ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Levocetirizine Dihydrochloride Syrup

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

1. Identification:

1.1. Levocetirizine 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 levocetirizine HCl.

2. pH

Operate the pH meter according 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: 4-6

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 Levocetirizine dihydrochloride

4.1.1 Mobile Phase: A mixture of 65 volumes of 0.05M potassium dihydrogen phosphate adjusts the pH to 6.0 with 10% w/v of sodium hydroxide and add 35 volume of acetonitrile.

4.1.2 Standard Preparation: Weigh accurately about 25.0 mg of working standard of levocetirizine dihydrochloride and transfer in 50 ml volumetric flask. Dissolve it with 50 ml mobile phase, by sonicating for about 5 minutes, cool and make up the volume with same solvent. Dilute 5ml of the resulting solution to 50 ml with mobile phase. Filter through 0.2 micron filter paper.

4.1.3 Test Preparation: Weigh accurately about the sample equivalent to 5 mg of levocetirizine dihydrochloride and transfer into 100 ml volumetric flask. by sonicating for about 5 minutes, cool and make up the volume with same solvent. Filter through 0.2 micron filter paper.

4.1.4 Chromatographic system:

Apparatus : HPLC

Column : (250 × 4.6) mm; 5 micron, ODS (C18)

Temperature : Ambient

Wavelength : 230 nm

Flow rate : 1.0 ml/ min.

Injection volume : 20 µl

4.1.5 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 levocetirizine HCl in the syrup by using the following formula.

4.1.6 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.