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SPECTROPHOTOMETRIC DETERMINATION OF LOMEFLOXACIN IN PHARMACEUTICAL DOSAGE FORM WITH CITRIC ACID–ACETIC ANHYDRIDE REAGENT

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ABSTRACT

A sensitive spectrophotometric method is presented for determining Lomefloxacin (LOM). The drug was extracted from formulations with chloroform from an alkaline medium and reacted with citric acid–acetic anhydride reagent to produce a bluish–violet colour having absorption maximum at 580 nm. Beer's law is obeyed between 2–12μg/mL for LOM. The results agree within ±1.0% with official method.

Key words: Spectrophotometry, Citric acid, Acetic anhydride, Lomefloxain, Tablets

INTRODUCTION

Lomefloxacin (LOM), (±–1–ethyl–6,8–difluoro–1,4–dihydro–7–(3–methyl–1–piperazinyl)–4–oxo–3–quinoline carboxylic acid) is a fluoroquinoline derivative, which is a useful antibacterial agent. It is official in Martindale Extra pharmacopoeia[1] and Merck index[2]. The reported procedures for its estimation include HPLC methods[3–6] and visible spectrophotometric methods[7–10]. The analytically important functional groups of LOM namely imino and tertiary nitrogen in piperazine portion and carboxyl group in the quinoline moiety have not been fully exploited in this regard. Hence attention was focused on developing simple spectrophotometric methods exploiting the varied functional groups of the drug. In the present paper the authors present a spectrophotometric method for the estimation of LOM in its formulations based on the formation of a coloured internal salt (λ_{max} :590 nm) and involvement of tertiary nitrogen in the piperazine moiety in LOM and cis–aconitic anhydride (dehydrated product of C–A)[11] This method has been successfully used for the determination of LOM in the pure state and in pharmaceutical dosage forms.

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EXPERIMENTAL

Instrument

All spectral and absorbance measurements were made on a Systronics 106 Model visible spectrophotometer with1cm matched glass cells.

Reagents

All chemicals used were of analytical reagent grade. C–A was prepared by dissolving 1.2 g citric acid monohydrate (E.Merck) in 5 mL anhydrous methanol and diluting upto 100 mL with acetic anhydride (E.Merck).

Preparation of standard and sample drug solution

LOM (50 mg) was treated with 20 mL of 0.1N sodium hydroxide solution and transferred into 125 mL of separator. The free base of the drug was extracted with 4 successive 25 mL portions of chloroform. The total chloroform extract was filtered through a pledget of cotton carrying 2 g of anhydrous sodium sulphate and made upto 250 mL with chloroform for getting working standard solution (200 µg/mL of LOM). Tablets powder equivalent to 50 mg of the drug (LOM) was taken and sample solution was prepared in a similar manner as mentioned under standard drug solution.

Method

Aliquots of standard drug solution (1.0 – 3.0 mL, 200 µg/mL) in free base form in chloroform were taken into a series of 25 mL graduated tubes and gently evaporated on boiling water bath to dryness. To this 10 mL of 6.245×10^{-2}M C – A reagent was added and the flasks were immersed in boiling water bath for 30 min. The tubes were cooled to room temperature and were made up to the mark with acetic anhydride. The absorbances of the colored solutions were measured at 590 nm against reagent blank within the stability period of 15–60 min. The amount of drug was computed from its calibration curve.

RESULTS AND DISCUSSION

Beer'law limits, molar absorptivity, regression equation and correlation coefficient obtained for LOM by least squares treatment of the results are given in Table 1. The precision and accuracy of the method was tested by measuring six replicate samples of the drug within Beer's law limits (200 µg of LOM) and the results are summarized in Table 1.

The percent recovery values obtained are listed in Table 2. Commercial tablets of LOM were analysed by the proposed and reference methods and compared statistically by means of student's t–test and by the variance ratio F–test and no significant difference was observed. It

indicates that none of the usual excipients employed in the dosage forms interfere in the analysis of LOM by the proposed method. The proposed method is sensitive, rapid, selective and accurate for the determination of LOM in pharmaceutical dosage forms.

Table 1. Optical characteristics, precision and accuracy of the proposed method

Parameters	**C–A/Ac_2O**
λ_{max} (nm)	590
Beer's Law limits (µg/mL)	8–24
Molar absorptivity (L $mol^{-1}cm^{-1}$)	8.764×10^3
Correlation coefficient (r)	0.9999
Sandell's sensitivity (µg/cm^2/ 0.001 absorbance unit)	0.04425
Regression Equation (y = a + bc)	
(i) Slope (b)	0.02270
(iii) Intercept (a)	0.00040
Relative Standard Deviation *	0.3224
% Range of error (confidence limits)	
(i) 0.05 level	0.270
(ii) 0.01 level	0.399
% Error in bulk sample **	– 0.028

*Y = a + bc, where c is concentration of analyte and Y is absorbance unit;**Average of six determinations considered ;***Average of three determinations considered .

Table 2. Assay of LOM in pharmaceutical dosage forms

Drug*	**Lable claim mg/tablet**	**Amount found**		**% Recovery by proposed method*** **
		Proposed method**	**Reference method**	
Tablet 1	400	398.3±1.18	386.42±2.06	99.7±0.29
Tablet 2	400	400.1±1.91	396.9±0.695	100.1±0.47
Tablet 3	400	397.8±2.14	398.6±1.24	99.4±0.53
Tablet 4	400	396.9±3.0	400.79±2.48	100.2±0.48

Drugs from different pharmaceutical companies; ** Average ± Standard deviation of 6 determinations; ***Recovery of 10 mg added to the preanalysed pharmaceutical dosage forms(average of 3 determinations)

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