Original Article

SPECTROSCOPIC METHOD FOR DETERMINATION OF DESLORATADINE IN BULK AND ITS TABLET DOSAGE FORMS.

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Abstract

A simple and sensitive spectroscopic method was developed for Desloratadine in pure and its tablet dosage forms. The method has been developed by using its free soluble property in Methanol at ambient temperature. At optimum condition of the experiment the absorbance read at 242 nm. The developed method was validated and applied to the determination of Desloratadine in tablets. The linearity range for the concentration of Desloratadine was found to be 2-10µg/ml (r2 >0.996, RSD=1.8), precision (RSD=1.83) and accuracy (RSD<1.8). The LOD (0.11) and LOQ (0.33). The product remains stable up to 8 hours when kept at room temperature. The potency of Desloratadine in marketed product was determined by this method with acceptable precision and reproducibility.

Keywords: UV-Spectrophotometry, Desloratadine, Antihistaminic Drug.

Introduction

Today modern pharmaceutical analysis has got more emphasis to satisfy our query for better understanding of physiochemical properties of pharmaceutical compounds, using advanced instrumental methods. It also plays an important tool for quality assurance of pharmaceutical product throughout the shelf life. The pharmaceutical industry is under increased scrutiny to constrain costs and yet consistently deliver to market safe, efficacious products that fulfill medical needs. As a part of this, drug analysis also plays an important role. Standard analytical procedure for newer drugs or formulation may not be available in the pharmacopoeia; hence it is essential to develop a new analytical method, which is accurate, precise, specific, linear, simple & rapid[1,2].

Desloratadine is an H_1 receptor antihistaminic drug was developed on 2005. It is a metabolite of Loratadine, a second generation antihistaminic drug. Extensive literature survey, it was revealed that there were a few methods reported for estimation of desloratadine from plasma and from pharmaceutical dosage forms. Therefore here an attempt was made to develop simple, cost effective and accurate spectroscopic methods and a more precise cost effective, sensitive and specific HPLC method for desloratadine.[3,4]

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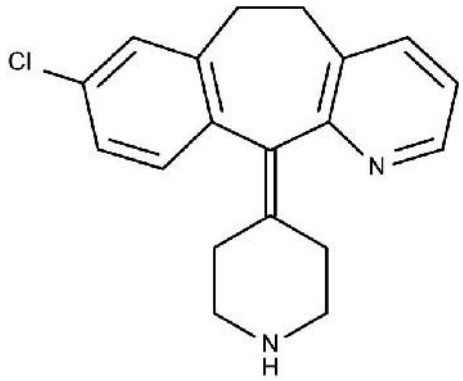

Structure of Desloratadine

Desloratadin[5] is a second generation tri cyclic anti histaminic drug which has a selective and peripheral H_1 antagonist action. It is active form of prodrug Loratadine. It is chemically 8-chloro-6, 11- di hydro-11(4-piperdinylidene)-5H (5,6 cycloheptyal (1,2-b) pyridine. Molecular weight is 310.84 gm/mol and white to off white powder. It is mainly used in the treatment of allergic disorder and in common cold. Sedation and dryness of mouth are common adverse drug reaction.

Caglar[6] has studied the sensitive spectrophotometric method for determination of Desloratadine in tablets. A simple, rapid and sensitive visible spectrophotometric method was developed for the first time for the analysis of Desloratadine in tablets. The method is based on deep blue colored radical anion formed by interaction of the drug with 7-7-8,-8 tetra acyanoquinodimethane in acetonitrile at ambient temperature.

Materials and Methods

Instrument

Absorption spectral measurements were carried out with a UV – Visible spectrophotometer (Shimadzu Model 1700, UV probe) was employed with spectral bandwidth of 1nm and wavelength accuracy of 0.3 nm (with automatic wavelength correction with a pair of 5 cm matched quartz cells).

Materials

Desloratadine pure drug was supplied by Sun pharmaceuticals, India as gift sample and used as such. Methanol was used as a solvent.

Linearity and Calibration

From the above standard solution 2ml was transferred in to a 100ml standard flask and made up to the mark with methanol to produce 20µg /ml solution. To the series of ten, 10ml volumetric flasks aliquots of 1-10 ml of 20µg/ml solutions were taken and made up to the mark with methanol. The absorbance was measured at 242 nm against methanol as blank. The calibration curve was plotted in the concentration range of 2- 20µg/ml. This was shown in Table -01 & Figure-01.

Selection of solvent and λ max

The solubility of Desloratadine was determined in a variety of solvents ranging from non polar to polar. The drug was found more soluble in methanol. Some concentration of drug was added in methanol and was scanned within the range of 200-400 nm. The λ max was found to be 242 nm. This solubility of drug was determined at 28±°C. The plots of Beer's law limit are shown in Fig.2.

Preparation of Standard stock Solution

An accurately weighed quantity of 25 mg Desloratadine (pure) was dissolved in minimum quantity of 25ml of methanol to produce 1mg/ml solution.

Quantification of Formulation

Twenty tablets of each formulation (DES-LOR) containing 5mg of Desloratadine were accurately weighed and powdered. Powdered tablet equivalent to 20mg of Desloratadine was transferred in to a 100ml volumetric flask, and to it 25ml of methanol was added and shaken vigorously for few minutes and repeated the extraction consequently by three times (3x 25ml) to produce 100ml with methanol, and then sonicated for 5 minutes. The solution was filtered through whattman filter paper NO: 41. From this clear solution 1ml was transferred 200ml standard flask and produce 2µg/ml solution. Like this absorbance measurements of 2µg /ml solution were made six times for each formulation. The absorbance was recorded at 242 nm. Results show in Table-02.

Validation of Method

The method was validated with reference to the ICH guide lines.[7]

Accuracy

The accuracy of the proposed methods was assessed by recovery studies at three different levels i.e. 80%, 100%, 120%. The recovery studies were carried out by adding known amount of standard solution of three drugs to preanalysed tablet solutions. The resulting solutions were then re-analysed by proposed methods; the results are shown in table-03.

Precision

Precision of the methods was studied as intra-day, inter day and repeatability. Intra-day study was performed by analyzing, the three different concentration of drug for three times in the same day. Inter-day precision was performed by analyzing six solutions of sample. The results are shown in table-04.

The limits of detection (LOD) and quantification (LOQ)

The limits of detection (LOD) and quantification(LOQ) calculated according to ICH guidelines using the formula: LOD = 3.3 S/b and LOQ = 10 S/b, where S is the standard deviation of blank absorbance values, and b is the slope of the calibration plot, are also presented in Table-05. The high values of molar absorptivity (ε), low values of Sandell sensitivity and LOD indicated the high sensitivity of the proposed methods. Results shown in Table-05.

Table-01
Optical characteristic and linearity data

Parameters	Desloratadine
Nm	242
Beer's law limits	2-10 µg/ ml
Correlation coefficient	0.996
Regression equation Y=mx+c	Y=0.0405-0.0086
Intercept(c)	-0.0086
Slope	0.0418
Std error	0.007685

Table-02
Analysis data of Tablet formulation

Drug	Label claim mg/tab	Amount found mg/tab	Label claim (%)	S.D.*	S.E*
Desloratadine	5	4.93	98.6	0.001414	0.007685

S.D: Standard deviation, S.E: Standard error,
*Average of four estimation of tablet formulation.

Table-03
Recovery studies

% conc	Amount added	Amount found	% Recovery	%RSD
80	8	7.9	98.7	1.90
100	10	9.93	99.3	1.69
120	12	11.9	99.1	1.87
	Mean		99.0	

Table-04
Precision Results

Parameters	Desloratadine
Intra-day (n = 3)	0.076
Inter-day (n = 3)	0.077

Figure-01
Linearity of Desloratadine

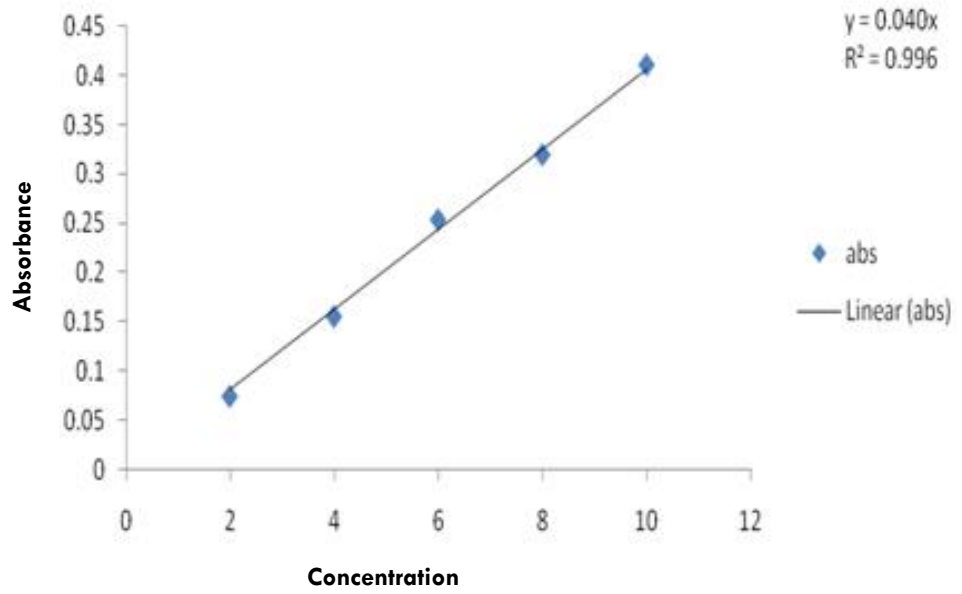

Figure-02
UV spectra of Desloratadine

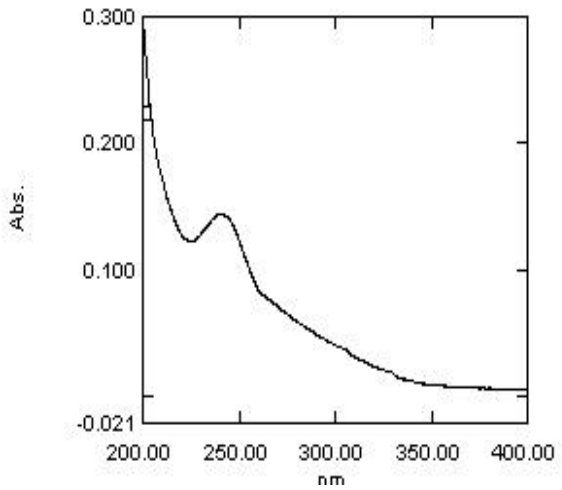

Figure-03
UV spectra of sample Desloratadine

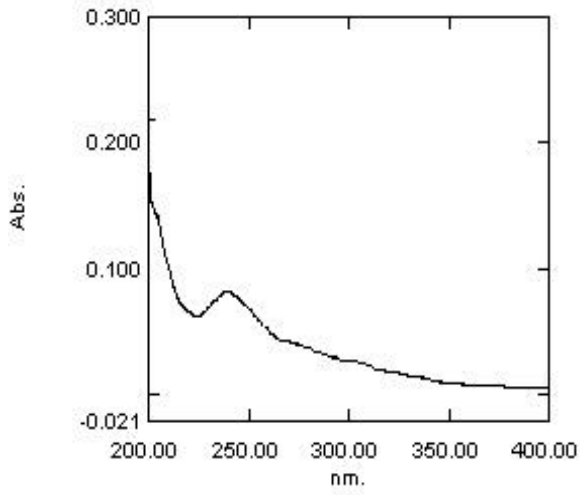

Table-05
The limits of detection (LOD) and quantitation (LOQ)

LOD in µg/ ml	0.11
LOQ in µg /ml	0.338

Result and Discussion

UV Spectroscopic method

The wavelengths 242nm (λmax for Desloratadine) was selected for analysis of the drugs in Methanol. Linearity was observed in the range 2-10 µg/ml (r2 =0.9976) for the amount of drugs estimated by the proposed methods was in good agreement with the label claim. The proposed methods were validated. The accuracy of the methods was assessed by recovery studies at three different levels. Recovery experiments indicated the absence of interference from commonly encountered pharmaceutical additives. The method was found to be precise as indicated by the repeatability, inter-day, intra-day analysis, showing %RSD less than 2. The results did not show any statistical difference between operators suggesting that methods developed were rugged. The results of precision shown in table-04. All statistical data proves validity of the methods and can be used for routine analysis of pharmaceutical formulation.

Acknowledgement

The authors are grateful to the Management of school of pharmaceutical sciences, VELS University, Chennai, for their continuous support and encouragement and for providing the necessary facilities.

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