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SPECTROPHOTOMETRIC DETERMINATION OF SPARFLOXACIN IN BULK AND DOSAGE FORMS

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ABSTRACT

A spectrophotometric study of sparfloxacin is described. The method is based on the charge-transfer complexation between sparfloxacin as n-electron donor with chloranilic acid as π-acceptor to form a violet-coloured complex having absorption maximum at 530 nm. Beer's plot is obeyed in the concentration range of 5-30 μg/ml. Results of the analysis of this method were validated statistically by recovery studies. The proposed method is simple, accurate and precise for the quantitative determination of sparfloxacin in bulk and tablet formulations.

Keywords: Sparfloxacin, spectrophotometry, chloranilic acid.

INTRODUCTION

Sparfloxacin, 5-amino-1-cyclopropyl-7-(cis-3, 5-dimethyl-1-piperazinyl)-6,8-difluoro-1, 4-dihydro-4-oxo-3-quinolinecarboxylic acid is a difluoroquinolone antibacterial agent belonging to the third generation quinolones. Clinically, it is very effective in the treatment of streptococci infections. Its mechanism of action involves the inhibition of DNA synthesis by promoting cleavage of bacterial DNA in the DNA-enzyme complexes of DNA gyrase and type iv topoisomerase, resulting in rapid bacterial death[1]. The drug is not official in any pharmacopoeia, hence no official method is available for the estimation of the drug in formulations. A number of analytical methods used for the determination of sparfloxacin in pure and dosage forms include electrochemistry[2-5]; UV-Visible spectrophotometry[6-9]; HPLC [10-13]. Chloranilic acid, 2,5-dichloro-3,6-dihydroxy-p-benzoquinone has been used largely as a spectrophotometric reagent for the determination of some organic compounds containing lone pair of electrons[14-18], but not hitherto in the assay of sparfloxacin. The present work describes the spectrophotometric determination of sparfloxacin in bulk and pharmaceutical formulations using

chloranilic acid as a chromogenic reagent.

MATERIALS AND METHODS

Materials:

Sparfloxacin (Micro Labs Limited.92, Sipcot, Hosur. India), all other chemicals were of analytical grade.

Freshly prepared 0.5 % (w/v) chloranilic acid solution in dioxan. A Hitachi UV/VIS spectrophotometer, model 2000 (Japan) was used for absorbance measurements.

Standard solution:

A stock solution of sparfloxacin (50 μg/ml) was prepared by dissolving the required amount in methanol. Standard solutions of the analyte (5-30 μg/ml) were prepared by serial dilution of the stock solution.

Proposed procedure:

An aliquot of the standard solution containing sparfloxacin was transferred into a 10 ml volumetric flask. A 1-ml volume of chloranilic acid solution (500 μg/ ml) was added and the contents were mixed thoroughly. After 30 min standing, the volume was made up with dioxan and the absorbance of the solution was measured at 530 nm against reagent blank.

Procedure for assay of dosage forms:

Ten tablets of the drug were weighed and ground to a fine powder. An adequate amount of the powder was transferred into a beaker. The powder was dissolved in methanol by stirring for 15 min. The mixture was filtered to a volumetric flask (100 ml) through Whatman filter paper No.41. The filtrate and washings were diluted to volume with methanol. A suitable volume of this solution was treated as described under proposed procedure and the drug content was evaluated. The results are given in Table 2.

Procedure for recovery of sparfloxacin:

To study the recovery of sparfloxacin, samples were prepared by mixing known amounts of pure sparfloxacin with portions of commercial preparation. The mixtures obtained were assayed by proposed method and the results are presented in Table 3.

RESULTS AND DISCUSSION

Spectrophotometric characteristic of the sparfloxacin-chloranilic acid system:

A violet-coloured complex with a ratio of sparfloxacin to reagent of 1:1 was formed when chloranilic acid solution was added to sparfloxacin solution. The complex exhibited a λ_{max} at 530 nm while the reagent showed a λ_{max} at 434 nm. An absorbance of the complex using an aliquot of the standard solution was measured at 530 nm at 30 min interval over a period of 2h. No change in the initial absorbance was observed indicating that the colour of the complex is fairly stable. Beer's law was obeyed in the range of 5-30 μg/ml. Beer's law range, molar absorptivity, slope, linear least-square analysis are given in Table 1.

Optimization of reaction conditions:

Effect of chloranilic acid concentration:

The effect of chloranilic acid concentration on the colour development was studied. It was observed that 1 ml of 0.5 % (w/v) chloranilic acid solution produced maximum colour intensity (Fig. 1).

Effect of reacting time:

The colour product developed rapidly after addition of the reagent attaining maximum intensity after 30 min at room temperature (Fig. 2). The colour was stable for over 2 h.

Stoichiometric relationship:

In order to establish the composition of the charge-transfer complex, the molar ratio method and Job's method of continuous variation using equimolar solutions of the drug (0.004 M) and reagent (0.004 M) were studied.

In the molar ratio method, the concentration of the drug was kept constant while varying the concentration of the reagent in the series of solutions prepared. In the Job's method, equimolar solutions of the drug and the reagent were mixed in complimentary proportions to a fixed total volume. The results obtained indicate that the composition of charge-transfer complex was (1:1) drug to reagent (Fig. 3).

In conclusion, the proposed spectrophotometric method was applied in the determination of sparfloxacin in bulk and pharmaceutical formulations. The method is simple, accurate, reproducible and the statistical analysis has good agreement with reported methods. The optimum conditions for the proposed method have been established and the method has shown a reasonable tolerance towards excipients. Finally, due to the minimum time required for the complexation to be complete, the proposed method can be employed for the routine analysis of sparfloxacin from bulk and tablet dosage form in quality control laboratories.

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Table 1: Optical characteristic and statistical data for the regression equation of the proposed method.

Parameters	**Values**
λ_{max} (nm)	530
Beer's law range (μg ml $^{-1}$)	5-30
Molar absorptivity (l mol $^{-1}$ cm $^{-1}$)	1.44 x 10 ^{4}
Colour stability (hours)	2
Regression equation (Y*)	
Slope	0.0347
Intercept	0.0160
Correlation coefficient (r)	0.9993
% Relative standard deviation (n = 5)	0.71

Y* = a + bC , where C is the concentration in μg ml $^{-1}$

Table 2 Determination of sparfloxacin in formulations by the proposed method.

Volume of sparfloxacin solution (ml)	Amount of sparfloxacin present (μg)	Amount of sparfloxacin found (μg)	Recovery (%)
0.2	4.0	3.9872	99.68
0.4	8.0	8.0322	100.40
0.6	12.0	12.0197	100.16
0.8	16.0	15.9774	99.86
1.0	20.0	20.2285	101.14

Mean ± SD: 100.248 ± 0.5700

Table 3: Recovery of sparfloxacin in tablet-pure sample mixture by the proposed method.

Amount of Sparfloxacin in tablets (mg)	Amount of pure sparfloxacin added (mg)	Amount of Sparfloxacin found,(mg)	Recovery (%)
200	0.0	200.5446	100.27
200	10	207.9718	99.03
200	15	213.4488	99.28
200	20	219.5189	99.78
200	25	227.7482	101.22

Mean ± SD: 99.916 ± 0.8709

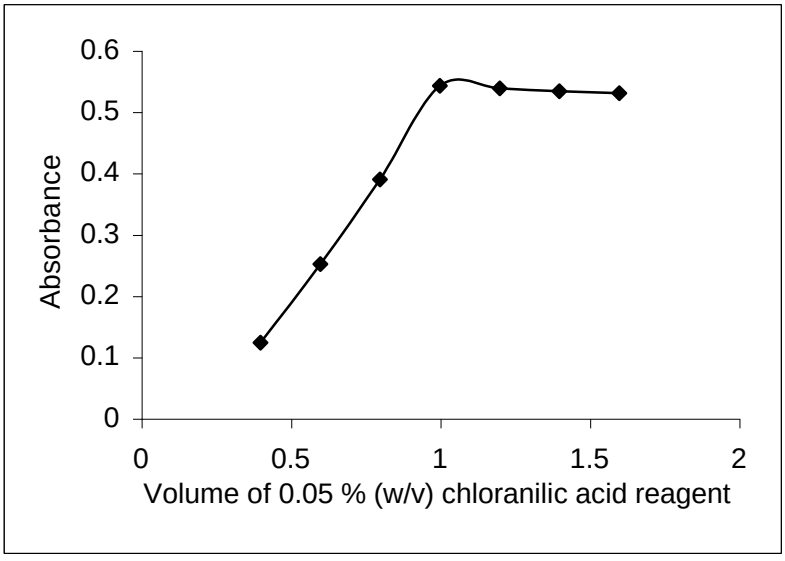

Fig. 1 : Effect of chloranilic acid reagent concentration on the development of charge-transfer complex of sparfloxacin at λ = 530 nm.

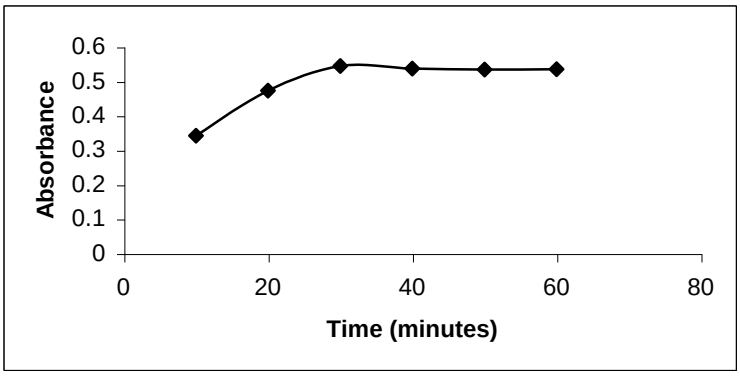

Fig. 2 : Effect of time on the development of charge-transfer complex of sparfloxacin and chloranilic acid at λ = 530 nm.

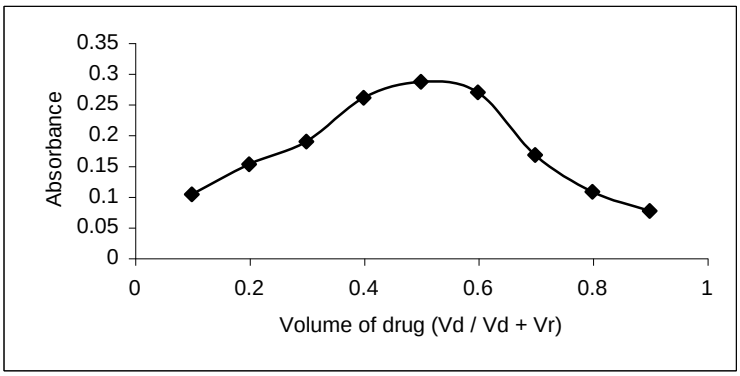

Figure 3 : Continuous variation plot for the charge-transfer complex, λ = 530 nm, where V_d and V_r are the volumes of added drug and reagent respectively.