

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.

$$\begin{aligned} &\text{Amount (mg) of reserpine (C}_{33}\text{H}_{40}\text{N}_2\text{O}_9) \\ &= \text{amount (mg) of Reserpine Reference Standard} \\ &\quad \times \frac{A_T}{A_S} \end{aligned}$$

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.

$$\begin{aligned} &\text{Amount (mg) of reserpine (C}_{33}\text{H}_{40}\text{N}_2\text{O}_9) \\ &= \text{amount (mg) of Reserpine Reference Standard} \\ &\quad \times \frac{Q_T}{Q_S} \times \frac{1}{20} \end{aligned}$$

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 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.

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 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

fibers, discard the first 10 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, dry Reserpine Reference Standard at 60°C in vacuum for 3 hours, weigh accurately an amount 100 times the labeled amount, dissolve in 1 mL of chloroform and 80 mL of ethanol (95), and add a solution of polysorbate 80 in diluted dilute acetic acid (1 in 200) (1 in 1000) to make exactly 200 mL. Pipet 1 mL of this solution, add a solution of polysorbate 80 in diluted dilute acetic acid (1 in 200) (1 in 1000) 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, transfer to glass-stoppered brown test tubes T and S, respectively, add exactly 5 mL each of ethanol (99.5), shake well, add exactly 1 mL each of diluted vanadium (V) oxide (1 in 2), shake vigorously, and allow to stand for 30 minutes. Perform the test with these solutions as directed under the Fluoroscopy, and determine the intensity of fluorescence, F_T and F_S , at the wavelength of excitation at 400 nm and at the wavelength of fluorescence at 500 nm.

Dissolution rate of Reserpine Tablets after 30 minutes should be not less than 70%.

$$\begin{aligned} & \text{Dissolution rate (\%)} \text{ to the labeled} \\ & \text{amount of reserpine (C}_{33}\text{H}_{40}\text{N}_2\text{O}_9\text{)} \\ & = W_s \times \frac{F_T}{F_S} \times \frac{1}{C} \end{aligned}$$

W_s : Amount (mg) of Reserpine Reference Standard.

C : Labeled amount (mg) of reserpine (C₃₃H₄₀N₂O₉) in each tablet.

Content uniformity Conduct this procedure without exposure to daylight, using light-resistant vessels. To one tablet of Reserpine Tablets add 2 mL of water, disintegrate by warming at 50°C for 15 minutes while shaking. After cooling, add exactly 2 mL of the internal standard solution per 0.1 mg of reserpine according to the labeled amount, add 2 mL of acetonitrile, warm at 50°C for 15 minutes while shaking, and after cooling, add water to make 10 mL. Centrifuge the solution, and use the supernatant liquid 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.

$$\begin{aligned} & \text{Amount (mg) of reserpine (C}_{33}\text{H}_{40}\text{N}_2\text{O}_9\text{)} \\ & = \text{amount (mg) of Reserpine Reference Standard} \\ & \times \frac{Q_T}{Q_S} \times \frac{\text{labeled amount (mg) of reserpine in each tablet}}{10} \end{aligned}$$

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

Assay Conduct this procedure without exposure to daylight, using light-resistant vessels. Weigh accurately and powder not less than 20 Reserpine Tablets. Weigh accurately a quantity of the powder, equivalent to about 0.5 mg of reserpine (C₃₃H₄₀N₂O₉), add 3 mL of water, and warm at 50°C for 15 minutes while shaking. After cooling, add exactly 10 mL of the internal standard solution, 10 mL of acetonitrile and warm at 50°C for 15 minutes while shaking. After cooling, add water to make 50 mL, centrifuge, and use the

supernatant liquid 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, and 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.

$$\begin{aligned} & \text{Amount (mg) of reserpine (C}_{33}\text{H}_{40}\text{NO}_9\text{)} \\ & = \text{amount (mg) of Reserpine Reference Standard} \\ & \times \frac{Q_T}{Q_S} \times \frac{1}{20} \end{aligned}$$

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

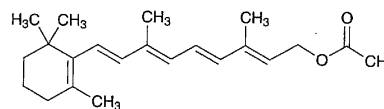
Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Retinol Acetate

Vitamin A Acetate

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C₂₂H₃₂O₂: 328.49

(2*E*,4*E*,6*E*,8*E*)-3,7-Dimethyl-9-(2,6,6-trimethylcyclohex-1-en-1-yl)nona-2,4,6,8-tetraen-1-yl acetate [127-47-9]

Retinol Acetate is synthetic retinol acetate or synthetic retinol acetate diluted with fixed oil. It contains not less than 2,500,000 Vitamin A Units per gram. A suitable antioxidant may be added. Retinol Acetate contains not less than 95% and not more than 105% of the labeled Units.

Description Retinol Acetate occurs as pale yellow to yellow-red crystals or an ointment-like substance, and has a faint, characteristic odor, but has no rancid odor.

When powdered, it is very soluble in chloroform and in diethyl ether, freely soluble in petroleum diethyl, soluble in 2-propanol and in ethanol (95), and practically insoluble in water.

It is affected by air and by light.

Identification (1) Prepare a solution of Retinol Acetate in chloroform containing 30 Vitamin A Units per mL according to the labeled Units, pipet 1 mL of the solution, and add 3 mL of antimony (III) chloride TS: a blue color develops immediately, then fades rapidly.

(2) Proceed with Retinol Acetate as directed in the Identification, Method 1 under the Vitamin A Assay, and perform the test: the color tone and the R_f value of the main spot from the sample solution correspond to those of the blue spot from retinol acetate from the standard solution, and no spot appears from the sample solution having the