Research Article

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DEVELOPMENT AND VALIDATION OF ANALYTICAL METHOD FOR ESTIMATION OF BALOFLOXACIN IN BULK AND PHARMACEUTICAL DOSAGE FORM

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ABSTRACT

A simple and highly sensitive spectrophotometric method has been developed for the estimation of Balofloxacin in bulk and marketed tablet dosage form. The proposed method is based on the principle that Balofloxacin exhibiting an absorption spectra of wavelength maxima 293 nm in methanol. This method has successfully used for the analysis of drug in marketed preparations in the range of 32-56 µg/ml with correlation coefficient of 0.997. The percentage recovery was found to be 99.04-101.47%. LOD and LOQ were found to be 0.621 and 1.88 µg/ml respectively. This method has been validated for linearity, accuracy and precision and found to be rapid, precise, accurate and economical and can be applied for routine estimation of Balofloxacin in solid dosage form.

Keywords: Balofloxacin, Spectrophotometric, Method Validation.

INTRODUCTION:

Balofloxacin (BLFX), 1-cyclopropyl-6-fluoro-8- methoxy-7-(3 methylaminopiperidin-1-yl)-4-oxoquinoline -3-carboxylic acid, is a broad spectrum fluorinated quinolone antibacterial. It exhibits excellent antibacterial activity against gram-positive bacteria such as multiple-drug-resistant staphylococci and pneumococci. It acts by binding to and inhibiting topoisomerase II (DNA-gyrase) and topoisomerase IV enzymes, which are responsible for the coiling and uncoiling of DNA, which is needed for bacterial cell repair and replication.(1,7,8) In literature, various analytical methods, such as RP-HPLC (Nakagawa T, et al. 1995(2), CHU Zhi-jie et al. 2008 (3), Mi Yaxian ,et al. 2010(4)), RP-HPLC with fluorescence detection (Yin S., et al. 2007(5)), HPLC-Electrospray ionization mass spectroscopy (Bian Z ,et al. 2007(6)) have been developed for determination of Balofloxacin. However, no UV spectrophotometric method is available for estimation of balofloxacin either in bulk or in dosage form. In this study, a simple UV spectrophotometric method was developed and validated in terms of linearity, accuracy, precision and specificity. The method was also used in the determination of the content of balofloxacin in marketed balofloxacin formulation (Baloforce TM).

MATERIALS AND METHODS

Apparatus and Materials

The present work was carried out on a Shimadzu UV- 1800 UV/Visible Spectrophotometer with 10 mm matched quartz cells. Whatman filter paper no. 42 was used for filtration purpose. Pure Balofloxacin was kindly gifted from Cirex Pharmaceuticals (P) Ltd., Hyderabad, Andhra Pradesh, India. Methanol used was of analytical grade.

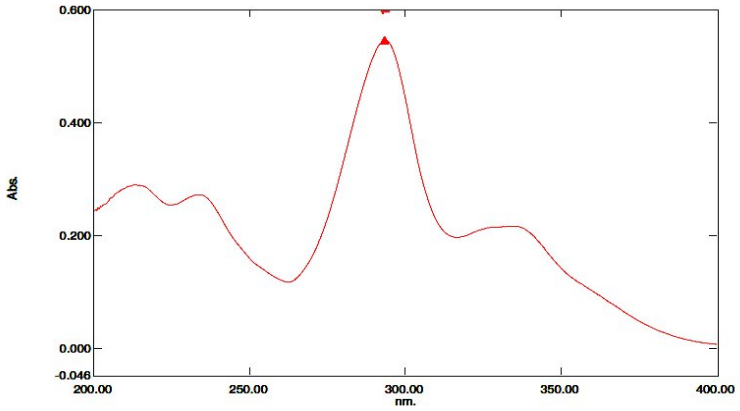
Preparation of standard solution:

Standard solution was prepared by dissolving 10 mg of BLFX in 10 ml of methanol (1000 µg/ml). From that 10 ml was taken and diluted upto 10 ml with methanol (100 µg/ml stock solution).

ABSORPTION SPECRTA OF DRUG SOLUTION WITH FERRIC ION

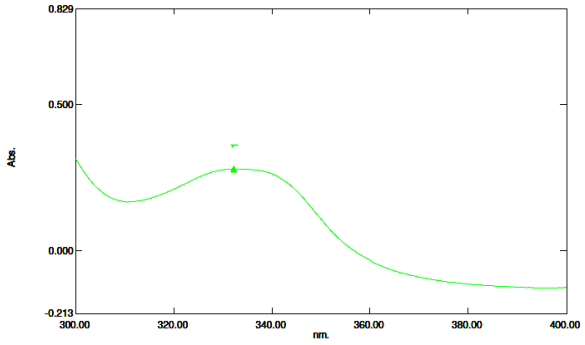
The absorption spectra of drug solution and ferric ion complex were recorded in wavelength region 200-800nm. Absorption spectra was taken by preparing the solution of drug(1 ml of two fold M stock solution) &metal ion(usually 10-15 fold molar excess to drug) with water by taking the metal ion solution as a blank. Observe the spectral data in Fig-1 and Fig-2

Data Set: BALOFLOXACIN – RawData- Fig -1



No.	Wavelength	Absorbance
1	293.40	0.546
2		

Data Set: BALOFLOXACIN+FERROUS 1 – RawData-Fig-2



No.	Wavelength	Absorbance
1	332.20	0.281

EFFECT OF P^H ON THE ABSORBANCE OF BALOFLOXACIN and METAL ION COMPLEX

To arrive the optimum P^H required for achieving the maximum and constant absorbance,the effect of P^H on the absorbance of the ferric ion-balofloxacin complex was studied by preparing the solutions. To prepare the solution mix the solution of drug (1 ml of two fold M stock solution) &metal ion(usually 10-15 fold molar excess to drug) & make up with conserned buffer solution & water. Then observe the absorbance by taking the buffers as blank.

EFFECT OF P^H ON METAL ION+DRUG

Drug40 mcg/ml	Metal ion 400mcg/ml	P^H of buffer	Volume of buffer in ml	Absorbance at 332
1ml	1ml	1.00	3ml	0.087
1ml	1ml	2.00	3ml	0.076
1ml	1ml	3.00	3ml	0.096
1ml	1ml	4.00	3ml	0.115
1ml	1ml	5.00	3ml	0.114
1ml	1ml	6.00	3ml	0.126
1ml	1ml	7.00	3ml	0.137
1ml	1ml	8.00	3ml	0.149
1ml	1ml	9.00	3ml	0.145

EFFECT OF REAGENT CONCENTRATION ON THE ABSORBANCE OF THE BALOFLOXACIN-FERRIC ION COMPLEX

The amount of reagent necessary for full color development was established by the following procedure. Absorbance was taken by preparing the solution of drug (1 ml of two fold M stock solutions) &metal ion (usually 10-15 fold molar excess to drug) with water by taking the metal ion solution as a blank at 332nm

Effect of Metal Ion conc. on the absorbance

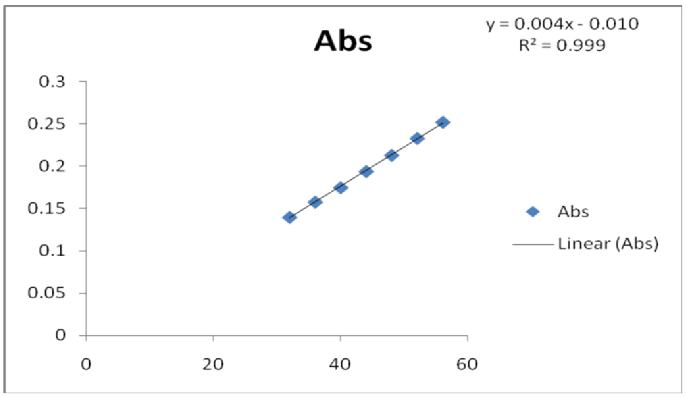
Conc. Of Balo. In mcg/ml	Vol. of metal ion	Water	Conc. Metal ion in mcg	Absorbance at 332nm
40mcg/ml	1ml	3ml	100	0.131
40mcg/ml	1.5ml	2.5ml	150	0.164
40mcg/ml	2ml	2ml	200	0.165
40mcg/ml	2.5ml	1.5ml	250	0.215
40mcg/ml	3ml	1ml	300	0.174
40mcg/ml	3.5ml	0.5ml	350	0.193
40mcg/ml	4ml	0ml	400	0.200

Effect of heating and time of heating on the absorbance of Balo-Fecl3 Complex

Conc. Of Balo in mcg	Time in minutes	Temperature Absorbance of Balo-Fecl3 at 332nm				
		30°	40°	50°	60°	70°
40mcg/ml	5	0.160	0.160	0.160	0.160	0.172
40mcg/ml	10	0.160	0.160	0.161	0.163	0.172
40mcg/ml	15	0.162	0.163	0.164	0.163	0.176
40mcg/ml	20	0.162	0.163	0.164	0.168	0.176
40mcg/ml	30	0.164	0.165	0.166	0.168	0.176
40mcg/ml	40	0.164	0.165	0.166	0.170	0.180

Beer's Law

Cocn.of Drug	Conc.of metal	Absorbance
32	100mcg	0.140
36	100mcg	0.158
40	100mcg	0.175
44	100mcg	0.194
48	100mcg	0.213
52	100mcg	0.233
56	100mcg	0.252



Precision

Cocn.of Drug	Conc.of metal	Absorbance
48	100µg	0.214
48	100 µg	0.214
48	100 µg	0.215
48	100 µg	0.214
48	100 µg	0.213
48	100 µg	0.213
	Average	0.213833333
	Stdev	0.000752773
	%RSD	0.35

Accuracy:

Conc. Of Drug added to placebo	Conc. Of Metal	Placebo	Absorbance	Amt.recovered	%Recovery
48	100mcg	0	0.211	47.37	98.69
48	100mcg	0	0.209	46.92	97.75
48	100mcg	0	0.212	47.6	99.15
			Average	47.29	98.53
52	100mcg	0	0.230	51.33	98.71
52	100mcg	0	0.229	51.11	98.28
52	100mcg	0	0.231	51.55	99.14
			Average	51.33	98.71
56	100mcg	0	0.254	56.22	100.4
56	100mcg	0	0.250	55.34	98.81
56	100mcg	0	0.251	55.55	99.21
			Average	55.70	99.47

REPEATABILITY:

Cocn.of Drug	Conc.of metal	Absorbance
48	100mcg	0.216
48	100mcg	0.213
48	100mcg	0.214
48	100mcg	0.215
48	100mcg	0.214
48	100mcg	0.215
	Average	0.2145
	Stdev	0.001049
	%RSD	0.49

STATISTICAL ANALYSIS OF THE EXP.DATA

PARAMETER	BALO-FECL3 332NM
Linearity	32-56mcg
LOD	0.621
LOQ	1.88
Slope	0.004
Intercept	0.010
Correlation co-efficient	0.999
Accuracy	
80%	98.53
100%	98.71
120%	99.47
Precision	0.35
Repeatability	0.49

CONCLUSION

From the above results it can be concluded that, the developed UV spectrophotometric method is simple, rapid, accurate, precise, specific and economical. Hence, this method can be applied for quantitative analysis of Balofloxacin in bulk and pharmaceutical formulation like tablet dosage form.

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