ISSN: 0975-766X
Available Online through
www.ijptonline.com
Research Article

A NEW SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION OF SPARFLOXACIN IN BULK AND PHARMACEUTICAL DOSAGE FORMS BY SODIUM DICHROMATE CHROMOGEN

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Received on 02-06-2010 ***Accepted on 25-06-2010***

ABSTRACT

A new, simple, sensitive and rapid spectrophotometric method was developed for the determination of sparfloxacin in bulk and pharmaceutical dosage forms. The method was based on the formation of a reddish brown chromophore with 0.03N sodium dichromate and concentrated hydrochloric acid showing the absorption maximum about 530nm. The proposed method has permitted the quantification of sparfloxacin over linearity in the range of 250–500 µg/ml. The method was validated as per the ICH guidelines.

Key words: Visible spectroscopy, Sparfloxacin, Chromophore

INTRODUCTION

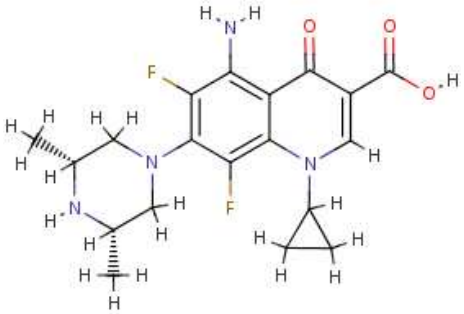

Fig.1. Chemical structure of Sparfloxacin

Sparfloxacin, it is an amino fluoroquinolone with broad spectrum of antibacterial activity against gram positive and gram negative anaerobic bacteria with bactericidal effect. Chemically, sparfloxacin is 5 – amino – 1 – cyclopropyl – 7 – (cis 3, 5 dimethyl – 1 – piperazinyl) -6, 8 difluoro 1, 4 dihydro – 4 –ono – 3 quinoline carboxylic acid. The chemical structure of Sparfloxacin was shown in fig.1. It elicits pharmacological action by inhibition of DNA replication of bacteria by inhibiting DNA gyrase activity. Sparfloxacin is official in Martindale Extra Pharmacopeia[1]. Literature survey revels that a variety of HPLC methods[2-3], visible and UV Spectrophotometric methods[4-9] were reported for the estimation of sparfloxacin. Here in, an attempt was made to develop a new sensitive spectrophotometric method for quantitative estimation of sparfloxacin. In present communication, we were developed a simple visible spectroscopic method with considerable precision, accuracy and sensitivity for the estimation of sparfloxacin in bulk and pharmaceutical dosage forms at 530 nm wavelength.

Experimental

Reagents and materials

The pure standard of sparfloxacin was obtained as a gift sample from Aurabindo pharmaceuticals, Hyderabad. The purity of the standard was found to be 99.97% and it was established by spectral conformation. A Shimadzu-UV-Vis double beam spectrophotometer-1601, with 1cm quartz matched cells was used for spectral measurements. All the chemicals which are used for performing of work are of A.R grade from S.D.FINE chemicals, Mumbai. The 0.03N sodium dichromate, concentrated hydrochloric acid and sparfloxacin tablets was employed for this study.

Preparation of working standard solution

Standard solution of sparfloxacin was prepared by dissolving 100 mg of sparfloxacin in 100ml of distilled water to get 1 mg/ml solution. For the stock solution series of dilutions (2.5-.5ml) were made to 10ml to obtain the concentrations of 250, 300,350, 400, 450, 500 µg/ml.

Preparation of sodium dichromate solution

Sodium dichromate (0.03N) solution was prepared by weighing accurately 1.49gm of sodium dichromate and dissolve in 100ml of distilled water.

Determination of absorption maximum

250 µg/ml of sparfloxacin was scanned against reagent blank and the λ_{max} was found to be 530nm.

Construction of linearity

Aliquots of standard solution ranging from 2.5-5 ml of Sparfloxacin were transferred in to six separate 10 ml volumetric flasks. To the flasks 1 ml of 0.03 N sodium chromate solution and I ml of concentrated hydrochloric acid was added. The flasks were kept aside for three minutes for color development. Appropriate volume of distilled water was added to each flask to bring the total volume to 10ml. and the absorbance of final reddish brown color chromophore was measured at 530nm against reagent blank. A calibration graph was plotted and regression equation was calculated. The measured absorbances were plotted against concentration that reveals the beer's law concentration lies between 250-500 µg/ml.

Preparation of sample stock solution

10 tablets of each formulation T_1 and T_2 containing 200mg of sparfloxacin are accurately weighed and powdered. A weight equivalent to 100mg of sparfloxacin was weighed from the powdered tablets and transferred into a 100ml volumetric flask. 30ml of distilled water was added and shaken on a mechanical shaker for 10min. The volume is made up to 100ml with the same and filtered through whatmann filter paper. The stock solution was used for further analysis.

Assay of marketed formulation

Aliquots of working standard solution (1000mcg/ml) of the drug ranging from 2.5 – 5ml (2500 – 5000mcg/ml) were transferred to a series of 10ml volumetric flasks. Then to each flask 1ml of 0.03N sodium dichromate and 1ml of concentrated hydrochloric acid was added and kept aside for three minutes

for color development. Appropriate volume of distilled water was added and the volume was made up to 10ml. The absorbance of the reddish brown colored chromophore of any concentration of the linearity range was measured at 530nm against reagent blank. The amount of sparfloxacin present in the sample solution was computed from the standard plot.

RESULTS AND DISCUSSIONS

The optimum conditions were established by changing one parameter at a time and keeping the others fixed and by observing the effect produced on the absorbance of the colored species. Various parameters involved in the color development like the concentration of the various reagents, volume and time involved for maximum color development were optimized. The reddish brown colored chromophore formed in method may be due to the oxidation of the amino group present in the quinolone ring of the drug by oxidizing agents like sodium dichromate. The optical characteristics such as beer's law limit, molar extinction coefficient, sandell's sensitivity and statistical parameters correlation coefficient, slope intercept of regression analysis and standard deviation, relative standard deviation were calculated for the proposed method and the results were incorporated in table.1.

Table 1: Optical characteristics of sparfloxacin.

S.NO	Parameter	Values	
		Sparfloxacin	**Sparfloxacin (Tablet)**
1.	λmax (nm)	530	530
2.	Beer's law (μg /ml)	250-500	250-500
3.	Regression equation*	-	-
	a. Slope b. Intercept c. Correlation Co-efficient	0.0008 -0.0066 0.9982	0.0010 -0.0072 0.9976
4	Molar extinction coefficient ($1 mole^{-1}.cm^{-1}$)	$0.337X 10^3$	$0.345 x10^3$

5	Sandell's sensitivity ($\mu g/cm^2$/0.001-absobance unit)	6.4338	6.761
6	% Range of errors** 95%Confidence interval 99%Confidence interval	± 0.0122 ± 0.0014	±0.0137 ±0.0018
7	% RSD	± 0.7437	±0.7223

*Y= a+bc, where c is the concentration of analyte and Y is the absorbance unit

Estimation of sparfloxacin in marketed formulation

The assay for the marketed tablets of sparfloxacin was established with present optimized spectrophotometric conditions and it was found to be more accurate and reliable. The results were shown in table-2.

Table 2: Assay of sparfloxacin

Brand name	Label claim (mg/tab)	Amount estimated* (mg/tab)	Mean ±S.D
TABLET-1	200	199.4817	199.4817 ± 0.0256
TABLET-2	200	199.5933	98.9567 ± 0.0377

*Mean of five values

Accuracy of the method

To study the accuracy, reproducibility of the proposed method, the recovery studies were carried out by addition of standard drug solution to preanalysed samples. Results of recovery studies were found to be satisfactory and were presented in table-3.

Table 3: Recovery of the method

Drug	Amount Added (mg)	Amount recovered* (mg)	% Recovery
Sparfloxacin	250	248.93±0.12	99.57
	300	299.45±0.31	99.81
	400	395.78±0.43	98.94

*Mean of three values

Precision of the method

The intraday and inter day variations of the method were established using six injections of three concentrations and they are analysed on the same day and three different days over a period of two weeks. The results were obtained was satisfactory and they are lying within the limits. The results were shown in table-4.

Table 4: Precision of method

Drug name	Concentration (µg/ml)	Observed concentrations			
		Intraday	%cv	Inter day	%cv
Sparfloxacin	250	249.09	0.77	249.66	1.72
	300	301.25	0.55	299.13	0.77
	350	351.25	0.69	349.15	0.92

*Mean of six values

Conclusion

The results indicate that the above proposed methods were simple, rapid and sensitive with reasonable precision and accuracy which makes it as choice for routine quality control analysis. There was no interference of excipients present in tablet formulation through out the experimental process that reflects the accuracy and precision of method.

Acknowledgement

The authors are thankful to Department of Pharmacy, College of Public Health and Medical sciences, Jimma University, Jimma, Ethiopia, for providing laboratory facilities and Aurabindo Pharmaceuticals, Hyderabad for providing gift samples of sparfloxacin.

REFERENCES

1. Extra Pharmacopoeia of Martindale. The Pharmaceutical Press, London, 31, 223.
2. Bhavani K and Srivastava C. M. R. The Eastern Pharmacist 1997; 476:121.
3. Borner K, Borner E, and Lode H. J. Chrom Biomed Appln 1992; 579: 285-289.
4. Tekchandani Chetna. Indian Drugs 1998: 35(4); 229-233.
5. Meyyanathan SN, Sebastian M and Suresh B. The Eastern Pharmacist 1998; 484:129-130.
6. Preetha Mathai, Tipre DN and Kasture AV. Indian Drugs 1998; 35(4): 239-240.
7. Meyyanathan SN, Sebastian M and Suresh B. Indian Drugs 1998; 35(6): 344-347.
8. Baburao Ch, Sreekanth N, Ramalingam P and Vijaya Kumar B. Acta Ciencia Indica 2008; XXXIV C (3): 423.
9. Sreekanth N, Bahlul Z. Awen and Babu rao Ch. Research journal of pharmaceutical, biological and chemical sciences 2010; 1(2): 9-13.

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