Spectrophotometric Determination of Sparfloxacin with Phloroglucinol

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A simple spectrophotometric method in the visible region is described for the estimation of sparfloxacin. The method is based on the formation of light green coloured species on treating diazotised sparfloxacin with phloroglucinol showing maximum absorption at 430 nm.

Sparfloxacin (SFC) is chemically 1-cyclopropyl-6,8-difluoro-1,4-dihydro-7-(3,5-dimethyl-1-piperazinyl)-4-oxo-5-amino-3-quinoline carbaxylic acid, which has broad spectrum of activity against gram positive and gram negative organisms¹⁻³. Few analytical methods, which include spectro-photometric⁴⁻⁷ and HPLC⁸⁻¹¹ have been reported for this drug. The mechanism involved in the present method is diazotisation of primary aromatic amine and coupling with phloroglucinol¹², which gives a light green colour.

A Systronics UV/Vis spectrophotometer model 117 with 10 mm matched quartz cells was used for all spectral measurements. All the chemicals used were of analytical grade. Aqueous solutions of HCI (5 N), sodium nitrite (0.1%), ammonium sulfamate (1%), and phloroglucinol (0.5%) were freshly prepared before using.

About 100 mg of SFC (pure) was accurately weighed, dissolved in 20 ml of alcohol and the total volume was made up to 100 ml with alcohol. The solution is further diluted to $100 \mu g/ml$ with distilled water.

Aliquots of working standard solution of SFC ranging from 0.5-6.0 ml (1 ml=100 μ g) were transferred in to a series of 10 ml graduated test tubes. To that 1.5 ml of aqueous solution of HCI (5 N) and 1.5 ml of sodium nitrite (0.1%) were added and kept aside for 5 min. Then 1 ml of aqueous ammonium sulphamate (1%) and 1 ml of phloroglucinol reagent (0.5%) were successively added and diluted to 10 ml with distilled water. The absorption of the lighter green coloured species formed was measured at 430 nm against

a reagent blank.

Sparfloxacin [SPARMAX (Micro Labs) and BLUSPAR (Blue Cross)] tablets were weighed and finely powdered. A quantity of the tablet powder equivalent to 100 mg of the drug is dissolved in 20 ml of alcohol and the total volume was brought to 100 ml with alcohol. Working sample solutions of 100 µg/ml were prepared with distilled water and the analytical procedure described above was followed.

TABLE 1: OPTICAL CHARACTERISTICS AND PRECISION.

Beer's law limit (µg/ml·¹)	5-30
Sandell's sensitivity (µg cm-² 0.001 absorbance unit)	0.083
Molar absorptivity (I mol-1 cm-1)	4.76 X 10 ³
% Relative standard deviation	0.707
% Range of error	
Confidence limit with 0.05 level	± 0.592
Confidence limit with 0.01 level	±0.0998
Correlation coefficient (r)	0.999
Regression equation (y*)	
Slope (a)	0.013
Intercept (I)	0.0004

 $Y^* = I + a C$, where "C" is concentration in μ g/ml and Y is absorbance unit.

^{*}For correspondence

TABLE 2: ESTIMATION OF SPARFLOXACIN IN PHARMACEUTICAL FORMULATIONS.

Sample	Labelled amount (mg)	Amount found (mg)		Percent recovery of
		Reported method ¹⁰	Proposed method	the proposed method*
1	200	198.9	199.4	99.7
2	200	199.5	199.0	99.5

^{*}Average of six determinations.

The optical characteristics such as, Beer's law limits, Sandell's sensitivity, molar extinction coefficient, correlation coefficient, % relative standard deviation and % range of error (0.05 and 0.01 confidence limits) were calculated and the results were summarized in Table 1.

The results showed that the present method have reasonable precision. Comparison of the results obtained with the proposed and the reference method for dosage forms (Table 2) confirm the suitability of this method for pharmaceutical dosage forms. Interference studies revealed that the common excipients and other additives usually present in the dosage form did not interfere in the proposed method. In conclusion the proposed method is simple, rapid, and sensitive with the reasonable precision and accuracy and it can be used for the determination of SFC in bulk as well as in its pharmaceutical formulations.

REFERENCES

1. Reynolds, J.E.F., Eds., In; Martindale, The Extra Pharmaco-

- poeia, 30th Edn., The Pharmaceutical Press, London, 1993, 202.
- Budavari, S., Eds., In; The Merck Index, 12th Edn., Merck and Co., Inc., Whitehouse Station, NJ, 1996, 1492.
- Krishnan, P. V. V., CIMS, Vol. 20, Bio-Gard Pvt. Ltd., Bangalore, 1997, 37.
- Borner, K., Borner, E. and Lode, H., J. Chromatogr. Biomed. Appl., 1992, 579, 285.
- 5. EL-Sayeed, Y. M., Anal. Lett., 1995, 28, 279.
- Lyon, D.J., Cheng, S.W., Chan, C.Y. and Cheng, A.F.B., Chem. Abstract, 1994, 121, 16300w.
- Bhavani, K. and Srivastava, C.M.R., Eastern Pharmacist, 1997, 40, 161.
- 8. Tekchandani, C., Indian Drugs, 1998, 35, 229.
- Kasture, A.V., Preetha, M. and Tipre, D.N., Indian Drugs, 1998, 35, 239.
- Meyyanathan, S.N., Sebastian, M. and Suresh, B., Eastern Pharmacist, 1998, 41, 129.
- Chowdary, K.P.R., Girish Kumar, K. and Devala Rao, G., Indian Drugs, 1999, 36, 239.
- 11. Reddy, M.N., Sankar, D.G., Rao, G.D. and Jagannath, G., Eastern Pharmacist, 1991, 36, 125.

Investigation of the Antidiarrhoeal Activity of Holarrhena antidysenterica

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Holarrhena antidysenterica (L)—Apocyanaceae, well known for its antidiarrhoeal activity was studied for its effect on diarrhoeagenic Escherichia coli. Different dilutions of the decoction of the plant were assayed for its effect on the adherence and toxin production of 2 groups of E.coli-

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