diethyl ether.

It is affected by light.

Identification (1) To 1 mg of Reserpine add 1 mL of vanillin-hydrochloric acid TS, and warm: a vivid red-purple color develops.

(2) Determine the absorption spectrum of a solution of Reserpine in acetonitrile (1 in 50,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Reserpine Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) Determine the infrared absorption spectrum of Reserpine, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of previously dried Reserpine Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers.

Optical rotation $[\alpha]_D^{20}$: -114 – $-127°$ (after drying, 0.25 g, chloroform, 25 mL, 100 mm).

Purity Related substances—Conduct this procedure without exposure to daylight, using light-resistant vessels. Dissolve 0.050 g of Reserpine in 50 mL of acetonitrile, and use this solution as the sample solution. Pipet 3 mL of the sample solution, add acetonitrile to make exactly 100 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 each peak area from these solutions by the automatic integration method: the total area of all peaks other than reserpine peak from the sample solution is not larger than the peak area of reserpine from the standard solution.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 268 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 25 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: A mixture of 0.05 mol/L potassium dihydrogenphosphate, pH 3.0 and acetonitrile (13:7).

Flow rate: Adjust the flow rate so that the retention time of reserpine is about 20 minutes.

Selection of column: Dissolve 0.01 g of Reserpine and 4 mg of butyl parahydroxybenzoate in 100 mL of acetonitrile. To 5 mL of this solution add acetonitrile to make 50 mL. Proceed with 20 μL of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of reserpine and butyl parahydroxybenzoate in this order with the resolution between these peaks being not less than 2.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of reserpine from 10 μL of the standard solution is about 20 mm.

Time span of measurement: About twice as long as the retention time of reserpine.

Loss on drying Not more than 0.5% (0.2 g, in vacuum, 60°C, 3 hours).

Residue on ignition Not more than 0.25% (0.2 g).

Assay Conduct this procedure without exposure to daylight, using light-resistant vessels. Weigh accurately about 0.01 g each of Reserpine and Reserpine Reference Standard, previously dried, and dissolve each in acetonitrile to make exactly 100 mL. Pipet 5 mL each of these solutions, add exactly 10 mL of the internal standard solution, 5 mL of acetonitrile and water to make 50 mL, and use these solutions as the sample solution and the standard solution, respectively. 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 reserpine to that of the internal standard.

$$\text{Amount (mg) of } C_{33}H_{40}N_2O_9 = \text{amount (mg) of Reserpine Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of butyl parahydroxybenzoate in acetonitrile (1 in 50,000).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 268 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 25 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: A mixture of 0.05 mol/L potassium dihydrogenphosphate, pH 3.0 and acetonitrile (11:9).

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

Selection of column: Proceed with 20 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of reserpine and the internal standard in this order with the resolution between these peaks being not less than 2.

Containers and Storage Containers—Well-closed containers.

Storage—Light-resistant.

Reserpine Injection

レセルピン注射液

Reserpine Injection is an aqueous solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of reserpine ($C_{33}H_{40}N_2O_9$: 608.68).

Method of preparation Prepare as directed under Injections with Reserpine.

Description Reserpine Injection is a clear, colorless or pale yellow liquid.

pH: 2.5 – 4.0

Identification Measure a volume of Reserpine Injection, equivalent to 1.5 mg of Reserpine according to the labeled amount, add 10 mL of diethyl ether, shake for 10 minutes, and take the aqueous layer. If necessary, add 10 mL of diethyl ether to the aqueous layer, and shake for 10 minutes to repeat the process. To the aqueous layer add water to make 50 mL, and determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 265 nm and 269 nm.

Assay Measure exactly a volume of Reserpine Injection, equivalent to about 4 mg of reserpine ($C_{33}H_{40}N_2O_9$). Separately, weigh accurately about 4 mg of Reserpine Reference Standard, previously dried in vacuum at 60°C for 3 hours. Transfer them to separate separator, add 10 mL each of water and 5 mL each of ammonia TS, and extract with one 20-mL portion of chloroform, then with three 10-mL portions of chloroform with shaking vigorously. Combine the chloroform extracts, wash with two 50-mL portions of diluted hydrochloric acid (1 in 1000), and combine the washings. Then wash the chloroform extract with two 50-mL portions of a solution of sodium hydrogen carbonate (1 in 100), and combine the all washings. Extract the combined washing with two 10-mL portions of chloroform, and combine the washings with the former chloroform extract. Transfer the chloroform solution to a 100-mL volumetric flask through a pledget of absorbent cotton previously wetted with chloroform, wash with a small amount of chloroform, dilute with chloroform to make 100 mL, and use these solutions as the sample solution and the standard solution, respectively. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution, respectively, at 295 nm as directed under the Ultraviolet-visible Spectrophotometry.

Amount (mg) of reserpine ($C_{33}H_{40}N_2O_9$)
= amount (mg) of Reserpine Reference Standard
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Hermetic containers, and colored containers may be used.
Storage—Light-resistant.

0.1% Reserpine Powder

Reserpine Powder

レセルピン散 0.1%

0.1% Reserpine Powder contains not less than 0.09% and not more than 0.11% of reserpine ($C_{33}H_{40}N_2O_9$: 608.68).

Method of preparation

Reserpine	1 g
Lactose	a sufficient quantity
	To make 1000 g

Prepare as directed under Powders, with the above ingredients.

Identification To 0.4 g of 0.1% Reserpine Powder add 20 mL of acetonitrile, shake for 30 minutes, and centrifuge. Determine the absorption spectrum of the supernatant liquid as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 265 nm and 269 nm, and between 294 nm and 298 nm.

Assay Conduct this procedure without exposure to daylight, using light-resistant vessels. Weigh accurately a quantity of 0.1% Reserpine Powder, equivalent to about 0.5 mg of reserpine ($C_{33}H_{40}N_2O_9$), disperse in 12 mL of water, add exactly 10 mL of the internal standard solution and 10 mL of acetonitrile, and dissolve by warming at 50°C for 15 minutes, then add water to make 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of Reserpine Reference Standard, previously dried at 60°C in vacuum for 3 hours, dissolve in acetonitrile to make exactly 100 mL. Pipet 5 mL of this solution, add exactly 10 mL of the internal standard solution, 5 mL of acetonitrile and water to make 50 mL, and use this solution as the standard solution. Proceed with the sample solution and the standard solution as directed in the Assay under Reserpine.

Amount (mg) of reserpine ($C_{33}H_{40}N_2O_9$)
= amount (mg) of Reserpine Reference Standard
$\times \frac{Q_T}{Q_S} \times \frac{1}{20}$

Internal standard solution—A solution of butyl parahydroxybenzoate in acetonitrile (1 in 50,000).

Containers and storage Containers—Well-closed containers.
Storage—Light-resistant.

Reserpine Tablets

レセルピン錠

Reserpine Tablets contain not less than 90% and not more than 110% of the labeled amount of reserpine ($C_{33}H_{40}N_2O_9$: 608.68).

Method of preparation Prepare as directed under Tablets, with Reserpine.

Identification Take a portion of powdered Reserpine Tablets, equivalent to 0.4 mg of Reserpine according to the labeled amount, add 20 mL of acetonitrile, shake for 30 minute, and centrifuge. Determine the absorption spectrum of the supernatant liquid as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 265 nm and 269 nm, and between 294 nm and 298 nm.

Dissolution test Take 1 tablet of Reserpine Tablets, and perform the test at 100 revolutions per minute with 500 mL of a solution of polysorbate 80 (1 in 20,000) in diluted dilute acetic acid (1 in 200) as the test solution according to Method 2 under the Dissolution Test. Take 20 mL or more of the dissolved solution 30 minutes after starting the dissolution test, filter through a filter laminated with polyester