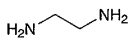
Assay Weigh accurately about 2 g of Ethyl Parahydroxybenzoate, previously dried, add exactly 40 mL of 1 mol/L sodium hydroxide VS, and boil for 30 minutes. Cool, and titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS until the solution shows the same color as that of phosphate buffer 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
= 166.18 mg of $C_9H_{10}O_3$

Containers and storage Containers—Well-closed containers.

Ethylenediamine

エチレンジアミン



$C_2H_8N_2$: 60.10
Ethane-1,2-diamine [*107-15-3*]

Ethylenediamine contains not less than 97.0% of $C_2H_8N_2$.

Description Ethylenediamine is a clear, colorless to pale yellow liquid. It has an ammonia-like odor.

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

It has a caustic nature and an irritating property.

It is gradually affected by air.

Specific gravity d_{20}^{20}: about 0.898

Identification **(1)** A solution of Ethylenediamine (1 in 500) is alkaline.

(2) To 2 mL of copper (II) sulfate TS add 2 drops of Ethylenediamine: a blue-purple color develops.

(3) To 0.04 g of Ethylenediamine add 6 drops of benzoyl chloride and 2 mL of a solution of sodium hydroxide (1 in 10), warm for 2 to 3 minutes with occasional shaking, collect the white precipitate formed, and wash with water. Dissolve the precipitate in 8 mL of ethanol (95) by warming, promptly add 8 mL of water, cool, filter the crystals, wash with water, and dry at 105°C for 1 hour: it melts between 247°C and 251°C.

Purity **(1)** Heavy metals—Place 1.0 g of Ethylenediamine in a porcelain crucible, evaporate to dryness on a water bath, cover loosely, ignite at a low temperature until charred, proceed according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(2) Residue on evaporation—Pipet 5 mL of Ethylenediamine, heat on a water bath to dryness, and dry to constant mass at 105°C: the mass of the residue does not exceed 3.0 mg.

Distilling range 114 - 119°C, not less than 95 vol%.

Assay Weigh accurately about 0.7 g of Ethylenediamine in a glass-stoppered conical flask, add 50 mL of water, and titrate with 1 mol/L hydrochloric acid VS (indicator: 3 drops of bromophenol blue TS).

Each mL of 1 mol/L hydrochloric acid VS
= 30.049 mg of $C_2H_8N_2$

Containers and storage Containers—Tight containers.
Storage—Light-resistant, and almost well-filled.

Eucalyptus Oil

Oleum Eucalypti

ユーカリ油

Eucalyptus Oil is the essential oil distilled with steam from the leaves of *Eucalyptus globulus* Labillardière or allied plants (*Myrtaceae*).

Description Eucalyptus Oil is a clear, colorless or pale yellow liquid. It has a characteristic, aromatic odor and a pungent taste.

It is neutral.

Identification Shake 1 mL of Eucalyptus Oil vigorously with 1 mL of phosphoric acid, and allow to stand: the solution congeals within 30 minutes.

Refractive index n_D^{20}: 1.458 - 1.470

Specific gravity d_{20}^{20}: 0.907 - 0.927

Purity **(1)** Clarity of solution—Mix 1.0 mL of Eucalyptus Oil with 5 mL of diluted ethanol (7 in 10): the solution is clear.

(2) Heavy metals—Proceed with 1.0 mL of Eucalyptus Oil according to Method 2, and perform the test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 40 ppm).

Assay Weigh accurately about 0.1 g of Eucalyptus Oil, and dissolve in hexane to make exactly 25 mL. Pipet 5 mL of this solution, add exactly 5 mL of the internal standard solution, then add hexane to make 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of cineol for assay, proceed as directed in the sample solution, 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. Calculate the ratios, Q_T and Q_S, of the peak area of cineol to that of the internal standard of each solutions, respectively.

$$\text{Amount (mg) of cineol } (C_{10}H_{18}O) = \text{amount (mg) of cineol for assay} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of anisol in hexane (1 in 250).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass column about 3 mm in inside diameter and about 5 m in length, having alkylene glycol phthalate ester for gas chromatography coated at the ratio of 10% on silanized siliceous earth for gas chromatography (150 to 180 μm in particle diameter).

Column temperature: A constant temperature of about 120°C.

Carrier gas: Nitrogen.

Flow rate: Adjust the flow rate so that the retention time of cineol is about 11 minutes.

Selection of column: Dissolve 0.1 g each of cineol and limonene in 25 mL of hexane. To 1 mL of this solution add hexane to make 20 mL. Proceed with about 2 μL of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of limonene and cineol in this order with the resolution between these peaks being not less than 1.5.

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Evodia Fruit

Evodiae Fructus

ゴシュユ

Evodia Fruit is the fruit of *Evodia rutaecarpa* Bentham or *Evodia officinalis* Dode (*Rutaceae*).

Description Flattened spheroidal or globular fruit, 2 - 5 mm in diameter; externally dark brown to grayish brown, with many oil sacs appearing as hollow pits, and often with peduncle, 2 - 5 mm in length, covered densely with hairs; matured pericarp split to reveal five loculi, and each loculus containing obovoid or globular seeds of a lustrous brown to blackish brown or bluish black color. Odor, characteristic; taste, acrid, followed by a lasting bitterness.

Identification To 1.0 g of pulverized Evodia Fruit add 20 mL of methanol, heat for 5 minutes on a water bath, cool, and filter. Evaporate the filtrate to dryness, add 3 mL of dilute acetic acid to the residue, warm for 2 minutes on a water bath, cool, and filter. Perform the following tests using the filtrate as the sample solution.

(1) Spot one drop of the sample solution on a filter paper, air-dry, spray Dragendorff's TS for spraying, and allow to stand: a yellow-red color develops.

(2) To 0.2 mL of the sample solution add 0.8 mL of dilute acetic acid. To this solution add gently 2 mL of 4-dimethylaminobenzaldehyde TS, and warm in a water bath: a purple-brown ring develops at the zone of contact.

Purity **(1)** Peduncle—The amount of peduncles contained in Evodia Fruit does not exceed 5.0%.

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

Total ash Not more than 8.0%.

Fennel

Foeniculi Fructus

ウイキョウ

Fennel is the fruit of *Foeniculum vulgare* Miller (*Umbelliferae*).

Description Cylindrical cremocarp, 3.5 - 8 mm in length, 1 - 2.5 mm in width; externally grayish yellow-green to grayish yellow; two mericarps closely attached with each other, and with five longitudinal ridges; cremocarp often with pedicel 2 - 10 mm in length. Characteristic odor and taste.

Under a microscope, ridges near the bentral side are far protruded than those on the dorsal side; one large oil canal between each ridge, and two oil canals on the bentral side.

Identification To 0.5 g of pulverized Fennel add 10 mL of hexane, allow to stand for 5 minutes with occasional shaking, filter, and use the filtrate as the sample solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot 5 μL of the sample solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (20:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): a main spot with a dark purple color appears at the *Rf* value of about 0.4.

Purity **(1)** Peduncle—The amount of peduncles contained in Fennel does not exceed 3.0%.

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

Total ash Not more than 10.0%.

Acid-insoluble ash Not more than 1.5%.

Essential oil content Perform the test with 50.0 g of pulverized Fennel as directed in the Essential oil content under Crude Drugs: the volume of essential oil is not less than 0.7 mL.

Powdered Fennel

Foeniculi Fructus Pulveratus

ウイキョウ末

Powdered Fennel is the powder of Fennel.

Description Powdered Fennel occurs as a greenish pale brown to greenish brown, and is a characteristic odor and taste.

Under a microscope, fennel powder reveals fragments of parenchyma cells of perisperm containing aleurone grain, fragments of parenchyma cells of endosperm containing fatty oil, fragments of sclerenchyma with characteristic single pits, fragments of oil canal within yellowish brown material, fragments of endocarp shown scalariform, spiral vessels, epidermis, stomata.