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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 2-14 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 0.21 and 0.63 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 ™).

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).

Calibration Curve

Different aliquots of this stock solution were taken in 10 ml volumetric flask and diluted upto the mark to get the working standard solution. The calibration curve was prepared by plotting absorbance versus concentration of BLFX. A spectrum of BLFX is shown in figure 1 and calibration curve is in figure 2. The results are shown in table 1.

Method Validation

The developed method was validated for its linearity, accuracy, precision and specificity. The linearity of measurement was evaluated by analyzing different concentrations of the standard solution of BLFX. The results are shown in table 1.To ascertain the accuracy of the proposed method, recovery studies were carried out by standard addition method**.** The results are shown in table 2. The precision of the proposed method was determined by analyzing different concentrations (2-14 µg/ml) at different time intervals on same day (Intra-day precision) and on three different days (Inter-day precision). Interference and non-interference of excipients and binders was confirmed by performing the specificity study. Specificity was performed by spiking placebo with standard drug. The LOD and LOQ were calculated from the equations, LOD =3.3 σ/S and LOQ = 10 σ/S, where σ is the standard deviation of the lowest standard concentration and S is the slope of the standard curve. The results are shown in **table 1.**

Estimation of Balofloxacin from tablet

Marketed preparation of Balofloxacin (BALOFORCE ^{TM}) selected for the purpose of analysis. Twenty tablets were accurately weighed and powdered. A quantity equivalent to 100 mg of BLFX was transferred in volumetric flask and sonicated in 45 ml of methanol at ambient temperature for 15 min. Then the volume was made upto the mark and the solution was filtered using Whatman filter paper no. 42 to obtain sample stock solution. 0.1 ml of filtrate was further diluted to 10ml with same solvent and absorbance of sample was measured against blank. The amount of BLFX was calculated from the calibration curve. The results of assay are shown in **table 3.**

Figure 1: Spectrum of Balofloxacin (10 µg/ml)

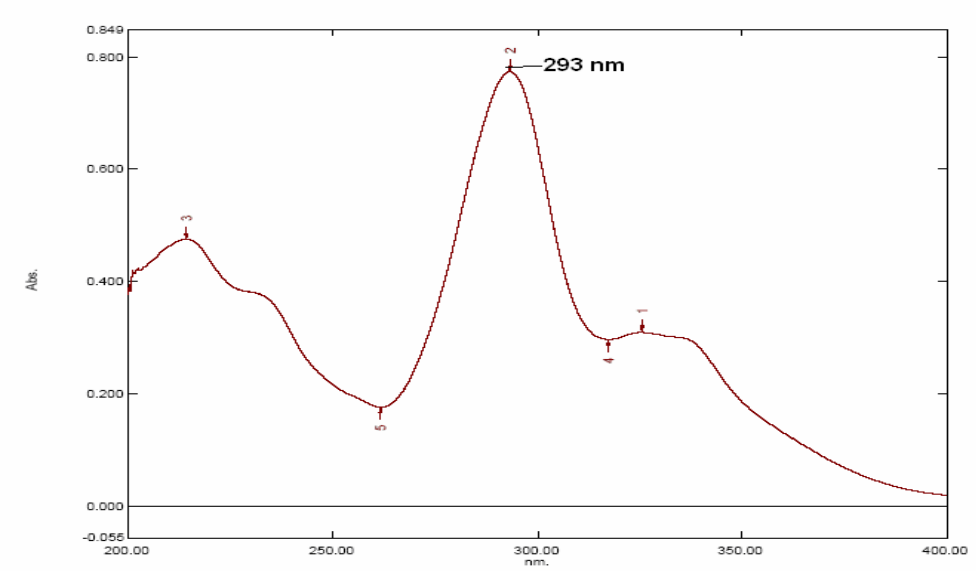

Figure 2: Calibration curve of Balofloxacin (2-14 µg/ml)

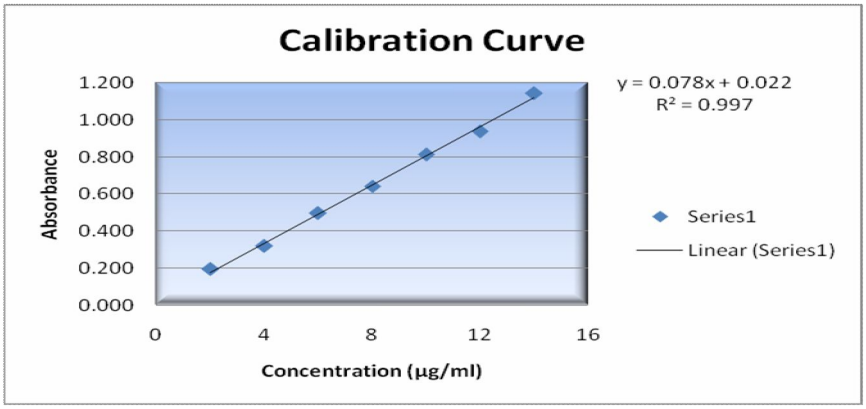

Table 1: Optical and Statistical data

Parameters	Values
Maximum wavelength (λmax)	293 nm
Calibration curve range (µg/ml)	2-14
Molar extinction coefficient (L mol^{-1} $cm^{-1)}$	$3.22*10^5$
Regression equation	y = 0.078*X +0.022
Slope	0.078
Intercept	0.022
Correlation co-efficient (r^2)	0.9993
Limit of Detection (LOD) (µg/ml)	0.210
Limit of Quantitation (LOQ) (µg/ml)	0.637

Table 2: Results of Accuracy Study

Amount of sample (µg/ml)	Amount of std. added (µg/ml)	Total amount (µg/ml)	Total amt found (µg/ml) Mean ±S.D.*	Accuracy (%)
4	0	4	4.11 ±0.374	102.75
4	2	6	6.09 ±0.090	101.47
4	4	8	7.92 ±0.356	99.04
4	6	10	10.10 ±0.243	101.00

* n = 3

Table 3: Estimation of Marketed Formulation

Formulation	Label claim	Assay (% of label claim) ± %RSD*
BALOFORCE™ tablet	100 mg	99.27% ± 1.8

*Mean of three determinations

RESULTS AND DISCUSSION

As shown in fig. 1, Balofloxacin showed wavelength maxima at 293 nm in methanol. As shown in fig. 2 and table 1, the calibration curve was found to be linear in the range of 2-14 µg/ ml with regression equation of y = 0.078*X +0.022; (r^2 = 0.997) which clearly indicates linearity of developed method. % Recoveries for Balofloxacin was found to be satisfied i.e. 99.04 to 101.47% as shown in table 2; clearly indicate that the developed method is accurate. Results of intra-day and inter-day precision is expressed in % RSD and found to be 0.461 and 1.06 respectively. As, % RSD is within the allowable limit of $\leq$ 2% it clearly indicate that the developed method is precise. Results of specificity study shows that the excipients present in the formulation do not interfere with the estimation of Balofloxacin. As, shown in table 3, assay result is in good agreements with the label claim. Hence, the proposed method can be successfully used for its analysis and quality control of marketed solid dosage preparation with good linearity, accuracy and precision.

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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