Spectrophotometric Estimation of Sparfloxacin in Tablets

B. S. KUCHEKAR*, S. V. THAKKAR, P. P. CHOTHE, M. R. HIREMATH AND D. B. SHINDE[1]

Govt. College of Pharmacy, Vidyanagar, Karad-415 124.

[1]Department of Chemical Technology,

Dr. Babasaheb Ambedkar Marathwada University, Aurangabad-431 004.

Two methods have been developed for estimation of sparfloxacin in tablets. Method A is based on the reduction of ferric ions to ferrous ions by the drug, which further in presence of potassium dichromate as oxidizing agent produces green chromogen measured at 680 nm against reagent blank. The chromoger obeyed linearity over 1.5 to 5.5 μg/ml. Method B is based on similar reaction using ferric nitrate and potassium ferricyanide which produces blue chromogen measured at 720 nm against reagent blank. The chromogen obeyed linearity over 1.5 to 4.0 μg/ml.

Chemically, sparfloxacin is 1-cyclopropyl-5-amino-6,8-difluoro-1,4-dihydro-7(3,5-dimethyl-1-piperazinyl)-4-oxo-3-quinolinecarboxylic acid[1]. It has potent and broad spectrum activity against various gram positive and gram negative microorganisms[2,3]. It is official in Martindale Extra Pharmacopoeia[4]. Literature survey reveals that the drug has been analyzed by HPLC[5-8], microbiological[9] and spectrophotometric methods[10].

The present work describes two simple colorimetric methods for estimation of sparfloxacin in tablets. The estimation is based on the reduction of ferric ions in its salt to ferrous ions by the drug which further in presence of oxidizing agents such as potassium dichromate and potassium ferricyanide produces green and blue chromogen measured at 680 nm and 720 nm, respectively against reagent blank.

A Systronic spectrophotometer 106 with 1 cm-matched cuvettes was used for spectrophotometric estimation. Ferric nitrate reagent was prepared by dissolving 4 g of ferric nitrate in 30 ml of 1 M hydrochloric acid and making the volume to 100 ml with distilled water. Solutions of potassium dichromate (0.15% in distilled water), potassium ferricyanide (0.15% in distilled water) were freshly prepared. All the chemicals were procured from Loba Chemie, Pvt. Ltd. Mumbai.

Twenty five milligrams of sparfloxacin was dissolved in

*For correspondence

1 ml of 0.1 N sodium hydroxide solution and diluted to 100 ml with distilled water. The standard drug solution (25 μg/ml) was prepared by diluting 10 ml of this solution to 100 ml with the same solvent. Twenty tablets of sparfloxacin were weighed and powdered in a glass mortar. The amount equivalent to 25 mg of sparfloxacin was transferred to a 100 ml volumetric flask, dissolved and prepared in the same way as standard solution.

In method A, aliquots of 0.6 ml to 2.2 ml portions of standard solution were transferred to a series of 10 ml corning test tubes. To each test tube, 0.5 ml of ferric nitrate reagent and 0.9 ml of potassium dichromate were added. The solution was heated on a boiling water-bath for 2 min to complete the reaction and cooled at room temperature. The volume of each test tube was adjusted to 10 ml with distilled water. The absorbance of the solution in each test tube was measured at 680 nm against reagent blank and the calibration curve was constructed. The linearity range was found to be 1.5 to 5.5 μg/ml. Similarly the absorbance of sample solution was measured and the amount of sparfloxacin was determined by referring to the calibration curve.

For method B, aliquots of 0.6 ml to 1.6 ml portion of standard solution were transferred to a series of 10 ml corning test tubes. To each test tube, 1.2 ml of ferric nitrate reagent and 0.3 ml of potassium ferricyanide were added. The solution was heated on a boiling water-bath for 2 min to complete the reaction and cooled at room temperature. The vol-

ume of each test tube was adjusted to 10 ml with distilled water. The absorbance of the solution in each test tube was measured at 720 nm against reagent blank prepared in similar manner without addition of drug. The graph was plotted and found linear over concentration range of 1.5 to 4.0 $\mu g/$ ml. The amount of sparfloxacin in sample solution was determined by referring to the calibration curve.

These methods involve the reduction of ferric ions to ferrous ions, which further reacts with potassium dichromate (Method A) to give a green coloured chromium salt or with potassium ferricyanide (Method B) to produce the blue coloured ferrous ferricyanide. After a systematic study, the optimum parameters found for maximum color development were incorporated into the assay procedures given above. The optical characteristics such as absorption maxima, beers law limit, correlation coefficient (r), slope (m), y-intercept (c), molar absorbtivity, Sandell's sensitivity and the percent range of error (95% and 99% level confidence limit) calculated from five measurements containing ¾ of upper Beers limit and the result are incorporated in Table 1. The molar absorptivity and Sandell's sensitivity show that the methods are sensitive and percent range of error shows precision of the methods. The analysis results of marketed formulations are in good agreement with the reported method[10]. To test the accuracy and reproducibility of the proposed method, recovery experiments were performed by adding known amount of drug to the preanalyzed formulation and reanalyzing the mixture by proposed method. The recovery was performed at 0%, 100%, 200% and 250% levels for Method A and 0%, 50%, 100%, 150% and 200% levels for Method B. The recoveries ranged from 96.8% to 99.8%.

The reproducibility, repeatability and accuracy of these methods were found to be good, which is evidenced by low standard deviation. The percent recovery obtained indicates non-interference from the excipients used in the formulations. In conclusion, the methods developed in the present investigation are simple, sensitive, accurate and precise. Hence they can be successfully applied in estimation of sparfloxacin in tablets.

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TABLE 1: OPTICAL CHARACTERISTICS AND PRECISION

Observation	Method A	Method B
Absorption maxima (nm)	680	720
Beer's law limit ($\mu g/ml$)	1.5-5.5	1.5-4.0
Co-relation coefficient	0.9995	0.9994
Molar absorbtivity (lit/mole/cm)	7.941×10^4	4.052×10^4
Sandell's sensitivity ($\mu g/cm^2/0.001$)	4.942×10^{-3}	9.685×10^{-3}
Regression equation (y=mx+c) Slope (m)	0.2023	0.1032
Intercept (c)	0.0685	0.2275
% Range of error Confidence limit with 95% level	0.0013	0.0005
Confidence limit with 99% level	0.0021	0.0008

REFERENCES

1) Budavari, S., Eds., In; Merck Index, 12th Edn., Merck and Co. Inc., Whitehouse Station, NJ, 1996, 8879.

2) Kojima, T., Inoue, M. and Mitsuhashi, S., Antimicrob. Agents Chemother., 1989, 33, 1980.

3) Lewin, C.S., Morrissey, I. and Smith, J.T., J. Antimicrob. Chemother., 1992, 30, 625.

4) Reynolds, J.E.F. and Prasad, B.A., Eds., In; Martindale, The Extra Pharmacopoeia, 30th Edn., The Pharmaceutical Press, London, 1993, 202.

5) Lyon, D.J., Cheung, S.W., Chan, C.Y. and Cheng, A.F.D., J. Antimicrob. Chemother., 1994, 34, 446.

6) Borner, K., Borner, E. and Lode, H., J. Chromatogr., 1992, 579, 285.

7) EL-Sayeed, Y.M., Anal. Lett, 1995, 28, 279.

8) Bhavani, K. and Srivastava, C.M.R., Eastern Pharmacist, 1997, 456, 161.

9) Chaudhari, J.J., Sharma, A.V., Thakore, K.H. and Trivedi, J.K., Indian drugs, 1999, 36, 474.

10) Tekchandani, C., Indian drugs, 1998, 35, 229.