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

Granisetron Mouth Dissolving Tablets

Analytical Profile No.: Grani 075/076/058

Granisetron Mouth Dissolving Tablets contain not less than 92.0 % and not more than 108.0 % of the stated amount of Granisetron.

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 Granisetron.

2. Dissolution: Determine by liquid chromatography

2.1 Dissolution parameters

Apparatus: Paddle

Medium: 500 ml Phosphate buffer pH 6.5

Speed and Time: 50 rpm and 10 minutes

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 accurately Granisetron Hydrochloride WS equivalent to 20 mg of Granisetron into 100 ml volumetric flask, add about 70 ml of diluents and sonicate for about 10 minutes to dissolve, cool at room temperature and make up the volume to 100 ml with same solvent. Dilute 1 ml of this solution 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 using injection volume 50 µl.

2.5 Procedure:

Proceed the method as described in assay method (using injection volume 50 µl) and obtain respective chromatograms. Measure the peak responses and calculate the per cent release.

2.7 Limit:

D. Not less than 75.0 % 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 20 ml volumetric flask, add about 10 ml of diluents and sonicate for 10-15 minutes, cool at room temperature and make up the volume up to mark with same solvent, mix well. Filter the solution through 0.2 µm membrane filter.

4. Assay: Determine by Liquid Chromatography

4.1 Buffer(Diluent):

Weigh 15.6 gm of Sodium dihydrogen orthophosphate (NaH_2PO_4) in 900 ml water, dissolve it, adjust pH to 2.0. Dilute with water to 1000 ml with water.

4.2 Test solution:

Weigh individually 20 tablets and crush them to fine powder. Weigh powder equivalent to 5 mg of Granisetron and transfer into 100 ml volumetric flask. Add about 70 ml of diluents and sonicate for about 10-15 minutes, cool at room temperature and make up the volume to 100 ml with same solvent. Filter the resulting solution through 0.2 µm membrane filter.

4.3 Reference Solution:

Weigh accurately Granisetron Hydrochloride WS equivalent to 50 mg of Granisetron into 50 ml volumetric flask, add about 30 ml of diluents and sonicate for about 10 minutes to dissolve, cool at room temperature and make up the volume to 50 ml with the same solvent. Dilute 1 ml of this solution to 20 ml with same solvent. Filter the solution through 0.2 µm membrane filter.

4.4 Chromatographic system

Column: C18, 15 cm x 4.6 mm, 5 µm

Flow rate: 1.2 ml per minute

Injection volume: 20 µl

Wavelength: 300nm

Column temperature: 35 ^{o}C

Detector: UV

Mobile phase: Buffer:Methanol:Tetrahydrofuran::75:24:1.1

4.5 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 theoretical plates, the tailing factor is not more than 2.0 and the relative standard deviation of replicate injections is not more than 2.0 %. Inject 20 µl test solution separately and obtain the respective chromatogram. Measure the peak responses. Calculate the content of Granisetron per tablet.

4.6: Other tests: As per pharmacopoieal requirements.