

NOTE

Visible Spectrophotometric Determination of Lomefloxacin in Bulk and Pharmaceutical Dosage Formulations

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Two simple and sensitive visible spectrophotometric methods are developed for the estimation of lomefloxacin in its dosage forms. In method A, lomefloxacin reacts with sodium nitroprusside and hydroxylamine hydrochloride in alkaline medium to produce a yellow-coloured chromogen having λ_{\max} at 440 nm. Beer's law is obeyed in the concentration range of 15–75 μg . In the method B, lomefloxacin reacts with chloranil and acetaldehyde to give a dark bluish coloured chromogen having λ_{\max} at 665 nm. Linearity is 3–15 μg . Commercial dosage forms of the drugs have been evaluated by the proposed methods and found satisfactory.

Key Words: Spectrophotometric analysis, Lomefloxacin.

Lomefloxacin (LOM) (\pm -1-ethyl-6,8-difluoro-1,4-dihydro-7-(3-methyl-1-piperazinyl)-4-oxo-3-quinoline carboxylic acid) is a fluoroquinolone derivative, which is useful as antibacterial agent. It is official in Martindale Extra Pharmacopoeia¹ and Merck Index². 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 focussed on developing simple spectrophotometric methods exploiting the varied functional groups of the drug. In the present paper, two spectrophotometric methods for the estimation of LOM in its formulations are presented.

All spectral and absorbance measurements were made on a Systronics 106 model visible spectrophotometer with 1 cm matched glass cells.

Sample preparation for Method A and Method B

A weighed amount of tablet powder equivalent to 100 mg of LOM was transferred into a separator with 10 mL distilled water. The separator was shaken to disperse the material and the contents were extracted with 3 \times 25 mL portions of chloroform. The total chloroform extracts were collected in a 100 mL volumetric flask and then diluted to the mark with same solvent to obtain stock solution (1 mg mL⁻¹) in chloroform.

Method A: 10 mL of the above stock solution (1 mg mL⁻¹) was taken and

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chloroform portion was evaporated to dryness and the residue was dissolved initially in 5 mL methanol and diluted to 10 mL with the same solvent. This solution was further diluted stepwise with methanol as given in the working standard drug solution and the amount of drug was estimated under the given assay procedure.

Method B: 10 mL of the above stock solution (1 mg mL^{-1}) was taken and the chloroform portion was evaporated to dryness and the residue was dissolved initially in 5 mL distilled water and diluted to 10 mL with it. This solution was further diluted stepwise with distilled water as given in the working standard drug solution and the amount of drug was estimated under the given assay procedure.

Assay procedure

Method A: Aliquots of standard LOM solution ($1.0\text{--}5.0 \text{ mL}$, $100 \mu\text{g mL}^{-1}$) were transferred into a series of 25 mL calibrated tubes and the volume in each tube was brought to 5.0 mL with distilled water. 1 mL each of ($1.678 \times 10^{-2} \text{ M}$) sodium nitroprusside and ($7.195 \times 10^{-2} \text{ M}$) hydroxylamine hydrochloride solution were successively added to each test tube and shaken for 2 min. Then 1.0 mL of ($9.43 \times 10^{-2} \text{ M}$) Na_2CO_3 solution was added and further shaken for 15 min. The contents were diluted to the mark with distilled water and the absorbances were measured at 440 nm against a reagent blank. The amount of LOM in sample solution was computed from its calibration curve.

Method B: Aliquots of standard drug LOM solution ($0.5 \text{ mL--}2.5 \text{ mL}$, $60 \mu\text{g mL}^{-1}$) were taken into a series of 10 mL calibrated tubes. Then, 0.5 mL of acetaldehyde (2% in 1,4-dioxane, v/v) and 0.1 mL chloranil ($4.067 \times 10^{-3} \text{ M}$) were added and allowed to stand for 5 min at room temperature. Finally, the solution was made up to the mark with 1,4-dioxane and the absorbances were measured at 665 nm against a reagent blank prepared simultaneously. The amount was computed from the appropriate calibration curve.

The optimum conditions were established by varying one parameter at a time and keeping the other fixed by observing the effect produced on the absorbance of the coloured species. The various parameters involved in the colour development, like the concentration of the various reagents and time involved for maximum colour development, were optimized. The optical characteristics and figures of merit are given in Table-1, together with regression equations (obtained by linear least square treatment) for the calibration plots. The precision and accuracy were found by analyzing six replicate samples containing the known amount of drug and the results are summarized in Table-1. Some commercially available dosage forms of LOM are analyzed by both the methods and the results obtained are given in Table-2. As an additional check on the accuracy of methods, recovery experiments were performed by adding known amounts of pure drug to preanalyzed dosage forms and per cent recovery values obtained are listed in Table-2.

Recovery experiments indicate the absence of interference from the commonly encountered pharmaceutical additives and excipients. The proposed methods can be employed for the routine determination of lomefloxacin in bulk sample and pharmaceutical formulations.

TABLE-1
OPTICAL CHARACTERISTICS, PRECISION AND ACCURACY
OF THE PPOPOSED METHOD

Optical characteristics	(MA)	(MB)
λ_{\max} (nm)	440	665
Beer's law limits ($\mu\text{g}/\text{mL}$)	15–75	3–15
Molar absorptivity ($\text{L mol}^{-1} \text{cm}^{-1}$)	2.087×10^3	1.0535×10^4
Correlation coefficient (r)	0.9999	0.9996
Sandell's sensitivity ($\mu\text{g}/\text{cm}^2/0.001$ absorbance unit)	0.1857	0.03681
Regression equation ($y = a + bc$)		
(i) Slope (b)	0.00541	0.02697
(ii) Intercept (a)	0.00050	-0.00230
Relative standard deviation *	0.2348	0.5416
% Range error (confidence limits)		
(i) 0.05 level	0.196	0.453
(ii) 0.01 level	0.290	0.670
% Error in bulk sample †	0.310	-0.115

*Average of six determinations considered.

†Average of three determinations.

TABLE-2
ASSAY OF LOM IN PHARMACEUTICAL DOSAGE FORMS

Drug*	Label claim (mg/tablet)	Amount found		% Recovery by proposed method‡
		Proposed method†	Reference method (LOM) ¹⁰	
Tablet 1	400	398.3 ± 1.18	386.42 ± 2.06	99.7 ± 0.29
Tablet 2	400	400.1 ± 1.91	396.90 ± 0.695	100.1 ± 0.47
Tablet 3	400	397.8 ± 2.14	398.60 ± 1.24	99.4 ± 0.53
Tablet 4	400	396.9 ± 3.00	400.79 ± 2.48	100.2 ± 0.48

* Drugs from different pharmaceutical companies.

† Average \pm standard deviation of 6 determinations.

‡ Recovery of 10 mg added to the preanalyzed pharmaceutical dosage forms (average of 3 determinations).

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