Containers and storage Containers—Tight containers.

Opium Tincture

アヘンチンキ

Opium Tincture contains not less than 0.93 w/v% and not more than 1.07 w/v% of morphine ($C_{17}H_{19}NO_3$: 285.34).

Method of preparation

Powdered Opium	100 g
35 vol% Ethanol	a sufficient quantity
To make	1000 mL

Prepare as directed under Tinctures, with the above ingredients. May be prepared with an appropriate quantity of Ethanol and Purified Water in place of 35 vol% Ethanol.

Description Opium Tincture is a dark red-brown liquid. It is affected by light.

Identification (1) To 1 mL of Opium Tincure add diluted ethanol (7 in 10) to make 10 mL, filter, and use the filtrate as the sample solution. Proceed as directed in the Identification (1) under Powdered Opium.

(2) Evaporate 1 mL of Opium Tincture to dryness on a water bath, and proceed with the residue as directed in the Identification (2) under Powdered Opium.

Alcohol number Not less than 3.5 (Method 1).

Assay Evaporate 50 mL of Opium Tincture, accurately measured, on a water bath to dryness. Add 10 mL of ethanol (99.5) to the residue, evaporate to dryness again, cool, and triturate with exactly 10 mL of water. Proceed with this solution as directed in the Assay under Powdered Opium.

Each mL of 0.05 mol/L sulfuric acid VS
= 28.534 mg of $C_{17}H_{19}NO_3$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Opium Alkaloids Hydrochlorides

塩酸アヘンアルカロイド

Opium Alkaloids Hydrochlorides consists of the hydrochlorides of some of the main alkaloids obtained from opium.

It contains not less than 47.0% and not more than 52.0% of morphine ($C_{17}H_{19}NO_3$: 285.34), and not less than 33.0% and not more than 38.5% of other opium alkaloids.

Description Opium Alkaloids Hydrochlorides occur as a white to light brown powder.

It is soluble in water, and slightly soluble in ethanol (95).

It is affected by light.

Identification (1) Dissolve 0.1 g of Opium Alkaloids Hydrochlorides in 10 mL of diluted ethanol (1 in 2), and use this solution as the sample solution. Separately, dissolve 0.06 g of Morphine Hydrochloride, 0.04 g of Noscapine Hydrochloride, 0.01 g of Codein Phosphate and 0.01 g of Papaverine Hydrochloride in 10 mL each of diluted ethanol (1 in 2), and use these solutions as the standard solution (1), (2), (3) and (4), respectively. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 20 μL each of the sample solution and the standard solutions on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of acetone, toluene, ethanol (99.5) and ammonia solution (28) (20:20:3:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): each spot from the sample solution is the same in color tone and *R*f value with the corresponding spot from the standard solution (1), (2), (3) and (4) (morphine, noscapine, codeine and papaverine).

(2) A solution of Opium Alkaloids Hydrochlorides (1 in 50) responds to the Qualitative Tests (2) for chloride.

pH Dissolve 1.0 g of Opium Alkaloids Hydrochlorides in 50 mL of water: the pH of the solution is between 3.0 and 4.0.

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Opium Alkaloids Hydrochlorides in 10 mL of water: the solution is clear, and has no more color than the following control solution.

Control solution: Dilute 6.0 mL of 1/60 mol/L potassium dichromate VS with water to make 1000 mL.

(2) Meconic acid—Dissolve 0.1 g of Opium Alkaloids Hydrochlorides in 2 mL of water, and pour into a polyethylene column 1 cm in inside diameter, packed with about 0.36 g of aminopropylsilanized silica gel for pretreatment (55 - 105 μm in particle diameter) and previously washed through with 5 mL of water. Then, wash the column with 5 mL of water, 5 mL of methanol and 10 mL of 0.1 mol/L hydrochloric acid in this order, then elute with 2 mL of 1 mol/L hydrochloric acid, and use the eluate as the test solution. To the test solution add 2 mL of dilute sodium hydroxide TS and 1 drop of iron (III) chloride TS: no red color develops.

Loss on drying Not more than 6.0% (0.5 g, 120°C, 8 hours).

Residue on ignition Not more than 0.5% (0.5 g).

Assay (1) Morphine—Weigh accurately about 0.2 g of Opium Alkaloids Hydrochlorides, and dissolve in water to make exactly 50 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution and water to make 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.025 g of morphine hydrochloride for assay, dissolve in exactly 10 mL of the internal standard solution, add water to make 50 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of morphine to that of the internal standard.

Amount (mg) of morphine ($C_{17}H_{19}NO_3$)
= amount (mg) of morphine hydrochloride for assay, calculated on the anhydrous basis
$\times \frac{Q_T}{Q_S} \times 0.8867 \times 5$

Internal standard solution—A solution of etilefrine hydrochloride (1 in 500).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 285 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 1.0 g of sodium lauryl sulfate in 500 mL of diluted phosphoric acid (1 in 1000), and adjust the pH to 3.0 with sodium hydroxide TS. To 240 mL of this solution add 70 mL of tetrahydrofuran, and mix.

Flow rate: Adjust the flow rate so that the retention time of morphine is about 10 minutes.

System suitability—

System performance: When the procedure is run with 20 μL of the standard solution under the above operating conditions, morphine and the internal standard are eluted in this order with the resolution between these peaks being not less than 3.

System repeatability: When the test is repeated 6 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of morphine to that of the internal standard is not more than 1.0%.

(2) Other opium alkaloids—Weigh accurately about 1 g of Opium Alkaloids Hydrochlorides, dissolve in 20 mL of water, add 25 mL of sodium hydroxide TS, and extract successively with 50 mL, 40 mL, 30 mL and 20 mL of chloroform. Combine the chloroform extracts, and wash the extract with 10 mL of water. Filter the chloroform through a filter paper previously moistened with chloroform. Extract the washing with two 5-mL portions of chloroform, filter the chloroform extracts through the filter paper, and combine all of the filtrates. Wash the filter paper with four 5-mL portions of chloroform, and combine the washings with the combined filtrate. Evaporate the chloroform on a water bath, add 2 mL of ethanol (99.5) after the odor of chloroform is no longer perceptible, and evaporate to dryness. Dry the residue at 105°C for 4 hours, and weigh as other opium alkaloids.

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Opium Alkaloids Hydrochlorides Injection

塩酸アヘンアルカロイド注射液

Opium Alkaloids Hydrochlorides Injection is an aqueous solution for injection.

It contains not less than 0.90 w/v% and not more than 1.10 w/v% of morphine ($C_{17}H_{19}NO_3$: 285.34).

Method of preparation

Opium Alkaloids Hydrochlorides	20 g
Water for Injection	a sufficient quantity
To make	1000 mL

Prepare as directed under Injections, with the above ingredients.

Description Opium Alkaloids Hydrochlorides Injection is a clear, colorless or light brown liquid.

It is affected by light.

pH: 2.5 - 3.5

Identification To 1 mL of Opium Alkaloids Hydrochlorides Injection add 1 mL of ethanol (99.5), mix, and use this solution as the sample solution, and proceed as directed in the Identification (1) under Opium Alkaloids Hydrochlorides.

Assay Pipet 2 mL of Opium Alkaloids Hydrochlorides Injection, add exactly 10 mL of the internal standard solution and water to make 50 mL, and use this solution as the sample solution. Proceed as directed in the Assay (1) under Opium Alkaloids Hydrochlorides.

Amount (mg) of morphine ($C_{17}H_{19}NO_3$)
= amount (mg) of morphine hydrochloride for assay, calculated on the anhydrous basis
$\times \frac{Q_T}{Q_S} \times 0.8867$

Internal standard solution—A solution of Etilefrine Hydrochloride (1 in 500).

Containers and storage Containers—Hermetic containers, and colored containers may be used.

Storage—Light-resistant.