

Dehydrocholic Acid Injection

Dehydrocholate Sodium Injection

デヒドロコール酸注射液

Dehydrocholic Acid Injection is an aqueous solution for injection. It contains not less than 95% and not more than 105% of the labeled amount of dehydrocholic acid ($C_{24}H_{34}O_5$; 402.52).

Method of preparation Dissolve Purified Dehydrocholic Acid in a solution of Sodium Hydroxide, and prepare as directed under Injections.

Description Dehydrocholic Acid Injection is a clear, colorless to light yellow liquid, and has a bitter taste.

pH: 9 - 11

Identification Transfer a volume of Dehydrocholic Acid Injection, equivalent to 0.1 g of Purified Dehydrocholic Acid according to the labeled amount, to a separator, and add 10 mL of water and 1 mL of dilute hydrochloric acid: a white precipitate is produced. Extract the mixture with three 15-mL portions of chloroform, combine all the chloroform extracts, evaporate the chloroform on a water bath, and dry the residue at 105°C for 1 hour: the residue so obtained melts between 235°C and 242°C.

Purity Heavy metals—Evaporate a volume of Dehydrocholic Acid Injection, equivalent to 1.0 g of Purified Dehydrocholic Acid according to the labeled amount, on a water bath to dryness. Proceed with the residue according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

Bacterial endotoxins Less than 0.30 EU/mg.

Assay Transfer an exactly measured volume of Dehydrocholic Acid Injection, equivalent to about 0.5 g of dehydrocholic acid ($C_{24}H_{34}O_5$), to a 100-mL separator, and add, if necessary, water to make 25 mL. Add 2 mL of hydrochloric acid, and extract with 25-mL, 20-mL and 15-mL portions of chloroform successively. Combine the chloroform extracts, wash with cold water until the washings become negative to acid, and evaporate the chloroform on a water bath. Dissolve the residue in 40 mL of neutralized ethanol and 20 mL of water by warming. Add 2 drops of phenolphthalein TS to this solution, titrate with 0.1 mol/L sodium hydroxide VS, adding 100 mL of freshly boiled and cooled water as the end point is approached, and continue the titration.

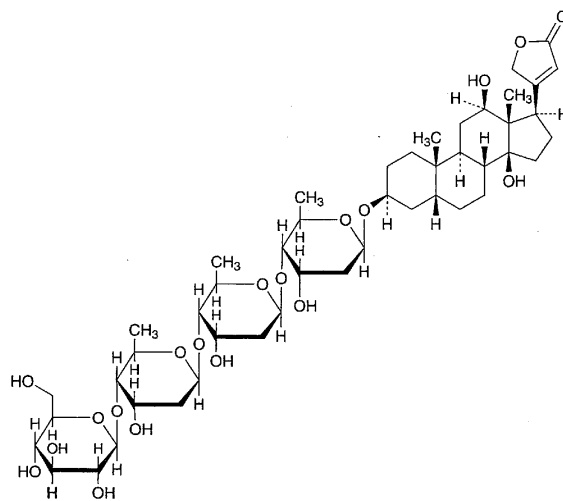
Each mL of 0.1 mol/L sodium hydroxide VS
= 40.25 mg of $C_{24}H_{34}O_5$

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

Storage—Light-resistant.

Deslanoside

デスラノシド



$C_{47}H_{74}O_{19}$: 943.08

3 β -[O- β -D-Glucopyranosyl-(1 \rightarrow 4)-O-2,6-dideoxy- β -D-ribo-hexopyranosyl-(1 \rightarrow 4)-O-2,6-dideoxy- β -D-ribo-hexopyranosyl-(1 \rightarrow 4)-2,6-dideoxy- β -D-ribo-hexopyranosyloxy]-12 β ,14-dihydroxy-5 β ,14 β -card-20(22)-enolide [17598-65-1]

Deslanoside, when dried, contains not less than 90.0% and not more than 102.0% of $C_{47}H_{74}O_{19}$.

Description Deslanoside occurs as colorless or white crystals or a white, crystalline powder. It is odorless.

It is freely soluble in anhydrous pyridine, sparingly soluble in methanol, slightly soluble in ethanol (95), and practically insoluble in water and in diethyl ether.

It is hygroscopic.

Identification Transfer 1 mg of Deslanoside to a small test tube about 10 mm in inside diameter, dissolve in 1 mL of a solution of iron (III) chloride hexahydrate in acetic acid (100) (1 in 1000), and underlay gently with 1 mL of sulfuric acid: at the zone of contact of two liquids a brown ring is produced, and the color of the upper layer near to the contact zone changes gradually to blue through purple, and the entire acetic acid layer shows a blue-green color through a deep blue color.

Purity (1) Clarity and color of solution—Dissolve 0.020 g of Deslanoside in 10 mL of ethanol (95) and 3 mL of water by warming, cool, and dilute to 100 mL with water: the solution is clear and colorless.

(2) Related substances—Dissolve 0.010 g of Deslanoside in exactly 5 mL of methanol, and use this solution as the sample solution. Dissolve 1.0 mg of Deslanoside Reference Standard in exactly 5 mL of methanol, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 20 μ L each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, methanol and water (84:15:1) to a distance of about 13 cm,

and air-dry the plate. Spray evenly dilute sulfuric acid on the plate, and heat the plate at 110°C for 10 minutes: the spots other than the principal spot from the sample solution are not larger than and not more intense than the spot from the standard solution.

Optical rotation $[\alpha]_D^{20}$: +6.5 – +8.5° (after drying, 0.5 g, anhydrous pyridine, 25 mL, 100 mm).

Loss on drying Not more than 8.0% (0.5 g, in vacuum, phosphorus (V) oxide, 60°C, 4 hours).

Residue on ignition Not more than 0.5% (0.1 g).

Assay Dissolve about 0.012 g each of Deslanoside and Deslanoside Reference Standard, previously dried and accurately weighed, in 20 mL each of methanol, add water to make exactly 100 mL, and use these solutions as the sample solution and the standard solution, respectively. Pipet 5 mL each of these solutions, transfer to light-resistant, 25-mL volumetric flasks, shake well with 5 mL each of 2,4,6-trinitrophenol TS and 0.5 mL each of a solution of sodium hydroxide (1 in 10), add diluted methanol (1 in 4) to make 25 mL, and allow to stand at a temperature between 18°C and 22°C for 25 minutes. Determine the absorbances, A_T and A_S , of the subsequent solutions of the sample solution and the standard solution, respectively, at 485 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared with 5 mL of diluted methanol (1 in 5) in the same manner as the blank.

$$\begin{aligned} \text{Amount (mg) of } C_{47}H_{74}O_{19} \\ = \text{amount (mg) of Deslanoside Reference Standard} \\ \times \frac{A_T}{A_S} \end{aligned}$$

Containers and storage Containers—Tight containers.

Deslanoside Injection

デスラノシド注射液

Deslanoside Injection is an aqueous solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of deslanoside ($C_{47}H_{74}O_{19}$: 943.08).

Method of preparation Dissolve Deslanoside in 10 vol% ethanol and prepare as directed under Injections. It may contain Glycerin. It may be prepared with a suitable amount of Ethanol and Water for Injection.

Description Deslanoside Injection is a clear and colorless liquid.

pH: 5.0 – 7.0

Identification (1) Place a volume of Deslanoside Injection, equivalent to 2 mg of Deslanoside according to the labeled amount, in a separator, add sodium chloride in the ratio of 0.2 g to each mL of this solution, and extract with three 10-mL portions of chloroform. Combine the chloroform extracts, mix uniformly, pipet 15 mL of this solution, and evaporate the chloroform under reduced pressure. Proceed with the residue as directed in the Identification under Deslanoside.

(2) Place a volume of Deslanoside Injection, equivalent to 1 mg of Deslanoside according to the labeled amount, in a separator, add sodium chloride in the ratio of 0.2 g to each mL of this solution, and extract with three 5-mL portions of chloroform. Combine the chloroform extracts, evaporate the chloroform under reduced pressure, dissolve the residue in 5 mL of methanol, and use this solution as the sample solution. Separately, dissolve 1 mg of Deslanoside Reference Standard in 5 mL of methanol, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 20 μ L each of these solutions on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, methanol and water (84:15:1) to a distance of about 13 cm, and air-dry the plate. Spray evenly dilute sulfuric acid upon the plate, and heat the plate at 110°C for 10 minutes: the spots from the sample solution and the standard solution show a black color and have the same R_f value.

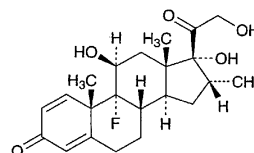
Assay Measure exactly a volume of Deslanoside Injection, equivalent to about 3 mg of deslanoside ($C_{47}H_{74}O_{19}$). Add 5 mL of methanol and water to make 25 mL. Use this solution as the sample solution, and proceed as directed in the Assay under Deslanoside.

$$\begin{aligned} \text{Amount (mg) of deslanoside (} C_{47}H_{74}O_{19} \text{)} \\ = \text{amount (mg) of Deslanoside Reference Standard} \\ \times \frac{A_T}{A_S} \times \frac{1}{4} \end{aligned}$$

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

Dexamethasone

デキサメタゾン



$C_{22}H_{29}FO_5$: 392.46

9-Fluoro-11 β ,17,21-trihydroxy-16 α -methylpregna-1,4-diene-3,20-dione [50-02-2]

Dexamethasone, when dried, contains not less than 97.0% and not more than 102.0% of $C_{22}H_{29}FO_5$.

Description Dexamethasone occurs as white to pale yellow crystals or crystalline powder. It is odorless.

It is sparingly soluble in methanol, in ethanol (95), in acetone and in 1,4-dioxane, and practically insoluble in water and in diethyl ether.

Melting point: about 245°C (with decomposition).

Identification (1) Dissolve 2 mg of Dexamethasone in 40 mL of ethanol (95), add 5 mL of 2,6-di-*tert*-butylcresol TS and 5 mL of sodium hydroxide TS, and heat under a reflux condenser on a water bath for 20 minutes: a green color develops.

(2) Dissolve 0.01 g of Dexamethasone in 1 mL of