DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Febuxostat Tablets

Analytical Profile No: Febu 073/074/ AP 017

Febuxostat Tablets contains not less than 90 % and not more than 110 % of the stated amount of Febuxostat.

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 solution of Febuxostat.

Tests:

2. Dissolution: *Determine by Liquid Chromatography*

2.1 Dissolution Parameter:

Apparatus:	Paddle
Medium:	900 ml of phosphate buffer pH 6.0
Speed and time:	75 rpm and 30 minutes
Temperature :	37˚C ± 0.5˚C

Withdraw the suitable volume of the medium and filter.

2.2 Test Solution: Use the filtrate, dilute if necessary with dissolution medium to obtain a solution having concentration similar to that of reference solution. Filter through 0.2 micron filter paper.

2.3 Reference Solution: Weigh accurately about 20 mg febuxostat RS and transfer into 50 ml volumetric flask. Add about 35 ml of methanol and dissolve by sonicating for few minutes. Allow the sample to cool to the room temperature and make up the volume to 50 ml with methanol. Dilute 2 ml of the above solution to 20 ml with the dissolution medium. Filter through 0.2 micron filter paper.

2.4 Chromatographic system: Proceed as directed under Assay

2.5 Procedure: Separately inject 20 µl of standard and sample solution and blank solution (dissolution medium) and obtain the respective chromatograms. Measure the peak responses and calculate the % release of the drug.

2.6 Limit: Not less than 70 % (D) of the stated amount.

3. Assay: Determine by Liquid Chromatography

3.1 Test Solution: Weigh individually 20 tablets and crush them into fine powder. Weigh accurately the powder equivalent to 20 mg of Febuxostat and transfer into 50 ml volumetric flask. Add about 35 ml of mobile phase and dissolve by sonicating for about 10 minutes. Allow the sample to cool to room temperature and make up the volume to 50 ml with the mobile phase. Filter or centrifuge the solution. Dilute 5 ml of the clear solution to 50 ml with mobile phase. Again filter the solution through 0.2 µm membrane filter.

3.2 Reference Solution: Weigh accurately about 20 mg febuxostat RS and transfer into 50 ml volumetric flask. Add about 35 ml of mobile phase and dissolve by sonicating for about 10 minutes. Allow the sample to cool to room temperature and make up the volume to 50 ml with the mobile phase. Dilute 5 ml of the resulting solution to 50 ml with mobile phase. Filter through 0.2 µm membrane filter.

3.3 Chromatographic system:

Column: a stainless steel column 15 cm x 4.6 mm, packed with octadecyl silane bonded to porous silica (5 µm),

Flow rate: 1.0 ml per minute,

Detector: UV

Wavelength: 220 nm

Injection volume: 20 µl

Column temperature: 30˚c

Mobile phase: a mixture of 25 volumes of 0.01 v/v orthophosphoric acid solution and 75 volumes of methanol

3.4 Procedure: Inject 20 µl of standard and sample solution separately and obtain the respective chromatogram. Measure the peak responses.

Calculate the content of febuxostat in each tablet.

4. Other tests: As per pharmacopoeial requirements.s