 0.5 g of pulverized Ipecac, in a glass-stoppered centrifuge tube, add 30 mL of 0.01 mol/L hydrochloric acid TS, shake for 15 minutes, centrifuge, and separate the supernatant liquid. Repeat this procedure twice with the residue using 30-mL portions of 0.01 mol/L hydrochloric acid TS. Combine all the extracts, add 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of emetine hydrochloride for component determination, previously dried in a desiccator (reduced below 0.67 kPa, phosphorus (V) oxide, 50°C) for 5 hours, dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 100 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_{TE} and A_{TC}, of emetine and cephaeline in the sample solution, and the peak area, A_{SE}, of emetine in the standard solution.

$$\text{Amount (mg) of total alkaloids (emetine and cephaeline)} = \text{amount (mg) of emetine hydrochloride for component determination} \times \frac{A_{TE} + A_{TC} \times 0.971}{A_{SE}} \times 0.868$$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength : 283 nm).

Column: A stainless steel column 4 to 6 mm in inside diameter and 10 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: Dissolve 2.0 g of sodium 1-heptane sulfonate in 500 mL of water, adjust the pH 4.0 with acetic acid (100), and add 500 mL of methanol.

Flow rate: Adjust the flow rate so that the retention time of emetine is about 14 minutes.

Selection of column: Dissolve 1 mg each of emetine hydrochloride for component determination and cephaeline hydrobromide in 10 mL of 0.01 mol/L hydrochloric acid TS. Perform the test with 10 μL of this solution under the above operating conditions. Use a column giving elution of cephaeline and emetine in this order, and clearly dividing each peak.

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

Powdered Ipecac

Ipecacuanhae Radix Pulverata

トコン末

Powdered Ipecac is the powder of Ipecac or its powder diluted with Potato Starch.

It contains not less than 2.0% and not more than 2.6% of the total alkaloids (emetine and cephaeline).

Description Powdered Ipecac occurs as a light grayish yellow to light brown powder. It has a slight odor, which is irritating to the nasal mucosa, and has a somewhat bitter and unpleasant taste.

Under a microscope, Powdered Ipecac reveals starch grains and needle crystals of calcium oxalate; fragments of parenchyma cells containing starch grains or the needle crystals; substitute fibers,thin-walled cork tissue; vessels and tracheids with simple or bordered pits; a few wood fibers and wood parenchyma. Starch grains inherent in Ipecac, mainly 2 - 8-compound grains, rarely simple grains 4 - 22 μm in diameter; and needle crystals of calcium oxalate 25 - 60 μm in length.

Identification To 0.5 g of Powdered Ipecac add 2.5 mL of hydrochloric acid, allow to stand for 1 hour with occasional shaking, and filter. Collect the filtrate into an evaporating dish, and add a small pieces of chlorinated lime: circumference of it turns red.

Purity Foreign matter—Under a microscope, groups of stone cells and thick-walled fibers are not observed.

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

Total ash Not more than 5.0%.

Acid-insoluble ash Not more than 2.0%.

Component determination Weigh accurately about 0.5 g of Powdered Ipecac, transfer into a glass-stoppered centrifuge tube, add 30 mL of 0.01 mol/L hydrochloric acid TS, shake for 15 minutes, centrifuge, and separate the supernatant liquid. Repeat this procedure twice with the residue using 30-mL portions of 0.01 mol/L hydrochloric acid TS. Combine

all the extracts, add 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of emetine hydrochloride for component determination, previously dried in a desiccator (reduced below 0.67 kPa, phosphorus (V) oxide, 50°C) for 5 hours, dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 100 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_{TE} and A_{TC}, of emetine and cephaeline in the sample solution, and the peak area, A_{SE}, of emetine in the standard solution.

Amount (mg) of total alkaloids (emetine and cephaeline)
= amount (mg) of emetine hydrochloride for component determination
$$\times \frac{A_{TE} + A_{TC} \times 0.971}{A_{SE}} \times 0.868$$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength : 283 nm).

Column: A stainless steel column 4 to 6 mm in inside diameter and 10 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: Dissolve 2.0 g of sodium 1-heptane sulfonate in 500 mL of water, adjust the pH 4.0 with acetic acid (100), and add 500 mL of methanol.

Flow rate: Adjust the flow rate so that the retention time of emetine is about 14 minutes.

Selection of column: Dissolve 1 mg each of emetine hydrochloride for component determination and cephaeline hydrobromide in 10 mL of 0.01 mol/L hydrochloric acid TS. Perform the test with 10 μL of this solution under the above operating conditions. Use a column giving elution of cephaeline and emetine in this order, and clearly dividing each peak.

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

Ipecac Syrup

Syrupus Ipecacuanha

トコンシロップ

Ipecac Syrup is a syrup containing not less than 0.12 g and not more than 0.15 g of the total alkaloids (emetine and cephaeline) per 100 mL.

Method of preparation Take coarse powder of Ipecac, prepare the fluidextract as directed under Fluidextracts using a mixture of Ethanol and Purified Water (3:1), and evaporate the mixture under reduced pressure or add a suitable amount of Ethanol or Purifiect Water if necessary to get a solution containing 1.7 to 2.1 g of the total alkaloids (emetine and cephaeline) per 100 mL. 70 mL of To this solution add 100 mL of Glycerin and Simple Syrup to make 1000 mL, as directed under Syrups.

Description Ipecac Syrup is a yellow-brown, viscous liquid. It has a sweet taste and a bitter aftertaste.

Identification Take 2 mL of Ipecac Syrup into an evaporating dish, mix with 1 mL of hydrochloric acid, and add small pieces of chlorinated lime: circumference of it turns orange.

Purity Ethanol—Take exactly 5 mL of Ipecac Syrup, add 5 mL of the internal standard solution and water to make 50 mL, and use this solution as the sample solution. Separately, pipet 5 mL of ethanol (99.5), and add water to make exactly 100 mL. To exactly 5 mL of this solution add exactly 5 mL of the internal standard solution and water to make 50 mL, and use this solution as the standard solution. Perform the test with 2 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and determine the rate of peak height of ethanol to that of the internal standard, Q_T and Q_S: Q_T is not larger than Q_S.

Internal standard solution—A solution of acetonitrile (5 in 100).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass-column about 3 mm in inside diameter and about 1.5 m in length, packed with ethylvinylbenzene-divinylbenzene porous co-polymer for gas chromatography (150 to 180 μm in particle diameter).

Column temperature: A constant temperature of between 105°C and 115°C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of ethanol is 5 to 10 minutes.

Selection of column: Proceed with 2 μL of the standard solution under the above operating conditions. Use a column giving elution of ethanol and the internal standard in this order, and clearly dividing each peak.

Component determination Take exactly 5 mL of Ipecac Syrup, add 0.01 mol/L hydrochloric acid TS to make exactly 50 mL, and use the solution as the sample solution. Separately, weigh accurately about 0.01 g of emetine hydrochloride for component determination, previously dried in a desiccator (reduced pressure under 0.67 kPa, phosphorus (V) oxide, 50°C) for 5 hours, dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas, A_{TE} and A_{TC}, of emetine and cephaeline in the sample solution, and the peak area, A_{SE}, of emetine in the standard solution.

Amount (mg) of total alkaloids (emetine and cephaeline)
= amount (mg) of emetine hydrochloride for component determination
$$\times \frac{A_{TE} + A_{TC} \times 0.971}{A_{SE}} \times \frac{1}{2} \times 0.868$$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 283 nm).