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SIMULTANEOUS ESTIMATION OF MOXIFLOXACIN HCL AND PREDNISOLONE ACETATE FROM EYE DROP FORMULATION BY Q ANALYSIS METHOD

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ABSTRACT
A simple and precise UV spectrophotometric 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 Q analysis equations at two wavelengths i.e 288nm (absortion maximum of Moxifloxacin HCl) and 273n m(isoabsoptivity point) 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%.

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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.

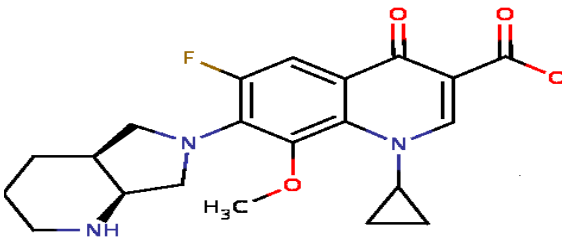


Figure 1: Moxifloxacin

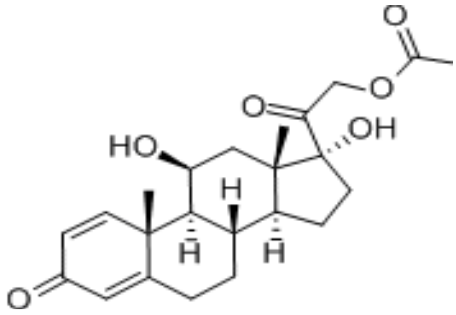


Figure 2: Prednisolone Acetate

METHOD AND MATERIAL

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 273.0 nm (Isoabsorptivity point)

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 273 nm against distill water as blank.

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

In Q analysis 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 273 nm (Isoabsorptivity point) and 288 nm (λ-max of Moxifloxacin HCl) were measured and absorptivity coefficients were calculated using calibration curve.

$$CMOX = \frac{Qm - Qy}{Qx - Qy} \times \frac{A}{ax1} \quad (1)$$

$$CPRD = \frac{Qm - Qx}{Qy - Qx} \times \frac{A}{ay1} \quad (2)$$

Where,

$$Qm = \frac{\text{Absorbance of sample at 288 nm}}{\text{Absorbance of sample at 273 nm}}$$

$$Qx = \frac{\text{Absorptivity of MOX at 288 nm}}{\text{Absorptivity of MOX at 273 nm}}$$

$$Qy = \frac{\text{Absorptivity of PRD at 288 nm}}{\text{Absorptivity of PRD at 273 nm}}$$

'A', is the absorbance of mixture at 273 nm

ax1 (922.4), ax2 (435.4) and ay1 (601.8), ay2 (435.2) are E (1%, 1 cm) of MOX and PRD at 288 nm and273 nm and Qm= A2/A1, Qy = ay2/ay1 and Qx = ax2/ax1.

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 CONCLUSION

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.. 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.

Table No. 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

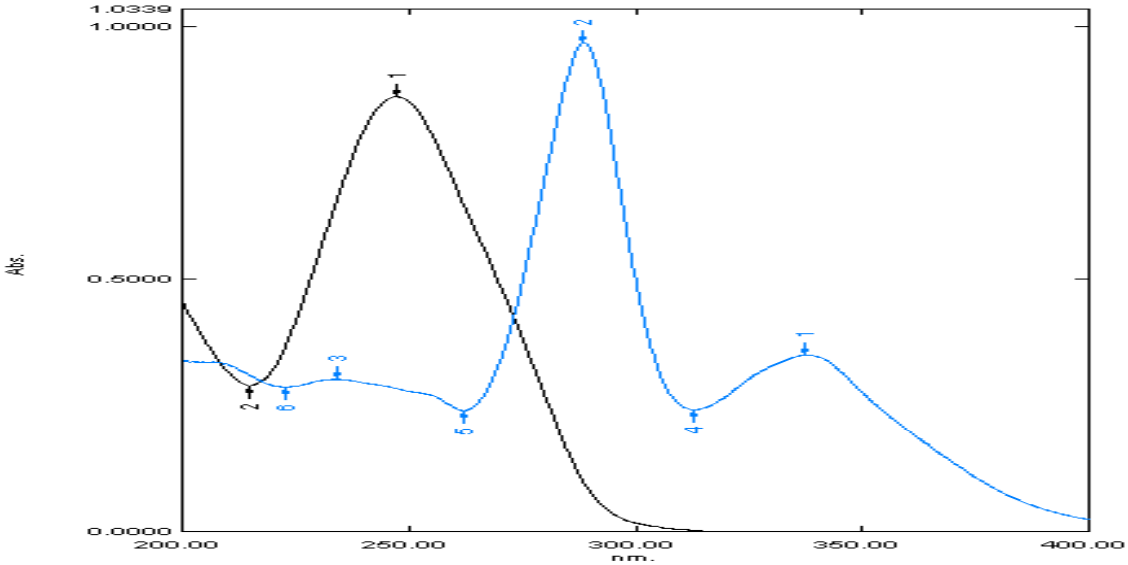
Table No. 2: Analysis of Eye drop formulation

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 No. 3: Validation Parameter

Parameter		MOX	PRD
% Recovery		0.33-1.27	0.23-1.28
Precision	Inter-day	0.53-0.82	0.57-0.95
	Intra-day	0.71-1.25	0.24-0.48
Repeatability		0.342	0.392

Figure 3: Overlain spectra of PRD and MOX



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REFERENCES

1. The Merck Index (2006): An Encyclopedia of chemicals, drugs and biological. 14th ed. *Merck Research Laboratories, Merck and Co., Inc, Whitehouse station, New Jersey* 329. Balfour JAB, Lamb HM. *Drugs*. 2000; 59:115–39.
2. Muijsers RBR, Jarvis B. (2002) *Drugs*. 62:967–73.
3. Vishwanathan K., Bartlett M. G., and Stewart James T. (2002). Determination of Moxifloxacin in human plasma by liquid chromatography electro sprays ionization tandem mass spectrometry, *J. Pharma. Biomed. Ana*, 2002, 30(4), 961-968.
4. Moller J.G., Stab H., Heinig R., and Blaschke G.(1998).Capillary electrophoresis with laser induced fluorescence: a routine method to determine Moxifloxacin in human body fluids in very small sample volumes, *J. of Chromato Biomedical science and applications.*1998, 716(1-2), 325-334.
5. A.Juan, Spectrofluorimetric determination of moxifloxacin in tablets, human urine and serum, *Analyst* 2000, 125(12), 2322-2325.
6. Dewani A.P., Barik B.B., Kanungo S.K., and Wattyani B.R. (2011). Development and Validation of RP-HPLC Method for the Determination of Moxifloxacin in Presence of Its Degradation Products; *American-Eurasian*

Journal of Scientific Research 6 (4):2011 ISSN 1818-6785, 192-200.

7. Dhillon V., and Chaudhary A.(2010). Validated HPTLC method for estimation of moxifloxacin hydrochloride in tablets; *Pharmaceutical methods; volume 1, Issue* 1; 2010, 54-56.
8. Dewani A. P.,.Bakal R. L, Shiradkar Dr. M.R., Chandewar Dr A.V.(2011). Absorption ratio method for the estimation of moxifloxacin HCl and ketorolac tromethamine in their combined dosage form by UV-Visible spectroscopy; *International Journal of Pharmaceutical Research & Development*; Vol 3(7): September 2011, 21 - 26.
9. Sweetman SC. Martindale (2007). The Complete Drug Reference. Edition 35. Pharmaceutical Press, London, UK 209.
10. British Pharmacopoeia (2009)*, Her Majesty's Stationary office, 3*, pp.4950
11. Doppenschmitt SA, Scheidel B, Harrison F, Surmann JP. (1995) Simultaneous determination of prednisolone, prednisolone acetate and hydrocortisone in human serum by high-performance liquid chromatography. *Journal of Chromatography Biomed Appl.* 1995 Dec 15;674(2):237
12. Ali MS., Ghori M., and Saeed A. (2002) Simultaneous determination of tetrahydrozoline hydrochloride, and prednisolone acetate by high-performance liquid chromatography. *Journal OF Chromatography Sci.* 2002 Sep;40(8):429-33
13. Ashok R., Prakash P.P. and Selvan R.Tamil.(2011). Development and validation of analytical method for estimation of prednisolone in bulk and tablets using UV-Visible spectroscopy; *International Journal of Pharmacy and Pharmaceutical Sciences; ISSN-0975-1491 Vol 3, Suppl 4, 2011,184-186.*
14. Text on Validation of Analytical Procedures Q2B in *I.C.H. Harmonized Tripartite Guidelines* (1996)**.**

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