lute acetic acid and water to make 50 mL, and use this solution as the control solution (not more than 20 ppm).

(4) Arsenic—Prepare the test solution with 1.0 g of Saccharin Sodium, according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

(5) Benzoate and salicylate—Dissolve 0.5 g of Saccharin Sodium in 10 mL of water, add 5 drops of acetic acid (31) and 3 drops of iron (III) chloride TS: no turbidity is produced, and no red-purple to purple color develops.

(6) Orthotoluene sulfonamide—Dissolve 10 g of Saccharin Sodium in 50 mL of water, and extract with three 30-mL portions of ethyl acetate. Combine all the ethyl acetate extracts, wash with 30 mL of a solution of sodium chloride (1 in 4), dehydrate with 5 g of anhydrous sodium sulfate, and evaporate ethyl acetate. Dissolve the residue in exactly 5 mL of the internal standard solution, and use this solution as the sample solution. Separately, dissolve 0.10 g of orthotoluene sulfonamide in ethyl acetate to make exactly 100 mL. Pipet 1 mL of this solution, evaporate on a water bath to dryness, dissolve the residue in exactly 5 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 1 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak height of orthotoluene sulfonamide to that of the internal standard: Q_T is not more than Q_S.

Internal standard solution—A solution of caffeine in ethyl acetate (1 in 500).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A column about 3 mm in inside diameter and about 1 m in length, packed with siliceous earth for gas chromatography (180 to 250 μm in diameter), coated with diethyleneglycol polyester succinate at the ratio of 3%.

Column temperature: Constant temperature between 195°C and 205°C.

Carrier gas: Nitrogen.

Flow rate: Adjust the flow rate so that the retention time of caffeine is about 6 minutes.

Selection of column: Proceed with 1 μL of the standard solution under the above operating conditions. Use a column giving elution of the internal standard and orthotoluene sulfonamide in this order with the resolution between these peaks being not less than 2.0.

(7) Readily carbonizable substances—Perform the test with 0.20 g of Saccharin Sodium. Allow the solution to stand between 48°C and 50°C for 10 minutes: the solution has no more color than Matching Fluid A.

Loss on drying Not more than 15.0% (1 g, 120°C, 4 hours).

Assay Weigh accurately about 0.5 g of Saccharin Sodium, previously dried, and dissolve in 20 mL of water in a separator. Add 2 mL of dilute hydrochloric acid, extract the produced precipitate with one 50-mL portion and four 20-mL portions of a mixture of chloroform and ethanol (99.5) (9:1), and filter each extract through absorbent cotton moistened with the mixture at each extraction. Wash the extremity of the separator and the absorbent cotton with the mixture, and combine the washings with the extract. Evaporate the solution on a water bath to dryness, dissolve the residue in 75 mL of hot water, cool, and titrate the solution with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS).

Each mL of 0.1 mol/L sodium hydroxide VS
= 20.517 mg of $C_7H_4NNaO_3S$

Containers and storage Containers—Well-closed containers.

Safflower

Carthami Flos

コウカ

Safflower is the tubulous flower of *Carthamus tinctorius* Linné (*Compositae*) without any treatment or with most of the yellow pigment removed, and pressed into a flat slab.

Description Red to red-brown corolla, yellow style and stamen, rarely mixed with immature ovary; total length about 1 cm; corolla, tubular and with 5 lobes; 5 stamens surrounding long pistil; pollen grains yellow and approximately spherical, about 50 μm in diameter, with fine protrusions on the surface. The pressed slab, about 0.5 cm in thickness, consists of a collection of numerous corollas. Odor, characteristic; taste, slightly bitter.

Identification Boil 0.2 g of Safflower with 10 mL of dilute ethanol under a reflux condenser for 15 minutes, and after cooling, filter. Place 3 mL of the filtrate in a small glass vessel about 3 cm in both internal diameter and height, hang a piece of filter paper, 20 mm by 300 mm, so that one end of the filter paper reaches the bottom of the vessel, and allow the paper to soak up the liquid for 1 hour. Transfer and immediately hang the paper in another glass vessel of the same type, containing 3 mL of water, and allow the paper to soak up the water for 1 hour: most of the upper part of the paper is colored light yellow, and the lower portion, light red.

Purity Foreign matter—The amount of ovaries, stems, leaves and other foreign matter contained in Safflower does not exceed 2.0%.

Total ash Not more than 18.0%.

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Saffron

Crocus

サフラン

Saffron is the stigma of *Crocus sativus* Linné (*Iridaceae*).

Description Thin cord-like stigma, externally dark yellow-red to red-brown, 1.5 - 3.5 cm in length, tripartite or separate; the end of partite part widened and the other end

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 μm 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 μL 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