

and 50 cm in length, packed with gel type strong acid ion-exchange resin for liquid chromatography (degree of crosslinkage: 6%) (11 μ m in particle diameter).

Column temperature: A constant temperature of about 75°C.

Mobile phase: Water.

Flow rate: Adjust the flow rate so that the retention time of lactulose is about 18 minutes.

System suitability—

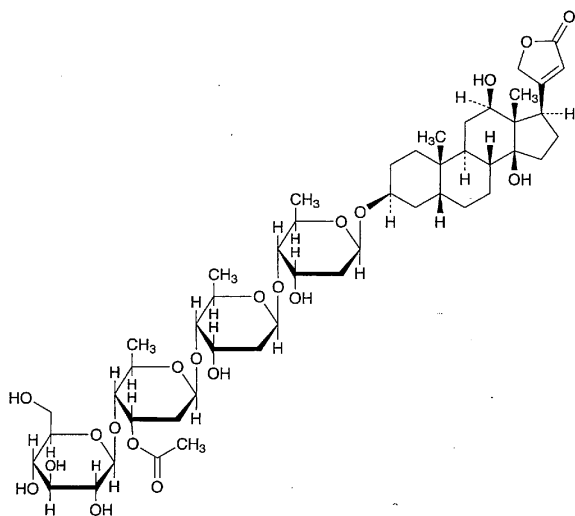
System performance: When the procedure is run with 10 μ L of the standard solution under the above operating conditions, lactulose and the internal standard are eluted in this order with the resolution between these peaks being not less than 8.

System repeatability: When the test is repeated 6 times with 20 μ L of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak heights of lactulose, galactose and lactose to the height of the internal standard are not more than 2.0%, respectively.

Containers and storage Containers—Tight containers.

Lanatoside C

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$C_{49}H_{76}O_{20}$: 985.12

3 β -[O- β -D-Glucopyranosyl-(1 \rightarrow 4)-O-3-acetyl-2,6-dideoxy- β -D-ribohexopyranosyl-(1 \rightarrow 4)-O-2,6-dideoxy- β -D-ribohexopyranosyloxy]-12 β ,14-dihydroxy-5 β ,14 β -card-20(22)-enolide [17575-22-3]

Lanatoside C, when dried, contains not less than 90.0% and not more than 102.0% of $C_{49}H_{76}O_{20}$.

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

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

It is hygroscopic.

Identification Place 1 mg of Lanatoside C to a small test tube having an internal diameter of about 10 mm, dissolve in 1 mL of a solution of iron (III) chloride hexahydrate in acetic acid (100) (1 in 10,000), and underlay gently with 1 mL of sulfuric acid: at the zone of contact of the two liquids, a brown ring is produced, and the color of the upper layer near the contact zone gradually changes to blue through purple. Finally the color of the entire acetic acid layer changes to blue-green through deep blue.

Purity Related substances—Dissolve 0.010 g of Lanatoside C in exactly 5 mL of methanol, and use this solution as the sample solution. Separately, dissolve 1.0 mg of Lanatoside C Reference Standard in exactly 5 mL of methanol, and use this solution as the standard solution. Perform the test as directed under the Thin-layer Chromatography with the sample solution and the standard solution. 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: any spots other than the principal spot from the sample solution are neither larger nor darker than the spot from the standard solution.

Optical rotation $[\alpha]_D^{20}$: +32 – +35° (after drying, 0.5 g, methanol, 25 mL, 100 mm).

Loss on drying Not more than 7.5% (0.5 g, in vacuum, phosphorus (V) oxide, 60°C, 4 hours).

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

Assay Weigh accurately about 0.05 g each of Lanatoside C and Lanatoside C Reference Standard, previously dried, and dissolve in methanol to make exactly 25 mL. Pipet 5 mL each of these solutions, add methanol to make exactly 100 mL, and use these solutions as the sample solution and the standard solution, respectively. Pipet 5 mL each of the sample solution and the standard solution into 25-mL light-resistant, volumetric flasks, and add 5 mL of 2,4,6-trinitrophenol TS and 0.5 mL of a solution of sodium hydroxide (1 in 10), shake well, and add methanol to make 25 mL. Allow these solutions to stand between 18°C and 22°C for 25 minutes, and determine the absorbances, A_T and A_S , of the solutions at 485 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared with 5 mL of methanol in the same manner as the blank solution.

$$\begin{aligned} & \text{Amount (mg) of } C_{49}H_{76}O_{20} \\ &= \text{amount (mg) of Lanatoside C Reference Standard} \\ & \quad \times \frac{A_T}{A_S} \end{aligned}$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Lanatoside C Tablets

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Lanatoside C Tablets contain not less than 90%