component determination and 4-hydroxy-3-methoxybenzyl nonylic acid amide in methanol to make 50 mL. When the procedure is run with $20\,\mu\text{L}$ of this solution under the above operating conditions, 4-hydroxy-3-methoxybenzyl nonylic acid amide and capsaicin are eluted in this order with the resolution between these peaks being not less than 1.5.

System repeatability: When the test is repeated 6 times with $20 \,\mu\text{L}$ of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of capsaicin is not more than 1.5%.

Powdered Capsicum

Capsici Fructus Pulveratus

トウガラシ末

Powdered Capsicum is the powder of Capsicum. Powdered Capsicum contains not less than 0.10% of total capsaicins (capsaicin and dihydrocapsaicin), calculated on the basis of dried material.

Description Powdered Capsicum occurs as a yellow-red powder. It has a slight, characteristic odor and a hot, acrid taste.

Under a microscope, Powdered Capsicum reveals fragments of parenchyma containing oil droplets and yellow-red chromoplasts; fragments of outer pericarp with thick cuticle; fragments of stone cells from inner surface of pericarp, with wavy curved side walls; fragments of thin vessels; fragments of seed coat with thick wall, and fragments of parenchyma consisting of small cells of endosperm containing fixed oil and aleuron grains.

Identification To 2.0 g of Powdered Capsicum add 5 mL of ethanol (95), warm on a water bath for 5 minutes, cool, centrifuge, and use the supernatant liquid as the sample solution. Separately, dissolve 1 mg of capsaicin for thin-layer chromatography in 1 mL of ethanol (95), and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot $10 \,\mu\text{L}$ each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of diethyl ether and methanol (19:1) to a distance of about 12 cm, and air-dry the plate. Spray evenly 2,6-dibromo-N-chloro-1,4-benzoquinone monoimine TS on the plate, and allow to stand in ammonia gas: a spot from the sample solution and blue spot from the standard solution show the same in color tone and Rf value.

Loss on drying Not more than 14.0% (6 hours).

Total ash Not more than 8.0%.

Acid-insoluble ash Not more than 1.2%.

Extract content Ether-soluble extract: not less than 9.0%.

Component determination Weigh accurately about 0.5 g of medium powder of Powdered Capsicum in a glass-stoppered centrifuge tube, add 30 mL of methanol, shake for 15 minutes, centrifuge, and separate the supernatant liquid. To the residue add 10 mL of methanol, shake for 5 minutes, centrifuge, and separate the supernatant liquid. Repeat this

procedure again, combine the extracts, add methanol to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of capsaicin for component determination, previously dried in a desiccator (in vacuum, phosphorus (v) oxide, 40°C) for 5 hours, and dissolve in methanol to make exactly 50 mL. Pipet 2 mL of this solution, add methanol to make exactly 25 mL, and use this solution as the standard solution. Perform the test with exactly 20 μ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, $A_{\rm TC}$ and $A_{\rm TD}$, of capsaicin and dihydrocapsaicin (the relative retention time to capsaicin is about 1.3) in the sample solution, and the peak area, $A_{\rm S}$, of capsaicin in the standard solution.

Amount (mg) of total capsaicins

= amount (mg) of capsaicin for component determination

$$\times \frac{A_{\rm TC} + A_{\rm TD}}{A_{\rm S}} \times 0.08$$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 281 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 25 cm in length, packed with phenylated silica gel for liquid chromatography (5 μ m in particle diameter).

Column temperature: A constant temperature of about 30°C.

Mobile phase: A mixture of diluted phosphoric acid (1 in 1000) and acetonitrile (3:2).

Flow rate: Adjust the flow rate so that the retention time of capsaicin is about 20 minutes.

System suitability—

System performance: Dissolve 1 mg each of capsaicin for component determination and 4-hydroxy-3-methoxybenzyl nonylic acid amide in methanol to make 50 mL. When the procedure is run with 20 μ L of this solution under the above operating conditions, 4-hydroxy-3-methoxybenzyl nonylic acid amide and capsaicin are eluted in this order with the resolution between these peaks being not less than 1.5.

System repeatability: When the test is repeated 6 times with $20 \,\mu\text{L}$ of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of capsaicin is not more than 1.5%.

Capsicum Tincture

トウガラシチンキ

Capsicum Tincture contains not less than 0.010 w/v% of total capsaicins (capsaicin and dihydrocapsaicin).

Method of preparation

Capsicum, in medium cutting
Ethanol a sufficient quantity

To make 1000 mL

Prepare as directed under Tinctures, with the above ingredients.