

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  $\mu$ L 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  $\mu$ m 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  $\mu$ L 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 stamens, 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  $\mu$ m 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