Department of Drug Administration
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

Ibandronate Sodium Tablets

Analytical Profile No.: Iban075/076/AP054

Ibandronate Sodium Tablets contain not less than 90.0 per cent and not more than 110.0 per cent of the stated amount of Ibandronic Acid.

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 of Ibandronic acid.

Tests:

2. Dissolution: Determine by liquid chromatography

2.1 Dissolution parameters

Apparatus: Paddle

Medium: 500 mlwater

Speed and Time: 50 rpm and 45 minutes

Temperature: 37+/-0.5℃

Withdraw the suitable volume of the medium and filter.

2.2 Test Solution:

Filter the sample solution promptly through filter paper of 0.2 µm pore size. Discard the first few ml of the filtrate.

2.3 Reference Solution:

Weigh accuratelyabout 16.87 mg of Ibandronate sodium WS into 100 ml volumetric flask. Add about 70 ml of water and sonicate for about 10 minutes, cool and make up the volume to 100 ml with the same solvent. Filter the solution through 0.2 µm membrane filter.

2.4 Chromatographic System

Use chromatographic system as described under assay.

2.5 Procedure:

Separately inject 20 µl of standard and sample solution and obtain the respective chromatograms. Measure the peak responses and calculate the per cent release of the drug by using following formula. Calculate the %release of Ibandronic Acid.

2.7 Limit:

D.Not less than 70.0 % of the stated amount.

3. Assay: Determine by Liquid Chromatography

3.1 Test solution:

Weigh individually 20 tablets and calculate average weight. Weigh and transfer powdered sample eq. to 28 mg of Ibandronate sodium into 100 ml volumetric flask, add 70 ml of water and sonicate for about 10 minutes with intermittent shaking, cool and make volume to 100 ml with same solvent. Filter the solution through 0.2 µm filter paper.

3.2 Reference Solution:

Weigh accurately about 28 mg of Ibandronate sodium WS (equivalent to 25mg of Ibandronic acid) in 100 ml volumetric flask. Add about 70 ml of water and sonicate for about 10 minutes, cool and make up the volume to 100 ml with same solvent. Filter the solution through 0.2 µm filter paper.

3.3 Chromatographic system

Column:	C18, 25 cm x 4.6 mm, 5 µm
Injection volume:	20 µl
Flow rate:	1.2 ml per minute
Wavelength:	195 nm
Column temperature:	Ambient
Detector:	Spectrophotometer

Mobile phase: a mixture of 95 volumes of buffer solution prepared by dissolving 1 g of 1-hexane sulphonic acid sodium salt in 1000 ml of water and 5 volumes of acetonitrile.

3.4 Procedure:

Inject reference solution and the test solution five/six times. The test is not valid unless the column efficiency determined from the major peak is not less than 2000 theoreticalplates, the tailing factor is not more than 2.0 and the relative standard deviation ofreplicate injections is not more than 2.0 %. Inject 20 µl of standard and sample solution separately and obtain the respective chromatogram. Measure the peak responses.

Calculate the content of Ibandronic Acid.

3.5 Other Tests: As per Pharmacopoieal requirements.