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NOTE

RP-HPLC Estimation of Desloratadine in Pharmaceutical Dosage Form

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A simple, rapid, sensitive and precise HPLC method has been developed for the estimation of desloratadine in pharmaceutical dosage form. In this method RP-C_{18} column (250 mm × 4.6 mm i.d., 5 µm particle size) with mobile phase consisting of acetonitrile, 0.05 M phosphate buffer and methanol in the ratio of 48:45:7 v/v/v in isocratic mode was used. The detection wavelength is 247 nm and the flow rate is 0.8 mL/min. In the range of 20-100 µg/mL, the linearity of desloratadine shows a correlation coefficient of 0.998. The percentage recovery ranges from 99.98-100.30 %. The proposed method was validated by determining sensitivity, accuracy, precision and linearity.

Key Words: Desloratadine, HPLC, Validation.

Desloratadine is an orally administered nonsedative, long acting antihistaminic with selective H_1-receptor antagonistic activity. Desloratadine is an active metabolite of loratadine. Chemically[1] desloratadine is 8-chloro-6,11-dihydro-11-(4-piperidinylidene)-5*H*-benzo[5,6]cyclohepta[1,2-b]pyridine. Literature survey reveals that various HPLC methods[2-10] have been reported for the estimation of desloratadine in pharmaceutical dosage forms. The present method is simple, rapid, sensitive, accurate and precise HPLC method for the determination of desloratadine in bulk as well as in tablet dosage form.

The separation was carried out on isocratic HPLC system (Shimadzu) with Shimadzu Binary HPLC pump, Shimadzu LC-10AT UV-Visible Detector, Spinchrom software and RP-C_{18} column (250 mm × 4.6 mm i.d.; particle size 5 µm).

HPLC conditions: The mobile phase consisting of acetonitrile (HPLC grade), 0.05 M phosphate (KH_2PO_4) buffer (pH adjusted to 3.0 with orthophosphoric acid) of AR grade and methanol (HPLC grade) were filtered through 0.45 µ membrane filter before use, degassed and were pumped from the solvent reservoir in the ratio

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of 48:45:7 v/v/v was pumped into the column at a flow rate of 0.8 mL/min. The detection was monitored at 247 nm and the run time was 7 min. The volume of injection loop was 20 µL prior to injection of the drug solution. The column was equilibrated for atleast 0.5 h. with the mobile phase flowing through the system.

Procedure: Stock solution of desloratadine was prepared by dissolving 50 mg of desloratadine in 50 mL standard volumetric flask containing 50 mL of methanol. 5 mL of the above solution was transferred to 50 mL volumetric flask and the volume was made up to the mark with mobile phase. Subsequent dilutions of this solution were made with mobile phase to get concentration of 20-100 µg/mL. The solutions were injected into the 20 µL loop and the chromatogram was recorded. The calibration curve was constructed by plotting concentration *vs.* peak area ratio. The linearity experiment was carried out in triplicate to ascertain accuracy and precision of the method.

Assay: Twenty tablets were weighed accurately and powdered. A quantity equivalent to 50 mg of desloratadine was weighed accurately and transferred to 50 mL volumetric flask. About 30 mL of methanol was added and kept in ultrasonic bath for 15 min. This solution is filtered through a membrane filter and the volume was made up to the mark to get 1 mg/mL concentration. From the above solution 5 mL was transferred to 50 mL volumetric flask and the volume was made upto 50 mL with mobile phase. From this solution, further dilutions were made to obtain concentration range of 20-100 µg/mL. 20 µL of the sample solution was injected under the chromatographic conditions and the chromatogram was recorded. The amount of desloratadine present in tablet formulation was determined by comparing the peak area from the standard. The results are presented in Table-1.

TABLE-1
VALIDATION SUMMARY

System suitability	Results
Linearity range (µg/mL)	20-100
Correlation coefficient	0.998
Assymmetry factor	1.17
Theoretical plates (N)	8181
LOD (µg/mL)	0.00721
LOQ (µg/mL)	0.0210
Percentage recovery (accuracy)	99.8

Validation of proposed method: Selectivity of the method was assessed on the basis of elution of desloratadine using the above mentioned chromatographic conditions. Precision was ascertained by the determination of intra-day and inter-day variabilities. To study the accuracy, reproducibility, precision of the proposed method, recovery studies were carriedout in triplicate by adding a known quantity of the sample was added to placebo and the percentage recovery was calculated. The results are presented in Table-2.

TABLE-2
ASSAY AND RECOVERY STUDIES

Formulation	Label claim (mg)	Amount found (mg)	Amount found* (%)	Recovery (%)	RSD (%)
Brand-1	5	4.96	99.26	99.93	0.601
Brand-2	5	4.95	99.14	99.99	0.377

*Mean of five determinations.

By applying the proposed method, the retention time of desloratadine was found to be 3.5 min. Linearity range was observed in concentration range of 20-100 µg/mL. The regression equation of desloratadine concentration over its peak area ratio was found to be Y = -34.148+14.438X (r = 0.998) where Y is the peak area ratio and X is the concentration of desloratadine (µg/mL). The assymmetry factor was found to be 1.17, which indicated assymmetric nature of peak. The number of theoretical plates was found to be 8181, which indicates efficient performance of the column. The limit of detection and limit of quantification was found to be 0.00721 and 0.0210 µg/mL, respectively indicates the sensitivity of the method. The high percentage of recovery indicates that the proposed method is highly accurate.

Conclusion

The proposed HPLC method was found to be highly accurate, sensitive and precise. Therefore this method can be applied for the routine quality control analysis of desloratadine in its tablet dosage form.

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