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Research Article

METHOD DEVELOPMENT AND VALIDATION FOR ESTIMATION OF MOXIFLOXACIN HCL IN TABLET DOSAGE FORM BY RP-HPLC METHOD

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ABSTRACT

A rapid, sensitive and specific RP-HPLC method involving UV detection was developed and validated for determination and quantification of Moxifloxacin HCl in tablet dosage form. Chromatography was carried out on a pre-packed Luna C-18, 5µ (250 x 4.6) mm column using filtered and degassed mixture of Buffer:Methanol (55:45) as mobile phase at a flow rate of 1.0 ml/min and effluent was monitored at 293 nm. The pH of the mobile phase was adjusted with acetic acid to 6.3±0.4. The method was validated in terms of linearity, precision, accuracy, and specificity, limit of quantification and limit of detection. The assay was linear over the concentration range of 20 mcg-60 mcg/ml. Accuracy of the method was determined through recovery studies by adding known quantities of standard drug to the pre analysed test solution and was found to be 99.3 %-100.2 % within precision RSD of 0.58 for Moxifloxacin HCl. The system suitability parameters such as theoritical plates, retention time factor and tailintg factor were found to be 7968, 5.855 and 1.207 respectively. The method does require only 10 mins as run time for analysis which prove the adoptability of the method for the routine quality control of the drug.

KEYWORDS: Moxifloxacin HCl, RP-HPLC, Estimation, Validation

INTRODUCTION

Moxifloxacin HCl is chemically 1-cyclopropyl-7-((s,s)-2,8-diazabicyclo(4.3.0)non-8-yl)-6-fluoro-8-methoxy-1,4-dihydro-4-oxo-3 quinoline carboxylic acid. Moxifloxacin is an antibiotic used to treat respiratory infections, including acute sinusitis, acute exacerbations of chronic bronchitis, and community-acquired pneumonia, as well as dermatological infections, as a second-line agent in tuberculosis. The literature survey[1-7] reveals that there is some HPLC methods have been reported. In this paper we describe a simple, inexpensive, sensitive and validated HPLC[8-12] method for the determination of Moxifloxacin HCl in bulk and pharmaceutical formulation.

MATERIALS AND METHODS

Working standard of Moxifloxacin HCl was obtained from well reputed research laboratories. HPLC grade Methanol, Merck grade Orthophosphoric acid and Triethylamine and Milli-Q water were procured from the market. The separation was carried out on isocratic HPLC system with pre-packed Luna C-18, 5 µ (250 x 4.6) mm column using filtered and degassed mixture of Buffer:Methanol (55:45) as mobile phase.

Buffer preparation: Transferred 2 ml of orthophosphoric acid into 1000 ml of water and the P^H was adjusted to 2.5 with triethylamine, filtered through 0.45µm nylon membrane filter and degassed.

Mobile phase: Buffer and methanol were mixed in the ratio of 55:45 and sonicated to degas.

Standard Preparation: Accurately Weighed and transferred Moxifloxacin HCl equivalent to 20 mg of Moxifloxacin Working Standard into a 50 ml clean dry volumetric flask, and 30 ml of mobile phase was added, sonicated for 5 minutes, and diluted to volume with mobile phase. Further diluted 5 ml to 50 ml with mobile phase.

Procedure

Flow rate 1.0 ml/min; detection wavelength 293 nm; injection volume 10 µl; column used Luna C18 (5 µm, 250x4.6 mm); column temperature: 25^{o} C; mobile phase: Buffer:Methanol (55:45).

Working standard of various concentrations was prepared by taking aliquots of standard solution and diluted to get required concentration for calibration plot and which was injected.

Sample preparation: Weighed and powered 20 tablets. Transferred the powder equivalent to 400 mg of Moxifloxacin into 100 ml of clean, dry, volumetric flask and, to this added 70 ml of mobile phase and sonicated for about 15 minutes, further made up the volume with mobile phase and then filtered through 0.45 micron filter. Further diluted 1 ml of the filtrate to 100 ml with mobile phase.

10 µl of the standard preparation and assay preparation were separately injected and chromatographed.

RESULTS AND DISCUSSION

Linearity was demonstrated by analysing six different concentrations of active compound. Peak areas were recorded for all the peaks and calibration plot was constructed by plotting peak area vs concentrations of Moxifloxacin HCl which were found to be linear in the range of 20 mcg/ml-60 mcg/ml. Coefficient of correlation was 0.9990 (Fig-1).

Accuracy was done by recovery study using standard addition method, known amount of standard Moxifloxacin HCl in to pre-analysed samples and subjected to proposed HPLC method. The results of recovery studies are shown in Table-1.

Precision: To demonstrate agreement among results, a series of measurements are done with Moxifloxacin HCl six replicate injections of the specific standard at various time intervals on the same day were injected into the chromatograph and the value of % RSD was found to be 0.58. In inter-day precision same standard was injected on different days and the found % RSD was found to be 0.95 %. The results were showed in the Table no-2.

CONCLUSION

The regression value was found to be 0.9990 for Moxifloxacin HCl, which shows the response is linear from 20 µg/ml to 60 µg/ml. Selectivity experiment showed that there is no interference or overlapping of the peaks either due to excipients or diluents with the main peak of Moxifloxacin HCl. The percentage RSD for precision is < 2 which confirms that method is sufficiently precise and the total run time required for the method is only 10 mins for eluting Moxifloxacin HCl. The proposed method is simple, fast, accurate, and precise and can be used for routine analysis in quality control of Moxifloxacin HCl.

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Table 1 : Analysis Of Tablet Containing Moxifloxacin HCl

Formulation	Drug	Spike level (%)	Amount std. Added (mcg/ml)	Amount found (mcg/ml)	Recovery (%)
Tablet	Moxifloxacin HCl	50%	20.8	20.6	99.3
		75%	31.2	31.2	100.2
		100%	41.5	41.6	100.2
		125%	51.9	51.8	99.8
		150%	62.3	62.4	100.1

Table 2: Precision

	Intra-day		Inter-day	
Amount found on	**Mean %**	**RSD (%)**	**Mean %**	**RSD (%)**
Moxifloxacin HCl	100.1	0.58	99.72	0.95

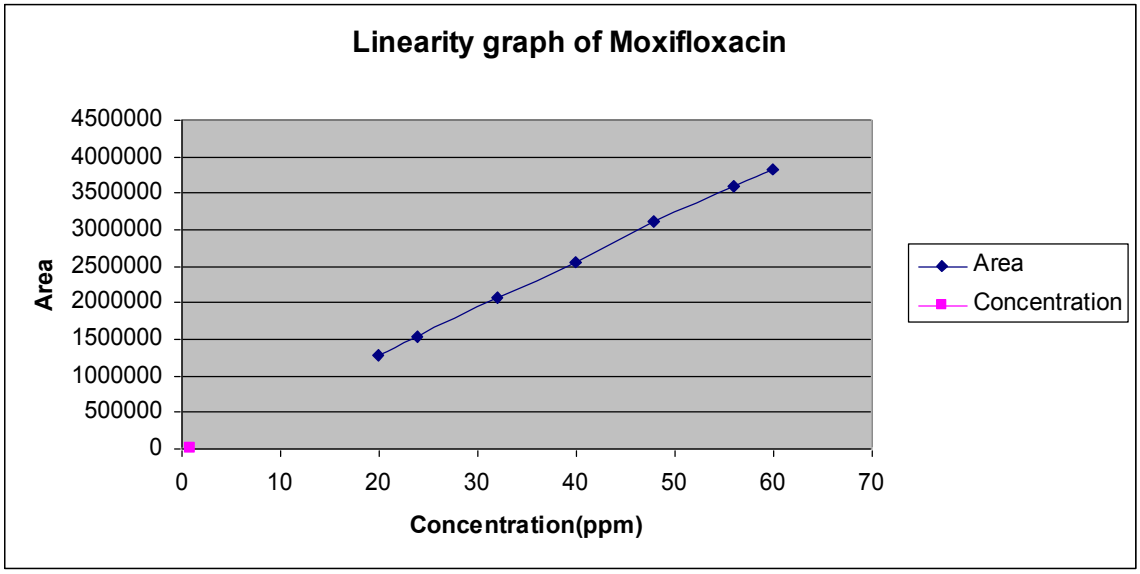

Figure-1: Linearity curve of Moxifloxacin HC

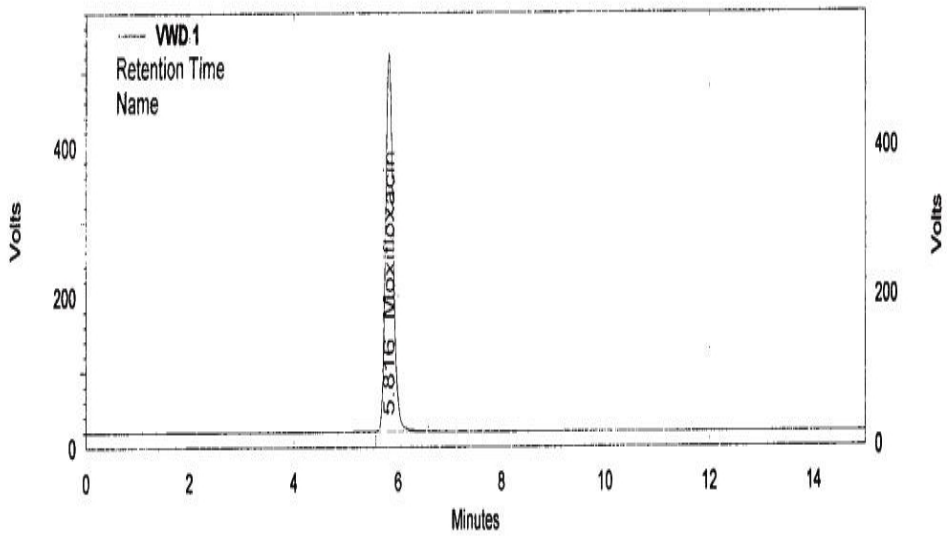

Figure-2: Chromatogram of Moxifloxacin HCl

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