Spectrophotometric and Conductometric Analysis of Cerium-Ofloxacin Complex

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A simple spectrophotometric method for the estimation of ofloxacin through the formation of complex with cerium has been developed. The yellow coloured complex has a λ_{max} at 243 nm and obeys Beer's law in the concentration range 0 to 22 mg/mL. The stoichiometry of the complex has been determined by spectrophotometric and conductometric methods. The formation of the complex is not affected by change in pH. Being a simple, rapid and economical method it can be used in routine laboratory work.

Key Words: Spectrophotometric, Conductometric analysis, Cerium-ofloxacin complex.

INTRODUCTION

Ofloxacin is a fluorinated carboxy quinolone bactericidal agent with a broad-spectrum activity. It is structurally related to nalidixic acid. Ofloxacin completely eliminates the aerobic gram-negative bacilli but does not affect the anaerobic gram-negative organisms. It is the most active compound tested against chlamydia trachomatis and is moderately active against *Mycobacterium* bacteria.

Earlier methods of estimation of ofloxacin include fluorimetric analysis¹. HPTLC² and complexation with folin ciocalteu reagent³. A new spectrophotometric method has been developed based on the formation of a coloured complex of ofloxacin with ceric ammonium nitrate. The stoichiometry of the complex has been determined. The results are further confirmed by conductometric analysis.

EXPERIMENTAL

Standard ofloxacin solution (0.01 M) was prepared by dissolving 361 mg of pure ofloxacin in rectified spirit by heating on boiling water bath. After cooling, the volume was made to 100 mL. Ceric ammonium nitrate (CAN) (0.01 M) was prepared by dissolving 548 mg in 100 mL of distilled water.

Spectral and absorbance meadurements were made on Shimadzu UV 160 A UV-Visible recording spectrophotometer. Conductometric titrations were performed using digital conductivity meter Century CC-601 and digital pH-meter Century CC-901.

Verification of Beer's law: Equal instalments of standard drug solution (100 mg/mL) were added to an excess of CAN, final volume raised to 10 mL and optical density measured at λ_{max} 243 nm. The value of optical density was plotted against concentration of ofloxacin.

Stoichiometric study

(i) Mole ratio method: A series of solutions were prepared where concentration of metal ion (CAN) was kept fixed. Concentration of ofloxacin was increased stepwise.

Exactly 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.4, 1.6, 1.8, 2.0, 2.2, 2.4 and 2.6 mL of drug solution was transferred into separate 10 mL volumetric flasks. 1 mL of CAN was added to each flask and volume was made up to 10 mL. Optical densities were recorded. The method was carried out with equimolar and non-equimolar solutions of reactants.

(ii) Job's method: The sum of molar concentrations of ofloxacin and CAN was kept constant and volume of ofloxacin vs. CAN was varied. Exactly 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0 mL of standard drug solution was transferred to separate 10 mL flasks and 5.0, 4.5, 4.0, 3.5, 3.0, 2.5, 2.0, 1.5, 1.0, 0.5 and 0.0 mL of CAN was added to the above flasks. Total volume was kept constant. Optical densities were recorded and plotted against volume of the variable component.

Conductometric titration: 10 mL of 0.01 M drug solution was taken and diluted to 50 mL with conductivity water and stirred. After 5 min conductivity was observed as blank. 0.01 M CAN solution was added in instalments of 0.5 mL and conductivity observed after each addition till a constant value was obtained. Conductance values were plotted against the volume of CAN added.

Stoichiometric studies

- (i) Mole ratio method: A series of solutions with fixed metal ion concentration and gradually increasing drug concentration were prepared. Total volume was kept 25 mL in each case and conductivities were recorded. Both equimolar and non-equimolar solutions were used for these determinations.
- (ii) Job's method: Series of solutions were prepared as described above; total volume was kept 25 mL and conductivity recorded and was plotted against the volume of the variable component. The effect of pH on the stability and conductivity of the complex was also determined.

RESULTS AND DISCUSSION

Golden yellow colour formed by addition of CAN to ofloxacin solution indicates the formation of a complex. The golden yellow colour of the complex is stable for more than 5 h. Thus CAN was found to be a satisfactory reagent for complexation.

The λ_{max} for cerium-ofloxacin complex was found to be at 243 nm (Fig. 1)

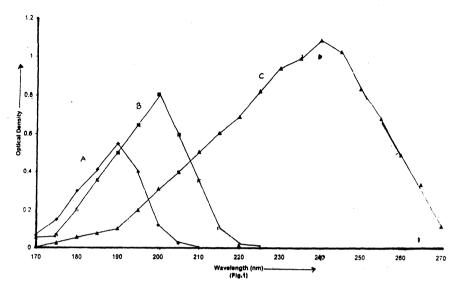


Fig. 1. Absorption Spectra of A.Ceric Ammonium Nitrate (0.01 M) B. Ofloxacin (0.01 M) C. Ce-Ofloxacin complex.

and Beer's law was obeyed in the concentration range 0-22 mcg/mL (Fig. 2). Spectrophotometric studies were carried out using Job's method and Mole ratio

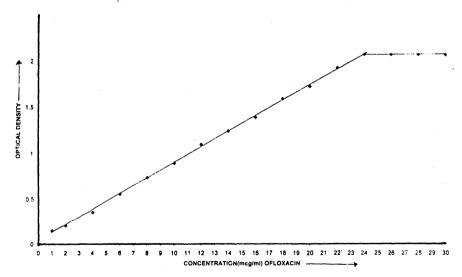


Fig. 2. Calibration Curve for Ceriun-ofloxacin Complex.

method. The plots for both these methods indicate the formation of only one complex between cerium and ofloxacin under the conditions of study and also confirmed a stoichiometric ratio 1:1 for the cerium-ofloxacin complex (Figs. 3 and 4).

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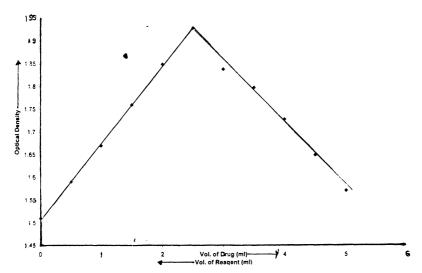


Fig. 3. Stoichiometry of Cerium-ofloxacin Complex Job's (Equimolar) Method. [Drug] = 0.01 M, [Reagent] = 0.01 M

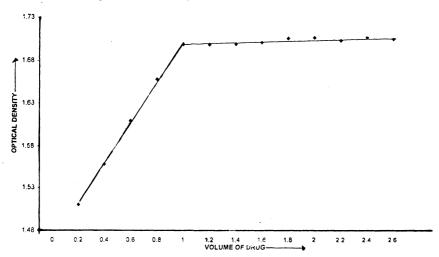


Fig. 4. Stoichiometry of Cerium-ofloxacin Complex Mole Ratio (Equimolar) Method. [Drug] = 0.01 M, [Reagent] = 0.01 M

Conductometric measurements further confirm the above results. They also show that formation of complex is not affected by changes in pH. The proposed method is simple, rapid and economical and gives reproducible results; hence it can be used in routine laboratory analysis.

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