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

Illaprazole Enteric Coated Tablets

Analytical Profile No.: ILL 074/075/AP 027

Illaprazole enteric coated tablets contain not less than 90 per cent and not more than 110 per cent of the stated amount of Illaprazole.

1. Identification: In the assay, the principle peak in the chromatogram obtained with the sample solution should correspond to the peak in the chromatogram obtained with the reference standard solution of Illaprazole.

2. Dissolution Test:

2.1 Acid stage:

Dissolution Parameters:

Apparatus: Paddle

Medium: 1000 ml of 0.1 M Hydrochloric acid

Speed and time: 100 rpm and 120 minutes

2.1.1 Test solution:

Withdraw a suitable volume of the medium and filter. After above mentioned procedure, remove each tablet from individual vessels and subject them to the test in buffer stage.

2.1.2 Reference solution:

Weigh accurately about 28 mg of working standard of Illaprazole and transfer into 100 ml volumetric flask. Dissolve with 0.1 N HCl and make up the volume to 100 ml with 0.1 N HCl. Pipette 2 ml of this solution and transfer into 50 ml volumetric flask and make up the volume to 50 ml with 0.1 M HCl.

2.1.3 Procedure:

Measure absorbance of both the standard and sample preparation at about 220 nm using 0.1 M HCl as blank. Calculate the percentage release of Illaprazole in acid medium.

2.1.4 Limit:

Not more than 10 per cent of the stated amount.

2.2 Buffer stage

Dissolution Parameters: Determine by UV spectroscopy.

Apparatus: Paddle

Medium: 1000 ml of tris-acetate buffer pH 8.5

Speed and time: 100 rpm and 45 minutes

2.2.2 Test solution:

Withdraw a suitable volume of the medium and filter.

2.2.3 Reference solution:

Weigh accurately about 28 mg of working standard of Illaprazole and transfer into 50 ml volumetric flask. Dissolve with methanol and make up the volume to 50 ml with methanol. Pipette 2 ml of this solution and transfer into 100 ml volumetric flask and make up the volume to 100 ml with dissolution medium.

2.2.4 Procedure:

Measure absorbance of both the standard and sample preparation at about 220 nm using dissolution medium as blank. Calculate the percentage release of Illaprazole in buffer medium.

2.2.6 Limit:

D. Not less than 75 per cent of the stated amount.

3. Uniformity of content

Determine by liquid chromatography as described under assay, using the following solution as sample solution.

3.1 Test solution:

Weigh 10 tablets individually and transfer each into 100 ml volumetric flask add about 60 ml of methanol to dissolve with the help of sonicator. Cool and make up the volume to 100 ml with methanol. Filter through 0.22 micron nylon membrane filter paper.

4. Assay: Determine by liquid chromatography

4.1 Test Solution:

Weigh individually 20 tablets and crush the tablet to fine powder. Weigh accurately the powder equivalent to 50 mg of Illaprazole and transfer into 100 ml volumetric flask. Add about 60 ml of methanol, sonicate for about 10 minutes and cool the solution to room temperature and make up the volume to 100 ml with methanol. Centrifuge the solution. Dilute 5 ml of the resulting solution to 25 ml with methanol. Filter the solution with 0.22 micron nylon membrane filter paper.

4.2 Reference solution:

Weigh accurately about 25 mg of working standard of Illaprazole and transfer into 50 ml volumetric flask. Dissolve in the methanol and make up the volume to 50 ml with methanol. Dilute 5 ml of the resulting solution to 50 ml with methanol. Filter through 0.22 micron nylon membrane filter paper.

4.3 Chromatographic system:

Column: C18, 15 cm x 4.6 mm, 5 µm

Flow rate: 1.0 ml per minute

Wavelength: 237nm

Injection volume: 10 µl

Detector: UV

Mobile phase:

A mixture of 50 volumes of buffer solution prepared by dissolving 2.727 g potassium dihydrogen orthophosphate and 1 g sodium hydroxide in 1000 ml water, and 50 volumes of acetonitrile.

4.4 Procedure:

Inject 10 µl of standard preparation five/six times. 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%. After the completion of the system suitability test parameter, inject 10 µl of each of the sample solution separately. Calculate the content of Illaprazole in each tablet.

5. Other tests: As per pharmacopoeial requirements.