Amount (mg) of scopolamine ($C_{17}H_{21}NO_4$)
= amount (mg) of Scopolamine Hydrobromide Reference Standard, calculated on the dried basis
$\times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.789$

Internal standard solution—A solution of brucine dihydrate in the mobile phase (1 in 2500).

Containers and storage Containers—Tight containers.
Storage—Light-resistant, and in a cold place.

Scopolia Extract Powder

ロートエキス散

Scopolia Extract Powder contains not less than 0.085% and not more than 0.110% of total alkaloids [hyoscyamine ($C_{17}H_{23}NO_3$: 289.37) and scopolamine ($C_{17}H_{21}NO_4$: 303.35)].

Method of preparation

Scopolia Extract	100 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

To Scopolia Extract add 100 mL of Purified Water, then warm and soften the mixture with stirring. Cool, add 800 g of starch, Lactose or their mixture little by little, and mix well. Dry preferably at a low temperature, and dilute with a sufficient additional quantity of starch, Lactose or their mixture to make 1000 g of homogeneous powder.

Description Scopolia Extract Powder is a brownish yellow to grayish yellow-brown powder. It has a faint, characteristic odor and a slightly bitter taste.

Identification **(1)** To 20 g of Scopolia Extract Powder add 15 mL of water and 8 mL of ammonia TS, mix homogeneously, add 100 mL of diethyl ether and 7 g of sodium chloride, stopper tightly, shake for 1 hour, add 5 g of Powdered Tragacanth, and shake vigorously. Allow to stand for 5 minutes, take the clearly separated diethyl ether layer, and filter. Proceed with the filtrate as directed in the Identification (1) under Scopolia Extract.

(2) Place 5.0 g of Scopolia Extract Powder in a glass-stoppered centrifuge tube, add 30 mL of ammonia TS, and centrifuge after irradiation of ultrasonic waves for 5 minutes. Transfer the supernatant liquid to a separator, add 40 mL of ethyl acetate, and shake. Drain off the ethyl acetate layer, add 3 g of anhydrous sodium sulfate to the ethyl acetate, shake, and filter after the ethyl acetate becomes clear. Evaporate the filtrate to dryness under reduced pressure, dissolve the residue in 1 mL of ethanol (95), and use this solution as the sample solution. Proceed as directed in the Identification (2) under Scopolia Rhizome.

Assay Weigh accurately about 4.0 g of Scopolia Extract Powder, place in a glass-stoppered, centrifuge tube, add 15 mL of ammonia TS, and shake. Add 25 mL of diethyl ether, stopper tightly, shake for 15 minutes, centrifuge to take the diethyl ether layer. Repeat this procedure three times with the water layer, using 25-mL portions of diethyl ether. Combine the extracts, and evaporate the diethyl ether on a water bath. Dissolve the residue in 5 mL of the mobile phase, add exactly 3 mL of the internal standard solution, and add the mobile phase to make exactly 25 mL. Filter this solution through a membrane filter of a porosity of not more than 0.8 μm, discard the first 2 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.025 g of Atropine Sulfate Reference Standard (separately determine its loss on drying), dissolve in the mobile phase to make exactly 25 mL, and use this solution as standard stock solution A. Weigh accurately about 0.025 g of Scopolamine Hydrobromide Reference Standard (separately determine its loss on drying), dissolve in the mobile phase to make exactly 25 mL, and use this solution as standard stock solution B. Pipet 5 mL of the standard stock solution A and 1 mL of the standard stock solution B, add exactly 3 mL of the internal standard solution, then add the mobile phase to make exactly 25 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the ratios, Q_{TA} and Q_{SA}, of the peak area of hyoscyamine (atropine), and ratios, Q_{TS} and Q_{SS}, of the peak area of scopolamine to that of the internal standard in each solution, calculate the amounts of hyoscyamine and scopolamine by the following equation, and designate the total as the amount of total alkaloids.

Amount (mg) of hyoscyamine ($C_{17}H_{23}NO_3$)
= amount (mg) of Atropine Sulfate Reference Standard, calculated on the dried basis
$\times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.855$

Amount (mg) of scopolamine ($C_{17}H_{21}NO_4$)
= amount (mg) of Scopolamine Hydrobromide Reference Standard, calculated on the dried basis
$\times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.789$

Internal standard solution—A solution of brucine dihydrate in the mobile phase (1 in 2500).

Operating conditions—

Detector: An ultraviolet absorption spectrometer (wavelength: 210 nm).

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

Column temperature: A constant temperature of about 20°C.

Mobile phase: A mixture of a solution obtained by dissolving 6.8 g of potassium dihydrogenphosphate in 900 mL of water, adding 10 mL of triethylamine, adjusting the pH to 3.5 with phosphoric acid, and adding water to make 1000 mL, and acetonitrile (9:1).

Flow rate: Adjust the flow rate so that the retention time of scopolamine is about 8 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and determine the resolution. Use a column giving elution of scopolamine, atropine and the internal standard in this order with the resolution between the peaks of scopolamine and atropine being not less than 11, and the resolution between the

peaks of atropine and the internal standard being not less than 4.

Containers and storage Containers—Tight containers.

Scopolia Extract and Carbon Powder

ロートエキス・カーボン散

Method of preparation

Scopolia Extract	5 g
Medicinal Carbon	550 g
Natural Aluminum Silicate	345 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

Prepare before use as directed under Powders, with the above ingredients. May be prepared with Scopolia Extract Powder in place of Scopolia Extract.

Description Scopolia Extract and Carbon Powder is easily dustable and black in color. It is tasteless.

Containers and storage Containers—Well-closed containers.

Compound Scopolia Extract and Diastase Powder

複方ロートエキス・ジアスターゼ散

Method of preparation

Scopolia Extract	8 g
Diastase	200 g
Precipitate Calcium Carbonate	300 g
Sodium Bicarbonate	250 g
Magnesium Oxide	100 g
Powdered Gentian	50 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

Prepare before use as directed under Powders, with the above ingredients. May be prepared with Scopolia Extract Powder in place of Scopolia Extract.

Description Compound Scopolia Extract and Diastase Powder is light yellow in color. It has a bitter taste.

Containers and storage Containers—Well-closed containers.

Scopolia Extract and Ethyl Aminobenzoate Powder

ロートエキス・アネスタミン散

Scopolia Extract and Ethyl Aminobenzoate Powder contains not less than 22.5% and not more than 27.5% of ethyl aminobenzoate ($C_9H_{11}NO_2$: 165.19).

Method of preparation

Scopolia Extract	10 g
Ethyl Aminobenzoate	250 g
Magnesium Oxide	150 g
Sodium Bicarbonate	500 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

Prepare as directed under Powders, with the above ingredients. May be prepared with Scopolia Extract Powder in place of Scopolia Extract.

Description Scopolia Extract and Ethyl Aminobenzoate Powder is slightly brownish white in color. It has a slightly bitter taste, leaving a sensation of numbness on the tongue.

Identification **(1)** To 2 g of Scopolia Extract and Ethyl Aminobenzoate Powder add 20 mL of diethyl ether, shake, and filter through a glass filter (G4). Wash the residue with three 10-mL portions of diethyl ether, combine the filtrate and the washings, evaporate to dryness, and perform the following test with the residue (ethyl aminobenzoate).

(i) Dissolve 0.01 g of the residue in 1 mL of dilute hydrochloric acid and 4 mL of water: the solution responds to the Qualitative Tests for primary aromatic amines.

(ii) Dissolve 0.1 g of the residue in 5 mL of water with the aid of dilute hydrochloric acid added dropwise, and add iodine TS dropwise: a brown precipitate is produced.

(iii) Warm 0.05 g of the residue with 2 drops of acetic acid (31) and 5 drops of sulfuric acid: the odor of ethyl acetate is perceptible.

(2) To the diethyl ether-insoluble residue obtained in (1) add 30 mL of water, shake gently, and filter: the filtrate responds to the Qualitative Tests for sodium salt and for bicarbonate.

(3) To the water-insoluble residue obtained in (2) add 10 mL of dilute hydrochloric acid, shake, and filter: the filtrate responds to the Qualitative Tests for magnesium salt.

(4) Place 30 g of Scopolia Extract and Ethyl Aminobenzoate Powder in a glass-stoppered conical flask, add 100 mL of water, shake for 30 minutes, and filter immediately by suction through a glass filter (G3). Transfer the residue in the flask to the same glass filter with the filtrate, and filter the residue by suction while pressing vigorously the residue on the same glass filter. Place 75 mL of the filtrate in a 300-mL beaker, and add cautiously 10 mL of diluted sulfuric acid (1 in 3). Add 0.2 mL of bromocresol green TS to this solution, and add dilute sulfuric acid dropwise while shaking thoroughly, until the color of the solution changes from green to yellow-green. After cooling, place this solution in a separator, wash with two 25-mL portions of a mixture of hexane and diethyl ether (1:1) by shaking well, and place the water layer in another separator. Make slightly alkaline with ammonia