

1 **Lanconazole Ointment**

2 ラノコナゾール軟膏

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4 Lanconazole Ointment contains not less than
5 93.0% and not more than 107.0% of the labeled
6 amount of lanconazole (C₁₄H₁₀ClN₃S₂: 319.83).

7 **Method of preparation** Prepare as directed under Oint-
8 ments, with Lanconazole.

9 **Identification** To a quantity of Lanconazole Ointment,
10 equivalent to 50 mg of Lanconazole, add 15 mL of hexane,
11 sonicate to disperse, add 10 mL of methanol, and shake for
12 10 minutes. Centrifuge this solution, discard the hexane
13 layer, and take the methanol layer. Wash the residue with a
14 small amount of methanol if necessary, and combine the
15 washing with the methanol layer. Dry the combined meth-
16 anol layer under reduced pressure, dissolve the residue in
17 40 mL of acetone, and use this solution as the sample solu-
18 tion. Separately, dissolve 10 mg of lanconazole in 10 mL
19 of acetone, and use this solution as the standard solution.
20 Perform the test with these solutions as directed under
21 Thin-layer Chromatography <2.03>. Spot 10 μL each of the
22 sample solution and standard solution on a plate of silica
23 gel with fluorescent indicator for thin-layer chromatog-
24 raphy. Develop the plate with a mixture of ethyl acetate,
25 toluene, methanol and ammonia solution (28)
26 (400:400:20:1) to a distance of about 15 cm, and air-dry the
27 plate. Examine under ultraviolet light (main wavelength:
28 254 nm): the principal spot obtained from the sample solu-
29 tion and the spot from the standard solution show the same
30 R_f value.

31 **Assay** Conduct this procedure using light-resistant ves-
32 sels. Weigh accurately a quantity of Lanconazole Oint-
33 ment, equivalent to about 15 mg of lanconazole
34 (C₁₄H₁₀ClN₃S₂), add 20 mL of tetrahydrofuran, sonicate to
35 disperse, and add exactly 10 mL of the internal standard
36 solution. Add methanol to make 100 mL, and use this solu-
37 tion as the sample solution. Separately, weigh accurately
38 about 15 mg of Lanconazole RS, previously dried at
39 105°C for 2 hours, dissolve in methanol, and add exactly
40 10 mL of the internal standard solution. Add methanol to
41 make 100 mL, and use this solution as the standard solution.
42 Perform the test with 10 μL each of the sample solution and
43 standard solution as directed under Liquid Chromatog-
44 raphy <2.01> according to the following conditions, and
45 calculate the ratios, Q_T and Q_S, of the peak area of lancon-
46 azole to that of the internal standard.

$$47 \quad \text{Amount (mg) of lanconazole (C}_{14}\text{H}_{10}\text{ClN}_3\text{S}_2) \\ 48 \quad = M_S \times Q_T / Q_S$$

49 M_S : Amount (mg) of Lanconazole RS taken

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

52 *Operating conditions*—

53 Proceed as directed in the operating conditions in the
54 Assay under Lanconazole.

55 *System suitability*—

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

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

66 **Containers and storage** Containers—Tight containers.

67 Storage—Light-resistant.

68 **Add the following to 9.01 Reference**
69 **Standards (1):**

70 **Lanconazole RS**

71 **Add the following to 9.41 Reagents,**
72 **Test Solutions:**

73 **Diisopropyl 1,3-dithiolan-2-ylidenemalonate**

74 C₁₂H₁₈O₄S₂ White crystals.

75 *Identification*—Determine the absorption spectrum of a
76 solution of diisopropyl 1,3-dithiolan-2-ylidenemalonate in
77 methanol (1 in 125,000) as directed under Ultraviolet-visi-
78 ble Spectrophotometry <2.24>: it exhibits a maximum be-
79 tween 304 nm and 308 nm.

80 *Melting point* <2.60> 54 – 57°C

81 **Lanconazole** C₁₄H₁₀ClN₃S₂ [Same as the name-
82 sake monograph]

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