ature at a rate of 10°C per minute to 200°C, and then program to increase the temperature at a rate of 2°C per minute to 260°C.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention times of the objective compounds are between 10 and 30 minutes.

Selection of column: Proceed with 1 μL of the standard solution under the above operating conditions. Use a column clearly separating each peak.

System repeatability: Repeat the test 6 times with the standard solution under the above operating conditions: the relative standard deviation of the peak area is not more than 10% for any objective compound.

Total ash Not more than 12.0%.

Acid-insoluble ash Not more than 2.0%.

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

Component determination Weigh accurately about 0.5 g of pulverized Senna Leaf in a glass-stoppered centrifuge tube, add 25 mL of diluted methanol (7 in 10), shake for 30 minutes, centrifuge, and separate the supernatant liquid. To the residue add 10 mL of diluted methanol (7 in 10) twice, shake for 10 minutes, centrifuge, and separate the supernatant liquid, respectively. Combine all the extracts, add diluted methanol (7 in 10) to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of sennoside A for component determination, previously dried in a desiccator (in vacuum at a pressure not exceeding 0.67 kPa, phosphorus (V) oxide) for not less than 12 hours, dissolve in a solution of sodium hydrogen carbonate (1 in 100) to make exactly 20 mL, and use this solution as standard stock solution A. Weigh accurately about 0.01 g of sennoside B for component determination, previously dried in a desiccator (in vacuum at a pressure not exceeding 0.67 kPa, phosphorus (V) oxide) for not less than 12 hours, dissolve in a solution of sodium hydrogen carbonate (1 in 100) to make exactly 20 mL, and use this solution as standard stock solution B. Pipet 5 mL of the standard stock solution A and 10 mL of the standard stock solution B, add methanol to make exactly 50 mL, and use this solution as the standard solution. Pipet 10 μL of the sample solution and the standard solution, and perform the test as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas, A_{TA} and A_{SA}, of sennoside A, and the peak areas, A_{TB} and A_{SB}, of sennoside B in each solution, calculate the amounts of sennoside A and sennoside B by the following equation, and designate the total as the amount of total sennosides.

Amount (mg) of sennoside A
= amount (mg) of sennoside A for component determination
$\times \frac{A_{TA}}{A_{SA}} \times \frac{1}{4}$

Amount (mg) of sennoside B
= amount (mg) of sennoside B for component determination
$\times \frac{A_{TB}}{A_{SB}} \times \frac{1}{2}$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength : 340 nm).

Column: A stainless steel column 4 to 6 mm in inside diameter and 15 to 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 50°C.

Mobile phase: Dissolve 2.45 g of tetra-*n*-heptylammonium bromide in 1000 mL of a mixture of diluted 1 mol/L acetic acid-sodium acetate buffer solution, pH 5.0 (1 in 10) and acetonitrile (17:8).

Flow rate: Adjust the flow rate so that the retention time of sennoside A is about 26 minutes.

Selection of column: Perform the test with 10 μL of the standard solution under the above operating conditions. Use a column giving elution of sennoside B and sennoside A in this order, clearly dividing each peak, and the number of theoretical plates of the peak of sennoside A being not less than 8000.

System repeatability: Repeat the test 6 times with the standard solution under the above operating conditions: the relative standard deviation of the peak areas of sennoside A is not more than 1.5%.

Powdered Senna Leaf

Sennae Folium Pulveratum

センナ末

Powdered Senna Leaf is the power of Senna Leaf. It contains not less than 1.0% of total sennosides (sennoside A and sennoside B), calculated on the basis of dried material.

Description Powdered Senna Leaf occurs as a light yellow to light grayish yellow-green powder. It has a slight odor and a bitter taste.

Under a microscope, Powdered Senna Leaf reveals fragments of vessels and vein tissue accompanied with crystal cell rows; fragments of thick-walled, bent, unicellular hairs; fragments of palisade and spongy tissue; clustered and solitary crystals of calcium oxalate, 10 to 20 μm in diameter.

Identification **(1)** Macerate 0.5 g of Powdered Senna Leaf in 10 mL of diethyl ether for 2 minutes, and filter. Add 5 mL of ammonia TS to the filtrate: a yellow-red color is produced in the water layer. To the residue of maceration add 10 mL of water, and macerate for 2 minutes. Filter, and add 5 mL of ammonia TS: a yellow-red color is produced in the water layer.

(2) To 2.0 g of Powdered Senna Leaf add 40 mL of a mixture of tetrahydrofuran and water (7:3), shake for 30 minutes, and centrifuge. Transfer the supernatant liquid to a separator, add 13 g of sodium chloride, and shake for 30 minutes. Separate the water layer with undissolved sodium chloride, and adjust the pH to 1.5 with 1 mol/L hydrochloric acid TS. Transfer this solution to another separator, shake with 30 mL of tetrahydrofuran for 10 minutes, separate the tetrahydrofuran layer, and use this solution as the sample solution. Separately, dissolve 1 mg of sennoside A for thin-layer chromatography in 1 mL of a mixture of tetrahydrofuran

and water (7:3), and use this solution as the standard solution. Spot 10 μL each of these solutions on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, 1-propanol, water and acetic acid (100) (40:40:30:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the sample solution and a red fluorescent spot from the standard solution show the same in color tone and *Rf* value.

Purity **(1)** Foreign matter—Under a microscope, stone cells and thick fibers are not observable.

(2) Total BHC's and total DDT's—Sodium chloride, anhydrous sodium sulfate and synthetic magnesium silicate for column chromatography used in this procedure are used after drying by heating at about 130°C for more than 12 hours and cooling in a desiccator (silica gel). Chromatographic column is prepared as follows: Place 20 g of synthetic magnesium silicate for column chromatography in a 200-mL flask, add 50 mL of hexane for Purity of crude drug, shake vigorously, and immediately pour the mixture into a chromatographic tube about 2 cm in inside diameter and about 30 cm in length. Drip until the depth of hexane layer at the upper part is about 5 cm, introduce 8 g of anhydrous sodium sulfate from the top, and further drip until a small quantity of hexane is left at the upper part.

Weigh accurately about 5 g of Powdered Senna Leaf, place in a glass-stoppered centrifuge tube, add 30 mL of a mixture of acetone for Purity of crude drug and water (5:2), stopper tightly, shake for 15 minutes, centrifuge, and separate the supernatant liquid. Repeat the same procedure twice with the residue using two 30-mL portions of a mixture of acetone for Purity of crude drug and water (5:2). Combine all the supernatant liquids, and evaporate in a vacuum at a temperature not higher than 40°C until the order of acetone is faint. Transfer the evaporated solution to a separator containing 100 mL of sodium chloride TS, and shake twice with two 50-mL portions of hexane for Purity of crude drug for 5 minutes each. Combine the hexane solutions, transfer to a separator containing 50 mL of sodium chloride TS, and shake for 5 minutes. Take the hexane layer, dry over 30 g of anhydrous sodium sulfate, and filter. Wash the residue on the filter paper with 20 mL of hexane for Purity of crude drug. Combine the filtrate and the washings, and evaporate in a vacuum at a temperature not higher than 40°C to about 5 mL. Transfer this solution to the chromatographic column and allow to pass with 300 mL of a mixture of hexane for Purity of crude drug and diethyl ether for Purity of crude drug (17:3) at a rate of not more than 5 mL per minute. After evaporating the eluate in a vacuum at a temperature not higher than 40°C, add hexane for Purity of crude drug to make exactly 5 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g each of α-BHC, β-BHC, γ-BHC, δ-BHC, *o,p'*-DDT, *p,p'*-DDT, *p,p'*-DDD and *p,p'*-DDE, dissolve in 5 mL of acetone for Purity of crude drug and add hexane for Purity of crude drug to make exactly 100 mL. Pipet 10 mL of this solution, and add hexane for Purity of crude drug to make exactly 100 mL. Pipet 1 mL of this solution, add hexane for Purity of crude drug to make exactly 100 mL, 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 Gas Chromatography according to the following conditions, and determine the peak areas corresponding to α-BHC, β-BHC, γ-BHC, δ-BHC, *o,p'*-DDT, *p,p'*-DDT, *p,p'*-DDD and *p,p'*-DDE from each solution, A_{TA} and A_{SA}; A_{TB} and A_{SB}; A_{TC} and A_{SC}; A_{TD} and A_{SD}; A_{TE} and A_{SE}; A_{TF} and A_{SF}; A_{TG} and A_{SG}; A_{TH} and A_{SH}. Calculate the content of each of α-BHC, β-BHC, γ-BHC, δ-BHC, *o,p'*-DDT, *p,p'*-DDT, *p,p'*-DDD and *p,p'*-DDE by means of the following equations, and determine the content of total BHC's and that of total DDT's: the content of total BHC's and that of total DDT's are each not more than 0.2 ppm.

Content (ppm) of α-BHC

$$= \frac{\text{amount (g) of } \alpha\text{-BHC}}{W} \times \frac{A_{TA}}{A_{SA}} \times 50$$

Content (ppm) of β-BHC

$$= \frac{\text{amount (g) of } \beta\text{-BHC}}{W} \times \frac{A_{TB}}{A_{SB}} \times 50$$

Content (ppm) of γ-BHC

$$= \frac{\text{amount (g) of } \gamma\text{-BHC}}{W} \times \frac{A_{TC}}{A_{SC}} \times 50$$

Content (ppm) of δ-BHC

$$= \frac{\text{amount (g) of } \delta\text{-BHC}}{W} \times \frac{A_{TD}}{A_{SD}} \times 50$$

Content (ppm) of *o,p'*-DDT

$$= \frac{\text{amount (g) of } o,p'\text{-DDT}}{W} \times \frac{A_{TE}}{A_{SE}} \times 50$$

Content (ppm) of *p,p'*-DDT

$$= \frac{\text{amount (g) of } p,p'\text{-DDT}}{W} \times \frac{A_{TF}}{A_{SF}} \times 50$$

Content (ppm) of *p,p'*-DDD

$$= \frac{\text{amount (g) of } p,p'\text{-DDD}}{W} \times \frac{A_{TG}}{A_{SG}} \times 50$$

Content (ppm) of *p,p'*-DDE

$$= \frac{\text{amount (g) of } p,p'\text{-DDE}}{W} \times \frac{A_{TH}}{A_{SH}} \times 50$$

W: Amount (g) of Powdered Senna Leaf

Content (ppm) of total BHC's
= content (ppm) of α-BHC + content (ppm) of β-BHC + content (ppm) of γ-BHC + content (ppm) of δ-BHC

Content (ppm) of total DDT's
= content (ppm) of *o,p'*-DDT + content (ppm) of *p,p'*-DDT + content (ppm) of *p,p'*-DDD + content (ppm) of *p,p'*-DDE

Operating conditions—

Detector: An electron capture detector

Sample injection system: A splitless injection system

Column: A fused silica capillary column about 0.3 mm in inside diameter and about 30 m in length, coated the inside wall with 7% cyanopropyl-7% phenylmethylsilicone polymer for gas chromatography in a thickness of 0.25 to 1.0 μm.

Column temperature: Maintain the temperature at 60°C for 2 minutes after injection, program to increase the temperature at a rate of 10°C per minute to 200°C, and then program to increase the temperature at a rate of 2°C per minute to 260°C.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention times of the objective compounds are between 10 and 30 minutes.

Selection of column: Proceed with 1 μL of the standard solution under the above operating conditions. Use a column clearly separating each peak.

System repeatability: Repeat the test 6 times with the standard solution under the above operating conditions: the relative standard deviation of the peak area is not more than 10% for any objective compound.

Total ash Not more than 12.0%.

Acid-insoluble ash Not more than 2.0%.

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

Component determination Weigh accurately about 0.5 g of Powdered Senna Leaf in a glass-stoppered centrifuge tube, add 25 mL of diluted methanol (7 in 10), shake for 30 minutes, centrifuge, and separate the supernatant liquid. To the residue add 10 mL of diluted methanol (7 in 10) twice, shake for 10 minutes, centrifuge, and separate the supernatant liquid, respectively. Combine all the extracts, add diluted methanol (7 in 10) to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of sennoside A for component determination, previously dried in a desiccator (in vacuum at a pressure not exceeding 0.67 kPa, phosphorus (V) oxide) for not less than 12 hours, dissolve in a solution of sodium hydrogen carbonate (1 in 100) to make exactly 20 mL, and use this solution as standard stock solution A. Weigh accurately about 0.01 g of sennoside B for component determination, previously dried in a desiccator (in vacuum at a pressure not exceeding 0.67 kPa, phosphorus (V) oxide) for not less than 12 hours, dissolve in a solution of sodium hydrogen carbonate (1 in 100) to make exactly 20 mL, and use this solution as standard stock solution B. Pipet 5 mL of the standard stock solution A and 10 mL of the standard stock solution B, add methanol to make exactly 50 mL, and use this solution as the standard solution. Pipet 10 μL of the sample solution and the standard solution, and perform the test as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas, A_{TA} and A_{SA}, of sennoside A, and the peak areas, A_{TB} and A_{SB}, of sennoside B in each solution, calculate the amounts of sennoside A and sennoside B by the following equation, and designate the total as the amount of total sennosides.

Amount (mg) of sennoside A
= amount (mg) of sennoside A for component determination
$\times \frac{A_{TA}}{A_{SA}} \times \frac{1}{4}$

Amount (mg) of sennoside B
= amount (mg) of sennoside B for component determination
$\times \frac{A_{TB}}{A_{SB}} \times \frac{1}{2}$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 340 nm).

Column: A stainless steel column 4 to 6 mm in inside diameter and 15 to 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 50°C.

Mobile phase: Dissolve 2.45 g of tetra-*n*-heptylammonium bromide in 1000 mL of a mixture of diluted 1 mol/L acetic acid-sodium acetate buffer solution, pH 5.0 (1 in 10) and acetonitrile (17:8).

Flow rate: Adjust the flow rate so that the retention time of sennoside A is about 26 minutes.

Selection of column: Perform the test with 10 μL of the standard solution under the above operating conditions. Use a column giving elution of sennoside B and sennoside A in this order with well separation of these peaks, and the number of theoretical plates of the peak of sennoside A being not less than 8000.

System repeatability: When the test is repeated 6 times with the standard solution under the above operating conditions, the relative standard deviation of the peak areas of sennoside A is not more than 1.5%.

Sesame Oil

Oleum Sesami

ゴマ油

Sesame Oil is the fixed oil obtained from the seeds of *Sesamum indicum* Linné (*Pedaliaceae*).

Description Sesame Oil is a clear, pale yellow oil. It is odorless or has a faint, characteristic odor, and has a bland taste.

It is miscible with diethyl ether and with petroleum ether.

It is slightly soluble in ethanol (95).

It congeals between 0°C and −5°C.

Congealing point of the fatty acids: 20 - 25°C

Identification To 1 mL of Sesame Oil add 0.1 g of sucrose and 10 mL of hydrochloric acid, and shake for 30 seconds: the acid layer becomes light red and changes to red on standing.

Specific gravity d_{20}^{20}: 0.914 - 0.921

Acid value Not more than 0.2.

Saponification value 187 - 194

Unsaponifiable matters Not more than 2.0%.

Iodine value 103 - 118

Containers and storage Containers—Tight containers.

Purified Shellac

精製セラック

Purified Shellac is a resin-like substance obtained from a purified secretion of *Laccifer lacca* Kerr (*Coccidae*).

Description Purified Shellac occurs as light yellow-brown to brown, lustrous, hard, brittle scutella. It has no odor or has a faint, characteristic odor.

It is freely soluble in ethanol (95) and in ethanol (99.5),