ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Fexofenadine Hydrochloride Suspension

Fexofenadine HCl suspension contains not less than 90 % and not more than 110 % of the stated amount of fexofenadine.

1. Identification:

1.1. Fexofenadine HCl

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 standard solution of Fexofenadine HCl.

2. pH

Operate the pH meter accordind to the manufacturer's instruction. Calibrate the pH meter using the standard buffer solution. Determine the pH of the syrup by using the calibrated pH meter.

Tolerance limit: 5-7

3. wt/ml

First dry the pycnometer in the hot air oven and cool it in dessicator for 5 to 10 minutes. Take the weight of the empty pycnometer together with its stopper (W_1). Fill the pycnometer with water, put the stopper, wipe it dry outside with the help of tissue paper. Now take the weight of pycnometer and water (W_2). Throw water, dry the pycnometer and fill the pycnometer with the syrup, put the stopper, wipe it dry outside with the help of tissue paper. Take the weight of pycnometer and suspension (W_3). The calculate wt/ml

wt/ml (g/ml) = $\frac{W3-W1}{W2-W1}$ **x volume of 1 g of water at various temperature**

4. Assay

4.1 Fexofenadine HCl

4.2 Acid solution: Dilute 1.7 ml of glacial acetic acid with water to 1 litre.

4.3 Buffer solution: Dilute 15 ml of a solution containing a mixture of acetonitrile and triethylamine (1:1) with acid solution to 1 litre. Adjust the pH to 5.5 with phosphoric acid.

4.4 Mobile phase: Prepare a filtered and degassed mixture of Buffer solution and acetonitrile in the ratio (64:36).

4.5 Diluent: Prepare a mixture of Acetonitrile and Acid solution (75:25)

4.6 Standard Preparation: Weigh accurately about 30 mg of working standard of Fexofenadine HCl and transfer into 100 ml volumetric flask, add about 50 ml of diluent and sonicate for about 10 minutes. Cool the solution to room temperature and make up the volume to 100 ml with

diluent. Dilute 2 ml of the resulting solution to 50 ml with mobile phase. Filter through 0.2 micron filter paper.

4.7 Test Preparation: Weigh accurately the sample equivalent to 30 mg of fexofenadine HCl and transfer into 100 ml volumetric flask. Add about 50 ml of diluent and sonicate for about 10 minutes. Cool the solution to room temperature and make up the volume to 100 ml with diluent. Centrifuge the sample solution. Dilute 2 ml of the resulting solution to 50 ml with mobile phase. Filter through 0.2 micron filter paper.

4.8 Chromatographic system:

Apparatus : HPLC

Column : (150 × 4.6) mm; 5 micron, ODS (C18)

Temperature : 35 ºC

Wavelength : 220 nm

Flow rate : 1.5 ml/ min.

Injection volume : 20 µl

4.9 Procedure: Inject the reference solution five/six times and sample solutions. 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%. Calculate the content of fexofenadine in the suspension by using the following formula.

4.10 Calculation:

$$\frac{Area\ of\ spl}{Area\ of\ Std} \times \frac{Conc.\ of\ std}{Conc.of\ spl} \times \frac{Potency\ of\ std}{100} \times \frac{100 - LOD}{100} \times 5\ x\ wt/ml$$

Note

1. Average fill volume and fill volume variation should be as per the Pharmacopoeia recognized by drug advisory committee.