

(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.

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

(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.

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

(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).

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

(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.

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

**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-

uid, having the odor of iodine.

**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) Put 1 mL of Dental 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).

(4) The colored solution obtained in the Assay (3) acquires a red-purple to purple color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 618 nm and 622 nm (zinc sulfate).

**Assay (1)** Iodine—Pipet 5 mL of Dental Iodine Glycerin, and add diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 200 mL, and use this solution as the sample solution. On the other hand, weigh accurately about 0.5 g of iodine for assay and about 0.4 g of potassium iodide for assay, previously dried at 105°C for 4 hours, and dissolve in diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 200 mL, and use this solution as the standard solution. Pipet 10 mL each of the sample solution and the standard solution, to each add exactly 20 mL of a mixture of chloroform and hexane (2:1), shake immediately, and separate the chloroform-hexane layer [use the water layer in (2)]. Filter through a pledget of cotton. Determine the absorbances,  $A_T$  and  $A_S$ , of the filtrates obtained from the sample solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2:1) as the blank.

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

(2) Potassium iodide—Separate the water layers of the sample solution and the standard solution obtained in (1), pipet 7 mL each of the water layers, and to each add exactly 1 mL of diluted hydrochloric acid (1 in 2), 1 mL of sodium nitrite TS and 10 mL of a mixture of chloroform and hexane (2:1), and shake immediately. Separate the chloroform-hexane layer, and filter through a pledget of cotton. Determine the absorbances,  $A_T$  and  $A_S$ , of the filtrates obtained from the sample solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2:1) as the blank.

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

(3) Zinc sulfate—Pipet 5 mL of Dental Iodine Glycerin, and add diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the sample solution. On the other hand, pipet 10 mL of Standard Zinc Stock Solution, add diluted ethanol (3 in 200) to make exactly 1000 mL, and use this solution as the standard solution. Pipet 10 mL each of the sample solution and the standard solution, to each add 10 mL of a mixture of chloroform and hexane (2:1), shake, and allow to stand. Pipet 3 mL each of the water layers, and to each add 2 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 10.0, 2 mL of zincon TS and water to make exactly 25 mL. Determine the absorbances,  $A_T$  and  $A_S$ , obtained from the sample solution and the standard solution, respectively, at 620 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution prepared in the same manner with 3 mL of water as the blank.

$$\begin{aligned} &\text{Amount (mg) of zinc sulfate (ZnSO}_4\cdot 7\text{H}_2\text{O)} \\ &= \text{amount (mg) of zinc in 10 mL of Standard} \\ &\quad \text{Zinc Stock Solution} \\ &\quad \times \frac{A_T}{A_S} \times 4.398 \end{aligned}$$

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

Storage—Light-resistant.

## Iodine, Salicylic Acid and Phenol Spirit

ヨード・サリチル酸・フェノール精

Iodine, Salicylic Acid and Phenol Spirit contains not less than 1.08 w/v% and not more than 1.32 w/v% of iodine (I: 126.90), not less than 0.72 w/v% and not more than 0.88 w/v% of potassium iodide (KI: 166.00), not less than 4.5 w/v% and not more than 5.5 w/v% of salicylic acid ( $\text{C}_7\text{H}_6\text{O}_3$ : 138.12), not less than 1.8 w/v% and not more than 2.2 w/v% of phenol ( $\text{C}_6\text{H}_6\text{O}$ : 94.11), and not less than 7.2 w/v% and not more than 8.8 w/v% of benzoic acid ( $\text{C}_7\text{H}_6\text{O}_2$ : 122.12).

### Method of preparation

Iodine Tincture	200 mL
Salicylic Acid	50 g
Phenol	20 g
Benzoid Acid	80 g
Ethanol for Disinfection	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 and Purified Water in place of Ethanol for Disinfection.

**Description** Iodine, Salicylic Acid and Phenol Spirit is a dark red-brown liquid, having the odor of phenol.

**Identification (1)** To a mixture of 1 mL of starch TS and 9 mL of water add 1 drop of Iodine, Salicylic Acid and