Government of Nepal
Ministry of Health and Population
Department of Drug Administration
National Medicines Laboratory
Quality and Method Validation Section

Analytical Profile of Linagliptin Tablets

Analytical Profile No.: Lina 073/074/ AP 012

Linagliptin Tablets contains not less than 90.0% and not more than 110.0% of the stated amount of Linagliptin.

Usual Strength: 2.5 mg, 5 mg

1. Identification:

In the Assay, the principle peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution.

2. Dissolution

2.1 Dissolution Parameters:

Apparatus: Basket

Medium: 900 ml of 0.1 N HCl

Speed and Time: 50 rpm and 45 minutes

Withdraw a suitable volume of the medium and filter.

2.2 Test Solution: Use the filtrate for 2.5 mg Linagliptin Tablets. For 5 mg Linagliptin tablet, dilute 5 ml of the resulting filtrate to 10 ml with the dissolution medium.

2.3 Reference Solution: Weigh accurately about 28 mg of linagliptin RS and transfer into 100 ml volumetric flask. Dissolve in 0.1 N HCl and make up the volume to 100 ml with same solvent. Dilute 2 ml of this solution to 20 ml with 0.1 N HCl. Again dilute 2 ml of the resulting solution to 20 ml with dissolution medium.

2.4 Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution. Calculate the percent release of Linagliptin.

2.5 Limit: Not less than 80% (Q) of the stated amount.

3. Uniformity of content:

Determine by liquid chromatography, as described in the Assay, using the following test solution.

Test Solution: Transfer 1 tablet into 100 ml volumetric flask. Add about 70 ml of 0.1N HCl, sonicate for about 10 minutes and cool the solution to room temperature and make up the volume to 100 ml with same solvent. Centrifuge the solution. **Dilute 3 ml of the resulting solution to 25 ml with 0.1N HCl for 2.5 mg linagliptin tablet. For 5 mg tablet, dilute 3 ml of the resulting solution to 50 ml with 0.1N HCl.** Prepare similarly for 9 more tablets.

4. Assay:

4.1 Test Solution: Weigh individually 20 tablets and crush the tablet to fine powder. Weigh accurately the powder equivalent to 10 mg of linagliptin and transfer into 100 ml volumetric flask. Add about 70 ml of 0.1N HCl, sonicate for about 10 minutes and cool the solution to room temperature and make up the volume to 100 ml with same solvent. Centrifuge the solution. Dilute 3 ml of the resulting solution to 100 ml with 0.1N HCl.

4.2 Reference Solution: Weigh accurately about 30 mg of linagliptin RS and transfer into 100 ml volumetric flask. Dissolve in the 0.1 M HCl and make up the volume to 100 ml with 0.1 M HCl. Dilute 2 ml of this solution to 20 ml with 0.1 M HCl. Again dilute 2 ml of the resulting solution to 20 ml with 0.1 M HCl.

4.3 Chromatographic system:

Column: 250 X 4.6 mm (C 18)

Flow rate: 1.0 ml/min

Wave length: 295 nm

Injection volume: 20 µl

Column Oven Temperature: 25˚c

Mobile Phase: 30 volume of Acetonitrile and 70 volume of 0.02 M Phosphate Buffer pH 4.0

0.02 M Phosphate Buffer pH 4.0: Weigh accurately about 2.7 g of potassium dihydrogen orthophosphate and dilute to 1000 ml with water.

4.4 Procedure: Inject the reference solution five times and test solutions. The test is not valid unless the column efficiency is not less than 2000 theoretical plates, tailing factor is not more than 2.0, and the relative standard deviation for replicate injections is not more than 2.0%. Measure the peak responses. Calculate the content of Metformin HCl and Sitagliptin.

5. Other tests: As per pharmacopoeial requirements.