

tinuously. After the chloroform layer has been decolorized, allow the mixture to stand for 5 minutes. If the color reappears, the mixture should be titrated further with 0.05 mol/L potassium iodate VS. Calculate the amount (mg) of potassium iodide from the number of mL (*a*) of 0.05 mol/L potassium iodate VS used as above and the number of mL (*b*) of 0.1 mol/L sodium thiosulfate VS used in the titration under the Assay (1).

Amount (mg) of potassium iodide (KI)

$$= 16.600 \times \left( a - \frac{b}{2} \right)$$

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

## Dilute Iodine Tincture

希ヨードチンキ

Dilute Iodine Tincture contains not less than 2.8 w/v% and not more than 3.2 w/v% of iodine (I: 126.90), and not less than 1.9 w/v% and not more than 2.1 w/v% of potassium iodide (KI: 166.00).

### Method of preparation

Iodine	30 g
Potassium Iodide	20 g
70 vol% Ethanol	a sufficient quantity
To make 1000 mL	

Prepare as directed under Medicated Spirits, with the above ingredients. It may be prepared with an appropriate quantity of Ethanol or Ethanol for Disinfection and Purified Water in place of 70 vol% Ethanol. It may also be prepared by adding 70 vol% Ethanol to 500 mL of Iodine Tincture to make 1000 mL.

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

Specific gravity  $d_{20}^{20}$ : about 0.93

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

(2) Evaporate 3 mL of Diluted 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 6.7 (Method 2). Perform the pretreatment (ii) in the Method 1.

**Assay (1)** Iodine—Pipet exactly 10 mL of Dilute 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 exactly 10 mL of Dilute 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 io-

date VS until the red-purple color in the chloroform layer disappears while agitating vigorously and continuously. After the chloroform layer has been decolorized, allow the mixture to stand for 5 minutes. If the color reappears, the mixture should be titrated further with 0.05 mol/L potassium iodate VS. Calculate the amount (mg) of potassium iodide from the volume (*a* mL) of 0.05 mol/L potassium iodate VS consumed as above and the volume (*b* mL) of 0.1 mol/L sodium thiosulfate VS consumed in the titration under Assay (1).

Amount (mg) of potassium iodide (KI)

$$= 16.600 \times \left( a - \frac{b}{2} \right)$$

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

## Compound Iodine Glycerin

複方ヨード・グリセリン

Compound Iodine Glycerin contains not less than 1.1 w/v% and not more than 1.3 w/v% of iodine (I: 126.90), not less than 2.2 w/v% and not more than 2.6 w/v% of potassium iodide (KI: 166.00), not less than 2.7 w/v% and not more than 3.3 w/v% of total iodine (as I), and not less than 0.43 w/v% and not more than 0.53 w/v% of phenol (C<sub>6</sub>H<sub>6</sub>O: 94.11).

### Method of preparation

Iodine	12 g
Potassium Iodide	24 g
Glycerin	900 mL
Mentha Water	45 mL
Liquefied Phenol	5 mL
Purified Water	a sufficient quantity
To make 1000 mL	

Dissolve Potassium Iodide and Iodine in about 25 mL of Purified Water. After adding Glycerin, add Mentha Water, Liquefied Phenol and sufficient Purified Water to make 1000 mL, mixing thoroughly. It may be prepared with an appropriate quantity of Concentrated Glycerin and Purified Water in place of Glycerin.

**Description** Compound Iodine Glycerin is a red-brown, viscous liquid. It has a characteristic odor.

Specific gravity  $d_{20}^{20}$ : about 1.23

**Identification (1)** The colored solution obtained in the Assay (1) acquires a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 510 nm and 514 nm (iodine).

(2) The colored solution obtained in the Assay (2) acquires a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 510 nm and 514 nm (potassium iodide).

(3) The colored solution obtained in the Assay (4) has a yellow color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 401 nm and 405 nm

(phenol).

(4) Take 1 mL of Compound Iodine Glycerin in a glass-stoppered test tube, add 10 mL of ethanol (95), and mix. Then add 2 mL of sodium hydroxide TS, add 1 mL of a solution of copper (II) chloride dihydrate in ethanol (95) (1 in 10), and shake: a blue color develops (glycerin).

**Assay (1) Iodine**—Measure the specific gravity of Compound Iodine Glycerin according to Method 2. Weigh exactly about 7 mL of it, add ethanol (95) to make exactly 200 mL, and use this solution as the sample solution. On the other hand, weigh accurately about 0.08 g of iodine for assay and about 0.17 g of potassium iodide for assay, previously dried at 105°C for 4 hours, dissolve in ethanol (95) to make exactly 200 mL, and use this solution as the standard solution. Pipet 3 mL each of the sample solution and the standard solution into 50-mL separators, to each add exactly 10 mL of a mixture of chloroform and hexane (2:1) and 15 mL of water successively, and shake immediately and vigorously. Separate the chloroform-hexane layers [use the water layers in (2)], and filter through a pledget of cotton. Determine the absorbances of the filtrates,  $A_T$  and  $A_S$ , at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2:1) as the blank.

$$\begin{aligned} & \text{Amount (mg) of iodine (I)} \\ &= \text{amount (mg) of iodine for assay} \\ & \times \frac{A_T}{A_S} \end{aligned}$$

(2) **Potassium iodide**—Separate the water layers of the sample solution and the standard solution obtained in (1), pipet 10 mL of each of the water layers, and to each add 1 mL of diluted dilute hydrochloric acid (1 in 2), 1 mL of sodium nitrite TS and exactly 10 mL of a mixture of chloroform and hexane (2:1). Shake immediately and vigorously, separate the chloroform-hexane layers, and filter through a pledget of cotton. Determine the absorbances,  $A_T$  and  $A_S$ , of both solutions at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2:1) as the blank.

$$\begin{aligned} & \text{Amount (mg) of potassium iodide (KI)} \\ &= \text{amount (mg) of potassium iodide for assay} \\ & \times \frac{A_T}{A_S} \end{aligned}$$

(3) **Total iodine**—Measure the specific gravity of Compound Iodine Glycerin according to Method 2. Weigh exactly about 5 mL of it, and add water to make exactly 50 mL. Pipet 5 mL of this solution into a 50-mL flask, and add 0.5 g of zinc powder and 5 mL of acetic acid (100). Shake until the color of iodine disappears, and heat under a reflux condenser on a water bath for 30 minutes. Wash the condenser with 10 mL of hot water, and filter through a glass filter (G3). Wash the flask with two 10-mL portions of warm water, and combine the filtrate and the washings. After cooling, add water to make exactly 50 mL, and use this solution as the sample solution. On the other hand, dissolve about 0.2 g of potassium iodide for assay, previously dried at 105°C for 4 hours and accurately weighed, in water to make exactly 50 mL. Pipet 5 mL of this solution, add 5 mL of acetic acid (100) and water to make exactly 50 mL, and use this solution as the standard solution. Pipet 4 mL each of the sample solution and the standard solution into 30-mL separators, and to each add 5 mL of water, 1 mL of diluted dilute hydrochloric acid (1 in 2), 1

mL of sodium nitrite TS and 10 mL of a mixture of chloroform and hexane (2:1). Shake well immediately, and proceed as directed in (2).

$$\begin{aligned} & \text{Amount (mg) of total iodine (I)} \\ &= \text{amount (mg) of potassium iodide for assay} \\ & \times \frac{A_T}{A_S} \times 0.7645 \end{aligned}$$

(4) **Phenol**—Measure the specific gravity of Compound Iodine Glycerin according to Method 2. Weigh exactly about 2 mL of it, add 3 mL of 0.1 mol/L sodium thiosulfate VS, and shake. Add 2 mL of dilute hydrochloric acid, and shake with two 10-mL portions of chloroform. Separate the chloroform layer, and shake with two 10-mL portions of 0.5 mol/L sodium hydroxide TS. Separate the water layer, add water to make exactly 500 mL, and use this solution as the sample solution. Dissolve about 0.5 g of phenol for assay, accurately weighed, in ethanol (95) to make exactly 100 mL, pipet 2 mL of this solution, proceed in the same manner as the sample solution, and use so obtained solution as the standard solution. Pipet 3 mL each of the sample solution and the standard solution, to each add 2 mL of dilute hydrochloric acid, and place in a water bath at 30°C. Allow to stand for 10 minutes, and add exactly 2 mL of a solution of sodium nitrite (1 in 100), shake, and allow to stand at 30°C for 60 minutes. Add dilute potassium hydroxide-ethanol TS to make exactly 25 mL, and determine the absorbances of these solutions,  $A_T$  and  $A_S$ , at 403 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution prepared in the same manner with 3 mL of water instead of the sample solution as the blank.

$$\begin{aligned} & \text{Amount (mg) of phenol (C}_6\text{H}_6\text{O)} \\ &= \text{amount (mg) of phenol for assay} \\ & \times \frac{A_T}{A_S} \times \frac{1}{50} \end{aligned}$$

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

## Dental Iodine Glycerin

歯科用ヨード・グリセリン

Dental Iodine Glycerin contains not less than 9.0 w/v% and not more than 11.0 w/v% of iodine (I: 126.90), not less than 7.2 w/v% and not more than 8.8 w/v% of potassium iodide (KI: 166.00) and not less than 0.9 w/v% and not more than 1.1 w/v% of zinc sulfate (ZnSO<sub>4</sub>·7H<sub>2</sub>O: 287.56).

### Method of preparation

Iodine	10 g
Potassium Iodide	8 g
Zinc Sulfate	1 g
Glycerin	35 mL
Purified Water	a sufficient quantity

To make 100 mL

Dissolve and mix the above ingredients.

**Description** Dental Iodine Glycerin is a dark red-brown liq-