DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Granisetron Syrup

Analytical Profile No.: Grani 075/076/AP059

Granisetron Syrup contains not less than 92.0 per cent and not more than 108.0 per cent 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.

Tests:

2. pH: 4 to 5

3. wt/ml: As per manufacturer's specification

4. Microbial test:

Method as per Indian Pharmacopoeia (latest edition)

Limit:

E. coli: should be absent

Total aerobic microbial count: NMT 100 cfu/ml

Total combined molds and yeasts count: NMT 50 cfu/ml

5. Assay: *Determine by Liquid Chromatography*

5.1 Buffer: Weigh 15.6 gm of Sodium dihydrogen orthophosphate (NaH_2PO_4) in 900 ml water, dissolve it, and adjust pH to 2.0 with dilute orthophosphoric acid. Dilute with water to 1000 ml.

5.2 Test solution: Weigh about 27 gm of syrup eq. to 5 mg of Granisetron in 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.

5.3 Reference Solution: Weigh accurately Granisetron Hydrochloride reference standard 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.

5.4 Chromatographic system

Column:	C18,15 cm x 4.6 mm, 5 µm
Injection volume:	20 µl
Flow rate:	1.2 ml per minute
Wavelength:	300 nm
Column temperature:	35 °C
Detector:	UV
Mobile phase:	Buffer : Methanol : Tetrahydrofuran :: 75 : 24 : 1.1
Diluent:	Buffer

5.5 Procedure: Inject reference solution five times and test solution. 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 of standard and sample solution separately and obtain the respective chromatogram. Measure the peak responses.

6. Other tests: As per pharmacopoieal requirement.