



DISTRIBUTION OF IRON (II) BETWEEN BUFFERED AQUEOUS SOLUTIONS AND CHLOROFORM SOLUTION OF *N,N'*-ETHYLENEBIS(4-BUTANOYL-2,4-DIHYDRO-5-METHYL-2-PHENYL-3H-PYRAZOL-3-ONEIMINE)

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ABSTRACT

The distribution of Fe(II) between buffered aqueous solution and chloroform solution of *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine)(H₂BuEtP) was investigated. The effect of 4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one(HBuP) in the distribution was also studied. Fe(II) concentration in aqueous raffinate was determined colorimetrically and distribution ratios and percentage extractions calculated by difference from Fe(II) in aqueous phase before and after equilibrations. The optimal pH for Fe(II) distribution slightly lowered to 8.00 in mixed ligands(H₂BuEtP/HBuP) organic phase from 8.25 in ligand (H₂BuEtP) alone organic phase. The pH range at which quantitative extraction of Fe(II) from aqueous buffered solution into both type of organic phases used for the study was very narrow. The extraction parameters; pH_{1/2}, log D from extraction plots, and log K_{ex} calculated using extraction equations derived from slope analysis showed that the values in both type of organic phases were very close even though mixed ligands(H₂BuEtP/HBuP) organic phase values were slightly higher and better. Slope analysis also indicated that the Fe(II) complexes extracted into both organic phases were Fe(BuEtP)_o and Fe(HBuEtP)(BuP)_o respectively.

Indexing terms/Keywords

Aqueous media; buffered; distribution; Fe(II); *N,N'*-ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine); organic phases.

Academic Discipline And Sub-Disciplines

Chemistry; Analytical chemistry

SUBJECT CLASSIFICATION

Applied Chemistry; Solvent Extraction

TYPE (METHOD/APPROACH)

Many organic phases has been extensively studied for the extraction and separation of various metal ions from aqueous media using solvent-solvent extraction technique. Results from these studies have been utilized in designing methods for treating metals from industrial effluents and recently in dating of materials through extraction and separation of radioactive nuclides that have a parent and daughter relationship.

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1. INTRODUCTION

Solvent extraction of metal ions using Schiff bases has generated lots of interesting results over the years. The successes recorded in these studies have been utilized in the synthesis of a wide range of metal complexes with varying properties; trivalent Samarium, Europium and Gadolinium complexes of tridentate salicylidene hydrazone derivatives of 4-acylpyrazolone-5¹ have shown that their solid complexes have fluorescence properties and the thiosemicarbazone² Schiff base derivatives exhibited biochemical³, photo chromic² and acid chromic properties due to tautomerism in their molecular structures. There are also reports of promising antitumor, antipyretic and anti-inflammatory activity of Schiff bases^{3,4}. These extractions have also been successfully applied in separation of metal ions in aqueous solutions owing to varying degree of extraction of metal at different pH, in the presence of mineral acids, common anions and auxiliary complex agents^{5,6}.

In the quest to discover more efficient extractant with a wide pH range of extraction of metal ions, new Schiff bases *N,N'*-Ethylenebis(1-phenyl-3-methyl-4-acylpyrazoloneimine) and its derivatives *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) (*H₂BuEtP*), *N,N'*-Bis(1-phenyl-3-methyl-4-acetylpyrazoloneimine)-1,2-propane (*H₂ADPP*) and *N,N'*-Ethylenebis(1-phenyl-3-methyl-4-propionylpyrazoloneimine) (*H₂PrEtP*) have been successfully synthesized, characterized using ultraviolet, infrared, ¹H and ¹³C NMR⁷ and have also shown excellent extraction abilities for Ni (II)⁸, Pb(II)⁹ and U(VI)^{10,11}.

In continuation of our evaluation of metal ions extraction with these synthesized 4-acylbis(1-phenyl-3-methyl pyrazoloneimines), we report the distribution of Fe(II) from aqueous solution into chloroform solution of *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) (*H₂BuEtP*) organic phase. The effect of 4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (*HBuP*) in the distribution of Fe(II) between the two phases was also investigated. The research is aimed at determining the potentials of *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) (*H₂BuEtP*) in the extraction of Fe(II) from aqueous solution with the objective of determining the pH range at which quantitative extraction of Fe(II) can be achieved.

2. MATERIALS AND METHOD

All reagents and chemicals used in the study were all analytical grade from BDH and Aldrich. 4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (*HBuP*) (figure. 1) and *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) (*H₂BuEtP*) (figure. 2) were synthesised by method described elsewhere⁷. The ligands were recrystallized from aqueous ethanol and its purity established by elemental analysis for C, H and N. The synthesised ligand *N,N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) melting point was determined with a melting point apparatus to be 234^oC. Measurement of IR and NMR spectral data were done at the Institut fur Anorganische Chemie, Technische Universitat Dresden, Germany. Stock solutions of 0.05M *H₂BuEtP* and 0.05M 4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (*HBuP*) were prepared by dissolving appropriate mass of the ligands in CHCl₃. A 2000mgL⁻¹ stock solution of Fe²⁺ was prepared by dissolving 1.404g of Ferrous Ammonium Sulphate [(NH₄)₂SO₄.FeSO₄.6H₂O] in 100ml volumetric flask containing 2ml of sulphuric [H₂SO₄] made up to the 100ml mark with deionized water. Buffer solutions were prepared with 0.1 M HCl/0.1 M NaCl (pH 1.0 – 3.0), 0.1 M acetic acid/0.1 M NaCl (pH 3.0 – 3.5), 0.1 M acetic acid/0.1 M Na-acetate (pH 3.6 – 5.6) and 0.1 M KH₂PO₄/0.1 M NaOH (pH 5.7 – 9.0). The actual pH of solutions were determined with a Labtech Digital pH meter.

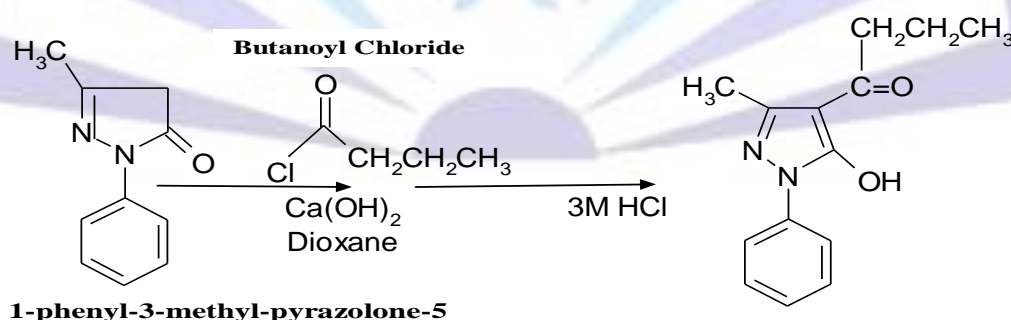


Fig 1: Reaction for the synthesis of 4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (*HBuP*)

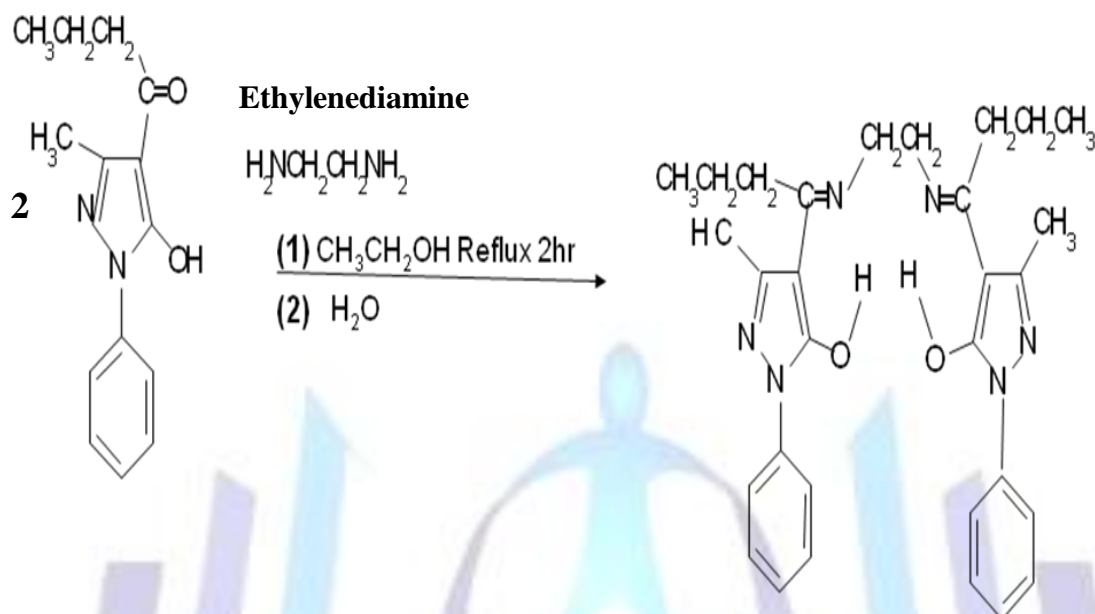


Fig 2: Reaction for the synthesis of *N, N'*-Ethylenebis(4-butanoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-oneimine) (H_2BuEtP)

2.1 Extraction Procedure

10 mL corked extraction bottles containing 2 mL aliquot of buffered solutions containing 200 mgL^{-1} of F (II) ions were prepared. Two millilitre (2 mL) solutions of 0.05 M concentration of H_2BuEtP or 0.05M H_2BuEtP :0.05 M $HBuP$ (9:1 ratio by volume) in chloroform was pipetted into the aqueous phases in the extraction containers. The immiscible phases were shaken mechanically for 40 minutes at a room temperature of $30\text{ }^\circ\text{C}$. A shaking time of 40 minutes was found suitable enough for equilibration. The two phases were allowed to settle and separated.

The concentration of Fe(II) in the aqueous phase was determined colorimetrically with a UV spectrophotometer (Spectronic 20 Genesys) at wavelength of 520nm^5 . The colour development for Iron(II) determination was by addition of 0.1ml of hydroxylamine hydrochloric acid, 0.5ml of 1,10-phenanthroline and 0.5ml of sodium acetate. Fe(II) ion concentration extracted into the organic phase was determined from the difference between the concentration of Fe(II) ion in aqueous phase before and after the extraction. Distribution ratio D was calculated as the ratio of metal ion concentration in the organic phase (C_o) to that in the aqueous phase (C). Thus $D = C_o/C$.

3. RESULTS AND DISCUSSION

The addition of H_2BuEtP organic phase to the aqueous phase containing Fe(II) at pH range of 8.00 – 8.25 resulted in the immediate formation of a purple coloured solution. This was not observed at other pH and indicates that at these 8.00 – 8.25 pH range, coloured Fe complex species formed. The coloured Fe complex with H_2BuEtP can be evaluated for use as a colorimetric reagent for Fe analysis. The presence of ligand H_2BuEtP alone, plots of $\log D$ against pH shown in figure 3 had a slope of 2, indicating that two hydrogen ions were displaced during the extraction reaction. Figure 4, showing plots of $\log D$ against $\log [H_2BuEtP]$ had a slope of 1 and Figure 6 plots of $\log D$ against $\log Fe(II)$ had slope of 0. These results indicate that 1 mole of the ligand H_2BuEtP , reacted with 1 mole of Fe(II) during the extraction reaction.

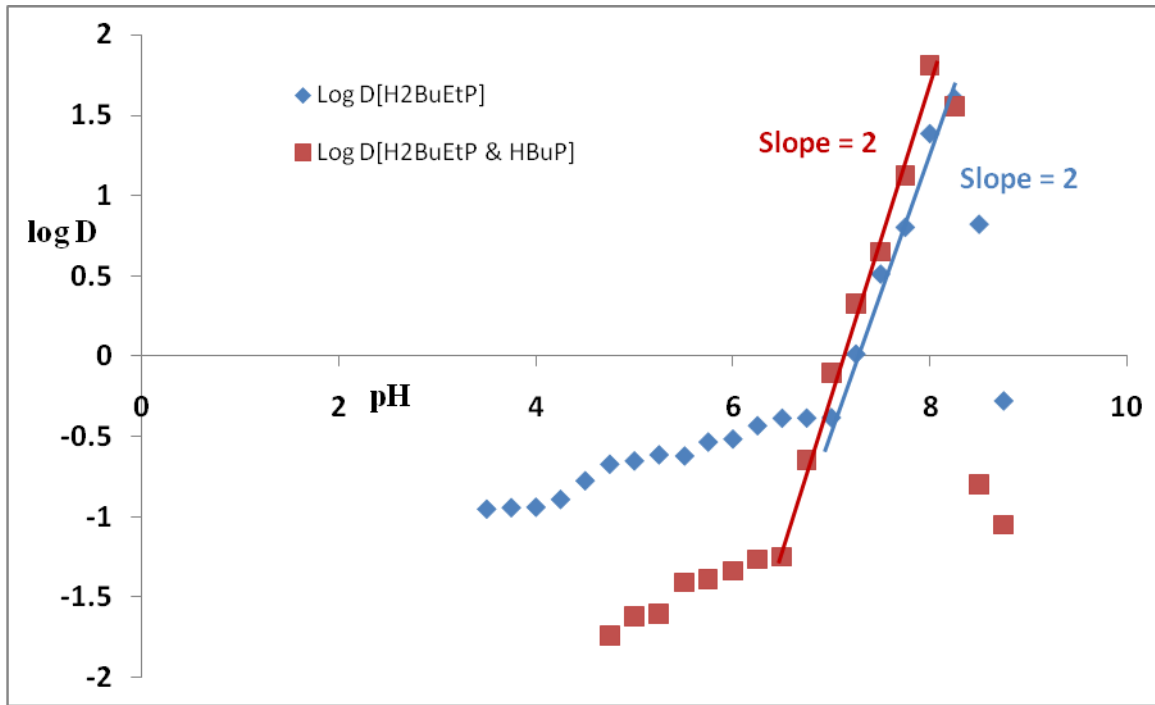


Fig 3: Plots of extraction of $3.58 \times 10^{-3} \text{M}$ (200mg/L)Fe(II) from buffer solutions into chloroform solution of 0.05 M H_2BuEtP and 0.05 M H_2BuEtP & 0.05M HBuP (9:1) volume ratio

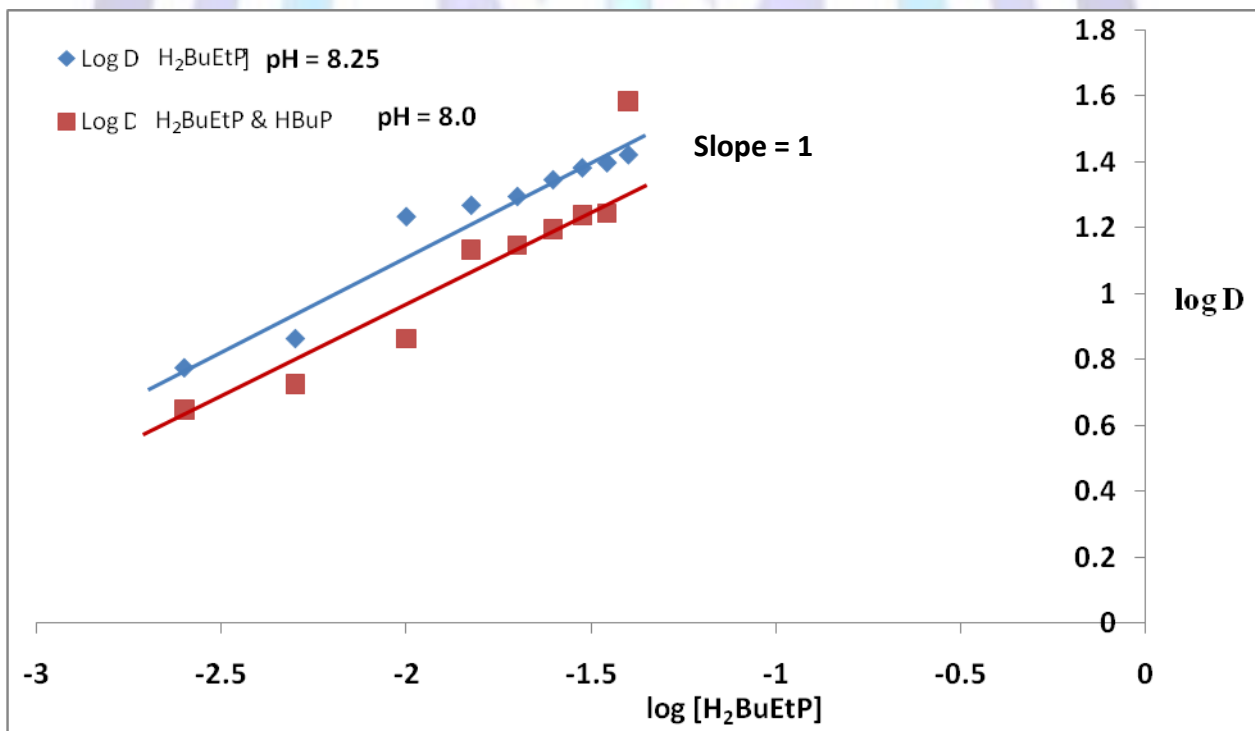


Fig 4: Log D-log $[\text{H}_2\text{BuEtP}]$ plot of extraction of $3.58 \times 10^{-3} \text{M}$ (200 mg/L) of Fe(II) from buffered solutions at pH 8.25 $[\text{H}_2\text{BuEtP}]$ and pH 8.00 $[\text{H}_2\text{BuEtP}$ and $\text{HBuP}]$ into chloroform solutions of varied ligand $[\text{H}_2\text{BuEtP}]$

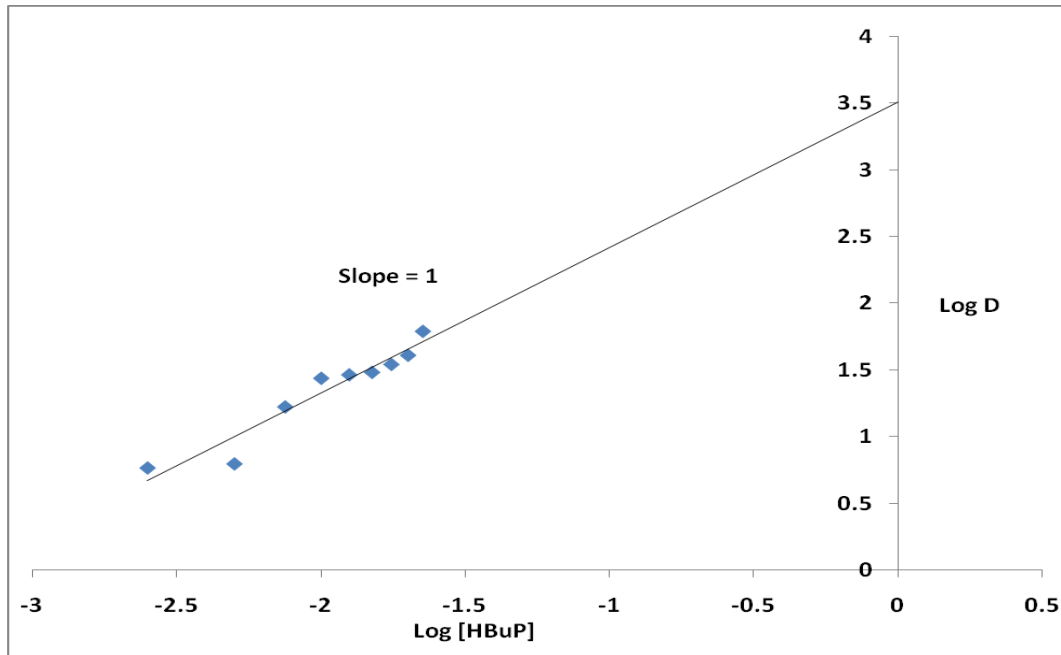


Fig 5: Log D-log [Ligand] plot of extraction of 200 mg/L of Fe(II) from buffered solutions at pH 8.0 into chloroform solutions of [HBuP] varied while [H₂BuEtP] was kept constant (2.5×10^{-2} M)

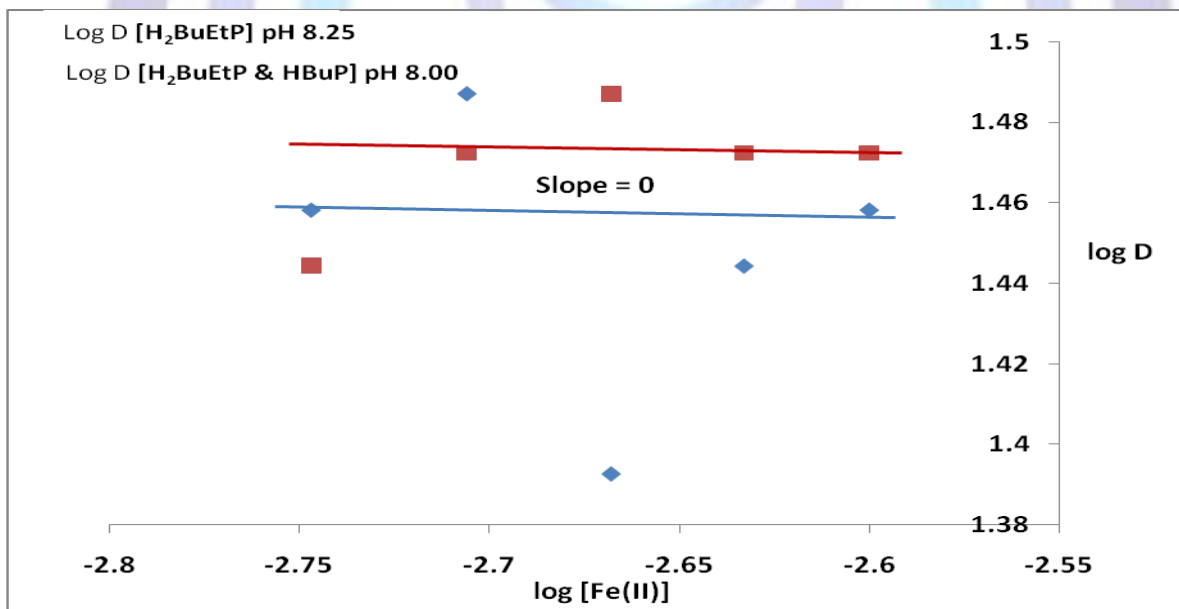


Fig 6: Log D-log [Fe(II)] plots of extraction of Fe(II) from buffer solutions at pH 8.25 and pH 8.00 into chloroform solutions of ligand H₂BuEtP and H₂BuEtP/HBuP



Extraction of Fe(II) from aqueous media into an organic solvent '(o)' containing a tetradentate ligand H₂BuEtP from all the results in figures 3, 4, and 6 can be represented by the following equations:



It shows that the reaction took place in the metal:ligand mole ratio of 1:1. Thus the extraction constant K_{ex1} can be represented by:

$$K_{\text{ex1}} = \frac{[\text{Fe}(\text{BuEtP})_{(o)}][\text{H}^+]^2}{[\text{Fe}^{2+}][\text{H}_2\text{BuEtP}_{(o)}]} \quad (2)$$

The distribution ratio $D_1 = [\text{Fe}(\text{BuEtP})_{(o)}]/[\text{Fe}^{2+}]$. Substitution into equation (2) gives:

$$\text{Log } D_1 = \text{log } K_{\text{ex1}} + \text{log}[\text{H}_2\text{BuEtP}] + 2\text{pH} \quad (3)$$

The partition coefficient (K_{D1}) of Fe(BuEtP)_(o) species is defined as $K_{D1} =$

$[\text{Fe}(\text{BuEtP})_{(o)}]/[\text{Fe}(\text{BuEtP})]$ for which a value of 1.5 ± 0.16 was determined and the pH at which 50 % extraction of a metal ion had occurred $\text{pH}_{1/2}$ was found to be 7.24 ± 0.10 from the graph in figure 3. $\text{Log } K_{\text{ex1}} = -13.45 \pm 0.2$ was calculated using equation 3.

The extraction parameters were similar to those obtained for the extraction of Ni²⁺ ion with this ligand H₂BuEtP as reported⁸, indicating that similar mechanism might be involved in their extraction mechanisms. However, even though their $\text{pH}_{1/2}$ values are close ($\text{pH}_{1/2\text{Fe}} = 7.24 \pm 0.10$ and $\text{pH}_{1/2\text{Ni}} = 7.14 \pm 0.10$), comparing other extraction parameters with those of Ni²⁺ ($\text{Log } K_{\text{exNi}} = -12.39 \pm 0.64 > \text{Log } K_{\text{exFe}} = -13.45 \pm 0.2$ and $\text{Log } D_{\text{Ni}} = 1.89 \pm 0.05 > \text{Log } D_{\text{Fe}} = 1.5 \pm 0.16$) showed that Ni²⁺ distributed better than Fe²⁺ in the presence of this ligand H₂BuEtP. The optimal pH for the extraction of Fe(II) with the ligand H₂BuEtP was 8.25 at which a 97.59% extraction of Fe(II) was achieved. However, it was observed that the pH range at which quantitative extraction occurred was very narrow (7.5 – 8.5). It is noteworthy to state that the mechanism of extraction of Fe(II) and Ni(II) is different from that observed in the extraction of Pb(II) and U(VI) with the same ligand. In the distribution of Pb(II), it was observed that the extracted complex was proposed as ion pair tris complex species with a wide pH range (5.9 – 8.0) at which quantitative extraction occurred⁹. In the case of U(VI), it was observed that anions from buffer reagents might have played a part in the formation and hydrophobicity of the extracted complex. The pH range at which quantitative extraction occurred was also wide (5.5 – 8.25)¹⁰. The partition coefficient K_{D1} for Fe(II) compared with those gotten for the other three metal ions Ni(II), Pb(II) and U(VI) in similar studies with the same ligand H₂BuEtP indicated that U(VI) was the least extracted into the organic phase, while Pb(II) was the most extracted; $\text{Log } D_{\text{U(VI)}} = 0.56 \pm 0.11 < \text{Log } D_{\text{Fe(II)}} = 1.5 \pm 0.16 < \text{Log } D_{\text{Ni(II)}} = 1.89 \pm 0.05 < \text{Log } D_{\text{Pb(II)}} = 1.92 \pm 0.25$.

Iron ions have been shown to form strong and weak bonds with nitrogen atom in ligands in their formation of complexes^{12,13}. Combining slope analysis results from figure 3, figure 4, figure 6 and the above information, the proposed structure of the extracted Fe(II) complex with the ligand H₂BuEtP is shown in figure 7.

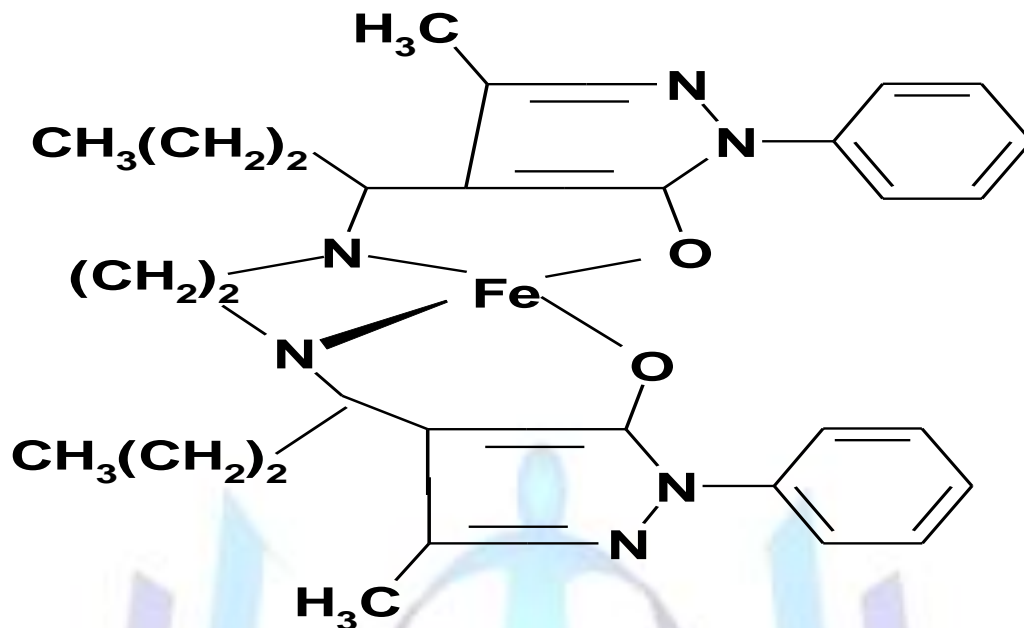


Fig 7: Proposed structure of Fe(BuEtP)

In the presence of HBuP and H₂BuEtP, results from figure 3 showed that two protons were also displaced during the reaction between Fe(II). Plots of Log D against log [H₂BuEtP] at fixed concentration (5 × 10⁻³ M) of HBuP (figure 4) had a slope of 1, indicating that one mole of H₂BuEtP was involved in the reaction. Also, plots of Log D against Log [HBuP] at fixed concentration 2.5 × 10⁻² M of the ligand H₂BuEtP also had a slope of 1 (figure 5) also showing that one mole of HBuP was involved in the reaction. Log D against Log [Fe(II)] plots in figure 6 had a slope of zero, showing that one mole of Fe(II) also reacted with the ligands in the extraction process. Putting all this information together, we could represent the reaction for the extraction of Fe(II) from aqueous solutions into organic chloroform solution of ligands H₂BuEtP and HBuP by equation 4



$$K_{\text{ex2}} = \frac{[\text{Fe}(\text{HBuEtP})\cdot\text{BuP}_{(o)}][\text{H}^+]^2}{[\text{Fe}^{2+}][\text{H}_2\text{BuEtP}_{(o)}]} \quad (5)$$

where [HBuP] is constant and incorporated in K_{ex2} . The distribution ratio $D_2 = [\text{Fe}(\text{HBuEtP})\text{BuP}_{(o)}]/[\text{Fe}^{2+}]$, on substitution into equation (6) gives,

$$\text{Log } D_2 = \text{Log } K_{\text{ex2}} + \text{log}[\text{H}_2\text{BuEtP}] + 2\text{pH} \quad (6)$$

The partition coefficient $[\text{Fe}(\text{HBuEtP})\cdot\text{BuP}_{(o)}]/[\text{Fe}(\text{HBuEtP})\cdot\text{BuP}]$ was determined from figure 3 to be equal to 1.68 ± 0.18 . Log K_{ex2} was calculated from equation 6 to be -13.27 ± 0.54 and the $\text{pH}_{1/2}$, determined from figure 3 to be 7.13 ± 0.10 . These results were not significantly different from those obtained with the ligand H₂BuEtP alone even though they are slightly better; $K_{D2\text{H}_2\text{BuEtP}/\text{HBuP}} = 1.68 \pm 0.18 > K_{D1\text{H}_2\text{BuEtP}} = 1.5 \pm 0.16$; $\text{Log } K_{\text{ex2H}_2\text{BuEtP}/\text{HBuP}} = -13.27 \pm 0.54 > \text{Log } K_{\text{ex1H}_2\text{BuEtP}} = -13.45 \pm 0.2$. The $\text{pH}_{1/2}$ was slightly reduced from 7.24 ± 0.10 in ligand H₂BuEtP alone to 7.13 ± 0.10 in mixed ligands (H₂BuEtP/HBuP) organic phase. The pH range at which quantitative extraction occurred was still narrow (7.25 – 8.25). However the optimal pH at which maximum extraction was achieved was slightly reduced from 8.25 in ligand H₂BuEtP alone to 8.0 in mixed ligands (H₂BuEtP/HbuP) organic phase.

The distribution of Fe(II) in the mixed ligands organic phase was lower than those obtained for U(VI), Pb(II) and Ni(II) with the same mixed organic phase; $\text{Log } K_{D\text{Fe}} = 1.68 \pm 0.18 < \text{Log } K_{D\text{U}} = 1.74 \pm 0.20 < \text{Log } K_{D\text{Pb}} = 1.82 \pm 0.22 < \text{Log } K_{D\text{Ni}} = 1.89 \pm 0.02$. The combined results for the so far studied four metals with this same mixed ligands organic phase indicated that

while quantitative extraction of Fe(II) occurred only above the neutral pH region (7.75 – 8.00), the other three previously studied metals were quantitatively extracted over a wider range, stretching from the acidic pH region to the alkaline pH regions Ni(II) (6.0 – 9.0); Pb(II) (5.75 – 8.00) and U(VI) (3.75 – 7.25)^{8,9,10}. This could be exploited in the separation of Fe(II) from Ni(II), Pb(II) and U(VI) in aqueous phases using the mixed ligands (H₂BuEtP/HbuP) organic phase.

Analysis of data showed that extraction involving a mixture of the ligands probably gave a protonated mixed ligand Fe(II) complex species. The suggested Iron complex species is shown in figure 8 having Fe-N bonds. The formation of the adduct complex Fe(HBuEtP).BuP_(o) resulted in increased distribution of Fe(II) into the organic phase due increased hydrophobicity and reduced polarity of the complex compared to the complex Fe(BuEtP) formed in the ligand H₂BuEtP alone organic phase

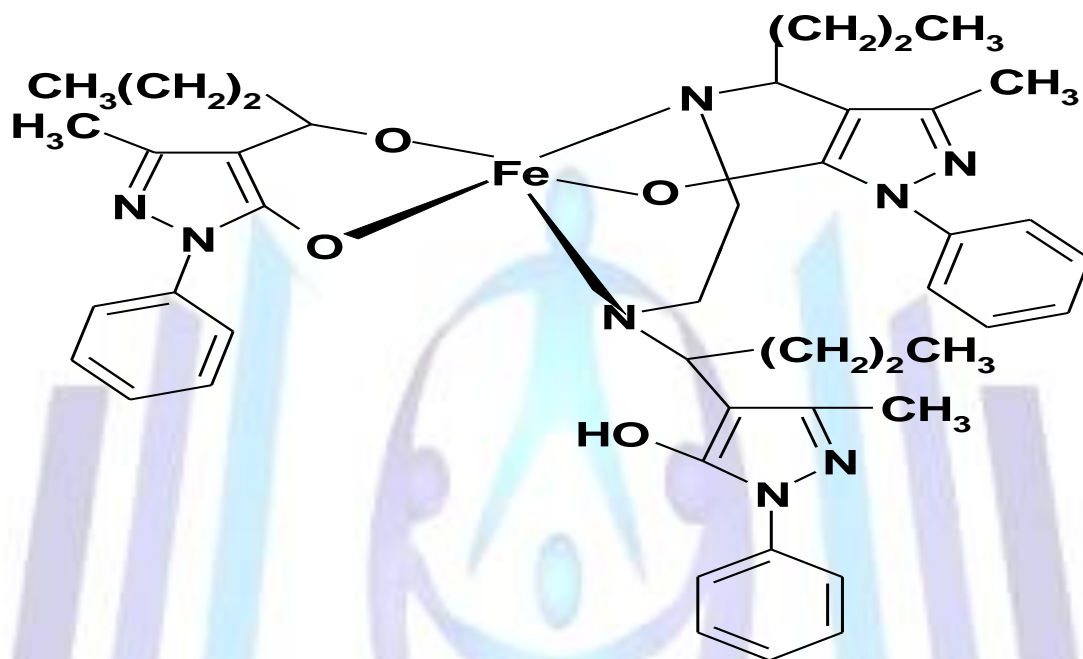


Fig 8: Proposed structure of Fe(HBuEtP).BuP

4. CONCLUSIONS

The ligand H₂BuEtP has potentials for use as a colorimetric reagent for the analysis of Fe.

The ligand H₂BuEtP can be used as an organic extractant for Fe(II) extraction from an aqueous solution, alone or in a mixed ligands organic system containing HBuP.

The optimal pH for extraction of Fe(II) with the ligand H₂BuEtP alone and mixed ligands (H₂BuEtP and HBuP) organic phase was 8.25 and 8.00 respectively.

Though the calculated extraction parameters showed that the ligand HBuP slightly improved the distribution of Fe(II) into organic phases, the synergistic effect of HBuP in the extraction of Fe(II) with H₂BuEtP was not significant.

Slope analysis showed the extracted Fe complex species in both type of organic phases were Fe(BuEtP)_o and Fe(HBuEtP)BuP_o respectively.

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Author' biography with Photo



Dr Jackson Godwin was born in 27th October, 1969 in Ekeni town in Southern Ijaw Local Government area of Bayelsa State, Nigeria in Africa. I attended Holy Trinity Primary School, Aggrey Road, Port Harcourt between 1975 – 1981, Government Secondary School, Borikiri, Port Harcourt, Rivers State, Nigeria between 1981 -1987 and University of Port Harcourt, Rivers State, Nigeria between 1987 – 1991 where I obtained a Bachelor of Science Degree in Pure Chemistry.

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I have been working since 2000 as an academic staff with Niger Delta University, Wilberforce, Bayelsa State, Nigeria with research area and ten(10) publications in Environmental and Analytical Chemistry.

I am Married to Barrister (Mrs) Timiebi Godwin with two children, Miss Ayebatokoni Emmanuella Godwin(6 years) and Master Ebimobowei Jewel Godwin(5 years).