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Ion-Pair Spectrophotometric Determination of Sparfloxacin

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A method was developed for the quantitative estimation of sparfloxacin in tablets. The method is based upon the reaction of sparfloxacin with methyl orange, forming a yellow coloured complex, which is extracted in chloroform and analyzed. The absorbance maxima (λ_{max}) was found to be 390.8 nm. Optimization of the reaction was carried out with factors such as buffer strength, stability of complex, molar ratio of drug : dye and extraction time. The proposed method was validated as per ICH guidelines. The recovery studies confirmed the accuracy and precision of the method.

Key words: Spectrophotometry, Ion-pair, Determination, Sparfloxacin, Methyl Orange, Validation.

INTRODUCTION

Chemically sparfloxacin (SFX) is 4-amino-1-cyclopropyl-6,8-difluoro-1,4-dihydro-4-oxo-7-(3,5-dimethyl piperazin-1-yl) quinoline-3-caboxylic acid. SFX is a second-generation member of quinolones and is greatly effective against both gram-positive and gram-negative bacteria. The antibacterial action is due to the ability to inhibit DNA gyrase and topoisomerase-IV that is required for DNA replication and transcription.

Various analytical methods are reported in literature, including HPLC, RPHPLC, LC-MS, HPTLC, spectrophotometry, derivative spectrophotometry, spectrofluorimetry, voltammetric, non-aqueous titrimetry, etc., but no method is official in any pharmacopoeia. This paper presents a simple, accurate, sensitive, reproducible and economic method for determination of SFX in bulk and tablet form.

EXPERIMENTAL

All the chemicals used were of analytical grade. The instruments include, UV-Vis spectrophotometer (Systronics 119), FTIR-8300 (Shimadzu) and digital pH meter (Century, CE-601).

Procedure for bulk drug: Accurately weighed 100 mg of SFX was dissolved in acetate buffer (pH 3.4) to give a stock solution having a concentration of 1000

μg/mL. From this stock solution, working standard solutions of drug (100 μg/mL) were prepared by appropriate dilutions.

Standard solutions of SFX (5, 10, 15, 20, 25, 30, 35 and 40) were transferred into a series of separating funnels. To each separating funnel, 0.5 mL methyl orange (1%) was added, mixed and kept for 5 min. The ion-pair complex was extracted by chloroform (3 × 5 mL); content of separating funnel was shaken gently for 2 min and allowed to separate for 5 min. The chloroform layer was separated out. Absorbances of the coloured extracts were measured at wavelength 390.8 nm against determination with reagent. The calibration curve was plotted between the absorbance *vs.* concentration (μg/mL) and correlation coefficient was also measured (Table-2).

TABLE-1
RESULTS OF ASSAY OF TABLETS OF SFX

S.No.	Formulation	Label claim (mg/tab)	Estimated (mg/tab)*	Found (%)	RSD
1	A	100	99.60	99.60	0.4247
2	B	200	200.20	100.20	

* Average of 5 measurements.

Procedure for tablet formulations: Twenty tablets were weighed and ground to fine powder. An accurately weighed powder equivalent to amount of drug claimed on tablet was transferred into a 100 mL volumetric flask. The powder was dissolved in acetate buffer (pH 3.4) by shaking for about 10 min. This solution was filtered through Whatmann filter paper no. 41 and the filtrate was diluted up to the mark with acetate buffer (pH 3.4). From this stock solution, working sample solutions were prepared by appropriate dilutions and analyzed by the developed method. Recovery studies carried out gave satisfactory results (Table-2).

TABLE-2
OPTICAL AND VALIDATION CHARACTERISTICS OF SFX

S.No.	Parameters		Value
1.	Beer's law limit (μg/mL)		1-40
2.	Sandell's sensitivity (mg/cm^2/0.001 absorbance unit)		0.0444
3.	Molar extinction coefficient (L mol^{-1} cm^{-1})		8.829×10^3
4.	Correlation coefficient		0.9957
5.	Regression equation (Y*)	Slope (a)	0.0171
		Intercept (b)	0.0417
6.	Precision (% CV)	Repeatability (n = 6	0.3210
		Intraday (n = 3)	0.1522
		Interday (n = 3	0.1785
7	Accuracy (% recovery)		99.77
8	Limit of detection (LOD)		0.1663
9	Limit of quantification (LOQ)		0.5040

Y* = b + ac, where "c" is concentration in μg/mL and y is absorbance unit.
CV = coefficient of variance; n = number of measurements.

RESULTS AND DISCUSSION

The proposed method was found to be accurate, simple and rapid for routine analysis of SFX. The drug reacts with methyl orange, forms a yellow coloured complex, which is extracted in chloroform and analyzed.

The method was optimized with the following parameters:

(a) Buffer strength: Various pH strengths of acetate buffer, *i.e.*, 2.8, 3.0, 3.4, 3.7 and 4.0 were tried for selection of buffer strength. The optimum buffer strength was found to be 3.4.

(b) Reaction time: Optimization of reaction time was done by measuring the absorbance at an interval of 5 min up to 60 min. A minimum of 5 min time was found to be sufficient to complete the reaction.

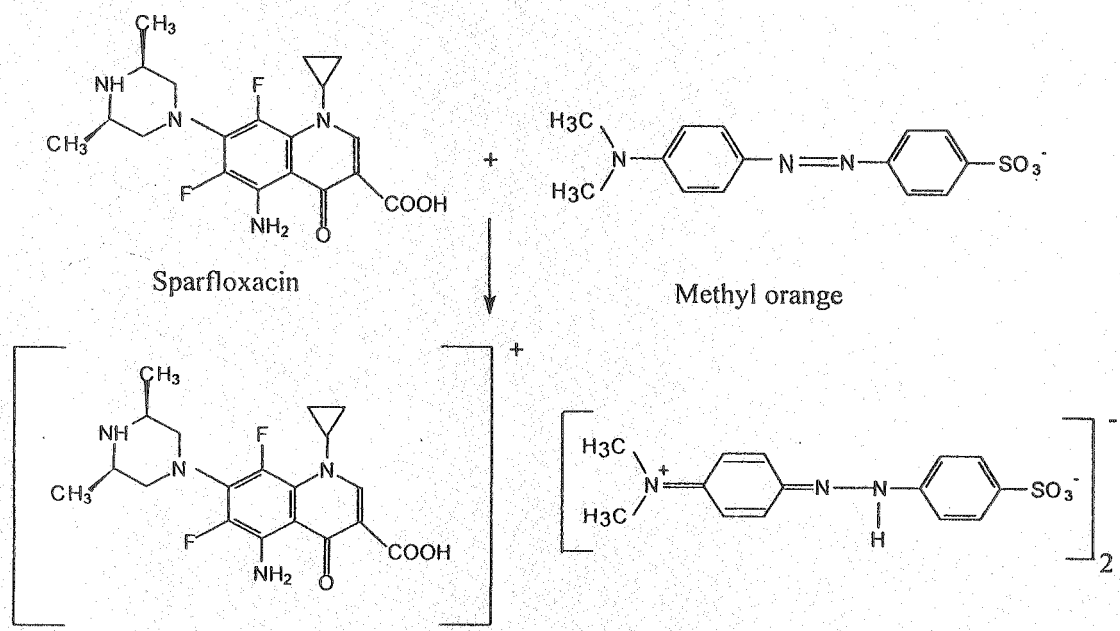
(c) Stability of complex: Stability of complex was observed and it remained stable for 70 min.

(d) Molar ratio of drug : dye: The molar ratio of drug : dye was determined by Job's method and found to be 1 : 2.

All method validation parameters of ICH guidelines were studied. The molar absorptivity and Sandell's sensitivity values show the sensitivity of drug while the precision is confirmed by % relative standard deviation (Table-1).

The results are in good agreement with labelled value (Table-1). The reproducibility, repeatability and accuracy of this method were found to be good, which is confirmed by the low relative standard deviation value. The proposed method can be successfully applied for the estimation of SFX in tablets.

Mechanism of the reaction:



Sparfloxacin : Methyl orange ion pair complex

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REFERENCES

1. J.O.N. Maryadele and S. Ann, The Merck Index, Merck Research Laboratories, NJ, USA, pp. 6730, 8810 (1996).
2. Goodman and Oilman, The Pharmacological Basis of Therapeutics, McGraw-Hill Publication, pp. 1182–1183 (2001).
3. A.I. Vogel, in: G.H. Jeffrey, J. Bassett, J. Mendham and R.C. Denney (Eds.), A Textbook of Quantitative Chemical Analysis, ELBS-Longman, p. 265 (1997).
4. H.R.N. Marona, J.A.S. Zuanazzi and E.E.S. Schapoval, *J. Antimicrob. Chemother.*, **44**, 301 (1999).
5. D.G. Sankar, J.M.R. Kumar, M.V.V.N. Reddy and T.K. Murthy, *Indian J. Pharm. Sci.*, **64**, 163 (2002).
6. R.N.M. Herida and E.S.S. Elfrides, *J. Antimicrob. Chemother.*, **43**, 136 (1999).
7. H.R. Marona and E.E Schapoyal. *J. Pharm. Biomed. Anal.*, **26**, 501 (2001).
8. M. Rizk, F. Belal, F. Ibrahim, S. Ahemad and N.M. El-Enany, *J. Pharm. Biomed. Anal.*, **24**, 211 (2000).
9. Y. Fangtian, Z. Tieli, J. Linpei, Z. Huichun and W. Shubin, *Spectrochim. Acta,* **55A**, 1119 (1999).
10. G.K. Kumar, K.P.R. Choudhary and G.R. Devala, *The Antiseptic,* **98**, 217 (2001).
11. H.R. Marona and E.E. Schapoval, *Eur. J. Pharm. Biopharm.*, **52**, 227 (2001).
12. S. Vangani, B.P. Udvadia, M. Dash, K. Ravikumar and R. Shivprakash, *J. Pharm. Biomed. Anal.*, **21**, 227 (1999).
13. D.J. Lyon, S.W. Cheung, C.Y. Chan and A.F Cheng. *J. Antimicrob. Chemother.*, **34**, 446 (1994).
14. Topic Q 2B, Validation of Analytical Procedures, International Conference on Harmonization (ICH) (2000).
15. S. Bolton, Pharmaceutical Statistics: Practical and Clinical Applications, Marcel-Dekker, Inc. New York, pp. 217, 265, 538 (1997).

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