## Method of preparation

Powdered Opium Powdered Ipecac		)0 g )0 g
Starch or a suitable diluent	a sufficient quantity	
	To make 100	00 g

Prepare as directed under Powders, with the above ingredients. Lactose should not be used.

**Description** Opium Ipecac Powder occurs as a light brown powder.

**Identification** (1) Proceed with 1 g of Opium Ipecac Powder as directed in the Identification (1) under Powdered Opium.

- (2) Proceed with 1 g of Opium Ipecac Powder as directed in the Identification (2) under Powdered Opium.
- (3) Shake frequently a mixture of 3 g of Opium Ipecac Powder and 5 mL of hydrochloric acid, and allow to stand for 1 hour. Filter the solution into an evaporating dish. Add 5 mg of chlorinated lime to the filtrate: an orange color is produced at the circumference of the chlorinated lime (emetine).

Assay Weigh accurately about 50 g of Opium Ipecac Powder in a glass stoppered flask, add 250 mL of dilute ethanol, warm in a water bath at 40°C for 1 hour with stirring, and filter through a glass filter (G3). Transfer the residue on the filter to the first glass-stoppered flask, add 50 mL of dilute ethanol, warm in a water bath at 40°C for 10 minutes with stirring, and filter through the glass filter. Repeat the extraction with three 50-mL portions of dilute ethanol. Combine all the filtrates in a mortar, evaporate on a water bath to dryness, add 10 mL of ethanol (99.5) to the residue, and evaporate again. After cooling, triturate the residue with an exactly measured 10 mL of water, add 2 g of calcium hydroxide and an exactly measured 40 mL of water, stir the mixture for 20 minutes, and filter. To 30 mL of the filtrate add 0.1 g of magnesium sulfate heptahydrate, shake for 1 minute, then add 0.3 g of calcium hydroxide, shake for 1 minute, allow to stand for 1 hour, and filter. To an exactly measured 20 mL of the filtrate add 5 mL of sodium hydroxide TS, and adjust the pH to between 9.0 and 9.2 with ammonium chloride. Extract the solution successively with 60 mL, 40 mL and 30 mL of a mixture of chloroform and ethanol (95) (3:1). Combine all the extracts, distil, then evaporate off the solvent on a water bath. Dissolve the residue in 20 mL of dilute sodium hydroxide TS and 10 mL of diethyl ether with shaking, add 0.5 g of ammonium chloride, shake vigorously with caution, and proceed as directed in the Assay under Powdered Opium.

Each mL of 0.05 mol/L sulfuric acid VS = 28.534 mg of  $C_{17}H_{19}NO_3$ 

Containers and storage Containers—Tight containers.

## **Orange Oil**

Oleum Aurantii

オレンジ油

Orange Oil is the essential oil obtained by expression from the peel of the edible fruit of *Citrus* species (*Rutaceae*).

**Description** Orange Oil is a yellow to yellow-brown liquid. It has a characteristic, aromatic odor, and a slightly bitter taste

It is miscible with an equal volume of ethanol (95) with turbidity.

**Refractive index**  $n_D^{20}$ : 1.472 – 1.474

**Optical rotation**  $\alpha_D^{20}$ : +85 - +99° (100 mm).

**Specific gravity**  $d_{20}^{20}$ : 0.842 – 0.848

**Purity** Heavy metals—Proceed with 1.0 mL of Orange 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.

## **Orange Peel Syrup**

トウヒシロップ

## Method of preparation

Orange Peel Tincture	200 mL
Simple Syrup	a sufficient quantity

To make 1000 mL

Prepare as directed under Syrups, with the above ingredients. An appropriate quantity of Sucrose and Purified Water may be used in place of Simple Syrup.

**Description** Orange Peel Syrup is a brownish yellow to reddish brown liquid. It has a characteristic odor, a sweet taste and a bitter aftertaste.

Specific gravity  $d_{20}^{20}$ : about 1.25

Identification To 25 mL of Orange Peel Syrup add 50 mL of ethyl acetate, shake for 5 minutes, allow to stand until clear ethyl acetate layer separate, and take the ethyl acetate layer, and evaporate on a water bath to dryness. Dissolve the residue in 10 mL of ethanol (95), filter if necessary, and use this solution as the sample solution. Separately, dissolve 10 mg of naringin for thin-layer chromatography in 10 mL of ethanol (95), and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot  $10 \mu$ L 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, ethanol (99.5) and water (8:2:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute 2,6-dibromo-N-chloro-1,4-benzoquinone monoimine TS on