International Journal of PharmTech Research
CODEN (USA): IJPRIF ISSN : 0974-4304
Vol. 3, No.1, pp 133-135, Jan-Mar 2011

Simple and Rapid Spectrophotometric Estimation of Gemifloxacin Mesylate in Bulk and Tablet Formulations

Danta Chhanda Charan[1]* and Sahu Satyabrata[1]

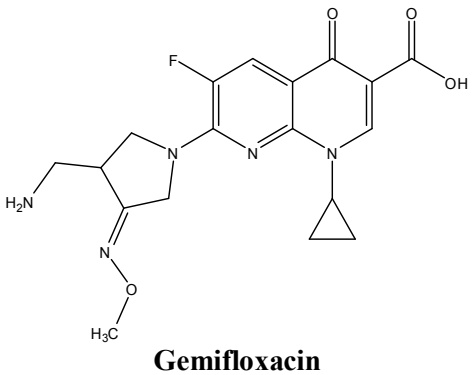
[1]Department of Medicinal Chemistry and Pharmaceutical Analysis, Dadhichi College of pharmacy, Vidya Vihar, Sundargram, Cuttack, Orissa-753002,India

*Corres.author: g49chhanda.itbhu@gmail.com, Mob. No. 91-9889595616

Abstract: A simple and highly sensitive spectrophotometric method has been developed for the estimation of gemifloxacin mesylate in bulk and marketed tablet dosage form. The proposed method is based on the principle that gemifloxacin mesylate exhibiting an absorption spectra of wavelength maxima 267 nm in acidic medium. This method has successfully used for the analysis of drug in marketed preparations in the range of 10-70 µg/ml with correlation coefficient of 0.9987. The percentage of recovery was found to be 99.4-99.5%. 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 gemifloxacin mesylate in solid dosage form.
Keywords: Gemifloxacin mesylate, Ultraviolet Spectrophotometry, Method Validation.

INTRODUCTION

Gemifloxacin mesylate (GEM) is chemically 7-[(4Z)-3-(aminomethyl)-4-methoxyimino-pyrrolidin-1-yl]-1-cyclopropyl-6-fluoro-4-oxo-1,4 dihydro -1,8-napththyridine-3-carboxylic acid, a fourth generation fluoroquinolone antibacterial agent having affinity towards bacterial topoisomerase IV. The compound has broad spectrum of activity against gram-positive and gram-negative bacteria. It is mainly used for the treatment of acute exacerbations of chronic bronchitis, pneumonia and urinary tract infection.[1-4] This drug is not official in any pharmacopoeia. The literature survey revealed that analytical methods reported for the estimation of Gemifloxacin mesylate include rapid determination by HPLC–tandem mass spectrometry[5], RP-HPLC and HPTLC[6] and microchip electrophoresis[7] in human plasma, and RP-HPLC[8] and ion-pair complex[9] for tablet formulation. However, there is no publication concerning the analysis of GEM in bulk and pharmaceutical formulation by simple UV method. The aim of the present work is to describe a simple and sensitive UV method for estimation of GEM in tablet formulation.



Gemifloxacin

MATERIALS AND METHODS

Apparatus

The present work was carried out on a Systronics-double beam UV Spectrophotometer 2203 with 2nm bandwidth and a pair of 1cm matched quartz cells. Whatman filter paper no. 42 was used for filtration purpose.

Working standard solution

Pure Gemifloxacin mesylate was the gifted sample from Lupin Ltd, Pune. All chemicals were of analytical reagent grade and solutions were prepared with triple distilled water. Standard solution was prepared by dissolving 100 mg of GEM in 0.05N H_2SO_4 solution and sonicated to dissolve and made up the volume to 100 ml (1 mg/ml stock solution). From that 10 ml was taken and diluted to again 100 ml (100 µg/ml stock solution). Different volumes of 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 GEM.

Estimation of Gemifloxacin mesylate from tablets

Tablet of three brands were selected for the purpose of analysis. Twenty tablets of each brand were powdered separately and powder equivalent to 100 mg of GEM was transferred in volumetric flask and sonicated in 45 ml of 0.05N H_2SO_4 solution at ambient temperature for 15 mins. Then made up the volume upto the mark and filtered the solution with Whatman filter paper no. 42. The absorbance of working solutions were measured against the blank and the amount of GEM was calculated from calibration curve. The LOD and LOQ were calculated according to ICH guideline as LOD = 3.3σ/S and LOQ = 10σ/S, where σ is the standard deviation of the lowest standard concentration and S is the slope of the standard curve. LOD is the lowest concentration of an analyte that an analytical process can reliably detect and LOQ is the lowest concentration of the standard that can be measured. The results are shown in Table 1. Interference or absence of interference of excipients and binders was confirmed by performing the recovery study, for which the standard addition method was employed. From the recovery results it is claimed that the method can be used for estimation of GEM in solid dosage form. The results are shown in Table 2.

Method Validation

The developed method was validated for its accuracy, linearity and precision. To ascertain the accuracy of the proposed method, recovery studies were carried out by standard addition method**.** The linearity of measurement was evaluated by analyzing different concentrations of the standard solution of GEM. The Beer-Lambert's concentration range was found to be 10-70µg/ml. The precision of the proposed method was determined by performing tablet assay at different time intervals (morning, afternoon and evening) on same day (Intra-day assay precision) and on three different days (Inter-day assay precision). Results of intra-day and inter-day precision is expressed in % RSD and found to be 0.335 and 0.557 respectively.

Table 1: Optical and Statistical data

Parameters	Values
Maximum wavelength λmax	267nm
Calibration curve range	10-70µg/ ml
Molar extinction coefficient	1.71×10^4 l mol^{-1} cm^{-1}
Sandell's sensitivity	5×10^{-5} µg/cm^2
Regression equation	Y=0.0529 X+0.0643
Slope	0.0529
Intercept	0.0643
Correlation co-efficient (r)	0.9987
Limit of detection (LOD)	0.037µg/ml
Limit of quantitation (LOQ)	0.058µg/ml

Table 2: Results of Recovery Study

Drug Taken From Tablets (mg)	Amount Found (mg)	% Labeled Claim *Mean±SD
5	4.97	99.5±0.34
5	4.97	99.5±0.34
5	4.96	99.4±0.33

*Average of six determinations; SD refers to standard deviation

RESULTS AND DISCUSSION

Gemifloxacin mesylate showed wavelength maxima at 267 nm in 0.05N H_2SO_4. The calibration curve was found to be linear in the range of 10-70 µg/ ml. The correlation coefficient was found to be 0.9987 and the percentage recovery was found to be 99.4-99.5%. The excipients present in the formulation do not interfere in the estimation of gemifloxacin mesylate. The proposed method can be successfully used for its analysis and quality control of marketed solid dosage preparations with good precision, sensitivity, linearity and accuracy.

ACKNOWLEDGEMENT

We are very much thankful to Lupin Pvt Ltd, Pune for providing gift sample and to Mr. M. K. Dash, the Chairman of Dadhichi College of Pharmacy, Vidya Vihar, Sundargram, Cuttack, Orissa for providing necessary facilities to carry out this research work.

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