Lanoconazole Cream

ラノコナゾールクリーム

Lanoconazole Cream contains not less than 95.0% and not more than 105.0% of the labeled amount of lanoconazole ($C_{14}H_{10}ClN_3S_2$: 319.83).

Method of preparation Prepare as directed under Creams, with Lanoconazole.

Identification Warm Lanoconazole Cream to soften, if necessary. To a quantity of Lanoconazole Cream, equivalent to 50 mg of Lanoconazole, add 10 mL of diluted hydrochloric acid (1 in 6) saturated with sodium chloride, previously warmed, shake vigorously for 15 minutes to disperse, and centrifuge. Filter the supernatant liquid, wash the residue with 1.5 mL of diluted hydrochloric acid (1 in 6) saturated with sodium chloride, filter, and combine the washing and the filtrate. To this solution add 2.5 g of sodium hydrogen carbonate to dissolve, and extract with 10 mL of diethyl ether. Wash the diethyl ether layer with three 10-mL portions of water, and dry under reduced pressure. Dissolve the residue in 15 mL of acetone, and use this solution as the sample solution. Separately, dissolve 10 mg of lanoconazole in 10 mL of acetone, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography *<2.03>*. Spot 10 μL each of the sample solution and standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, toluene, methanol and ammonia solution (28) (400:400:20:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the principal spot obtained from the sample solution and the spot from the standard solution show the same *R*f value.

Assay Conduct this procedure using light-resistant vessels. Weigh accurately a quantity of Lanoconazole Cream, equivalent to about 15 mg of lanoconazole ($C_{14}H_{10}ClN_3S_2$), add 80 mL of methanol, sonicate to disperse, and add exactly 10 mL of the internal standard solution. Add methanol to make 100 mL, filter through a membrane filter with a pore size of 0.45 μm if necessary, and use this solution as the sample solution. Separately, weigh accurately about 15 mg of Lanoconazole RS, previously dried at 105℃ for 2 hours, dissolve in methanol, and add exactly 10 mL of the internal standard solution. Add methanol to make 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and standard solution as directed under Liquid Chromatography *<2.01>* according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of lanoconazole to that of the internal standard.

$$\text{Amount (mg) of lanoconazole } (C_{14}H_{10}ClN_3S_2) = M_S \times Q_T / Q_S$$

M_S: Amount (mg) of Lanoconazole RS taken

Internal standard solution—A solution of diisopropyl 1,3-dithiolan-2-ylidenemalonate in methanol (1 in 1000).

Operating conditions—

Proceed as directed in the operating conditions in the Assay under Lanoconazole.

System suitability—

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, lanoconazole and the internal standard are eluted in this order with the resolution between these peaks being not less than 3.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratio of the peak area of lanoconazole to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Add the following to 9.01 Reference Standards (1):

Lanoconazole RS

Add the following to 9.41 Reagents, Test Solutions:

Diisopropyl 1,3-dithiolan-2-ylidenemalonate

$C_{12}H_{18}O_4S_2$ White crystals.

Identification—Determine the absorption spectrum of a solution of diisopropyl 1,3-dithiolan-2-ylidenemalonate in methanol (1 in 125,000) as directed under Ultraviolet-visible Spectrophotometry *<2.24>*: it exhibits a maximum between 304 nm and 308 nm.

Melting point <2.60> 54 – 57℃

Lanoconazole $C_{14}H_{10}ClN_3S_2$ [Same as the namesake monograph]