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Simultaneous estimation of moxifloxacin HCl and prednisolone acetate from eye drop formulation

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Abstract

A simple, fast, and precise simultaneous spectrometric method for the estimation of Moxifloxacin HCl and Prednisolone acetate in API and in Formulation was developed. The proposed method is based on the formation and solving of simultaneous equations using 288 nm and 247 nm as two analytical wavelengths. Moxifloxacin HCl shows absortion maximum at 288 nm and Prednisolone acetate shows absorbance at 247 nm in water (co-solvent Methanol). Beer's law was obeyed in the concentration range of 2- 12µg/ml for Moxifloxacin HCl and 2-12 µg/ml for Prednisolone acetate. The method allows rapid analysis of binary pharmaceutical formulation with accuracy. The results of analysis have been validated statistically and recovery studies confirmed the accuracy of the proposed method. The developed method was found to be very precise as % C.V calculated came out to be less than 2%.

Key-Words: Moxifloxacin HCl, Prednisolone acetate, Simultaneous spectrometric analysis, Method validation

Introduction

Moxifloxacin (MOX) (Figure 1) is chemically 1-Cyclopropyl-b-fluoro-1,4-dihydro- 8-methoxy-7-[(4aS,7aS)-octahydro 6H-pyrrolo [3,4–6] pyridine-6-yl]-4-oxo-3-quinoline carboxylic acid[1], is a broad spectrum antibiotic that is active against both Gram-positive and Gram-negative bacteria. The bactericidal activity of the drug is mediated by the inhibition of DNA gyrase (topoisomerase II) and topoisomerase IV, essential enzymes involved in bacterial DNA replication, transcription, repair and recombination. Moxifloxacin is prescribed for the bacterial infections of the respiratory tract including sinusitis, community acquired pneumonia and acute exacerbations of chronic bronchitis.[2]

Several analytical methods, such as High performance liquid chromatography [HPLC] [3], Liquid chromatography mass spectrometry (LC/MS)[4], Capillary electrophoresis[5] spectrofluorimetry[6], High performance thin layer chromatography [HPTLC][7] and Spectrophotometric method[8] of Moxifloxacin in bulk and pharmaceutical formulation have been reported.

Prednisolone acetate **(PRD) (Figure 2) is** chemically, 11β 17, 21-trihydroxypregna-1,4- diene-3,20-dione 21-acetate', is a hydrocortisone type corticosteroid[9]. It is used for infections of the eye[9].

Prednisolone acetate is official in B.P[10]. BP describes liquid chromatography method for its estimation. Literature survey reveals RP-HPLC[11, 12] and spectrophotometric methods[13] for determination of PRD with other drugs.

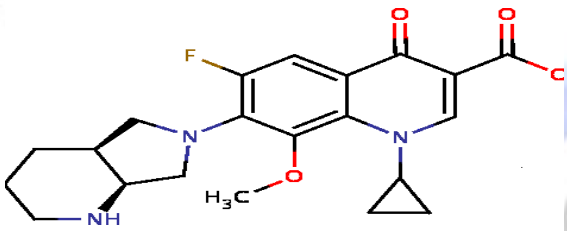

Fig. 1: Moxifloxacin

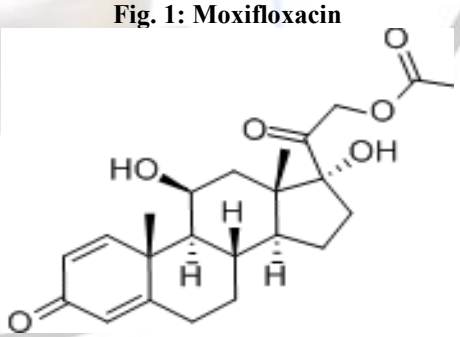

Fig. 2: Prednisolone Acetate

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Material and Methods

Instrument

Double beam UV - visible spectrophotometer (Shimadzu UV-1800) with 1 cm matched quartz cells.

Drug Sample

Moxifloxacin HCl and Prednisolone Acetate were obtained as gift sample from M/s Vindas Chemical Pvt. Ltd. Pithampure.

Chemicals and Reagent

Methanol A.R. Grade (Loba Chemie, Mumbai) and Distill Water.

Preparation of standard solutions

Accurately weighed Moxifloxacin HCL (10 mg) and Prednisolone Acetate (10 mg) were transferred in 100 ml volumetric flask separately, dissolved in 50 ml methanol and diluted up to mark with distill water. The final solutions contained 100 µg per ml of the solution.

Determination of wavelength of maximum absorbance

Aliquots portion 1.0 mL of MOXIFLOXACIN HCL and 1.0 mL of PREDNISOLONE ACETATE were transferred to 10 mL volumetric flask, diluted to mark with distill water to obtain concentration of 10 µg/mL for MOXIFLOXACIN HCL and 10 µg/mL of PREDNISOLONE ACETATE. The resultant solutions were scanned in UV range (400 nm – 200 nm) in 1.0 cm cell against solvent blank Maximum absorbance was obtained at 288.0 nm (λ_{max} of MOX) and 247.0 nm (λ_{max} of PRD)

Preparation of calibration curve for Moxifloxacin HCL and Prednisolone Acetate

Standard solutions of Moxifloxacin HCL (2, 4, 6, 8, 10 and 12 µg) and standard solutions of Prednisolone Acetate (2, 4, 6, 8, 10 and 12 µg) were taken and absorbances of the solutions were measured at 288 nm and 247 nm against distill water as blank.

Estimation of of Moxifloxacin HCL and Prednisolone Acetate in API mixture and formulation

In simultaneous equation method, six working standard solutions having concentration 2,4,6,8,10,12 µg/ml for Prednisolone acetate and six working standard solutions having concentration 2,4,6,8,10,12 µg/ml for Moxifloxacin HCL were prepared in methanol and the absorbance at 247 nm (λ-max of Prednisolone acetate) and 288 nm (λ-max of Moxifloxacin HCL) were measured and absorptivity coefficients were calculated using calibration curve.

$$CMOX = \frac{(A2 \times 601.8) - (A1 \times 854.6)}{-288668.68} \quad \text{..... (1)}$$

$$CPRD = \frac{(A1 \times 830.2) - (A2 \times 922.4)}{-288668.68} \quad \text{..... (2)}$$

Where CMOX and CPRD are concentration of Moxifloxacin HCl and Prednisolone acetate respectively in gm/liter in the sample solution. A1 and A2 are the absorbances of the mixture at 288 nm and 247 nm respectively.

Recovery Studies and Validation of the Method according to I.C.H Guidelines[14]

To study the accuracy, precision and repeatability of the above-proposed method, recovery studies were carried out by addition of standard drug solution to pre-analyzed samples taking into consideration percentage purity of added bulk drug sample. Precision of the method was studied by carrying out interday, intraday analysis.

Results and Discussion

The optical characteristics such as Beer's law limits, correlation coefficient, slope and intercept of regression equation are summarized in Table 1.The values obtained for determination of Moxifloxacin HCl and Prednisolone acetate in formulation by developed method are summarized in Table 2.To evaluate the validity and repeatability of the method, known amounts of pure drug was added to pre-analyzed formulation and mixture were analyzed by developed method and percent recoveries are given in Table 3. Interference studies reveal that the common excipients and other additives usually present in the dosage form do not interfere in the developed method. The low value of standard deviation and % C.V (less than 2% at each step of validation) as given in Table 3 confirms the precision of the method.

In conclusion, the developed spectrophotometric method is simple, sensitive, accurate and reproducible and can be used for routine simultaneous determination of Moxifloxacin HCl and Prednisolone acetate in bulk as well as in formulation mixture.

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Table 1: Optical Parameters and Regression Characteristics of Moxifloxacin HCl and Prednisolone acetate in Water

Parameters	Moxifloxacin HCl 288nm	Prednisolone acetate 247nm
Beer's law limit (µg/ml)	2-12	2-12
Absorptivity	922.6	854.6
Regression equation (y= a + bc) slope (b) intercept (a)	y = 0.091x + 0.007	y = 0.091x - 0.021
Correlation coefficient	R^2 = 0.999	R^2 = 0.998

Table 2: Analysis of Eye drop formulation

Drug	Amount Taken [µg/mL]	Amount Found [µg/mL]	% Amount Found
MOX	Mean	3.99	99.93
	± SD	0.044	1.123
	% RSD	1.124	1.124
PRD	8	8.06	100.77
	± SD	0.050	0.629
	% RSD	0.624	0.624

Table 3: Validation Parameter

Parameter		MOX	PRD
% Recovery		0.93-1.21	0.95-1.31
Precision	Inter-day	0.59-0.87	0.21-0.72
	Intra-day	0.71-1.25	0.26-0.52
Repeatability		0.73	0.89

Fig. 3: Overlain spectra of PRD and MOX