Volume : 2 | Issue : 7 | July 2013 • ISSN No 2277 - 8179

Research Paper

Development of a New Spectrophotometric Method for the Determination of Balofloxacin in Tablet Dosage Forms

Chemistry

KEYWORDS : Balofloxacin, Spectrophotometric, Analytical method validation, Cerric sulphate

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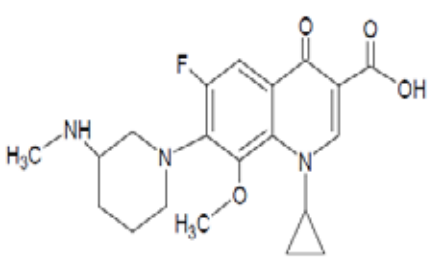
ABSTRACT

A simple and accurate visible spectrophotometric method was developed and validated for the determination of balofloxacin in bulk and marketed tablet dosage form using 1% w/v of ceric sulphate as chromogenic agent. The brown coloured complex formed by cerium (IV) solution with balofloxacin was stable up to 30 min and was measured for absorbance at its λ max of 492nm.The Beers law concentration range was found to 10-100 µg/ml with good correlation coefficient of 0.9922. The method was optimized and validated for linearity, accuracy and precision. The validated method was accurate as found from recovery of 97.14 % to 98.88 % when applied for marketed tablet formulations.

Introduction

Balofloxacin (BLFX), 1-cyclopropyl-6-fluoro-8-methoxy-7-(3-methylaminopiperidin 1-yl)-4-oxoquinoline-3-carboxylic acid (Punam & Vandana, 2011), is a broad spectrum fourth generation fluoroquinolone 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 (Ross, Elkinton, and Riley, 1992). Several analytical methods such as UV spectrophotometric method (Punam & Vandana, 2011), (Ashok Reddy & Chandra Sekhar, 2012), HPLC method in biological fluids (Nakagawa, Ishigai, Hiramatsu, Kinoshita, Ishitani, Ohkubo, and Okazaki, 1995), HPLC method in human plasma with solid extraction (Deng, Xiao, Zhang, Zhang, and Tang, 2007),RP-HPLC (Nyola & Jeyabalan 2012), RP-HPLC with fluorescence detection(Yin, Tao, Jing-wang, Yun-biao & Long-shan, 2007) HPLC-electrospray ionization mass spectroscopy (Bian, Tian, Zhang, Xu, Li, and Cao, 2007) have been developed for determination of balofloxacin. In the present study a simple, rapid and reliable visible spectrophotometric method has been developed and validated for linearity, accuracy, precision and specificity. The method was extended for determination of balofloxacin in the marketed tablet formulation (Baloforce™ Tablet manufactured by Mankind Pharma, New Delhi, India).



Balofloxacin

Method:

Labindia Analytical UV 3000 uv/vis spectrophotometer was used for recording the absorbance. Balofloxacin - Reference standard was kindly received from Cirex Pharmaceuticals (P) Ltd, Hyderabad, India. All the solvents used were of analytical grade.

Preparation of standard drug solution

A 1 mg/ml of stock solution was prepared in a 10 ml volumetric flask by dissolving 100 mg of balofloxacin in 0.1N HCl, diluting to the mark with the same acid.

Preparation of 1% w/v of ceric sulphate reagent (Reagent)

A 1% w/v of ceric sulphate reagent was prepared by weighing 1 gm of the reagent in 100 ml volumetric flask and diluting up to the mark with 5M H_2SO_4.

Standard Calibration Plot

Aliquots of the stock solution ranging from 0.1 ml to 1 ml were taken in separate 10 ml volumetric flasks and 2 ml each of reagent was added, further diluted to the mark with water to get the working standard solution of concentration 10 – 100 µg/ml. The absorbance of each solutions was measured at 492 nm against reagent blank (Fig 1). A standard calibration curve was prepared by plotting absorbance versus concentration of balofloxacin (Fig 2).

Further, with optimized conditions, the proposed method was validated for linearity, accuracy, precision, sensitivity, reproducibility and stability of colour. Recovery studies were carried out by mixing standard solutions of the drug at three different levels (75%, 100%, 125%) with previously analyzed tablet samples of balofloxacin. The results of the validation study and recovery study is presented in Table 1 and Table 2 respectively.

Estimation of Balofloxacin from tablets

10 tablets of (*Baloforce*) containing 100 mg of the active ingredient in each tablet was weighed and powdered. Tablet powder, equivalent to 25 mg of balofloxacin was transferred to 25 ml of volumetric flask and sonicated using 5 ml of 0.1N HCl at ambient temperature for 15 min. The resulting solution was filtered using whatman filter paper no. 42 and volume of solution diluted up to the mark with 0.1N HCl. Different aliquot volumes of this solution was taken in 10 ml volumetric flasks to which 2 ml of 1% w/v of the ceric sulphate solution was added and diluted to mark with water. The absorbance of the sample solutions was recorded against reagent blank prepared in a similar manner, but excluding the sample. The amount of balofloxacin was calculated from the calibration curve (Fig 2).

Results and Discussion

Balofloxacin, complexes with the ceric (IV) in 1% w/v of ceric sulphate forming a yellowish orange solution. The developed method was found to show linearity over the concentration range of 10 – 100 µg/ml. Accuracy was determined from recovery study at 3 different levels of 75 %, 100% and 125% by adding standard solution of balofloxacin to previously analysed tablet samples of balofloxacin. Average recovery of 97.14 % to 98.88 % indicated accuracy of the method. Sandell's sensitivity was determined and found to be 58.823 x 10^{-2} µg/cm^2. The colour was stable up to 30 min. The results of assay showed that the amount of drug determined by new method was in good agreement with the label claim of formulation.

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

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Table 1: Validation Parameters

Parameters	Observations
Linearity	10-100 µg/ml
Precision	1.471 % RSD
Sandell's sensitivity	58.823 x 10^{-2} µg/cm
Equation of linearity graph Slope: Intercept:	0.001x + 0.001 0.001 0.001
Stability of Colour	30 min

Table 2: Results of Recovery Study

Std Conc (in µg/ml)	Sample Conc (in µg/ml)	Absorbance at 492 nm	Total concentration as calculated from linearity graph	% Std Recovery (in %)
7.5	10	0.0341	17.0	97.14%
10.0	10	0.0402	20.22	98.91%
12.5	10	0.0439	22.22	98.88%

Fig 1: Absorption spectra

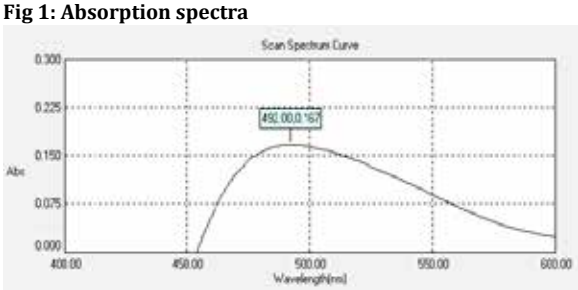
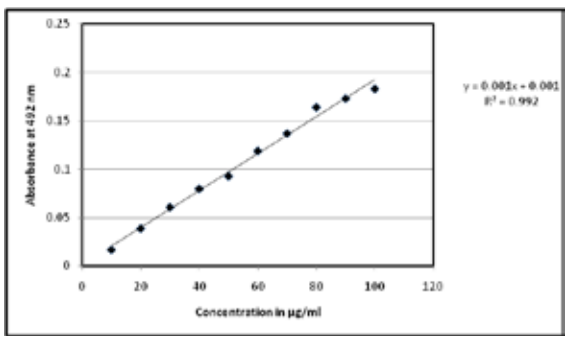


Fig 2: Standard Calibration Plot



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