Pranlukast for Syrup

シロップ用プランルカスト

Pranlukast for Syrup is a preparation for syrup, which is suspended before use.

It contains not less than 95.0% and not more than 105.0% of the labeled amount of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$: 490.51).

Method of preparation Prepare as directed under Syrups, with Pranlukast Hydrate.

Identification To an amount of Pranlukast for Syrup, equivalent to 10 mg of Pranlukast Hydrate, add 100 mL of ethanol (99.5), shake thoroughly, and centrifuge. To 1 mL of the supernatant liquid add ethanol (99.5) to make 10 mL. Determine the absorption spectrum of this solution as directed under Ultraviolet-visible Spectrophotometry *<2.24>*: it exhibits a maximum between 256 nm and 260 nm, and a shoulder between 310 nm and 318 nm.

Uniformity of dosage units *<6.02>* Perform the test according to the following method: Pranlukast for Syrup in single-dose packages meets the requirement of the Content uniformity test.

To the total amount of the content of 1 package of Pranlukast for Syrup, add 25 mL of dimethylsulfoxide, shake, and add acetonitrile to make exactly 100 mL. After allowing to stand, pipet V mL of the supernatant liquid, equivalent to 2 mg of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$), add exactly 5 mL of the internal standard solution, add a mixture of acetonitrile and dimethylsulfoxide (3:1) to make 10 mL, and use this solution as the sample solution. Then, proceed as directed in the Assay.

Amount (mg) of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$)
$= M_S \times Q_T / Q_S \times 1 / V \times 10 \times 1.0187$

M_S: Amount (mg) of Pranlukast RS taken, calculated on the anhydrous basis

Internal standard solution—A solution of isoamyl parahydroxybenzoate in a mixture of acetonitrile and dimethylsulfoxide (3:1) (1 in 2500).

Dissolution *<6.10>* When the test is performed at 50 revolutions per minute according to the Paddle method, using 900 mL of a solution, prepared by dissolving 1 g of polysorbate 80 in 2nd fluid for dissolution test to make 2000 mL, as the dissolution medium, the dissolution rate in 30 minutes of Pranlukast for Syrup is not less than 70%.

Start the test with an accurately weighed amount of Pranlukast for Syrup, equivalent to about 0.1 g of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$), withdraw not less than 10 mL of the medium at the specified minute after starting the test, and filter through a membrane filter with a pore size not exceeding 0.45 μm. Discard not less than 5 mL of the first filtrate, pipet 2 mL of the subsequent filtrate, add the dissolution medium to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 10 mg of Pranlukast RS (separately, determine the water *<2.48>* in the same manner as Pranlukast Hydrate), and dissolve in a solution, prepared by dissolving 1 g of polysorbate 80 in 2nd fluid for dissolution test to make 100 mL, to make exactly 100 mL. Pipet 5 mL of this solution, add 2nd fluid for dissolution test to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, at 260 nm of the sample solution and standard solution as directed under Ultraviolet-visible Spectrophotometry *<2.24>*.

Dissolution rate (%) with respect to the labeled amount of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$)
$= M_S / M_T \times A_T / A_S \times 1 / C \times 1125 \times 1.0187$

M_S: Amount (mg) of Pranlukast RS taken, calculated on the anhydrous basis
M_T: Amount (g) of Pranlukast for Syrup
C : Labeled amount (mg) of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$) in 1 capsule

Assay Weigh accurately an amount of Pranlukast for Syrup, equivalent to about 0.1 g of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$), add 25 mL of dimethylsulfoxide, shake , and add acetonitrile to make exactly 100 mL. After allowing to stand, pipet 2 mL of the supernatant liquid, add exactly 5 mL of the internal standard solution, add 3 mL of a mixture of acetonitrile and dimethylsulfoxide (3:1), and use this solution as the sample solution. Separately, weigh accurately about 20 mg of Pranlukast RS (separately, determine the water *<2.48>* in the same manner as Pranlukast Hydrate), dissolve in a mixture of acetonitrile and dimethylsulfoxide (3:1) to make exactly 50 mL. Pipet 5 mL of this solution, add exactly 5 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 5 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 pranlukast to that of the internal standard.

Amount (mg) of pranlukast hydrate ($C_{27}H_{23}N_5O_4.^1/_2H_2O$)
$= M_S \times Q_T / Q_S \times 5 \times 1.0187$

M_S: Amount (mg) of Pranlukast RS taken, calculated on the anhydrous basis

Internal standard solution—A solution of isoamyl parahydroxybenzoate in a mixture of acetonitrile and dimethylsulfoxide (3:1) (1 in 2500).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 260 nm).

Column: A stainless steel column 6 mm in inside diameter and 15 cm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: A mixture of 0.02 mol/L potassium dihydrogen phosphate TS, acetonitrile and methanol (5:5:1).

Flow rate: Adjust so that the retention time of pranlukast is about 10 minutes.

System suitability—

System performance: When the procedure is run with 5 μL of the standard solution under the above operating conditions, pranlukast 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 5 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratio of the peak area of pranlukast to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Tight containers.