

Spectrophotometric determination of Fe (II) using ternary complex with SCR and 1,10-phenanthroline

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ABSTRACT

Fe (II) forms a coloured ternary complex with SCR in presence of 1,10-Phen which have molar absorptivity of $3.56 \times 10^4 \text{ mole}^{-1} \text{ cm}^{-1}$ at pH 5.0 and λ_{max} 520 nm. The complex contains three molecules of 1,10-Phen coordinated to Fe (II) ion and one molecule of SCR through ion association giving the overall formula Fe (II) (SCR) (Phen)₃. Beer's law range is 0.0223-1.6084 ppm and Sandell's sensitivity 0.0045 ν/cm^2 .

Key words: Spectrophotometric studies, SCR (Solochrome cyanine R), 1,10-phenanthroline.

INTRODUCTION

Recently studies have been made on binary¹⁻² and ternary³⁻⁵ complexes of SCR for photometric determination of several cations. Use of cationic surfactant was implemented first time by Mal'at⁶ and West⁷. Attempts have been made for spectrophotometric determination of Fe (II) with other chromophoric reagents^{8, 9}.

The present paper describes the spectrophotometric studies on ternary complex of Fe (II) with Solochrome Cyanine R (Tri sodium salt of 2'-sulpho-3,3'-dimethyl-4-hydroxyfuchson-5,5'-dicarboxylic acid, abbreviated as SCR, synonym: Eriochrome Cyanine R) and 1,10-Phenanthroline (abbreviated as 1,10-Phen) at pH 5.0. 1,10-Phen has extensively been used as one of the constituents in the study of a variety of binary^{10,11} and ternary¹²⁻¹⁵ complexes. In the present investigation it is observed that there is much increase in the absorbance which gives better condition for complex formation and characterisation. The proposed spectrophotometric method is suitable and sensitive for the determination of minute amounts of Fe (II).

EXPERIMENTAL

The solution of Fe (II) was prepared by dissolving weighed quantity of Ferrous Ammonium Sulphate in redistilled water containing small amount of dil. H₂SO₄. Fe (II) solution was standardized by usual volumetric method. A stock solution of SCR (0.001 M) was prepared by weighed amount in redistilled water. Freshly prepared solution was always used. The solution of 1,10-Phen (0.01 M) was prepared by dissolving weighed amount in redistilled water. Working solutions were prepared by appropriate dilution with redistilled water. 0.2 M solutions of sodium acetate and acetic acid were used for the preparation of acetate buffer. All the chemicals used were of AR grade.

Absorbance of solutions were measured with a SPEKOL Spectrophotometer. An ELICO direct reading pH indicator with a glass-calomel electrode system was employed for pH measurement.

The total volume of the mixtures was kept at 25 ml. All measurements were made at room temperature (25°C). The order of addition of

reactants was Fe (II) solution, SCR solution, buffer solution and finally 1,10-Phen solution. The order of addition of reactants has no significant effect on absorbance and stability of the colour.

RESULTS AND DISCUSSION

Fig. 1 shows absorption spectra of solutions containing SCR; SCR and 1,10-Phen; Fe (II) and SCR; Fe (II) and 1,10-Phen; Fe (II), SCR and 1,10-Phen (in the ratio of 1:1:1 and 1:1:4) at pH 5.0 and the formation of only one ternary complex was ascertained. Hyperchromic shift in absorbance was observed which gives sensitivity and absorptivity of the method. The colour formation was observed to be immediate and remained constant up to 24 hrs.

For determining the pH stability range of complex a number of mixtures containing Fe (II) and SCR in 1:1 ratio and large excess of 1,10-Phen solution were prepared at different pH and their spectra were recorded (Fig. 2A). Absorbance of the mixtures were then plotted against pH (Fig.2B). The pH range of stability is from 4.4 to 5.4 at λ_{\max} of the ternary complex and this pH range is considered as the pH stability range.

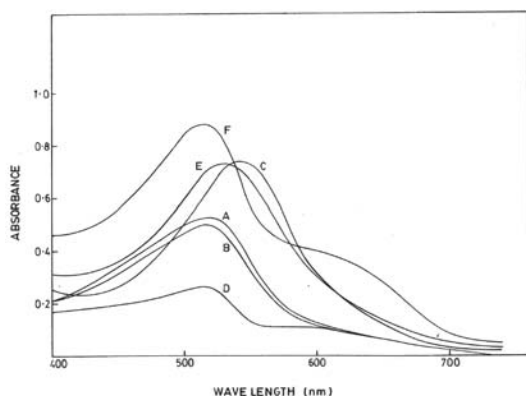


Fig. 1: Absorption spectra of solutions containing SCR (A), SCR + Phen (B), Fe (II) + SCR (C), Fe (II) + Phen (D), Fe (II) + SCR + Phen (in the ratio of 1:1:1) (E), Fe (II) + SCR + Phen (in the ratio of 1:1:4) (F); Fe (II) ($4.0 \times 10^{-5} \text{M}$), SCR ($4.0 \times 10^{-5} \text{M}$), Phen ($4.0 \times 10^{-5} \text{M}$); pH 5.0

The continuous variation¹⁶ and mole ratio¹⁷ methods (Fig.3) were used to ascertain the composition of Fe (II) and SCR (1:1) in the ternary system investigated in presence of suitable amount of 1,10-Phen. Mixtures containing Fe (II) and SCR in ratio 2:1, 1:1, 1:2, 1:3 and 1:4 ratio and different amounts of 1,10-Phen at pH 5.0 were prepared and the spectra of each mixture was recorded. Absorbance of these mixtures at λ_{\max} of the ternary complex were plotted against the amount of 1,10-Phen. From these plots the concentration range of 1,10-Phen over which the absorbance of the ternary complex remained constant was determined. Absorption spectra of the mixtures containing constant amount of Fe (II) and SCR solutions and varying amount of 1,10-Phen were recorded at pH 5.0 (Fig.4). The number of 1,10-Phen in the ternary complex has been determined by measuring the absorbance of mixtures containing Fe (II) and SCR in the stoichiometric ratio and different amount of 1,10-Phen at λ_{\max} and plotting the absorbance against the amount of 1,10-Phen. It was observed that three fold excess of 1,10-Phen is required for maximum colour formation. The ternary complex investigated is thus formulated as Fe (II) (SCR) (Phen)₃.

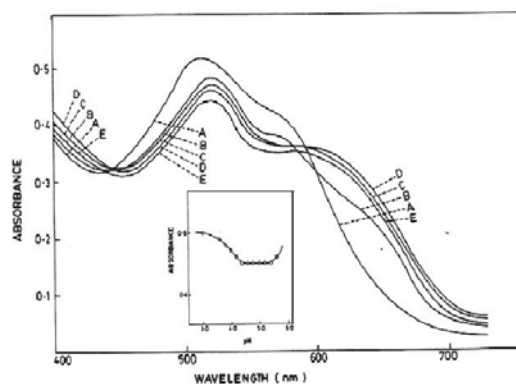


Fig. 2: (A) Absorption spectra of Fe (II)-SCR-Phen ternary complex at different pH; Fe (II) ($4.0 \times 10^{-5} \text{M}$), SCR ($4.0 \times 10^{-5} \text{M}$), Phen ($8.0 \times 10^{-5} \text{M}$); pH 3.0 (A), 4.0 (B), 4.4 (C), 5.0 (D), 5.4 (E). (B) Variation of absorbance at 520nm of Fe (II)-SCR-Phen ternary complex with pH; Fe (II) ($4.0 \times 10^{-5} \text{M}$), SCR ($4.0 \times 10^{-5} \text{M}$), Phen ($8.0 \times 10^{-5} \text{M}$).

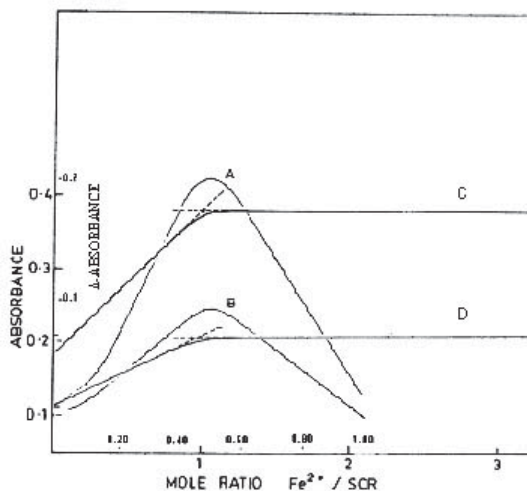


Fig. 3: Job's plots (A, B) and mole ratio plots (C, D) for Fe (II)-SCR-Phen ternary system; A, Fe(II) ($2.0 \times 10^{-4} \text{M}$), SCR ($2.0 \times 10^{-4} \text{M}$), Phen ($2.0 \times 10^{-4} \text{M}$) at 515nm; B, Fe (II) ($1.33 \times 10^{-4} \text{M}$), SCR ($1.33 \times 10^{-4} \text{M}$), Phen ($1.33 \times 10^{-4} \text{M}$) at 500nm; C, Fe (II) ($2.0 \times 10^{-4} \text{M}$), SCR ($4.0 \times 10^{-5} \text{M}$), Phen ($2.0 \times 10^{-4} \text{M}$) at 515nm; D, Fe(II) ($1.33 \times 10^{-4} \text{M}$), SCR ($2.66 \times 10^{-5} \text{M}$), Phen ($1.33 \times 10^{-4} \text{M}$) at 500 nm; pH 5.0

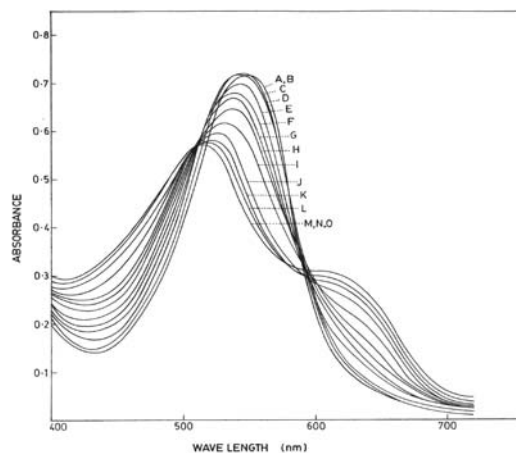
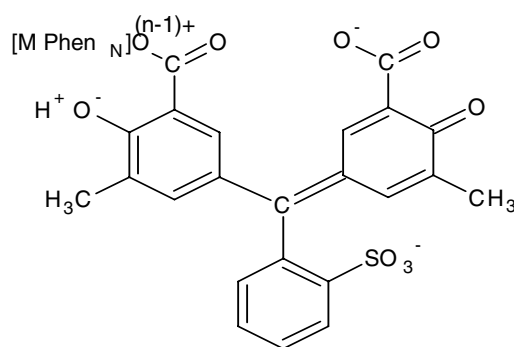


Fig. 4: Absorption spectra of Fe (II)- SCR-Phen ternary complex in presence of different amounts of Phen; Fe (II) ($3.2 \times 10^{-5} \text{M}$), SCR ($9.6 \times 10^{-4} \text{M}$), Phen A (0), B ($0.64 \times 10^{-5} \text{M}$), C ($1.28 \times 10^{-5} \text{M}$), D ($1.6 \times 10^{-5} \text{M}$), E ($1.92 \times 10^{-5} \text{M}$), F ($2.4 \times 10^{-5} \text{M}$), G ($3.2 \times 10^{-5} \text{M}$), H ($4.0 \times 10^{-5} \text{M}$), I ($4.8 \times 10^{-5} \text{M}$), J ($5.6 \times 10^{-5} \text{M}$), K ($6.4 \times 10^{-5} \text{M}$), L ($8.0 \times 10^{-5} \text{M}$); M ($9.6 \times 10^{-5} \text{M}$), N ($1.12 \times 10^{-4} \text{M}$), O ($1.28 \times 10^{-4} \text{M}$); pH 5.0

In the present investigation on the basis of experimental results obtained and mechanism proposed by Dagnall and West^{18, 19}, it appears that Fe (II) is primarily associated with 1,10-Phen molecule in a purely co-ordinate bonding arrangement and produces a change in the SCR molecules which is closely allied to release of the available strongly bonded protons. However, in the present investigation it appears that the last proton is not released and only a charge transfer is involved because in the Fe (II)-SCR-1,10-Phen ternary complex only colour intensification takes place. The ternary complex molecule can thus be represented by the following structure.



M= Fe (II)
N= 3
n= 2

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The spectrophotometric methods based on the formation of such type of ternary complexes are very sensitive and selective used for micro-determination of Fe (II).

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