

is whitish in color and adheres well to the skin.

**Containers and storage** Containers—Well-closed containers.

Storage—Light-resistant.

## Salicylic Acid Spirit

サリチル酸精

Salicylic Acid Spirit contains not less than 2.7 w/v% and not more than 3.3 w/v% of salicylic acid ( $C_7H_6O_3$ : 138.12).

### Method of preparation

Salicylic Acid	30 g
Glycerin	50 mL
Ethanol	a sufficient quantity
To make 1000 mL	

Prepare as directed under Medicated Spirits, with the above ingredients.

**Description** Salicylic Acid Spirit is a clear, colorless liquid.  
Specific gravity  $d_{20}^{20}$ : about 0.86

**Identification** The solution obtained in the Assay has a red-purple color. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 520 nm and 535 nm (salicylic acid).

**Alcohol number** Not less than 8.8 (Method 2).

**Assay** Measure exactly 10 mL of Salicylic Acid Spirit, add 10 mL of ethanol (95) and water to make exactly 100 mL. Pipet 3 mL of this solution, and dilute with hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 100 mL. Use this solution as the sample solution. Dissolve about 0.3 g of salicylic acid for assay, previously dried in a desiccator (silica gel) for 3 hours and accurately weighed, in 10 mL of alcohol and water to make exactly 100 mL. Pipet 3 mL of this solution, dilute with hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 100 mL, and use this solution as the standard solution. Pipet 10 mL each of the sample solution and the standard solution, to each add 5 mL of a solution of iron (III) nitrate enneahydrate (1 in 200), dilute with hydrochloric acid-potassium chloride buffer solution, pH 2.0, to exactly 25 mL. Determine the absorbances,  $A_T$  and  $A_S$ , of both solutions at 530 nm, using a blank solution prepared in the same manner with water instead of the sample solution.

$$\begin{aligned} & \text{Amount (mg) of salicylic acid (C}_7\text{H}_6\text{O}_3\text{)} \\ & = \text{amount (mg) of salicylic acid for assay} \\ & \quad \times \frac{A_T}{A_S} \end{aligned}$$

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

## Compound Salicylic Acid Spirit

複方サリチル酸精

Compound Salicylic Acid Spirit contains not less than 1.8 w/v% and not more than 2.2 w/v% of salicylic acid ( $C_7H_6O_3$ : 138.12), and not less than 0.43 w/v% and not more than 0.53 w/v% of phenol ( $C_6H_6O$ : 94.11).

### Method of preparation

Salicylic Acid	20 g
Liquefied Phenol	5 mL
Glycerin	40 mL
Ethanol	800 mL
Water or Purified Water	a sufficient quantity
To make 1000 mL	

Prepare as directed under Medicated Spirits, with the above ingredients.

**Description** Compound Salicylic Acid Spirit is a clear, colorless to light red liquid.

Specific gravity  $d_{20}^{20}$ : about 0.88

**Identification (1)** To 1 mL of Compound Salicylic Acid Spirit add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make 200 mL, and to 5 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).

(2) To 1 mL of Compound Salicylic Acid Spirit add 20 mL of water and 5 mL of dilute hydrochloric acid, and extract with 20 mL of diethyl ether. Wash the diethyl ether extract with two 5-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, allow to stand for 10 minutes, and add 3 mL of sodium hydroxide TS: a yellow color is produced (phenol).

(3) To 0.5 mL of Compound Salicylic Acid Spirit add 5 mL of dilute hydrochloric acid, extract with 5 mL of chloroform, and use the extract as the sample solution (1). To 2 mL of Compound Salicylic Acid Spirit add 5 mL of dilute hydrochloric acid, extract with 5 mL of chloroform, wash the extract with two 5-mL portions of sodium hydrogen carbonate TS, and use the chloroform extract as the sample solution (2). Separately, dissolve 0.01 g each of salicylic acid and phenol in 5 mL each of chloroform, and use both solutions as the standard solution (1) and the standard solution (2). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L each of the sample solutions and the standard solutions 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 spots from the sample solution (1) and the standard solution (1) show the same  $R_f$  value, and the spots from the sample solution (2) and the standard solution (2) show the same  $R_f$  value. Spray evenly iron (III) chloride TS upon the plate: the spot from the standard solution (1) and the corresponding spot from the sample solution (1)