

**Assay** Weigh accurately about 1 g of Hydroxypropylmethylcellulose Phthalate, dissolve in 50 mL of a mixture of ethanol (95), acetone and water (2:2:1), and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} & \text{Amount (\% of carboxybenzoyl group (C}_8\text{H}_5\text{O}_3\text{))} \\ &= \frac{0.01 \times 149.1 \times V}{W} - \frac{2 \times 149.1 \times P}{166.1} \end{aligned}$$

*P*: amount (%) of phthalic acid obtained in the Purity (3)

*V*: amount (mL) of 0.1 mol/L sodium hydroxide VS consumed

*W*: amount (g) of the sample, calculated on the anhydrous basis

**Containers and storage** Containers—Tight containers.

## Ichthammol

イクタモール

Ichthammol, calculated on the dried basis, contains not less than 2.5% of ammonia (NH<sub>3</sub>: 17.030), not more than 8.0% of ammonium sulfate [(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>: 132.14], and not less than 10.0% of total sulfur (as S: 32.07).

**Description** Ichthammol is a red-brown to blackish brown, viscous fluid. It has a characteristic odor.

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

**Identification (1)** To 4 mL of a solution of Ichthammol (3 in 10) add 8 mL of hydrochloric acid: a yellow-brown to blackish brown, oily or resinous mass is produced. Cool the mass with ice to solidify, and discard the water layer. Wash the residue with diethyl ether: a part of the mass dissolves but it does not dissolve completely even when it is washed until almost no color develops in the washing. Perform the following tests with this residue.

(i) To 0.1 g of the residue add 1 mL of a mixture of diethyl ether and ethanol (95) (1:1): it dissolves.

(ii) To 0.1 g of the residue add 2 mL of water: it dissolves. To 1 mL of this solution add 0.4 mL of hydrochloric acid: a yellow-brown to blackish brown oily or resinous substance is produced.

(iii) To 1 mL of the solution obtained in (ii) add 0.3 g of sodium chloride: a yellow-brown or blackish brown oily or resinous substance is produced.

(2) Boil 2 mL of a solution of Ichthammol (1 in 10) with 2 mL of sodium hydroxide TS: the gas evolved changes moistened red litmus paper to blue.

**Loss on drying** Not more than 50% (0.5 g, 105°C, 6 hours).

**Residue on ignition** Not more than 0.5% (1 g).

**Assay (1)** Ammonia—Weigh accurately about 5 g of Ichthammol, transfer to a Kjeldahl flask, and add 60 mL of water, 1 mL of 1-octanol and 4.5 mL of a solution of sodium hydroxide (2 in 5). Connect the flask to a distilling tube with a spray trap and a condenser, and immerse the lower outlet

of the condenser in the receiver containing exactly 30 mL of 0.25 mol/L sulfuric acid VS. Distil slowly, collect about 50 mL of the distillate, and titrate the excess sulfuric acid with 0.5 mol/L sodium hydroxide VS (indicator: 3 drops of methyl red TS). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} & \text{Each mL of 0.25 mol/L sulfuric acid VS} \\ &= 8.515 \text{ mg of NH}_3 \end{aligned}$$

(2) Ammonium sulfate—Weigh accurately about 1 g of Ichthammol, add 25 mL of ethanol (95), stir thoroughly, and filter. Wash with a mixture of diethyl ether and ethanol (95) (1:1) until the washings are clear and colorless. Dry the filter paper and the residue in air, dissolve the residue in 200 mL of hot water acidified slightly with hydrochloric acid, and filter. Boil the filtrate, add 30 mL of barium chloride TS slowly, heat for 30 minutes on a water bath, and filter. Wash the precipitate with water, dry, and ignite to constant mass. Weigh the residue as barium sulfate (BaSO<sub>4</sub>: 233.39).

$$\begin{aligned} & \text{Amount (mg) of ammonium sulfate [(NH}_4\text{)}_2\text{SO}_4\text{]} \\ &= \text{amount (mg) of barium sulfate (BaSO}_4\text{)} \times 0.5662 \end{aligned}$$

(3) Total sulfur—Weigh accurately about 0.6 g of Ichthammol, transfer to a 200-mL Kjeldahl flask, and add 30 mL of water and 5 g of potassium chlorate, then add slowly 30 mL of nitric acid, and evaporate the mixture to about 5 mL. Transfer the residue to a 300-mL beaker with the aid of 25 mL of hydrochloric acid, and evaporate again to 5 mL. Add 100 mL of water, boil, filter, and wash with water. Heat the combined filtrate and washings to boil, add gradually 30 mL of barium chloride TS, heat the mixture on a water bath for 30 minutes, and filter. Wash the precipitate with water, dry, and ignite to constant mass. Weigh the residue as barium sulfate (BaSO<sub>4</sub>).

$$\begin{aligned} & \text{Amount (mg) of total sulfur (S)} \\ &= \text{amount (mg) of barium sulfate (BaSO}_4\text{)} \times 0.13739 \end{aligned}$$

**Containers and storage** Containers—Tight containers.

## Immature Orange

*Aurantii Fructus Immaturus*

キシッ

Immature Orange is the immature fruit or the fruit cut crosswise of *Citrus aurantium* Linné var. *daidai* Makino, *Citrus aurantium* Linné or *Citrus natsuda-daidai* Hayata (*Rutaceae*).

**Description** Nearly spherical fruit, 1–2 cm in diameter, or semispherical, 1.5–4.5 cm in diameter; external surface, deep green-brown to brown, and without luster, with numerous small dents associated with oil sacs; the outer portion of transverse section exhibits pericarp and mesocarp about 0.4 cm in thickness, yellow-brown in color in the region contacting epidermis, and light grayish brown color in the other parts; the central portion is radially divided into 8 to 16 small loculi; each loculus is brown and indented, often containing immature seeds. Odor, characteristic; taste, bitter.

**Identification** To 0.5 g of pulverized Immature Orange add

10 mL of methanol, boil gently for 2 minutes, and filter. To 5 mL of the filtrate add 0.1 g of magnesium in ribbon form and 1 mL of hydrochloric acid, and allow to stand: a red-purple color develops.

**Total ash** Not more than 7.0%.

## Human Normal Immunoglobulin

人免疫グロブリン

Human Normal Immunoglobulin is a liquid for injection containing immunoglobulin G in serum globulins of humans.

It conforms to the requirements of Human Normal Immunoglobulin in the Minimum Requirements for Biological Products.

**Description** Human Normal Immunoglobulin is a clear, colorless or yellow-brown liquid.

## Imperata Rhizome

*Imperatae Rhizoma*

ボウコン

Imperata Rhizome is the rhizome of *Imperata cylindrica* Beauvois (*Gramineae*), from which rootlets and scale leaves have been removed.

**Description** Long and thin cylindrical rhizome, 0.3–0.5 cm in diameter; sometimes branched; externally yellowish white, with slight longitudinal wrinkles, and with nodes at 2–3-cm intervals; difficult to break; fractured surface fibrous. Cross section irregularly round; thickness of cortex is slightly smaller than the diameter of the stele; pith often forms a hollow. Under a magnifying glass, a transverse section reveals cortex, yellowish white, and with scattered brown spots; stele, yellow-brown in color. Odorless, and tasteless at first, but later slightly sweet.

**Identification** To 1 g of pulverized Imperata Rhizome add 20 mL of hexane, allow the mixture to stand for 30 minutes with occasional shaking, and filter. Evaporate the filtrate to dryness, dissolve the residue in 5 mL of chloroform, place 0.5 mL of this solution in a test tube, and, after mixing with 0.5 mL of acetic anhydride by shaking, add carefully 0.5 mL of sulfuric acid to make two layers: a red-brown color develops at the zone of contact, and the upper layer acquires a blue-green to blue-purple color.

**Purity** (1) Rootlet and scaly leaf—The amount of the rootlets and scaly leaves contained in Imperata Rhizome does not exceed 3.0%.

(2) Foreign matter—The amount of foreign matter other than rootlets and scaly leaves contained in Imperata Rhizome does not exceed 1.0%.

**Total ash** Not more than 5.0%.

**Acid-insoluble ash** Not more than 1.5%.

## Influenza HA Vaccine

インフルエンザ HA ワクチン

Influenza HA Vaccine is a liquid for injection containing hemagglutinin of influenza virus.

It conforms to the requirements of Influenza HA Vaccine in the Minimum Requirements for Biological Products.

**Description** Influenza HA Vaccine is a clear liquid or a slightly whitish turbid liquid.

## Iodine Tincture

ヨードチンキ

Iodine Tincture contains not less than 5.7 w/v% and not more than 6.3 w/v% of iodine (I: 126.90), and not less than 3.8 w/v% and not more than 4.2 w/v% of potassium iodide (KI: 166.00).

### Method of preparation

Iodine	60 g
Potassium Iodide	40 g
70 vol% Ethanol	a sufficient quantity

To make 1000 mL

Prepare as directed under Tinctures, with the above ingredients. It may be prepared with an appropriate quantity of Ethanol or Ethanol for Disinfectant and Purified Water in place of 70 vol% Ethanol.

**Description** Iodine Tincture is a dark red-brown liquid, and has a characteristic odor.

Specific gravity  $d_{20}^{20}$ : about 0.97

**Identification** (1) To a mixture of 1 mL of starch TS and 9 mL of water add 1 drop of Iodine Tincture: a dark blue-purple color develops.

(2) Evaporate 3 mL of Iodine Tincture to dryness on a water bath, and heat gently over a free flame: a white residue is formed which responds to the Qualitative Tests for potassium salt and iodide.

**Alcohol number** Not less than 6.6 (Method 2). Perform the pretreatment (ii) in the Method 1.

**Assay** (1) Iodine—Pipet 5 mL of Iodine Tincture, add 0.5 g of potassium iodide, 20 mL of water and 1 mL of dilute hydrochloric acid, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: 2 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS  
= 12.690 mg of I

(2) Potassium iodide—Pipet 5 mL of Iodine Tincture into an iodine flask, add 20 mL of water, 50 mL of hydrochloric acid and 5 mL of chloroform. Cool to room temperature, and titrate with 0.05 mol/L potassium iodate VS until the red-purple color disappears from the chloroform layer, with agitating the mixture vigorously and con-