

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

TS, add immediately 30 mL of diethyl ether, and shake well. Wash the diethyl ether layer with two 10-mL portions of a saturated solution of sodium chloride, separate the diethyl ether layer, add 3 g of anhydrous sodium sulfate, shake, and filter through a pledget of cotton. Evaporate the filtrate to dryness, dissolve the residue in 0.2 mL of ethanol (95), and use this solution as the sample solution. Separately, dissolve 0.020 g of atropine sulfate for thin-layer chromatography and 0.010 g of scopolamine hydrobromide in 10 mL each of ethanol (95), and use these solutions as standard solution (1) and standard solution (2). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L each of the sample solution and the standard solutions on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol, acetone and ammonia solution (28) (73:15:10:2) to a distance of about 10 cm, and dry the plate at 80°C for 10 minutes. After cooling, spray evenly Dragendorff's TS for spraying on the plate: two principal spots from the sample solution show the same in color tone and *R_f* value with each yellow-red spot from the standard solutions, respectively.

Assay Weigh accurately about 0.3 g of *Scopolia Extract* and *Ethyl Aminobenzoate Powder*, transfer to a Soxhlet extractor, extract with 100 mL of diethyl ether for 1 hour, and evaporate the diethyl ether on a water bath. Dissolve the residue in 25 mL of 1 mol/L hydrochloric acid TS, and add water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 250 mL, and use this solution as the sample solution. Weigh accurately about 0.075 g of *Ethyl Aminobenzoate Reference Standard*, previously dried in a desiccator (silica gel) for 3 hours, dissolve in 25 mL of 1 mol/L hydrochloric acid TS, and add water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 250 mL, and use this solution as the standard solution. Pipet 5 mL each of the sample solution and the standard solution, to each add 10 mL of 1 mol/L hydrochloric acid TS, then add 1 mL of a solution of sodium nitrite (1 in 200), prepared before use, and allow to stand for 5 minutes with occasional shaking. Add 5 mL of ammonium amidosulfate TS, shake well, and allow to stand for 10 minutes. Add 2 mL of *N*-(1-naphthyl)-*N'*-diethylethylenediamine oxalate-acetone TS, mix immediately, and add water to make exactly 50 mL. Allow to stand for 2 hours, determine the absorbances, *A_T* and *A_S*, of these solutions at 550 nm, as directed under the Ultraviolet-visible Spectrophotometry using a blank prepared in the same manner with 5 mL of water in place of the sample solution.

$$\begin{aligned} & \text{Amount (mg) of ethyl aminobenzoate (C}_9\text{H}_{11}\text{NO}_2\text{)} \\ &= \text{amount (mg) of Ethyl Aminobenzoate Reference} \\ & \quad \text{Standard} \\ & \quad \times \frac{A_T}{A_S} \end{aligned}$$

Containers and storage Containers—Well-closed containers.

Scopolia Extract, Papaverine and Ethyl Aminobenzoate Powder

ロートエキス・パパベリン・アネスタミン散

Scopolia Extract, *Papaverine* and *Ethyl Aminobenzoate Powder* contains not less than 10.8% and not more than 13.2% of ethyl aminobenzoate (C₉H₁₁NO₂: 165.19).

Method of preparation

| | |
|----------------------------------|-----------------------|
| <i>Scopolia Extract</i> | 15 g |
| <i>Papaverine Hydrochloride</i> | 15 g |
| <i>Ethyl Aminobenzoate</i> | 120 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*, *Papaverine* and *Ethyl Aminobenzoate Powder* is brownish yellow to grayish yellow-brown in color. It has a slightly bitter taste, leaving a sensation of numbness on the tongue.

Identification (1) To 4 g of *Scopolia Extract*, *Papaverine* 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 20 mL of chloroform, shake well, filter, and further wash the residue with 10 mL of chloroform. Combine the filtrate and the washing, transfer this solution to a separator, and add 10 mL of 0.1 mol/L hydrochloric acid TS. After shaking, separate the chloroform layer, add 2 g of anhydrous sodium sulfate, shake, and filter through a pledget of cotton. Evaporate the filtrate to dryness, dry the residue at 105°C for 3 hours, and perform the following tests (*papaverine hydrochloride*):

(i) To 1 mg of the residue add 1 drop of formaldehyde solution-sulfuric acid TS: a colorless or light yellow-green color, changing to red-purple, is produced.

(ii) Dissolve 1 mg of the residue in 3 mL of acetic anhydride and 5 drops of sulfuric acid, heat in a water bath for 1 minute, and view under ultraviolet light: the solution shows a yellow-green fluorescence.

(3) Place 20 g of *Scopolia Extract*, *Papaverine* and *Ethyl Aminobenzoate Powder* in a glass-stoppered conical flask, add 80 mL of water, shake for 15 minutes, and filter by suc-