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.

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

(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.

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

(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.

$$\text{Amount (mg) of zinc sulfate } (ZnSO_4.7H_2O) = \text{amount (mg) of zinc in 10 mL of Standard Zinc Stock Solution} \times \frac{A_T}{A_S} \times 4.398$$

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 ($C_7H_6O_3$: 138.12), not less than 1.8 w/v% and not more than 2.2 w/v% of phenol (C_6H_6O: 94.11), and not less than 7.2 w/v% and not more than 8.8 w/v% of benzoic acid ($C_7H_6O_2$: 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 appropiate 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

Phenol Spirit: a dark blue-purple color develops (iodine).

(2) To 1 mL of Iodine, Salicylic Acid and Phenol Spirit add 5 mL of ethanol (95) and water to make 50 mL. To 1 mL of this solution add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make 50 mL, and to 15 mL of this solution add 5 mL of a solution of iron (III) nitrate enneahydrate (1 in 200): a red-purple color is produced (salicylic acid).

(3) Shake 1 mL of Iodine, Salicylic Acid and Phenol Spirit with 1 mL of sodium thiosulfate TS, add 20 mL of water and 5 mL of dilute hydrochloric acid, and extract with 25 mL of diethyl ether. Wash the diethyl ether extract with two 25-mL portions of sodium hydrogen carbonate TS, and extract with 10 mL of dilute sodium hydroxide TS. Shake 1 mL of the extract with 1 mL of sodium nitrite TS and 1 mL of dilute hydrochloric acid, and add 3 mL of sodium hydroxide TS: a yellow color is developed (phenol).

(4) Shake 1 mL of Iodine, Salicylic Acid and Phenol Spirit with 1 mL of sodium thiosulfate TS, add 20 mL of water and 5 mL of dilute hydrochloric acid, extract with 10 mL of diethyl ether, and use the diethyl ether extract as the sample solution. Dissolve 0.025 g of salicylic acid, 0.01 g of phenol and 0.04 g of benzoic acid in 5 mL each of diethyl ether, respectively, and use these solutions as the standard solutions (1), (2) and (3). Perform the test with the sample solution and the standard solutions as directed under the Thin-layer Chromatography. Spot 5 μL of each solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, acetone and acetic acid (100) (45:5:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the 3 spots from the sample solution show the same Rf value as the corresponding spots of the standard solutions (1), (2) and (3). Spray evenly iron (III) chloride TS on the plate: the spot from standard solution (1) and the corresponding spot from the sample solution acquires a purple color.

Assay (1) Iodine—Pipet 4 mL of Iodine, Salicylic Acid and Phenol Spirit, add ethanol (95) to make exactly 50 ml, and use this solution as the sample solution. On the other hand, weigh accurately about 1.2 g of iodine for assay and about 0.8 g of potassium iodide for assay, previously dried at 105°C for 4 hours, and dissolve in ethanol (95) to make exactly 100 ml. Pipet 4 ml of this solution, add ethanol (95) to make exactly 50 ml, and use this solution as the standard solution. Pipet 3 ml each of the sample solution and the standard solution, to each add exactly 25 ml of a mixture of chloroform and hexane (2:1), and shake. Further add exactly 10 ml of water, shake and separate the chloroform-hexane layers [use the water layers in (2)]. Filter through a pledget of absorbent cotton, and determine the absorbances of the filtrates from the sample solution and the standard solution, respectively, 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.

$$\text{Amount (mg) of iodine (I)} = \text{amount (mg) of iodine for assay} \times \frac{A_T}{A_S} \times \frac{1}{25}$$

(2) Potassium iodide—Separate the water layers of the sample solution and the standard solution obtained in the Assay (1), pipet 8 ml each of the water layers, and add 1 ml of diluted dilute hydrochloric acid (1 in 2) and 1 ml of sodium nitrite TS. Immediately after shaking, add exactly 10 ml of a mixture of chloroform and hexane (2:1), shake, and proceed in the same manner as for the Assay (1).

$$\text{Amount (mg) of potassium iodide (KI)} = \text{amount (mg) of potassium iodide for assay} \times \frac{A_T}{A_S} \times \frac{1}{25}$$

(3) Salicylic acid, phenol and benzoic acid—Pipet 2 mL of Iodine, Salicylic Acid and Phenol Spirit, add 20 mL of diluted methanol (1 in 2) and 0.1 mol/L soium thiosulfate VS until the color of iodine disappears, add exactly 20 mL of the internal standard solution, then add diluted methanol (1 in 2) to make 200 mL, and use this solution as the sample solution. Weigh accurately about 0.2 g of salicylic acid for assay, previously dried in a desiccator (silica gel) for 3 hours, about 0.08 g of phenol for assay, and 0.32 g of benzoic acid, previously dried in a desiccator (silica gel) for 3 hours, dissolve in diluted methanol (1 in 2) to make exactly 50 mL. Pipet 25 mL of this solution, add exactly 20 mL of the internal standard solution and diluted methanol (1 in 2) to make 200 mL, and use this solution as the standard solution. Perform the test with 3 μL of the sample solution and the standard solution as directed under the Liquid Chromatography according to the operating conditions in the Assay under Compound Salicylic Acid Spirit. Calculate the ratios, Q_{Ta}, Q_{Tb} and Q_{Tc}, of the peak areas of salicylic acid, phenol and benzoic acid to those of the internal standard of the sample solution, and the ratios, Q_{Sa}, Q_{Sb} and Q_{Sc}, of the peak areas of salicylic acid, phenol and benzoic acid to those of the internal standard of the standard solution.

$$\text{Amount (mg) of salicylic acid } (C_7H_6O_3) = \text{amount (mg) of salicylic acid for assay} \times \frac{Q_{Ta}}{Q_{Sa}} \times \frac{1}{2}$$

$$\text{Amount (mg) of phenol } (C_6H_6O) = \text{amount (mg) of phenol for assay} \times \frac{Q_{Tb}}{Q_{Sb}} \times \frac{1}{2}$$

$$\text{Amount (mg) of benzoic acid } (C_7H_6O_2) = \text{amount (mg) of benzoic acid} \times \frac{Q_{Tc}}{Q_{Sc}} \times \frac{1}{2}$$

Internal standard solution—A solution of theophylline in methanol (1 in 1000).

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

Ipecac

Ipecacuanhae Radix

トコン

Ipecac is the root and rhizome of *Cephaelis ipecacuanha* (Broterol) A. Richard or *Cephaelis acuminata* Karsten (*Rubiaceae*).

It contains not less than 2.0% of the total alkaloids