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

Febuxostat Tablets

Analytical Profile No.: Febu 073/074/ AP 017

Febuxostat Tablets contain 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 test solution corresponds to the peak in the chromatogram obtained with the reference solution.

2. Dissolution: Determine by liquid chromatography

2.1 Dissolution Parameters:

Apparatus: Paddle

Medium: 900 ml of Phosphate buffer pH 6.0 prepared by dissolving 6.0 g of monobasic potassium phosphate in 1000 ml of water and adjusting the pH to 6.0 ± 0.05 with sodium hydroxide solution.

Speed and Time: 75 rpm and 30 minutes

Withdraw a suitable volume of the medium and filter

2.1 Test Solution:

Dilute the filtrate, if necessary, with dissolution medium.

2.2 Reference Solution:

Weigh accurately about 20 mg of febuxostat WS 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 this solution to 20 ml with the dissolution medium.

2.3 Procedure: Use the chromatographic system as described in the Assay.

Inject the reference solution and the test solution.

Calculate the % release of Febuxostat.

2.5 Limit:

D. Not less than 70 % of the stated amount of Febuxostat.

3. Assay: Determine by liquid chromatography

3.1 Test Solution:

Weigh individually 20 tablets & crush the tablet 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 this solution to 50 ml with mobile phase.

3.2 Reference Solution:

Weigh accurately about 20 mg of febuxostat WS 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.

3.3 Chromatographic system

Column: C18, (150*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Wavelength: 220 nm

Injection volume: 20 µl

Column temperature: 30 °C

Detector: UV

Mobile phase:

Mixture of 25 volumes of Orthophosphoric acid solution and 75 volumes of Methanol

Orthophosphoric acid solution. Dilute 10 ml of the orthophosphoric acid to 1000 ml with water.

3.4 Procedure: Inject the reference solution. 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%.

Inject the reference solution and the test solution.

Calculate the content of Febuxostat in the tablets.

4. Other tests: As per pharmacopoeial requirements.