

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 μ L 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

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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

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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 μ L 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 *R_f* 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

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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.

Identification To 0.5 g of Powdered 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 the sample solution as directed under the Thin-layer Chromatography. Spot 5 μ L of the sample solution on a plate prepared with silica gel with fluorescent indicator for thin-layer chromatography. Then 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 dark purple color appears at the *R_f* value of about 0.4.

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 Powdered Fennel as directed in Essential oil content under Crude Drugs: the volume of essential oil is not less than 0.45 mL.

Containers and storage Containers—Tight containers.

Fennel Oil

Oleum Foeniculi

ウイキョウ油

Fennel Oil is the essential oil distilled with steam from the fruit of *Foeniculum vulgare* Miller (*Umbeliferae*) or of *Illicium verum* Hooker fil. (*Illiciaceae*).

Description Fennel Oil is a colorless to pale yellow liquid. It has a characteristic, aromatic odor and a sweet taste with a slight, bitter aftertaste.

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

It is practically insoluble in water.

When cold, white crystals or crystalline masses may often separate from the oil.

Identification Dissolve 0.30 g of Fennel Oil in 20 mL of hexane, pipet 1 mL of this solution, add hexane to make exactly 10 mL, and use this solution as the sample solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot 5 μ L 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 *R_f* value of about 0.4.

Refractive index n_D^{20} : 1.528 – 1.560

Specific gravity d_{20}^{20} : 0.955 – 0.995

Purity (1) Clarity of solution—To 1.0 mL of Fennel Oil add 3 mL of ethanol (95): the solution is clear. To this solution add 7 mL of ethanol (95): the solution remains clear.

(2) Heavy metals—Proceed with 1.0 mL of Fennel 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).

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Foeniculated Ammonia Spirit

アンモニア・ウイキョウ精

Method of preparation

| | |
|---------------|-----------------------|
| Ammonia Water | 170 mL |
| Fennel Oil | 30 mL |
| Ethanol | a sufficient quantity |
| <hr/> | |
| | To make 1000 mL |

Prepare as directed under Medicated Spirits, with the above ingredients. A sufficient quantity of ammonia solution (28) and Purified Water may be used in place of Ammonia Water.

Description Foeniculated Ammonia Spirit is a clear, pale yellow to yellow liquid, having a characteristic odor. It has a slightly sweet, pungent taste.

Specific gravity d_{20}^{20} : about 0.85

Alcohol number Not less than 7.8 (Method 2).

Containers and storage Containers—Tight containers.

Formalin

ホルマリン

Formalin contains not less than 35.0% and not more than 38.0% of formaldehyde (CH_2O : 30.03.)

It contains 5% to 13% of methanol to prevent polymerization.

Description Formalin is a clear, colorless liquid. Its vapor is irritating to the mucous membrane.

It is miscible with water and with ethanol (95).

When stored for a long time, especially in a cold place, it may become cloudy.

Identification (1) Dilute 2 mL of Formalin with 10 mL of water in a test tube, and add 1 mL of silver nitrate-ammonia TS: a gray precipitate is produced, or a silver mirror is formed on the wall of the test tube.

(2) To 5 mL of sulfuric acid in which 0.1 g of salicylic acid has been dissolved add 2 drops of Formalin, and warm the solution: a persistent, dark red color develops.

Purity Acid—Dilute 20 mL of Formalin with 20 mL of water, and add 5.0 mL of 0.1 mol/L sodium hydroxide VS and 2 drops of bromothymol blue TS: a blue color develops.

Residue on ignition Not more than 0.06 w/v% (5 mL, after evaporation).

Assay Weigh accurately a weighing bottle containing 5 mL of water, add about 1 g of Formalin, and weigh accurately again. Add water to make exactly 100 mL. Pipet 10 mL of this solution, add exactly 50 mL of 0.05 mol/L iodine VS and 20 mL of potassium hydroxide TS, and allow to stand for 15 minutes at an ordinary temperature. To this mixture add 15 mL of dilute sulfuric acid, and titrate the excess iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL