Asian Journal of Chemistry; Vol. 25, No. 11 (2013), 5965-5967

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.14204

Determination of Cefoperazone Sodium in Pharmaceutical Formulations by Fe^{3+}-Phenanthroline Spectrophotometry

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(*Received*: 18 June 2012; *Accepted*: 22 April 2013) AJC-13378

A new method for determination cefoperazone by spectrophotometry was developed based on Fe^{3+} as the oxidizer of cefoperazone and phenanthroline as the colouring reagent of Fe^{2+}, which was produced from Fe^{3+}. Under the optimum conditions, the relationship between the absorbance and the concentration of cefoperazone was linear in the range of 0.50-12.50 mg/L, the regression equation was A = 0.1179 c (c: mg/L) +0.0003, the relative coefficient was 0.9985. The proposed methods have been applied to the determination of cefoperazone content in pharmaceutical formulations with satisfactory results.

Key Words: Spectrophotometry, Cefoperazone, Phenanthroline.

INTRODUCTION

Cefoperazone is one of the third-generation semi-synthetic cephalosporins antibiotic active against a wide range of Gram-positive and Gram-negative bacteria, including β-lactamases produced by *Enterobacteriaceae* and *Pseudomonas* spp[1]. Several analytical techniques have been used for quantification of cefoperazone in pharmaceutical formulations and human biological fluids. These include spectrophotometry[2-6], spectrofluorimetry[7,8], colourimetry[9], capillaryzoneelectrophoresis[10], voltammetry[11,12] and high-performance liquid chromatography[13-18].

This study aimed to describe a fully validated, simple and sensitive method. The use of Fe^{3+}-phenanthroline for the determination of cefoperazone method has not been reported before. In the weak acid medium Fe^{3+} could be reduced to form Fe^{2+} by cefoperazone. Then in the pH 5 Clark-Lubs buffer solution Fe^{2+} reacted with phenanthroline to form an orange complex. It has a maximum absorbance at 510 nm. So a new method for determination of cefoperazone was established on the base of these reactions. It has been applied to determine the content of cefoperazone in pharmaceutical formulations with satisfactory results.

EXPERIMENTAL

TU-1901 spectrophotometer (Beijing Purkinje General Instrument Co. Ltd.) were used. All reagents used were of analytical reagent grade and the solutions were prepared with distilled water unless otherwise specified.

A stock standard solution of cefoperazone (the China National Institute for the control of pharmaceutical and biological products, the quality percentage is 98.6 %) at a concentration of 250 mg/mL was used. Working standard solutions were obtained by appropriate dilution of the stock standard solution. Fe^{3+}: 500 mg/L, it was prepared by dissolving 4.2672 g $(NH_4)_2SO_4 \cdot Fe_2(SO_4)_3 \cdot 24H_2O$ with 10 mL 4 mol/L HCl and diluted with distilled water in a 1 L volumetric flask; 1,10-phenanthroline solution (Phen): 0.01 mol/L; F^-: 10 g/L; Clark-Lubs buffer solutions over the pH range of 1.0-4.4 were prepared according to ref.[19]. In this case 0.2 mol/L KCl solution, 0.2 mol/L HCl and 0.2 mol/L potassium acid phthalate were the buffer components.

Accurately remove appropriate amount of 50 mg/L cefoperazone sodium standard solution in a 10 mL colourimetric tube equipped with plug. Followed by adding 1 mL of 500 mg/L ferric solution, then this solution in the colour comparison tube was reacted in boiling water bath for 70 min. After removing from the electric-heated thermostatic water bath, the solution was immediately cooled with water, treated with 1 mL 0.01 moL/L phen solution, 2 mL pH 5.0 C-L buffer solution and 1 mL 10 g/L F^- solution which was added to mask the residual Fe^{3+}, then completed to volume with distilled water, mixed well and stood for 5 min. The absorbance was measured at 510 nm against a reagent blank in a 1 cm quartz cell.

RESULTS AND DISCUSSION

Determination of wavelength: Under the experimental conditions, the absorption spectra of cefoperazone + Fe^{3+}+ 1,10-phenanthroline system and Fe^{3+} + 1,10-phenanthroline system were scanned against the reagent blank respectively in the wavelength range of 440-540 nm (Fig. 1). As could be seen that the absorbance at 510 nm of cefoperazone + Fe^{3+} + 1,10-phenanthroline system was larger than that of the Fe^{3+} + 1,10-phenanthroline system and the absorbance was increased with the concentration of the cefoperazone. So 510 nm was the selected for the further study.

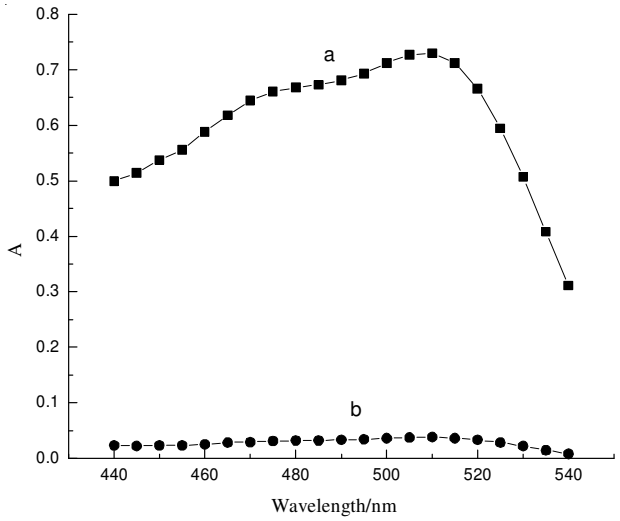

Fig. 1. The absorption spectrum (against water blank) a) cefoperazone + Fe^{3+} + Phen; b) Fe^{3+}+ Phen

Effect of pH: The effect of pH on the absorbance was examined over the range from pH 2-10. Clark-Lubs buffer solution was chosen to study the effect of reaction medium. The result was shown in Fig. 2. The absorbance showed a maximum and stable value at pH 2-9. The influence of different amounts of Clark-Lubs buffer solution (pH 5) on the system was also studied. The presence of 0.50-3.00 mL of Clark-Lubs solution gave a maximum and constant absorbance. Thus, pH 5 as one of the test conditions was selected and the amount of buffer solution was chosen to 1.5 mL.

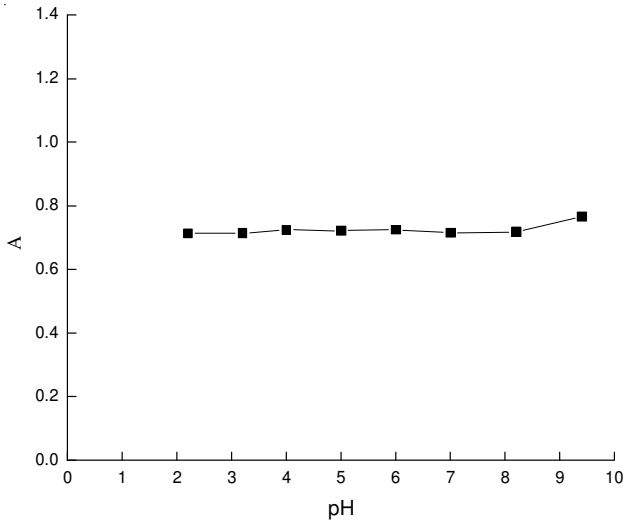

Fig. 2. Effect of pH

Effect of reaction temperature: For oxidation-reduction reaction between cefoperazone sodium and Fe^{3+} was not reacted at a lower temperature, a higher temperature could make this reaction proceeded fast. So we must consider the effect of the reaction temperature. Only by changing the reaction temperature, the result was shown in Fig. 3. It could be seen that the absorbance increased with the rising of temperature. In order to facilitate control and obtain a higher reaction rate, 100 ºC was selected.

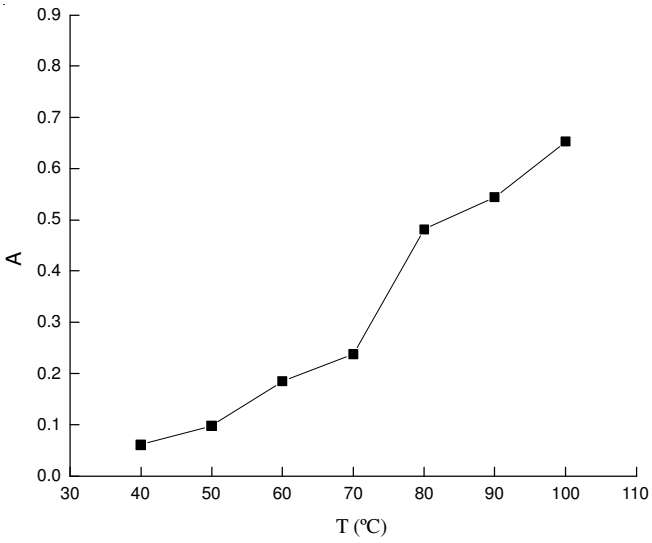

Fig. 3 Reaction temperature

Effect of reaction time: The reaction time of cefoperazone sodium and Fe^{3+} was also important parameter. After heating 30, 40, 50, 60, 65, 70, 75, 80 min respectively, the solution was cooled to room temperature and coloured with 1,10-phenanthroline. The result showed that after 1 h water bath heating the absorbance got a maximum value and kept stable (Table-1). Therefore heating 70 min at 100 ºC was chose for the further experiment.

TABLE-1
EFFECT OF REACTION TIME

Time /min	30	40	50	60	65	70	75	80
A	0.535	0.588	0.627	0.678	0.676	0.676	0.671	0.676

Effect of the amount of Fe^{3+}: The effect of Fe^{3+} and 1,10-phenanthroline was studied respectively. The result was shown in Fig. 4. When the amount of Fe^{3+} was in the range of 1-3 mL, the absorbance was maximum and stable. So the amount of 500 mg/L Fe^{3+} was fixed at 1.00 mL.

Effect of the amounts of 1,10-phenanthroline: Under the experimental conditions, the effect of the amount of added 1,10-phenanthroline in the systems has been investigated on the absorbance and the result was illustrated in Fig. 5. As indicated in this figure, when the amount of 1,10-phenanthroline was in the range of 0.50 mL-3.00 mL, the absorbance was maximum and unchangeable. So in this work, 1.50 mL 0.01 mol/L phenanthroline solution was adopted.

Characteristics of the method: Under the optimum conditions, the calibration graph was linear in the range 0.50 mg/L-

12.50 mg/L. The calibration equation is A = 0.11791c (mg/L) + 0.0003 (c: mg/L) with a correlation coefficient of 0.999. The limit of detection (DL), defined as DL = 3SB/m, was 0.09 mg/L.

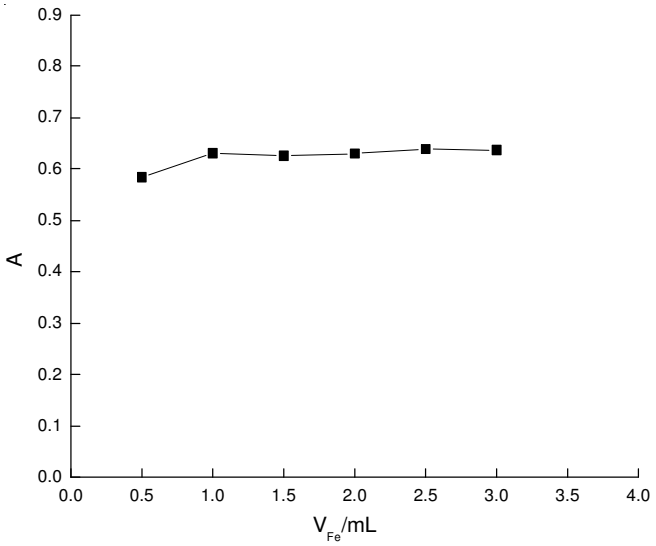

Fig. 4. Effect of the amounts of Fe^{3+}

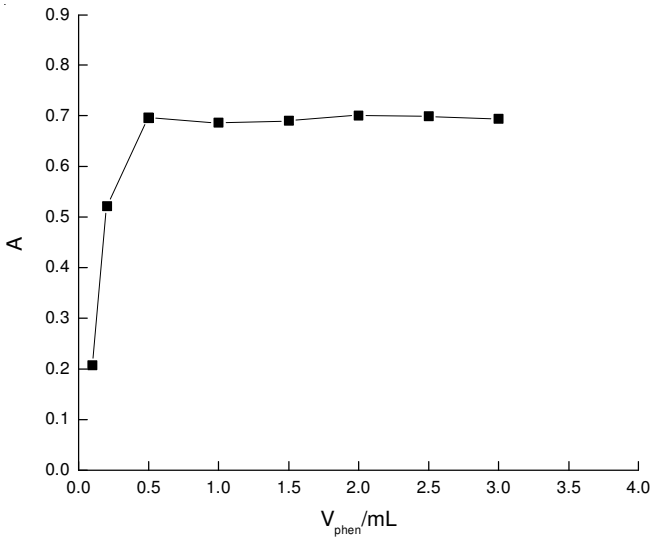

Fig. 5. Effect of the amount of phen

Sample analysis and recovery experiment: Accurately weigh a certain amount of cefoperazone sodium pharmaceutical formulations obtained from local pharmacy, completely dissolved into the 250 mL volumetric flask and diluted to volume with water. Appropriate amount of the cefoperazone sodium solution was placed into a 10 mL colourimetric tube and the experimental procedure was followed the step in 2.2 mentioned above. The analytical results were shown in Table-2. The recovery examination was carrying out by adding certain amounts of standard solution to this sample solution and HPLC method was also adopted to compare with result obtained from the proposed method[18]. It can be seen that a recovery in the range of 99.0 % to 100.6 % can be obtained.

TABLE-2
DETERMINATION RESULTS OF CEFOPERAZONEIN DIFFERENT SAMPLES (n = 6)

Order	Sample	RSD (%)	Method (%)	HPLC (%)	Added (mg)	Found (mg)	Recovery (%)
1	110423	1.04	97.29	96.74	5.00	4.96	99.2
2	110628	0.97	98.97	98.27	5.00	4.95	99.0
3	110826	0.57	99.01	98.81	5.00	5.03	100.6

Conclusion

A simple and rapid method for the determination of cefoperazone in the pharmaceutical formulations has been proposed. The method does not require a pre-treatment process. The method has the advantage of simple, reproducible, selective and sensitive. It is well suited for the determination of trace cefoperazone in the pharmaceutical formulations and got a satisfactory result.

ACKNOWLEDGEMENTS

The authors acknowledged the financial support from the Foundation of College Student Innovation Training Program of Huaihai Institute of Technology in 2012, the Subject Foundation of Huaihai Institute of Technology (No. Z2011020) and the Foundation of Jiangsu Provincial Research Institute of Marine Resources in 2011(JSIMR11B26).

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