ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT
DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory

Linagliptin Tablets

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

Linagliptin Tablets contain not less than 90 percent and not more than 110 percent of the stated amount of Linagliptin.

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: Determine by liquid chromatography

2.1 Dissolution Parameters:

Apparatus: Basket

Medium: 900ml of 0.1 M HCl

Speed and Time: 50 rpm and 45 minutes

Withdraw a suitable volume of the medium and filter

2.2 Test Solution: Dilute the filtrate, if necessary, with dissolution medium

2.3 Reference Solution:

Weigh accurately about 28 mg of linagliptin WS and transfer into 100 ml volumetric flask. Dissolve in solvent mixture and make up the volume to 100 ml with solvent mixture. Dilute 2 ml of this solution to 20 ml with solvent mixture. 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 content of Linagliptin.

2.6 Limit:

D. Not less than 80 % of the stated amount of Linagliptin.

3. Uniformity of content(if required):

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

3.1Test Solution:

Weigh 10 tablets individually and transfer each into 100 ml volumetric flask. Add about 70 ml of solvent mixture, sonicate for about 10 minutes and cool the solution to room temperature and make up the volume to 100 ml with solvent mixture. Centrifuge the solution. Dilute 3 ml of the resulting solution to 25 ml with solvent mixture for 2.5 mg linagliptin tablet. For 5 mg tablet, dilute 3 ml of the resulting solution to 50 ml with solvent mixture.

4. Assay: Determine by liquid chromatography

4.1 Solvent Mixture: 0.1 M HCl

4.2 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 solvent mixture, sonicate for about 10 minutes and cool the solution to room temperature and make up the volume to 100 ml with solvent mixture. Centrifuge the solution. Dilute 3 ml of the resulting solution to 100 ml with solvent mixture.

4.3 Reference Solution: Weigh accurately about 30 mg of linagliptin WS and transfer into 100 ml volumetric flask. Dissolve in solvent mixture and make up the volume to 100 ml with solvent mixture. Pipette 2 ml of this solution and dilute to 20 ml with solvent mixture. Again dilute 2 ml of the resulting solution to 20 ml with solvent mixture.

4.4 Chromatographic system:

Column: C18 (250 X 4.6 mm)

Column Temperature: 25˚c

Flow rate: 1.0 ml/min

Wavelength: 295 nm

Injection volume: 20 µl

Detector: UV

Mobile Phase:

A mixture of 30 volumes of Acetonitrile and 70 volumes 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.

4.5 Procedure:

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 theoretical plates. The tailing factor is not more than 2.0 and the relative standard deviation for replicate injections in not more than 2.0%.

Inject the reference solution and the test solution.

Calculate the content of Linagliptin in the tablets.

5. Other tests: As per pharmacopoeial requirements.