DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

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 test solution should correspond to the peak in the chromatogram obtained with the reference solution of Desloratidine.

Tests:

2. Dissolution Test:

2.1 Dissolution Parameters:

Apparatus:	Paddle
Medium:	900 ml 0.1 N HCl
Speed and time:	50 rpm and 60 minutes
Temperature :	37˚C ± 0.5˚C

Withdraw the suitable volume of the medium and filter.

2.2 Test Solution*:* Use the filtrate.

2.3 Reference Solution: Weigh accurately 27.5 mg of Desloratidine reference standard and transfer in 50 ml of volumetric flask, 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 5 ml 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. (5.5 ppm)

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.

2.5 Limit: Not less than 80% (D) of the stated amount

3. Uniformity of content: *Determine by Liquid Chromatography*

3.1 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.

3.2 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 solvent. Dilute 5 ml of this solution to 25 ml with mobile phase and filter the solution through 0.2 µm filter paper.

3.3 Chromatographic system: Proceed as directed under Assay.

3.4 Procedure: Proceed as directed under Assay using 5 µl injection volumes. Calculate the content of Desloratadine in each tablet.

3.5 Limit: 85 – 115 % of the stated amount.

4. Assay: *Determine by Liquid Chromatography*

4.1 Test solution: Weigh individually 20 tablets and crush them into fine powder. Weigh accurately the powdered sample equivalent to 25 mg of the Desloratidine and transfer into 50 ml volumetric flask. Add about 30 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 supernatant solution to 25 ml with mobile phase. Filter the resulting solution through 0.2 µm membrane filter paper.

4.2 Reference solution: Weigh accurately about 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. Filter the final solution through 0.2 µm membrane filter.

4.3 Chromatographic system:

Column: a stainless steel column 25 cm x 4.6 mm, C18 (5 µm)

Flow rate: 1.0 ml per minute,

Detector: 278 nm

Injection volume: 20 µl

Column temperature: 35˚ C

Mobile phase: a mixture of 80 volumes of 0.1 % triethylamine in water adjusted to pH 2.5 with orthophosphoric acid and 20 volumes of Acetonitrile.

4.4 Procedure: Inject 20 µl of standard preparation five times. 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 %. After the completion of the system suitability test parameter, inject 20 µl of each of the sample solution separately. Inject blank solution to check any interference and perform bracketing of standard preparation after injecting test solution. Calculate the content of Desloratadine in each tablet.

5 Other tests: As per pharmacopoeial requirements.