

Analysis of Lomefloxacin by Spectrophotometry: Development of Simple Quantitative Analytical Method

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Abstract

An attempt has been made to develop a simple, sensitive and rapid spectrophotometric method of analysis for lomefloxacin in pharmaceutical dosage form using different media such as water, 0.1N NaOH, 0.1N HCl and Chloride (Cl) buffer as solvent system. These solvent systems were used to dissolve lomefloxacin and 0.105 mg/mL stock solutions were prepared for each solvent system. Lomefloxacin solution was scanned with UV-spectrophotometer and the absorption maximum (λ_{max}) was found to be 287 nm. These solvent systems were successfully applied for analysis of five eye drop dosage form of lomefloxacin encoded as pp1, pp2, pp3, pp4 and pp5 marketed by five different pharmaceutical companies and the results were found to be satisfactory and reproducible. These solvent systems could be used for routine analysis of lomefloxacin in both research laboratories and pharmaceutical industries.

Keywords: Lomefloxacin, spectrophotometry, analysis, reproducible.

Introduction

Lomefloxacin is one of the third generation fluoroquinolones with some specific activity in upper respiratory tract infections and community acquired pneumonia. It is also used in meningitis, osteomyelitis, urinary tract infections, sexually transmitted diseases, bacteraemia, nosocomially acquired infections, gastrointestinal infections and in combination with other agents in the treatment of tuberculosis (Goodman and Gilman, 1996). A number of analytical methods (Tozo and Salgado, 2006.; Santoro *et al.*, 2006, Lyon *et al.*, 1994.; Wright *et al.*, 1998.) have been developed for the analysis of lomefloxacin for research purposes. Among these, the widely used methods for the analysis of lomefloxacin were based on spectroscopy with specific solvent system. The purpose of the present study thus was to develop handy and easily operable spectrophotometric method for the analysis of lomefloxacin in pharmaceutical dosage forms which would be simple, rapid, cost-effective and reproducible. We developed four solvent systems by long trial and error method. We, therefore, proposed a spectrometric method which is simple, sensitive and rapid for the estimation of lomefloxacin in pharmaceutical preparations (as eye drops) that can also be used for

quantitative estimation in research laboratory for research purpose and in pharmaceutical industries for routine analysis of lomefloxacin.

Materials and Methods

Drugs and chemicals: Standard lomefloxacin hydrochloride (99.99%) was supplied by Eskayef Pharma Ltd. It was collected in an air tight vial, stored in a cool and dry place and was used without further purification. *Ortho*-phosphoric acid, sodium dihydrogen phosphate and other reagents were of analytical grade, purchased from Merck, Germany. Demineralized water was used throughout the experiment.

Spectrophotometric Conditions for the proposed methods: Spectrometric apparatus - The spectrophotometric system consisted of a Shimadzu Double Beam UV-VIS 160A Spectrophotometer from Shimadzu Co., Japan. Sonicator of model-Minor from Decon Ultrasonics Co., UK.

Preparation of drug solutions: Stock solution - 10.4 mg of standard lomefloxacin was accurately weighed and taken in a 100 mL volumetric flask containing 50 mL of double distilled water. It was dissolved and diluted to 100

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mL with water. This was solution of 0.104 mg/mL and it was used as stock solution for subsequent experiments.

Determination of wavelength of maximum absorption (λ_{max}): The stock solution (0.104 mg/mL) was diluted 10 times to give a solution of 0.0104 mg/mL or 10.4 $\mu\text{g/mL}$ solution and 5 mL of this solution was taken in a cuvette and scanned from 200 to 400 nm with Shimadzu Double Beam UV-VIS 160A Spectrophotometer. The double distilled water, 0.1N NaOH, 0.1N HCl and Cl buffer were used as the blank. Lomefloxacin was found to absorb maximum radiation at 287 nm.

Calibration curve: The series of standard solutions prepared by diluting the stock solution with double distilled water, 0.1N NaOH, 0.1N HCl and Cl buffer, separately and the concentrations were 2.08 $\mu\text{g/mL}$, 3.12 $\mu\text{g/mL}$, 4.16 $\mu\text{g/mL}$, 5.2 $\mu\text{g/mL}$, 6.24 $\mu\text{g/mL}$ and 10.4 $\mu\text{g/mL}$. Absorbance of the above solutions were measured with Shimadzu Double Beam UV-VIS 160A Spectrophotometer and calibration curve was constructed by plotting absorbance versus concentration (Figures 1a-d).

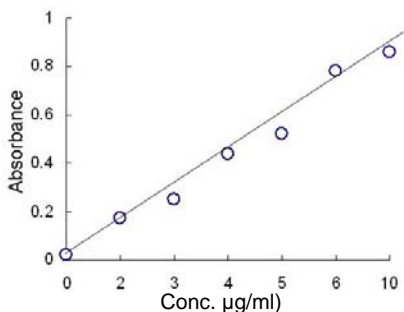


Figure 1a. Standard curve of Lomefloxacin using distilled water as solvent.

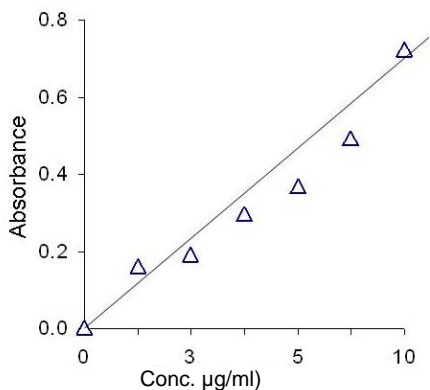


Figure 1b. Standard curve of Lomefloxacin using 0.1N NaOH as solvent.

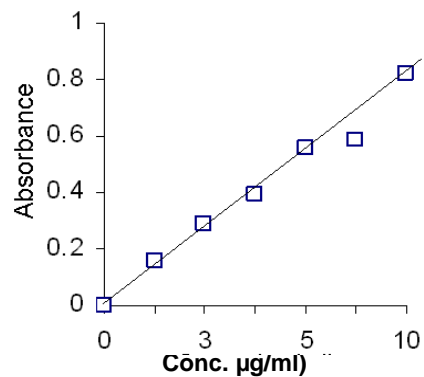


Figure 1c. Standard curve of Lomefloxacin using 0.1N HCl as solvent.

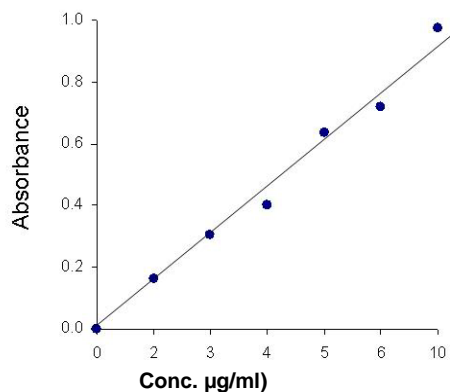


Figure 1d. Standard curve of Lomefloxacin using Cl buffer as solvent.

Assay in the dosage form: Five different marketed lomefloxacin eye drops formulations (coded as pp1, pp2, pp3, pp4 and pp5) were selected for analysis. 1 mL (equivalent to 3 mg lomefloxacin) of each pharmaceutical product was taken in 100 mL volumetric flask and then the volume was made up to the mark by adding double distilled water, 0.1N NaOH, 0.1N HCl and Cl buffer, separately. After that 5 mL was diluted to 50 mL, so that the concentration was 3 $\mu\text{g/mL}$. Absorbance of the sample solutions were measured using a spectrometer (Figure 2-5). The potencies of the five different marketed lomefloxacin were then determined from the calibration curve (Table 1).

Method validation and recovery experiment: Method validation and recovery experiments were conducted following our previous paper (Wahed *et al.*, 2007) and that of international norms (ICH, Q2B, 1995) and other investigators (Paul *et al.*, 2002, Marona *et al.*, 2001). Eye drops solution equivalent to 100 mg were taken in five 100 mL volumetric flask and 0.0, 10, 20, 30 and 40 mg

standard lomefloxacin were added to the volumetric flasks, respectively. Then the content of the each of volumetric flask was diluted with double distilled water, 0.1N NaOH, 0.1N HCl and Cl buffer, separately and the

potency was determined by the proposed method. The data of the recovery experiment were statistically analyzed to study the reproducibility and validity of the proposed method (Table 1) using following equation.

Table 1. Estimation of lomefloxacin in eye drops by proposed spectrometric method (Water as solvent, *Data are expressed as mean ± SD, where, n = 5)

Name of sample	Concentration (mg/mL)	Potency (%)* (Water)	Potency (%)* (0.1N NaOH)	Potency (%)* (0.1N HCl)	Potency (%)* (Cl buffer)
Standard	0.01-0.03	99.99	99.99	99.99	99.99
pp1	0.03	104.97 ± 0.21	94.59 ± 0.41	101.57 ± 0.32	93.53 ± 0.55
pp2	0.03	110.82 ± 3.70	95.00 ± 2.00	95.94 ± 4.11	98.93 ± 2.50
pp3	0.03	110.09 ± 2.51	96.22 ± 3.08	96.33 ± 3.50	101.93 ± 3.74
pp4	0.03	118.87 ± 2.59	96.22 ± 3.23	100.42 ± 4.01	101.93 ± 3.34
pp5	0.03	106.07 ± 2.55	94.59 ± 6.32	95.07 ± 4.34	96.75 ± 3.23

$$\% \text{ Recovery} = \frac{N\sum XY - \sum X.\sum Y}{N\sum X^2 - \sum X.\sum X} \times 100$$

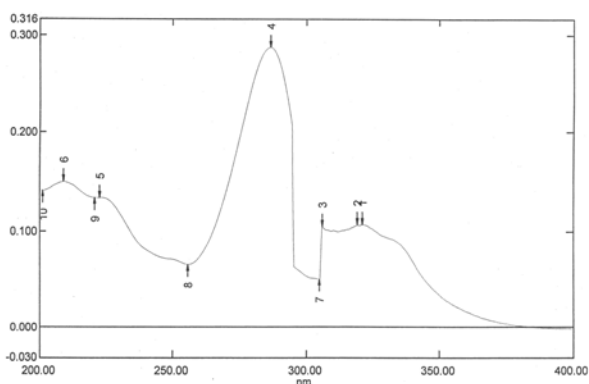


Figure 2. UV spectrum for analysis of lomefloxacin using water as solvent (PP-1)

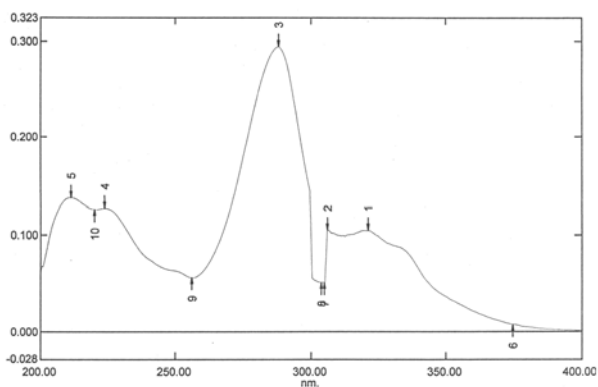


Figure 4. UV spectrum for analysis of lomefloxacin using 0.1N HCl as solvent (PP-1)

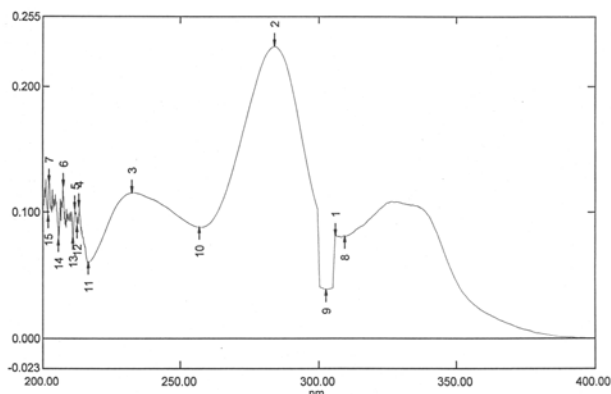


Figure 3. UV spectrum for analysis of lomefloxacin using 0.1N NaOH as solvent (PP-1)

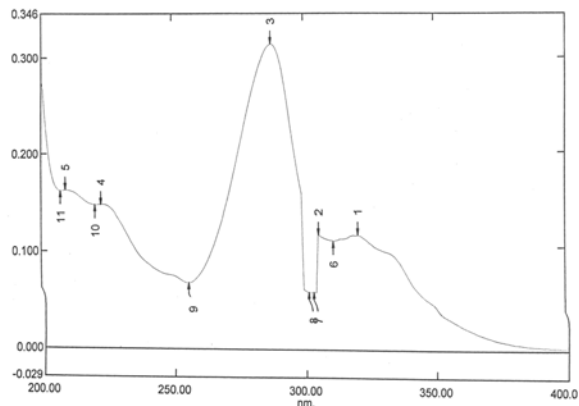


Figure 5. UV spectrum for analysis of lomefloxacin using Cl buffer as solvent (PP-1)

Results and Discussion

Double distilled water, 0.1N NaOH, 0.1N HCl and Cl buffer were established as solvents by long trial and error method for the analysis of lomefloxacin. The proposed method is simple, rapid and handy because the solvent systems were easy to prepare. It does not require any complex calculation. The standard calibration obtained by plotting known concentrations of lomefloxacin against absorbance values was found to be linear (Figure 1). Lambert-Beer's law was found to be obeyed in the concentration range of 10 to 50 mg/mL. The proposed method has also been successfully applied for the estimation of lomefloxacin in commercial eye drop preparations (coded as pp1, pp2, pp3, pp4 and pp5), the result of which are represented in Table 1. In order to confirm the reproducibility and validity of the proposed method recovery experiments were conducted following our previous papers and that of other investigators (Rahman and Shahabuddin, 2007; Paul *et al.*, 2002, Marona *et al.*, 2001). The recovery was almost 100%

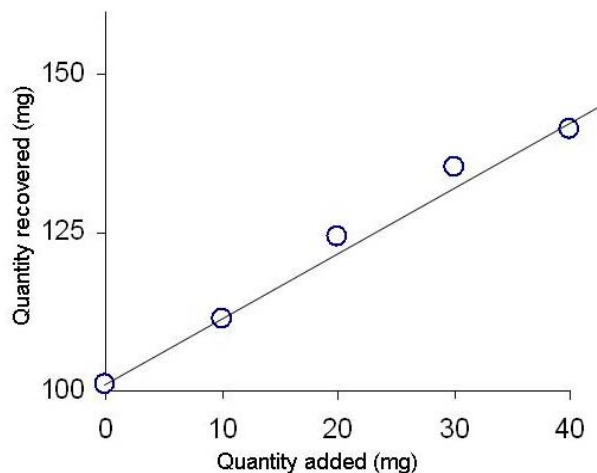


Figure 6. Data of the recovery experiment of lomefloxacin.

(99.14%) which showed that the developed method suffered no interference from common excipients used in the formulation (Figure 6). The lower values of standard deviation reflected the validity and reproducibility of the proposed method. The values of different statistical parameters indicate that the proposed method was accurate enough to give a valid and acceptable result. The calculated p value was extremely low ($p < 0.0001$) which

means that the difference of labeled potency and determined potency by the proposed method is insignificant. The shortest length of confidence interval with 95% and 99% indicates the accuracy and validity of the proposed method. The percent recovery was calculated by equation described in the text.

Conclusion

The present method offers several advantages in terms of simplicity, rapidity and accuracy over many of the known procedures and can be applied for the quality control analysis of lomefloxacin in pharmaceutical preparations.

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