

15 minutes, centrifuge, and separate the supernatant liquid. Repeat this procedure three times with the residue using 20-mL portions of diethyl ether. Combine all the extracts, and evaporate the diethyl ether on a water bath. Dissolve the residue in 10 mL of the mobile phase, add exactly 10 mL of the internal standard solution, and add the mobile phase to make exactly 100 mL. Filter this solution through a membrane filter with a porosity 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 0.075 g of strychnine nitrate for assay (determine the loss on drying before use), and dissolve in the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, then add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the ratio, Q_T and Q_S , of the peak area of strychnine to that of the internal standard in each solution.

$$\begin{aligned} &\text{Amount (mg) of strychnine (C}_{21}\text{H}_{22}\text{N}_2\text{O}_2) \\ &= \text{amount (mg) of strychnine nitrate for assay,} \\ &\quad \text{calculated on the dried basis} \\ &\quad \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414 \end{aligned}$$

Internal standard solution—A solution of barbital sodium in the mobile phase (1 in 500).

Operating conditions—

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

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

Column temperature: Room temperature.

Mobile phase: Dissolve 6.8 g of potassium dihydrogenphosphate in water to make 1000 mL, and mix with acetonitrile and triethylamine (45:5:1), and adjust the mixture with phosphoric acid to a pH of 3.0.

Flow rate: Adjust the flow rate so that the retention time of Strychnine is about 17 minutes.

Selection of column: Proceed with 5 μL of the standard solution under the above operating conditions. Use a column giving elution of the internal standard and strychnine in this order, and clearly dividing each peak.

Nux Vomica Extract

ホミカエクス

Nux Vomica Extract contains not less than 6.15% and not more than 6.81% of strychnine ($\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2$; 334.41).

Method of preparation After defatting 1000 g of coarse powder of Nux Vomica with hexane, digest by the percolation method, using a mixture of 750 mL of Ethanol, 10 mL of Acetic Acid and 240 mL of Purified Water as the first solvent, and 70 vol% ethanol as the second solvent. Combine

the extracts, and prepare the dry extract as directed under Extracts. May be prepared with an appropriate quantity of Ethanol and Purified Water.

Description Nux Vomica Extract occurs as yellow-brown to brown powder. It has a characteristic odor, and an extremely bitter taste.

Identification Extract 0.5 g of Nux Vomica Extract with 0.5 mL of ammonia TS and 10 mL of chloroform with occasional shaking. Filter the chloroform extract, evaporate the filtrate on a water bath until most of the chloroform is removed, and proceed as directed in the Identification under Nux Vomica.

Assay Weigh accurately about 0.2 g of Nux Vomica Extract, place in a glass-stoppered centrifuge tube, add 15 mL of ammonia TS, and shake. Add 20 mL of diethyl ether, stopper tightly, shake for 15 minutes, centrifuge to disperse the diethyl ether layer. Repeat this procedure three times with the water layer, using 20-mL portions of diethyl ether. Combine the extracts, and evaporate the diethyl ether on a water bath. Dissolve the residue in 10 mL of the mobile phase, and add exactly 10 mL of the internal standard solution, and add the mobile phase to make exactly 100 mL. Proceed as directed in the Assay under Nux Vomica.

$$\begin{aligned} &\text{Amount (mg) of strychnine (C}_{21}\text{H}_{22}\text{N}_2\text{O}_2) \\ &= \text{amount (mg) of strychnine nitrate for assay,} \\ &\quad \text{calculated on the dried basis} \\ &\quad \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414 \end{aligned}$$

Internal standard solution—A solution of barbital sodium in the mobile phase (1 in 500).

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Nux Vomica Extract Powder

ホミカエクス散

Nux Vomica Extract Powder contains not less than 0.61% and not more than 0.68% of strychnine ($\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2$; 334.41).

Method of preparation

| | |
|--|-------|
| Nux Vomica Extract | 100 g |
| Starch, Lactose or their mixture a sufficient quantity | |
| To make 1000 g | |

To Nux Vomica Extract add 100 mL of Purified Water, then warm, and soften 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 the homogeneous powder.

Description Nux Vomica Extract Powder occurs as a yellow-brown to grayish brown powder. It has a slight, characteristic odor and a bitter taste.

Identification (1) To 3 g of Nux Vomica Extract Powder