DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Flunarizine Tablets

Analytical Profile No: Fluna 077/078/AP 090

Flunarizine Tablets contains not less than 95.0% and not more than 105.0% of the stated amount of Flunarizine.

Usual Strength: 5 mg & 10 mg

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.

Tests:

2. Dissolution:

2.1 Dissolution Parameters: *Determine by UV-Vis spectrophotometer*

Apparatus: Paddle

Medium: 900 ml of 0.1 N Hydrochloric acid

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 33 mg of Flunarizine dihydrochloride WS in 100 ml volumetric flask. Add about 70 ml of dissolution medium, sonicate to dissolve, cool to room temperature and make up the volume to 100 ml with same solvent. Further dilute 2 ml of this solution to 100 ml with same solvent.

2.4 Procedure: Measure the absorbance of the reference and test solutions at the wavelength of maxima at 253 nm using dissolution medium as blank.

Calculate the content of Flunarizine.

2.5 Limit: Not less than 70 percent (D) of the stated amount of Flunarizine.

3. Uniformity of Content

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

3.1 Test Solution: Place a tablet in a 100 ml volumetric flask, add 70 ml of mobile phase, sonicate to disperse whole tablet with intermittent shaking. Cool, make up the volume to 100 ml with same solvent and filter.

4. Assay: *Determine by liquid chromatography*

4.1 Test Solution: Transfer an accurately weighed 5 intact tablets in to 500 ml volumetric flask, add about 350 ml of mobile phase, sonicate with intermittent shaking to disperse the tablets, cool and make volume to 500 ml with same solvent. Further dilute 5 ml of this solution to 10 ml with same solvent (for 10 mg tablet).

4.2 Reference Solution: Weigh accurately about 59.0 mg of Flunarizine Dihydrochloride WS in 100 ml volumetric flask. Add about 70 ml of mobile phase, sonicate to dissolve and make up the volume to 100 ml with same solvent. Further dilute 5 ml of this solution to 50 ml with same solvent.

4.3 Chromatographic system:

Column: C18, (150 x 4.6 mm), 5 µ particle size

Flow rate: 2.0 ml/min

Wavelength: 254 nm

Injection volume: 10 µl

Detector: UV

Column temperature: 40 °C

Mobile Phase: A mixture of 45 volumes of buffer and 55 volumes of Acetonitrile

Buffer: Dissolve 6.8 g of Potassium dihydrogen orthophosphate in 1000 ml water and mix

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

Calculate the content of Flunarizine in the tablets.

5. Other tests: As per pharmacopoeial requirement.