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

Desloratidine Tablets

Analytical Profile No.: Des 073/074/AP 004

Desloratidine Tablets contain not less than 90 % and not more than 110 % of the stated amount of Desloratidine.

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 standard solution of Desloratidine.

2. Dissolution Test:

2.1 Dissolution Parameter:

Apparatus: Paddle

Medium: 900 ml 0.1 N HCl

Speed and Time : 50 rpm and 60 minutes

2.2 Test Solution:

Place 1 tablet in each dissolution vessel and run the apparatus as per above condition and collect the sample solution from each jar at specified time. Filter the resulting solution.

2.3 Reference Solution:

Weigh accurately 27.5 mg of Desloratidine reference standard and transfer in 50 ml of volumetric flask and add 40 ml of dissolution medium and sonicate for 5 minutes, allow cooling at room temperature and make up the final volume with same media. Further dilute 5ml of the

solution to 50 ml with the dissolution media. Again dilute 5 ml of the resulting solution to 50 ml with the dissolution medium.

2.4 Procedure:

Measure the absorbance of the standard and sample solution at about 280 nm using 0.1 N HCl as blank.

Calculate the % release of the drug in each tablet.

2.6 Limit:

D. Not less than 80% of the stated amount

3. Assay: Determine by liquid chromatography

3.1 Test Solution:

Weigh individually 20 tablets and crush the tablet to fine powder. Weigh accurately the powder equivalent to 25 mg of the Desloratidine and transfer into 50 ml volumetric flask. Add about 30 ml of methanol dissolve by sonicating for about 5 minutes, cool to room temperature and make up the volume to 50 ml with mobile phase. Centrifuge the resulting solution. Dilute 5 ml of the resulting solution to 25 ml with mobile phase.

3.2 Reference Solution:

Weigh accurately 25 mg of Desloratidine reference standard and transfer in 50 ml volumetric flask, add 10 ml of methanol and dissolve by sonicating for about 5 minutes, cool to room temperature and make up the volume to 50 ml with mobile phase. Centrifuge the resulting solution. Dilute 5 ml of the resulting solution to 25 ml with mobile phase.

3.3 Chromatographic Condition:

Column: C18 (25 cm X 4.6 mm)

Flow rate: 1.0 ml/min

Wavelength: 278 nm

Injection volume: 20 µl

Column Temperature: 35˚ C

Detector: UV

Mobile Phase:

80 volume of 0.1 % Triethylamine in water pH adjusted to 2.5 with ortho phosphoric acid and 20 volume of Acetonitrile. Mix the solution and cool to room temperature and filter the solution through 0.2 micron filter paper using vacuum pump.

3.4 Procedure:

Inject 5 µl of standard solution of Desloratidine as per above mentioned chromatographic condition. In the chromatogram obtained from the standard preparation, the column efficiency determined from the major peak should not be less than 2000 theoretical plates, the tailing factor should be not more than 2.0 and the relative standard deviation of five replicate injections should be not more than 2.0 %. Inject 5 µl of the sample preparation and chromatograph as per above mentioned chromatographic condition.

Calculate the content of Desloratidine HCL per tablet.

4. Uniformity of content (if required):

4.1 Test Solution:

Weigh 10 tablets individually and place one tablet individually in 50 ml volumetric flask, add about 30 ml of mobile phase. Dissolve by sonicating for about 10 minutes and make up the volume to 50 ml with mobile phase. Filter the solution through 0.2 µm filter paper.

4.2 Reference Solution:

Weight about 26.1 mg of Desloratidine reference standard in a 50 ml volumetric flask, add about 30 ml of mobile phase and sonicate for about 5 minutes. Cool at room temperature and make up the volume to mark with same solvents. Dilute 5 ml of this solution to 25 ml with mobile phase and filter the solution through 0.2 µm filter paper.

4.3 Chromatographic condition: Same as Assay

4.4 Procedure:

Proceed as described in assay, using 5 µl injection volumes.

Calculate the content of Desloratidine HCL per tablet.

5. Other tests: As per pharmacopoeial requirement