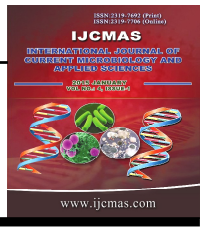
Int.J.Curr.Microbiol.App.Sci (2015) 4(1): 855-861

International Journal of Current Microbiology and Applied Sciences
ISSN: 2319-7706 **Volume 4 Number 1 (2015) pp. 855-861**
http://www.ijcmas.com



Original Research Article

Determination of Balofloxacin in Pharmaceutical dosage form by zero and first order derivative Spectrophotometric method

Mohammed Imran Anees[1*], Archana Ashok Kamble[2] and Mirza Shahed Baig[3]

Y.B.Chavan College of Pharmacy, Dr. Rafiq Zakaria Campus, Aurangabad, Maharashtra, India
**Corresponding author*

A B S T R A C T

Estimation of Balofloxacin in API and pharmaceutical formulation by different analytical methods zero order, first order derivative spectroscopy. The absorbance value were measure in zero order, first order derivative spectroscopy methods at 298nm, 285.7nm**,** respectively Calibration curves were linear between the concentration ranges of 2-10 mg/ml for both of derivative method. The % RSD value is less than 2% and the recovery were near 100% for both methods. Parameter such as linearity, accuracy, precision, limit of detection, limit of quantitation. The method was successfully developed for quantitative determination of Balofloxacin in pharmaceutical preparations. All the developed methods were applied on tablet formulation and the results were found within the limit as per ICH guideline.

Keywords

Balofloxacin, zero order, first order derivative spectroscopy, UV method

Introduction

Balofloxacin (BALO) is 1-cyclopropyl-6-fluoro-8methoxy-7-(3-methylaminopiperidin -1-yl)-4 oxoquinoline-3-carboxylic acid1, is a broad spectrum fourth generation fluoroquinolone antibacterial, having a formula as $C_{20}H_{24}FN_3O_4$ and molecular mass 389.42 g / mol. 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 (DNAgyrase) and topoisomerase IV enzymes, which are responsible for the coiling and uncoiling of DNA, which is needed for bacterial cell repair and replication, Several analytical methods such as UV Spectrophotometric method, HPLC determination in biological fluids, HPLC determination in human plasma with solid extraction, RPHPLC, RP-HPLC with fluorescence detection, HPLC Electrospray ionization mass spectroscopy, have been developed for determination of Balofloxacin. Thus there is need to develop simple rapid and cost effective method for routine analysis. The objective of present study was to develop simple, sensitive, accurate rapid and cost effective method for estimation of Balofloxacin. The present study reports newly developed zero & first order derivative spectroscopy for estimation of BALO in bulk & pharmaceutical preparations. The developed UV spectroscopic methods is easy to handle &

simple 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 (B- CIN).

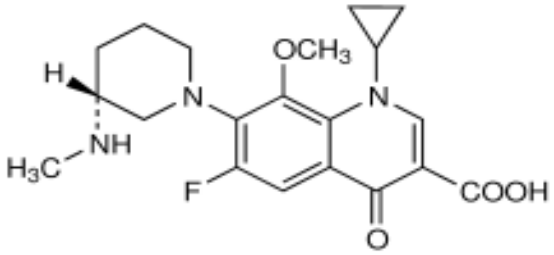


Fig .1 structure of Balofloxacin

Material and methods

Apparatus

A Shimadzu model 1800 double beam UV-Visible spectrophotometer with spectral width of 2 nm, wavelength accuracy of ± 0.1 nm and slit width of 2 nm, instrument scan speed of 600 nm min-1, a pair of 1 cm matched quartz cell was used to measure absorbance of all the solutions. Spectra were automatically obtained by UV-Probe system software. Wavelength range 190 to1100 nm.

Reagent and Material

The standard sample of Balofloxacin drug was kindly supplied as gift sample by Cirex Pharmaceutical LTD. Hatnoor Mandal-Andra Pradesh,India. All chemicals and reagents were used of AR grade. Acetonitrile (AR grade) from Fischer Ltd, Mumbai, Methanol (AR grade) from Fischer Ltd, Mumbai, Commercial tablets of BLFX was procured from local market and used for analysis of marketed formulation. B-CIN 100mg tablet are manufactured by Lupin Pharmaceutical Aurangabad, India.

Selection of detection wavelength

Solution of drug in was scanned over the range of 200-400 nm. The absorbance value were measure in zero order, first order derivative spectroscopy at 298nm, 285.7nm respectively were selected as the wavelength for detection.

Preparation of standard stock solutions

Stock solution of the drugs 100 µg/ml is prepared by dissolving 10 mg BALO drug in separate 100 ml volumetric flask and the volume is make up to the mark by Methanol & final concentration of solution containing 100ug/ml

Selection of Analytical Concentration Range:

For each drug appropriate aliquots were pipette out from the standard stock solution into series of 10 ml volumetric flask. The volume was made up to the mark with methanol to get set of solution for each drug having concentration 2, 4, 6, 8, 10µg/ml. The absorbance of each of these solutions were measured at selected wavelength and plotted against concentration. The range was found to be 2-10 µg/ml for both the drugs.

Tablet Analysis

Twenty tablets of BALO were weighed; their average weigh was determined and finally crushed to powder sample. From the triturate, tablet powder equivalent to 100mg of BALO was weighed and transfer to 100 ml volumetric flask and dissolved in 50ml methanol and the content was kept in ultrasonicated for 30 min. finally the volume was made up to the mark with methanol. The solution was filtered through Whatman filter paper no.41.This tablet solution was further diluted to obtain 1µg/ml of BALO. The mixed sample solution was analyzed to obtained spectra and absorbance value at 298 nm was noted. The concentration of BALO was calculated from the equation.

Method Development

Development of simple zero & first order spectroscopic method: The zero order spectra of Balofloxacin were obtained in scanning range between 200-400nm and calibration curve were plotted with absorbance versus concentrations and regression equations were calculated for both the methods. λ_{max} obtained for zero order was 298nm & for first order was 285.7nm (Fig.2,3)

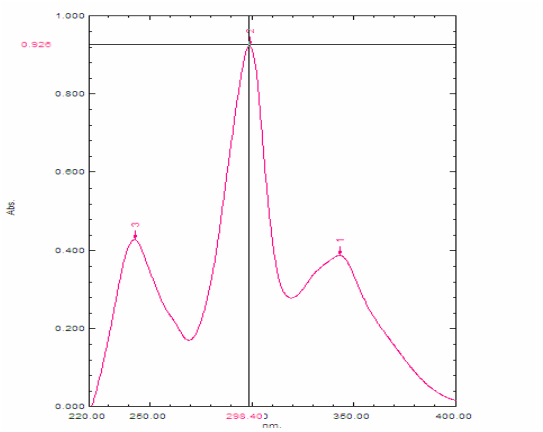


Fig.2 Spectra of BALO by zero order

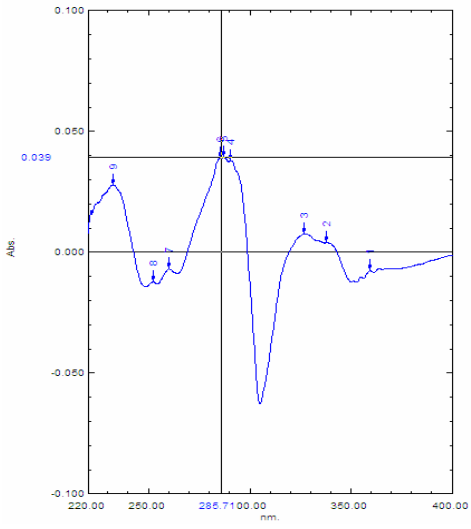


Fig.3 spectra of BALO by first order

Method Validation

Linearity

For quantitative analysis of Balofloxacin, the calibration curves were plotted for each concentration ranges. The linearity ranges for zero & first derivative found to be for Balofloxacin 2-10µg/ml. (Fig.4, 5)

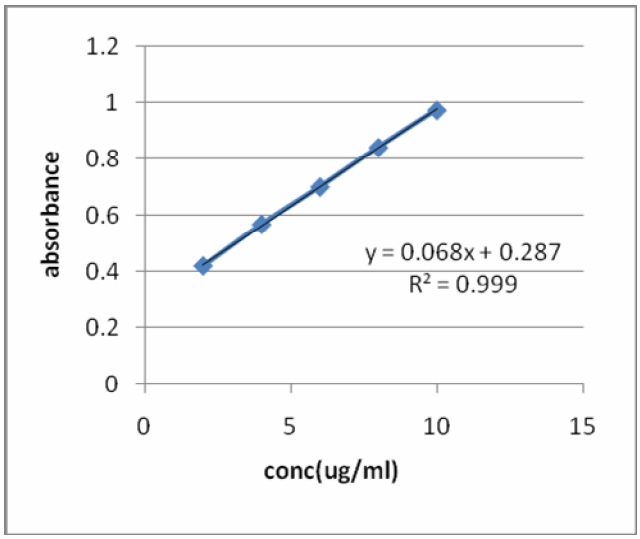


Fig.4 Calibration curve of BALO at 298nm

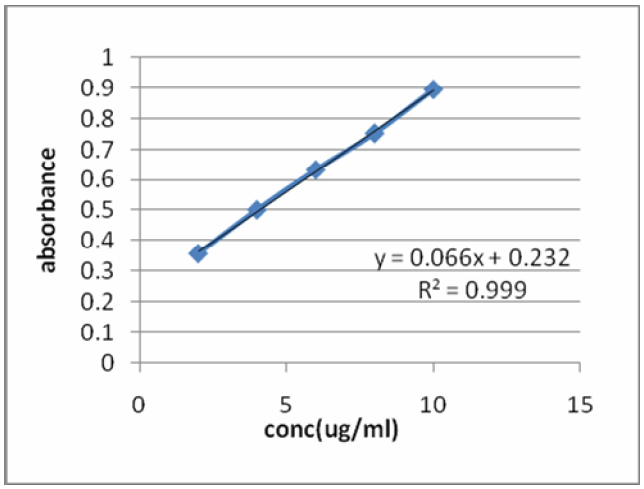


Fig.5 Calibration curve of BALO at 285.7nm

Accuracy

The accuracy was determined by standard addition method. Three different levels (80%, 100% and 120%) of standards were spiked to commercial tablets in triplicate. The mean of percentage recoveries and the % RSD was calculated given in (Table 1)

UV method	Level of % Recovery	Amt. Present (mg/tab)	Amt. of standard added (mg/tab)	Total Amt recovered (mg)	% Recovery	% mean	%RSD
ZERO ORDER	80	100	80	180	99.65		
	100	100	100	200	100.01	99.83	0.187
	120	100	120	220	99.74		
FIRST ORDER	80	100	80	180	98.40		
	100	100	100	200	99.56	99.42	0.962
	120	100	120	220	100.3		

Table.1 Result of recovery studies of BALO

Precision

The reproducibility of proposed method was determined by performing tablet assay at different time intervals (2 hour interval) on same day (Intra-day precision) and on three different days (Inter-day precision) at concentration BALO at the concentration 1 µg/ml (Table 2)

Method	Zero-order Spectrophotometric Method	First-order Spectrophotometric Method
Mean Recovery (Inter Day)	0.83	0.76
% RSD for Inter day		
Mean Recovery (Intra Day)		
% RSD for Intra day	0.68	

Table.2 Interday and Intraday Precision of Balofloxacin

Specificity

The spectrum of Balofloxacin in bulk drug with drug formulation (B-CIN) solutions shows that the wavelengths of maximum and minimum absorbance do not change.

Limit of detection (LOD) and Limit of Quantitation (LOQ)

LOD is the lowest amount of an analyte in a sample that can be detected, but not necessarily quantitated an exact value. LOQ is the lowest amount of an analyte in a sample that can be quantitatively determined with suitable precision and accuracy.

LOD = 3.3 σ/S

LOQ = 10 σ/S Where, σ = standard deviation of the y-intercepts of regression line S = slope of the calibration curve

Method characteristics	Zero order	First order
Linearity	2-10 µg/mL	2-10 µg/mL
Regressions equation	y = 0.068x + 0.287	y = 0.066x + 0.232
R^2	0.999	0.999
LOD(µg/mL)	0.9	1.06
LOQ(µg/mL)	0.4	0.29

Table 3: Results of Linearity, LOD and LOQ of BALO

Table.4 Validation parameter of Balofloxacin

Parameters	Zero order	First order
	BALO (λ_{max}=298nm)	BALO (λ_{max}=285.7 nm)
Linearity range (µg/ml)	2 – 10	2 – 10
Slope	0.068	0.066
Intercept	0.287	0.232

gression coefficient	0.999	0.999
nit of detection (µg/ml)	0.9	1.06
nit of quantitation (µg/ml)	0.4	0.29
curacy (%recovery)	99.83	99.42
ecision (%RSD)		
Intraday	0.006	0.001
Inter day	0.003	0.053

Result and Discussion

The developed zero & first order derivative methods for BALO are found to be linear, correlation coefficient is near to 0.999(Fig 4,5) respectively In .accuracy study for zero & first order derivative method % recovery was found to be 99.83 & 99.42 respectively. % RSD for both methods was less than 2 % (Table 1) .Good precision was found for both methods % RSD for zero order intraday precision& interday precision was found to be 0.68& 0.30 respectively, % RSD for first order intraday precision & interday precision was found to be 0.12& 0.53 respectively (Table 2).Linearity range for both methods was found to be 2-10ug/ml. Regressions equation for zero order derivative was y = 0.068x + 0.287 & for first order y = 0.066x + 0.232 (Table 3). Limit of detection of BALO by zero order was 0.9ug/ml & for first order was1.06 µg/ml. Limit of quantitation of BALO by zero order was 0.4 & for first order was 0.29 (Table 3)

The developed methods were found to be simple, sensitive, accurate and precise and validated as per ICH guidelines .The method was successfully used for determination of drugs in their pharmaceutical formulation.

Acknowledgement

The authors are thankful to Mrs. Fatma Rafiq Zakaria, Chairman, Dr Rafiq Zakaria Campus for providing necessary facility to conduct the research work.

References

1. Skoog D.R., Hollar F.J.,Nieman 2005 T.A. Principles of Instrumental Analysis. 5th edition. Thomson Brooks/Cole; 1-3,300,726.
2. Jeffery,G.H., Bassat,J. Mendham,J. Denny,R.C. 1989 :Vogel's Textbook of Qualitative Chemical Analysis**.** 5thedition. Atkin. ELB Swithlongman Publication. Harlow; 3.
3. 3. B. K. Sharma. Instrumental methods of chemical analysis. 2000, Introduction to analytical chemistry. 19th edition. Goel publishing house ; 200-203, 1-4.
4. U.S. Pharmacopoeia. United States Pharmacopoeial Convention, Inc. (1994); 1982-84.
5. International Conference on Harmonization. Draft Guideline on Validation of Analytical Procedures 1995: Definitions and Terminology. Federal Register.; 60: 112-160.
6. Reviewer Guidance, Validation of Chromatographic Methods 1994, Center for Drug Evaluation and Research, Food and Drug Administration. .
7. Guideline for Submitting Samples and Analytical Data for Methods Validation,1987. Food and Drug Administration.
8. Bekett A.H.,Stenlake J.B. Practicle Pharmaceutical chemistry.2004, 4th edition. CBS Publishers New Delhi ; Part-II: 275-314.
9. Kemp W. Organic Spectroscopy. 2nd edition. ELBS publication (1989); 194.
10. Chatwal and Anand. Instrumental methods of chemical analysis. 2007, 5th edition. Himalaya Publishing House ; 2.149-2.150.
11. Sharma B.K., in: Spectroscopy. 6thedition.Goel Publication Co. Meerut (1983); 1.
12. Tony Owen. Fundamentals of modern UV-visible spectroscopy 2000. Agilent technologies. 73.
13. Kasture A.V.,Wadodkar S.G.,Mahadik K.R.,More H.N. 1998, Pharmaceutical Analysis (Instrumental

Methods). Nirali Prakashan. Pune, 2: 1-3.
14. Howell J.A. Handbook of Instrumental techniques for Analytical Chemistry. 1st edition (2004); 490.
15. www.google.com/http: /-/-www.medschat.com/-wiki/-Antibacterial/-
16. The Indian Pharmacopeia, Government of India, Controller of publication, New Delhi 2010 vol.2; 1012
17. British pharmacopeia, 6th edition, 2010; Vol II, Vol III, London: Her Majesty's Stationary Office 1460.
18. The Merck Index "An encyclopedia of chemicals, drugs and biologicals" Merck and Co. Inc., whitehouse station, NJ, USA,14th edition, 2006, 6291.
19. British pharmacopeia, 6th edition, 2010; Vol I, Vol III, London: Her Majesty's Stationary Office 410.