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Research Article

THREE SIMPLE SPECTROMETRIC METHODS FOR NORFLOXACIN IN BULK AND TABLET DOSAGE FORM

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Abstract

Three simple, precise and economical UV methods have been developed for the estimation of Norfloxacin in pharmaceutical dosage form. Method A is Absorbance maxima method, Norfloxacin has the absorbance maxima at 277 nm, Method B is the first order derivative spectra, the absorbance was measured at λmaxima =287 nm and λminima = 264 nm and the difference was measured for the respective concentration of standard and was plotted against concentration and regression equation was calculated. Method C applied was Area under curve (AUC), in the wavelength range of 262-289nm. Linearity for detector response was observed in the concentration range of 1-6µg/ml for all three methods. The proposed methods were successfully applied for the determination of Norfloxacin in commercial pharmaceutical preparation.The Results were validated statistically as per ICH Q2 R1 guideline and was found to be satisfactory.

Key words: Norfloxacin, UV spectrophotometry, Absorbance maxima, First order derivative, Area under curve.

Introduction:

Norfloxacin [NOR] is a second generation fluoroquinolone (quinolone) developed by Kyorin Seiyaku K.K. (Kyorin). Norfloxacin is a synthetic chemotherapeutic agent. Occasionally used to treat common as well as complicated urinary tract infections. Norfloxacin is a last resort when all other antibiotics have failed. There are currently only three approved uses in the adult population (one of which is restricted.) and the other ineffective due

to bacterial resistance. It is sold under various brand names with the most common being Noroxin. In form of ophthalmic solutions it is known as Chibroxin. Chibroxin (ophthalmic) is approved for use in children older than one year of age. Norfloxacin interacts with a number of other drugs, as well as a number of herbal and natural supplements. Such interactions increase the risk of anticoagulation and the formation of non-absorbable complexes, as well as increasing the risk of toxicity. Norfloxacin is associated with a number of serious and life threatening adverse reactions as well as spontaneous tendon ruptures and irreversible peripheral neuropathy. Such reactions may manifest long after therapy had been completed and in severe cases may result in life long disabilities. Hepatoxicity resulting in fatalities has also been reported with the use of Norfloxacin.

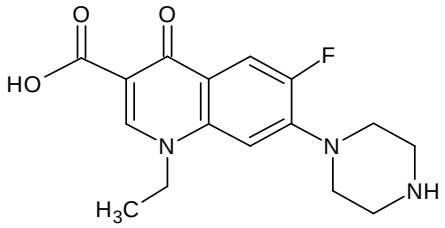

Fig. 1: Chemical structure of Norfloxacin

Method:

Materials & Reagents:

Norfloxacin- obtained as a gift sample from Aarati Drugs Ltd., Mumbai. All other chemicals of analytical grade were used.

Instrumentation:

A Jasco double beam UV–visible spectrophotometer, Model: V-530, with a fixed bandwidth(2nm) and 1-cm quartz cell. The V-530 double-beam spectrophotometer employs a single monochromator with holographic concave grating.

Solvent: Dil. Glacial acetic acid

Procedure:

Preparation of standard stock solution:

100 mg of NOR was taken in 100 ml volumetric flask, dissolved and volume was made upto mark with Dil. Glacial acetic acid. This solution was further diluted to get standard solution of concentration 100 µg/ml of NOR.

Preparation of Calibration Curve:

100mg of NOR standard powder was weighed and diluted to 100ml in Dil. Glacial acetic acid to get the concentration of 1000 g/ml. Stock solution was suitably diluted to different concentrations and linearity was studied at 277nm (λmax) against reagent blank. Linear relationship was observed in the range of 1-6 µg/ml.

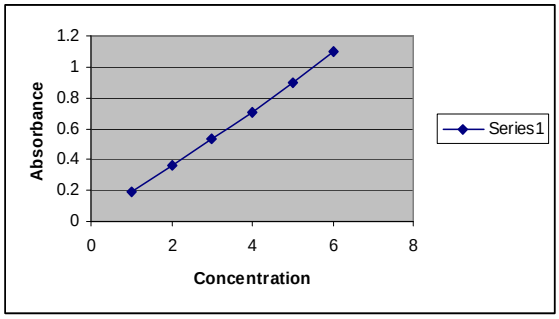

Fig. 2: Calibration Curve for Norfloxacin

Table 1: Absorbance values of Norfloxacin

Concentration µg/ml	Absorbance
1	0.1890
2	0.3540
3	0.5379
4	0.7102
5	0.8999
6	1.10

Method A: Absorption Maxima Method

For the selection of analytical wavelength, standard solution of NOR was scanned in the spectrum mode from 400 nm to 200 nm. From the spectra of drug [Fig.2], λ max of NOR, 277 nm was selected for the analysis. Aliquots of standard stock solution were made and calibration curve was prepared in the concentration range of 2-10 µg/ml at 277 nm.

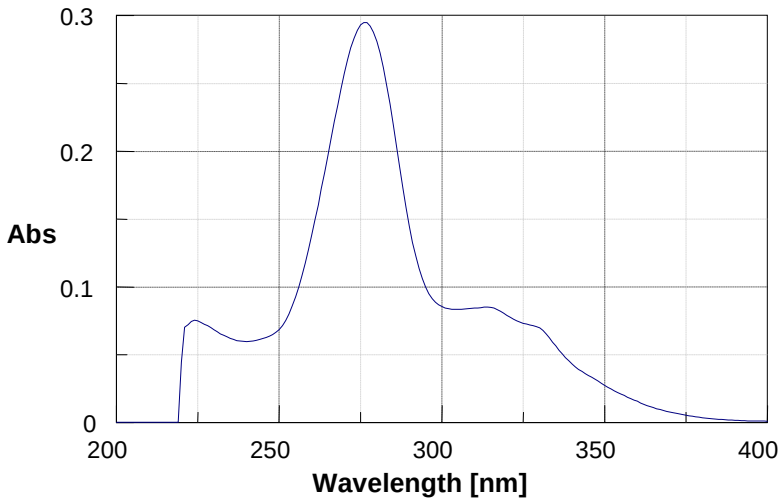

Fig. 3: λ max of Norfloxacin

Method B: First Order Derivative Spectroscopy

In this method, standard solution of NOR was scanned in the spectrum mode from 400 nm to 200 nm and the absorption spectra thus obtained were derivatized from first to fourth order. First order derivative spectra were selected for analysis of drug. First order derivative spectra of drug [Fig. 3], showed λ_{maxima} = 287 nm and λ_{minima} = 264 nm with 277 nm as zero crossing point and amplitude difference was measured for the respective concentration of standard and was plotted against concentration and regression equation was calculated. The concentration range of 1-6 µg/ml for METRO was chosen for the derivative analysis.

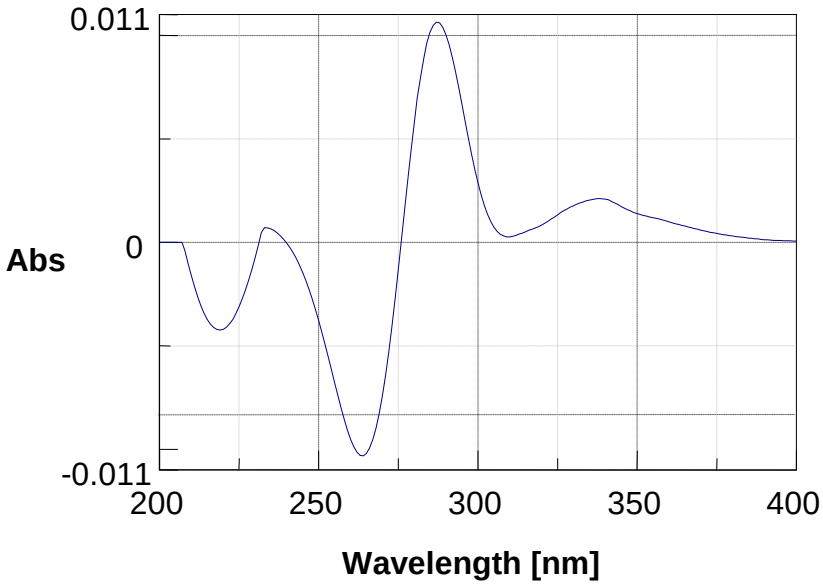

Fig. 4: First order derivative spectra of Norfloxacin

Method C: Area under Curve Method

From the spectra of drug obtained after scanning of standard solution of NOR, area under the curve in the range of 262-289 nm was selected for the analysis. The calibration curve was prepared in the concentration range of 1-6 µg/ml at their respective AUC range.

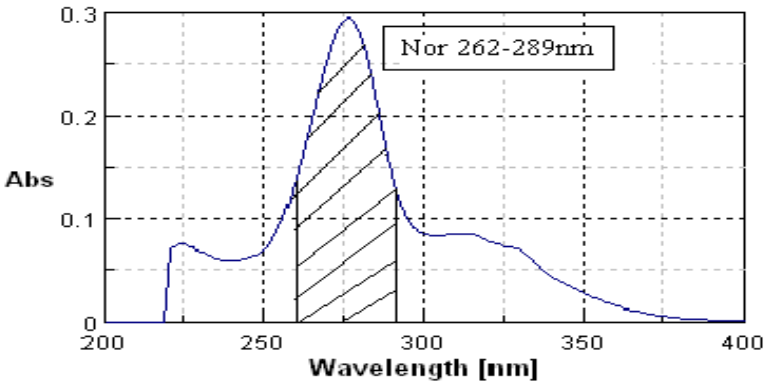

Fig.5: Area under curve of Norfloxacin

Application of the proposed methods for the determination of NOR in tablet dosage form:

For the estimation of drugs in the tablet formulation, 20 tablets were weighed and weight equivalent to 100 mg of NOR was transferred to 100 ml volumetric flask and volume was made up to the mark with water. In Method-A, the concentration of NOR was determined by measuring the absorbance of the sample at 277 nm in zero order spectrum mode. By using the calibration curve, the concentration of the sample solution was determined.

In Method-B, the concentration of NOR was determined by measuring the amplitude difference, First order derivative spectra of drug showed λ_{maxima} = 287nm and λ_{minima} = 264nm with 277 nm as zero crossing point and amplitude difference was measured for the respective concentration of standard and was plotted against concentration and regression equation was calculated.

For Method-C, the concentration of NOR was determined by measuring area under curve in the range of 262-289 nm. By using the calibration curve, the concentration of the sample solution can be determined.

Validation of the developed methods:

The methods were validated with respect to accuracy, linearity, precision and selectivity.

Accuracy: To ascertain the accuracy of proposed methods, recovery studies were carried out by standard addition method at three different levels (80%, 100% & 120%).

Linearity: The linearity of measurement was evaluated by analyzing different concentration of the standard solution of NOR.

Precision: The reproducibility 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 precision). Result of intra-day and inter-day precision is expressed in % RSD.

Results and Discussion:

The methods discussed in the present work provide a convenient and accurate way for analysis of NOR in its pharmaceutical dosage form. Absorbance maxima of NOR at 277 nm (Method A); in the first order derivative spectra, showed maxima and minima at 287 nm and 264 nm respectively (Method B) and area under curve in range of 262-289 nm (Method C) were selected for the analysis. Linearity for detector response was observed in the concentration range of 1-6µg/ml for all three methods. Percent label claim for NOR in tablet analysis, by all the methods,was found in the range of 99.72 % to 100.12% [Table 1]. Standard deviation and coefficient of variance for six determinations of tablet formulation, by all the methods, was found to be less than ± 2.0 indicating the precision of the methods. Accuracy of proposed methods was ascertained by recovery studies and the results are expressed as % recovery. Percent recovery for NOR, by all the methods, was found in the range of 99.6 % to 100.98 % values of standard deviation and coefficient of variation was satisfactorily low indicating the accuracy of all the methods [Table 2]. Percent RSD for Intraday assay precision was found to be 0.42, 0.305 and 1.07 for Method A, B and C, respectively. Interday assay precision was found to be 0.690, 0.564, 0.946for Method A, B and C, respectively [Table 3]. Based on the results obtained, it is found that the proposed methods are accurate, precise, reproducible &

economical and can be employed for routine quality control of Norfloxacin in bulk drug and its pharmaceutical dosage form.

Table 2: Results of Analysis of Tablet Formulation (N=6).

Method	Label Claim mg	Amount of drug estimated (mg/tab)	% Label Claim*± S.D.	% Recovery
A	400	398.9	398.9 ± 0.0103	99.72
B	400	400.12	100.12 ± 0.0221	100.3
C	400	400.5	100.5 ± 0.0422	100.12

Table 3: Result of Recovery studies.

Excess drug added to the analyte (%)	Recovery (%)			%RSD			SE		
	Method A	Method B	Method C	Method A	Method B	Method C	Method A	Method B	Method C
80	99.6	99.8	100.2	0.345	0.238	0.193	0.610	0.478	0.21
100	100.12	100.98	100.7	0.110	0.225	0.178	0.402	0.250	0.196
120	100.8	99.5	100.6	0.091	0.234	0.15	0.54	0.12	0.23

Table 4: Result of Intra-day and Inter-day precision.

Sr. No.	Intra-day precision			Inter-day precision		
	SD	%RSD	SE	SD	%RSD	SE
Method A	0.12	0.42	0.0863	0.120	0.690	0.160
Method B	0.172	0.305	0.168	0.191	0.564	0.786
Method C	0.296	1.07	0.175	0.13	0.946	0.197

a) RSD: Relative Standard deviation, b) SE: Standard error

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