

tasteless, and numbs the tongue.

It is freely soluble in ethanol (95), in ethanol (99.5), in acetone and in diethyl ether, slightly soluble in hot water, and very slightly soluble in water.

Identification (1) Dissolve 0.25 g of Propyl Parahydroxybenzoate in 5 mL of dilute ethanol, and add 1 drop of iron (III) chloride TS: a red-purple color develops.

(2) Boil 0.5 g of Propyl Parahydroxybenzoate with 10 mL of sodium hydroxide TS for about 30 minutes, allowing the solution to evaporate to about 5 mL. After cooling, acidify with dilute sulfuric acid, collect the precipitate formed, wash thoroughly with a small amount of water, and dry in a desiccator (silica gel): the precipitate melts between 213°C and 217°C.

(3) To 0.05 g of Propyl Parahydroxybenzoate add 2 drops of acetic acid (31) and 5 drops of sulfuric acid, and heat the mixture for 5 minutes: the odor of propyl acetate is perceptible.

Melting point 96 – 99°C

Purity (1) Chloride—Heat 2.0 g of Propyl Parahydroxybenzoate with 50 mL of water, allow to stand in ice water for 1 hour with occasional shaking, then add water to make 100 mL, and filter. Perform the test with 25 mL of the filtrate. Prepare the control solution with 0.50 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.035%).

(2) **Sulfate**—Perform the test with 40 mL of the filtrate obtained in Purity (1). Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.024%).

(3) **Heavy metals**—Dissolve 1.0 g of Propyl Parahydroxybenzoate in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 2.0 mL of Standard Lead Solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL (not more than 20 ppm).

(4) **Parahydroxybenzoic acid and salicylic acid**—Dissolve 0.50 g of Propyl Parahydroxybenzoate in 30 mL of diethyl ether, shake with 20 mL of a solution of sodium hydrogen carbonate (1 in 100), wash the separated aqueous layer with two 20-mL portions of diethyl ether, shake the aqueous layer with 5 mL of dilute sulfuric acid and 30 mL of diethyl ether, and allow to stand. Shake gently the separated diethyl ether layer with 10 mL of water, remove the aqueous layer after allowing the mixture to stand, filter the diethyl ether solution, wash the vessel and the filter paper with a small amount of diethyl ether, evaporate the diethyl ether from the combined filtrate and washings on a water bath, and dry the residue in a desiccator (silica gel) to constant mass: the mass of the residue is not more than 5.0 mg. Warm the residue with 5 mL of water, filter, and to the filtrate add 2 to 3 drops of dilute iron (III) chloride TS: no purple color develops.

(5) **Readily carbonizable substances**—Perform the test with 0.50 g of Propyl Parahydroxybenzoate. The solution has no more color than Matching Fluid D.

Loss on drying Not more than 0.5% (2 g, silica gel, 3 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 2 g of Propyl Parahydrox-

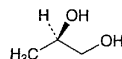
ybenzoate, previously dried, add exactly 40 mL of 1 mol/L sodium hydroxide VS, and boil for 30 minutes. Cool, and titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS until the solution shows the same color as that of phosphate buffer solution, pH 6.5, to which the same indicator has been added (indicator: 5 drops of bromothymol blue TS). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 180.20 mg of C₁₀H₁₂O₃

Containers and storage Containers—Well-closed containers.

Propylene Glycol

プロピレングリコール



and enantiomer

C₃H₈O₂: 76.09

(*RS*)-Propane-1,2-diol [57-55-6]

Description Propylene Glycol is a clear, colorless, viscous liquid. It is odorless, and has a slightly bitter taste.

It is miscible with water, with methanol, with ethanol (95) and with pyridine.

It is freely soluble in diethyl ether.

It is hygroscopic.

Identification (1) Mix 2 to 3 drops of Propylene Glycol with 0.7 g of triphenylchloromethane, add 1 mL of pyridine, and heat under a reflux condenser on a water bath for 1 hour. After cooling, dissolve the mixture in 20 mL of acetone by warming, shake with 0.02 g of activated charcoal, and filter. Concentrate the filtrate to about 10 mL, and cool. Collect the separated crystals, and dry in a desiccator (silica gel) for 4 hours: the crystals melt between 174°C and 178°C.

(2) Heat gently 1 mL of Propylene Glycol with 0.5 g of potassium hydrogen sulfate: a characteristic odor is evolved.

Specific gravity d_{20}^{20} : 1.035 – 1.040

Purity (1) Acid—Mix 10.0 mL of Propylene Glycol with 50 mL of freshly boiled and cooled water, and add 5 drops of phenolphthalein TS and 0.30 mL of 0.1 mol/L sodium hydroxide VS: the solution has a red color.

(2) **Chloride**—Perform the test with 2.0 g of Propylene Glycol. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.007%).

(3) **Sulfate**—Perform the test with 10.0 g of Propylene Glycol. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.002%).

(4) **Heavy metals**—Perform the test with 5.0 g of Propylene Glycol according to Method 1. Prepare the control solution with 2.5 mL of Standard Lead Solution (not more than 5 ppm).

(5) **Arsenic**—Prepare the test solution with 1.0 g of Propylene Glycol according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

(6) **Glycerin**—Heat 1.0 g of Propylene Glycol with 0.5 g of potassium hydrogen sulfate and evaporate to dryness: no odor of acrolein is perceptible.

Water Not more than 0.5% (2 g, direct titration).

Residue on ignition Weigh accurately about 20 g of Propylene Glycol in a tared crucible, and heat to boiling. Stop heating, and immediately ignite to burn. Cool, moisten the residue with 0.2 mL of sulfuric acid, and heat strongly with care to constant mass: the mass of the residue is not more than 0.005%.

Distilling range 184 – 189°C, not less than 95 vol%.

Containers and storage Containers—Tight containers.

Prunella Spike

Prunellae Spica

カゴソウ

Prunella Spike is the spike of *Prunella vulgaris* Linné var. *lilacina* Nakai (*Labiatae*).

Description Spikes in nearly cylindrical and wheat ear-like shape, 3 – 6 cm in length, 1 – 1.5 cm in diameter, externally grayish brown; spikes composed of a floral axis having numerous bracts and calyxes; corollas often remaining on the upper part; a calyx usually enclosing four mericarps; bract, cordate to eccentric, and exhibiting white hairs on the vein, as on the calyx; light in texture. Almost odorless and tasteless.

Purity (1) Stem—The amount of the stems contained in Prunella Spike does not exceed 5.0%.

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

Total ash Not more than 13.0%.

Acid-insoluble ash Not more than 5.0%.

Pueraria Root

Puerariae Radix

カッコン

Pueraria Root is the root of *Pueraria lobata* Ohwi (*Leguminosae*), from which periderm has been removed.

Description Usually cut into small pieces of irregular hexagons of about 0.5 cm cube, or cut into longitudinally plate-like pieces 20 – 30 cm in length, 5 – 10 cm in width, and about 1 cm in thickness; externally light grayish yellow to grayish white; transverse section showing concentric annulate ring or part of it formed by abnormal growth of cambium. Under a magnifying glass, phloem light grayish yellow in color; in xylem, numerous vessels appearing as small dots; medullary rays slightly dented; vertical section showing longitudinal patterns formed alternately by fibrous xylem and parenchyma; easily breakable lengthwise, and its section extremely fibrous. Odorless; taste, slightly sweet.

Under a microscope, a transverse section reveals fiber bun-

dles accompanied by crystal cells in phloem; distinct vessels and xylem fibers in xylem; starch grains numerous in parenchyma, mainly composed of polygonal simple grains, rarely 2- to 3-compound grains, 2 – 18 μm , mostly 8 – 12 μm , in size, with hilum or cleft in the center, and also with striae.

Identification To 2.0 g of pulverized Pueraria Root add 10 mL of methanol, shake for 3 minutes, filter, and use the filtrate as the sample solution. Separately, dissolve 1 mg of puerarin 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 2 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 ethyl acetate, methanol and water (12:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one of the spots from the sample solution and a blue-white spot from the standard solution show the same color tone and *R_f* value.

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

Total ash Not more than 6.0%.

Pyroxylin

ピロキシリン

Pyroxylin is a nitric acid ester of cellulose. It is usually moistened with 2-propanol or some other solvent.

Description Pyroxylin occurs as a white cotton-like substance or white flakes.

It is freely soluble in acetone, and very slightly soluble in diethyl ether.

Upon heating or exposure to light, it is decomposed with the evolution of nitrous acid vapors.

Identification Ignite Pyroxylin: it burns very rapidly with a luminous flame.

Purity (1) Clarity of solution—Dissolve 1.0 g of Pyroxylin, previously dried at 80°C for 2 hours, in 25 mL of a mixture of diethyl ether and ethanol (95) (3:1): the solution is clear.

(2) Acid—Shake 1.0 g of Pyroxylin, previously dried at 80°C for 2 hours, with 20 mL of water for 10 minutes: the filtrate is neutral.

(3) Water-soluble substances—Evaporate 10 mL of the filtrate obtained in (2) on a water bath to dryness, and dry at 105°C for 1 hour: the mass of the residue is not more than 1.5 mg.

(4) Residue on ignition—Weigh accurately about 2 g of Pyroxylin, previously dried at 80°C for 2 hours, and moisten with 10 mL of a solution of castor oil in acetone (1 in 20) to gelatinize the sample. Ignite the contents to carbonize the sample, heat strongly at about 500°C for 2 hours, and allow to cool over silica gel: the amount of the residue is not more than 0.30%.

Containers and storage Containers—Tight containers.

Storage—Light-resistant, packed loosely, remote from fire, and preferably in a cold place.