narrowed gradually. Odor, strong and characteristic; taste, bitter; colors the saliva yellow on chewing.

Under a microscope, when softened by immersion in water, the upper end has numerous tubular protrusions about 150 $\mu$m in length, with a small number of pollen grains.

**Identification** Add 1 drop of sulfuric acid to Saffron: the color changes to dark blue which gradually turns red-brown through purple.

**Purity** (1) Aniline dyes—Shake 0.05 g of Saffron with 10 mL of chloroform: the solution is colorless, or only slightly yellow.

(2) Glycerol, sugar or honey—Saffron has no sweet taste. Press it between two pieces of paper: no spot is left on the paper.

(3) Yellow style—The yellow style in Saffron does not exceed 10.0%.

**Loss on drying** Not more than 12.0% (6 hours).

**Total ash** Not more than 7.5%.

**Content of the active principle** Crocin—Dry Saffron in a desiccator (silica gel) for 24 hours, and powder. To exactly 0.100 g of the powder add 150 mL of warm water, warm the mixture between 60°C and 70°C for 30 minutes with frequent shaking, cool, and filter. Pipet 1 mL of the filtrate, add water to make exactly 10 mL, and use this solution as the sample solution. Separately, dissolve exactly 0.098 g of carbazochrome sodium sulfonate for content of the active principle in water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances of the sample solution and the standard solution at 438 nm as directed under the Ultraviolet-visible Spectrophotometry: the absorbance of the sample solution is larger than that of the standard solution.

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

Storage—Light-resistant.

# Salicylated Alum Powder

サリチル・ミョウバン散

Salicylated Alum Powder contains not less than 2.7% and not more than 3.3% of salicylic acid ($C_7H_6O_3$: 138.12).

**Method of preparation**

| | |
|---|---|
| Salicylic Acid, finely powdered | 30 g |
| Dried Aluminum Potassium Sulfate, very finely powdered | 640 g |
| Talc, very finely powdered | a sufficient quantity |
| | To make 1000 g |

Prepare as directed under Powders, with the above ingredients.

**Description** Salicylated Alum Powder occurs as a white powder.

**Identification** (1) The colored solution obtained in the Assay has a red-purple color and exhibits absorbance maximum between 520 nm and 535 nm (salicylic acid).

(2) Shake 0.3 g of Salicylated Alum Powder with 5 mL of methanol, filter, and use the filtrate as the sample solution. Separately, dissolve 0.01 g of salicylic acid in 5 mL of methanol, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 $\mu$L each of the sample solution and the standard solution on the 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 the plate under ultraviolet light (main wavelength: 254 nm): the spot from the sample solution and that from the standard solution show the same $Rf$ value. Spray evenly iron (III) chloride TS upon the plate: the spot from the standard solution and the corresponding spot from the sample solution reveal a purple color.

**Assay** Weigh accurately about 0.33 g of Salicylated Alum Powder, add 80 mL of ethanol (95), and shake vigorously. Dilute with ethanol (95) to make exactly 100 mL, filter, and discard the first 10 mL of the filtrate. Use the subsequent filtrate as the sample solution. Dissolve about 0.1 g of salicylic acid for assay, previously dried in a desiccator (silica gel) for 3 hours and accurately weighed, in sufficient ethanol (95) to make exactly 100 mL. Pipet 10 mL of this solution, dilute with ethanol (95) to make exactly 100 mL, and use the solution as the standard solution. Pipet 10 mL each of the sample solution and standard solution into stoppered test tubes respectively, to each add exactly 5 mL of a solution of iron (III) nitrate enneahydrate (1 in 200), and dilute with hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 25 mL. Determine the absorbance, $A_T$ and $A_S$, of both solutions at 530 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared in the same manner with ethanol (95), instead of the sample solution, as the blank.

$$\text{Amount (mg) of salicylic acid } (C_7H_6O_3) = \text{amount (mg) of salicylic acid for assay} \times \frac{A_T}{A_S} \times \frac{1}{10}$$

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

# Salicylic Acid Adhesive Plaster

サリチル酸絆創膏

**Method of preparation**

Adhesive Plaster consists of a mixture of the below ingredients with carefully selected rubber, resins, zinc oxide and other substances. It has adhesive properties. It spreads evenly on a fabric.

| | |
|---|---|
| Salicylic Acid, finely powdered | 500 g |
| Adhesive plaster base | a sufficient quantity |
| | To make 1000 g |

**Description** The surface of Salicylic Acid Adhesive Plaster

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.

$$\text{Amount (mg) of salicylic acid } (C_7H_6O_3) = \text{amount (mg) of salicylic acid for assay} \times \frac{A_T}{A_S}$$

**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 chrloroform, 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 μL 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 *Rf* value, and the spots from the sample solution (2) and the standard solution (2) show the same *Rf* 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)