ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Desloratidine Tablets

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

1. Identification:

1.1. Desloratidine:

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: Desloratidine

2.1 Dissolution Parameter:

2.1.1 Medium : 900 ml 0.1 N HCL
2.1.2 Apparatus : Paddle
2.1.3 Rotation : 50 RPM
3.1.4 Temperature : 37˚C ± 0.5˚C
3.1.5 Time : 60 minutes

2.1.6. Dissolution Medium Preparation:

Dissolve 51 ml of concentrated Hydrochloric acid in 6000 ml of water.

2.1.7 Standard Preparation:

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.

2.1.8. Sample preparation

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.

2.1.9. 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 by using following formula:

2.1.10 Calculation:

Desloratidine (%):

$$= \frac{Abs\ of\ spl}{Abs\ of\ std} \times \frac{conc.of\ std}{conc.of\ spl} \times \frac{100 - Std.LOD\ /\ water}{100} \times std\ potency\ \% \times 100\ \%$$

2.1.11 Tolerance Limit: Not less than 80 % D of the stated amount

3. Assay: Desloratidine

3.1 Chromatographic Condition:

Column	: C18 (25 cm X 4.6 mm)
Temperature	: 35˚ C
Detector	: 278 nm
Flow rate	: 1.0 ml/min
Injection volume	: 20 µl

3.2 Mobile Phase:

80 volume of 0.1 % Triethylamine in water pH adjusted to 2.5 with othophosphoric 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.

3.3 Diluent:

Same as mobile phase.

3.4 Method of Analysis

3.4.1 Standard preparation:

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 diluent. Centrifuge the resulting solution. Dilute 5 ml of the resulting solution to 25 ml with diluent.

3.4.2 Sample preparation:

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 diluent. Centrifuge the resulting solution. Dilute 5 ml of the resulting solution to 25 ml with diluent.

3.4.3 System suitability:

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 not more be than 2.0 %. Inject 5 µl of the sample preparation and chromatograph as per above mentioned chromatographic condition. Calculate the result from the formula given below.

3.4.1.4 Calculation:

Desloratidine HCL per tablet:

$$=\frac{Area\ of\ spl}{Area\ of\ Std}\times\frac{Conc.\ of\ std}{Conc.of\ spl}\times\frac{Potency\ of\ std}{100}\times\frac{100-Water\ \%}{100}\times Average\ Wt.$$

4. Uniformity of content:

4.1 Desloratidine:

4.1.1 Standard preparation:

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

4.1.2 Sample preparation:

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

4.1.3 Mobile phase: Same as Assay

4.1.4 Chromatographic condition: Same as Assay

4.1.5 Procedure:

Proceed the process as described in assay method, using 5 µl injection volumes and calculate uniformity of content using the formula given below.

4.1.6 Calculation:

Desloratidine % per tablet:

$$=\frac{Area\ of\ spl}{Area\ of\ Std}\times\frac{Conc.\ of\ std}{Conc.of\ spl}\times\frac{Potency\ of\ std}{100}\times\frac{100-Water\ \%}{100}\times\frac{Average\ Wt}{Label\ Claim}\times 100\ \%$$

Note

1. Weight variation and friability test should be as per the Pharmacopoeia recognized by Government of Nepal.