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Synthesis, Characterization and Antimicrobial Studies of Schiff Base Derived from the Reaction of 2-Acetyl-6-Bromopyridine and 1-(2-Aminoethyl)Piperidine and Co(II), Cr(II), Fe(II) and Ni(II) Metal Complexes

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Abstract

Schiff bases are well-recognized for their strong coordination tendencies and diverse biological properties. When complexed with metal ions, they often exhibit improved antimicrobial effects. work focuses on synthesizing a novel Schiff base derived from 2-acetyl-6-bromopyridine and 1-(2-aminoethyl)piperidine, forming its Co(II), Cr(II), Fe(II), and Ni(II) complexes, characterizing these compounds, and assessing their antimicrobial potential. The Schiff base was obtained through a condensation reaction carried out under reflux conditions. Its metal complexes were synthesized and characterized using techniques such as FT-IR, UV-Vis spectroscopy, magnetic susceptibility, and molar conductance measurements. Their antimicrobial efficacy was evaluated against selected bacterial and fungal species. Spectroscopic results confirmed that the ligand coordinated with metal ions through the azomethine and pyridine nitrogen atoms, resulting in octahedral geometries. The metal complexes demonstrated markedly greater antimicrobial activity than the uncoordinated ligand. Complexation with metal ions significantly enhanced the antimicrobial effectiveness of the Schiff base, highlighting its potential as a candidate for antimicrobial agent development.

Keywords: 2-acetyl-6-bromopyridine, 1-(2-aminoethyl)piperidine, antimicrobial, Schiff Bases, Metal Complexes.

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1. Introduction

The increasing prevalence of antimicrobial resistance (AMR) represents a major global health concern in the 21st century. This growing threat has intensified the demand for innovative antimicrobial agents, particularly those capable of addressing multi-drug resistant infections (Chen *et al.*, 2024; Loukili *et al.*, 2024; Haddou *et al.*, 2023). Among the promising candidates are Schiff bases-organic compounds produced through the condensation of primary amines with carbonyl groups-which have shown notable biological activities, such as antimicrobial, antifungal, and anticancer effects (Raza *et al.*, 2022; Titi *et al.*, 2021; Loukili *et al.*, 2022; Abrigach & Touzani, 2016; Hadda *et al.*, 2016). When these Schiff bases coordinate with metal ions, the resulting metal complexes often exhibit altered chemical and biological properties, making them attractive frameworks for drug development (Uddin *et al.*, 2025; Aljazzar *et al.*, 2024; Mohammad *et al.*, 2023).

Of particular interest are Schiff bases derived from pyridine-containing compounds, as these structures offer versatile coordination behavior and enhanced biological functionality. A noteworthy example is the Schiff base formed by the reaction of 2-acetyl-6-bromopyridine with 1-(2-aminoethyl)piperidine. This ligand integrates the electron-rich pyridine ring and the reactive amine group, boosting its metal-binding ability and making it suitable for various applications in bioinorganic chemistry (Kumar *et al.*, 2023; Arif *et al.*, 2022).

Transition metals such as cobalt (Co), chromium (Cr), nickel (Ni), and iron (Fe) readily form stable complexes with Schiff base ligands. These metal complexes often display improved solubility, structural stability, and functional activity compared to the free ligands (Patel *et al.*, 2023; Zerrouki *et al.*, 2011; Chohan *et al.*, 2010). The incorporation of metal ions can significantly affect the electronic characteristics of the ligand, potentially enhancing its bioactivity, including antimicrobial efficacy (Khan *et al.*, 2021). Indeed, Schiff base complexes with these metals have been reported to interfere with bacterial cell membranes and disrupt essential biological processes, making them promising candidates for antimicrobial therapy (Sharma *et al.*, 2022).

This study focuses on the synthesis and structural characterization of a Schiff base obtained from the reaction between 2-acetyl-6-bromopyridine and 1-(2-aminoethyl)piperidine. It also details the formation of Co(II), Cr(III), Ni(II), and Fe(III) complexes with this ligand. Comprehensive characterization was conducted using techniques such as UV-Vis, FTIR, NMR spectroscopy, and X-ray diffraction to elucidate the structural and bonding features of the compounds. The antimicrobial potential of the free ligand and its metal complexes was assessed against several bacterial strains to explore their suitability as therapeutic agents.

2. Materials and Methods

2.1 Chemicals, Reagents, and Apparatus

All chemicals and reagents utilized in this study were of analytical grade and used as received, without further purification. Glassware was thoroughly cleaned with detergent, rinsed with distilled water, and oven-dried at 110 °C prior to use. Weighing procedures were conducted using a Mettler electronic balance, model H3OAR.

2.2 Synthesis of the Schiff Base Ligand

The Schiff base ligand was synthesized through a condensation reaction between 2-acetyl-6-bromopyridine (0.3 g, 0.01 mol) and 1-(2-aminoethyl)piperidine (0.2 g, 0.01 mol) in 25 mL of ethanol. The mixture was heated under reflux at 75–85 °C for three hours. The resulting product was a yellowish oily solution, which transformed into a yellow, hygroscopic solid upon drying at 55 °C for 12 hours in an oven. This solid was then dissolved in methanol, heated to 60 °C, and evaporated under reduced pressure to yield a light brown solid. The ligand was characterized using melting point analysis, conductivity measurement, Job's method, FT-IR, and UV-Visible spectroscopy (Gwaram *et al.*, 2012).

2.3 Synthesis of Metal(II) Complexes

Complexes of the Schiff base with various metal(II) ions; namely Ni(II), Co(II), Cu(II), Mn(II), Cr(II), Fe(II), and Zn(II) were synthesized in situ. The reaction involved 2-acetyl-6-bromopyridine (0.2 g, 1.65 mmol) and 1-(2-aminoethyl)piperidine (0.21 g, 1.65 mmol) dissolved in 20 mL of dimethylformamide (DMF) and refluxed. After two hours, ethanolic solutions of the respective metal(II) halides were introduced, and the mixtures were refluxed further for 2–5 hours. The resulting precipitates were collected by filtration, washed with cold ethanol, and dried under vacuum. These synthesized complexes were subsequently characterized using Job's method, FT-IR, and UV-Visible spectroscopy. Physical data of the products are summarized separately.

2.4 Physical Measurements

FTIR spectra were recorded using a Cary Agilent Technologies model 630 spectrophotometer within the 400–4000 cm^{-1} range using KBr pellets. UV-Visible spectra were acquired in DMF using a model 4802 double-beam UV/Vis spectrophotometer. Melting and decomposition points were determined using a Stuart SMP10 apparatus. Molar conductivity measurements were conducted in DMF (10^{-3} M) using a Jenway 4010 conductivity meter. Magnetic susceptibility measurements were performed using an MK1 magnetic susceptibility balance, and effective magnetic moments (μ_{eff}) were calculated using the equation $\mu_{eff} = 2.828 \times (\chi_m T)^{1/2}$ Bohr Magnetons (B.M). Bacterial and fungal isolates for antimicrobial testing were sourced and identified in the Department of Microbiology at Umaru Musa

Yar'adua University, Katsina.

3. Results and Discussion

3.1 Physical Properties of the Schiff base and its Metal (II) Complexes

The Schiff base and its metal (II) complexes were prepared in good yield, the physical properties of the synthesized Schiff base and its metal complexes were analyzed and presented in **Table 1.** The percentage yield of the Schiff base was 60 % while that of the complexes were 58, 49,62 and 55%. The Schiff base was light Brown while the Ni(II) , Co(II), Cr(II) and Fe(II) complexes were found to be Green, Pink, Cream and Brown respectively. It was found that the melting point of the Schiff base was $130^{O}C$ and the decomposition temperature of the metal (II) complexes were 210, 170, 250 and $255^{O}C$, this indicated that they are thermally stable complexes.

Table 1. Physical Properties of Ligand and its Metal(II) Complexes

S/N	Compounds	COLOUR	YIELD %	MOLECULAR WEIGHT	M.P/D.T (^{0}C)
1	Ligand (L)	Light Brown	60	310.24	130
2	$[Ni(L)]Cl_2$	Green	58	439.83	210
3	$[Co(L)]Cl_2$	Pink	49	440.0	170
4	$[Cr(L)]Cl_2$	Cream	62	433.14	250
5	$[Fe(L)]Cl_2$	Brown	55	436.98	255

L = Schiff Base $[(C_{14}H_{20}BrN_3)(Cl_2)$

3.2 Solubility Test

Solubility analysis showed that the ligands and their metal complexes showed different behavior in some common organic solvents. All the compounds were found to be soluble in DMSO and few were slightly soluble in methanol, ethanol, and DMF. Their solubility in DMSO might be due to the high dielectric constant of the solvent (Babalola *et al.,* 2023). While in acetone and chloroform, the compounds were slightly soluble and some were insoluble (Table 2).

Table 2. Solubility test of the ligands and their metal (II) complexes in some common solvents.

S/N	Compound	Methanol	Ethanol	Chloroform	Acetone	DMF	DMSO
1	$(C_{14}H_{20}BrN_3)Cl_2$	S	S	SS	SS	S	S
2	$[Ni(L)]Cl_2$	S	S	SS	SS	S	S
3	$[Co(L)]Cl_2$	S	S	SS	IS	S	S
4	$[Cr(L)]Cl_2$	SS	SS	IS	SS	SS	S
5	$[Fe(L)]Cl_2$	S	SS	SS	SS	S	S

L = Schiff Base $[(C_{14}H_{20}BrN_3)(Cl_2)$

3.3 Magnetic Moment Values

Table 3 of the complexes showed that Ni(II), Co(II),Cr(II), and Fe (II) are paramagnetic. The observed magnetic moment value for Ni(II), complex was found to be 2.83 BM, which fall within the expected range of 2.83-3.50 BM, suggested the consistency with its octahedral environment (Deng *et al.*, 2024) The value for Co(II)complex was found to be 3.87 BM which suggested octahedral geometry of the Co(II)complex. Cr (II),Complex were found to have magnetic values of 4.92 BM respectively, And Fe(II) Complex were found to have magnetic values of 4.89 BM respectively, which are within the range of octahedral geometry values (5.6 - 6.0) of the Metal(II) complexes.

Table 3. Magnetic Susceptibility data of Metal(II) Complexes

S/N	Compounds	X_g (erg G^{-2} g^{-1})	X_M (ergG^{-2} mol $^{-1}$)	U_{eff}(B.M)	Magnetism
1	$[Ni(L)]Cl_2$	6.33x10^{-9}	2.56x10^{-7}	2.83	Paramagnetism
2	$[Co(L)]$ Cl_2	4.05X10^{-9}	1.78X10^{-7}	3.87	Paramagnetism
3	$[Cr(L)]Cl_2$	1.6x10^{-9}	6.9x10^{-7}	4.92	Paramagnetism
4	$[Fe(L)]$ Cl_2	7.83x10^{-9}	3.42x10^{-7}	4.89	Paramagnetism

L = Schiff Base $[(C_{14}H_{20}BrN_3)(Cl_2)$

3.4 Molar Conductivity

The molar conductivity values (**Table 4**) of the complexes (Λm = 10.25 - 25.28 $\Omega^{-1}cm^2 mol^{-1}$) indicated that these complexes are non-electrolytes due to low value of their molar conductance. (Spinu et al., 2008). Non-electrolyte complexes are found within 1 - 50 Ω^{-1} cm^2 mol^{-1}, while electrolyte complexes are found above 50 Ω^{-1} cm^2 mol^{-1} (Geary, 1971). Based on these data, it is clear that complexes are considered as 1:1 $[M(L)Cl_2]$, indicating the non ionic nature of the complexes. The +2 oxidation states of M(II) is satisfied by two negatively charged monodentate Cl^- ligands in the complexes of $[M(L)Cl_2]$ residing in coordination sphere, hence no any counter ion outside the coordination sphere.

Table 4. Molar Conductance of the complexes

S/N	Compounds	Concentration Mol/dm^3	Molar Conductance $\Omega^{-1}cm^2mol^{-1}$
1	$[Ni(L)Cl_2]$	1×10^{-3}	16.10
2	$[Co(L)Cl_2]$	1×10^{-3}	25.28
3	$[Cr(L)Cl_2]$	1×10^{-3}	10.25
4	$[Fe(L)Cl_2]$	1×10^{-3}	10.32

L = Schiff Base $[(C_{14}H_{20}BrN_3)(Cl_2)$

3.5 FTIR Analysis

The infrared spectra for the present compounds taken in the range 400-4000 cm^{-1} helped to indicate regions of absorption vibrations. The main stretching modes are for υ(C=N) υ(C=C) υ(M-N)υ(C-H). The IR spectral data of the Schiff base and its complexes are presented in **Table 5.** The spectra of the complexes were compared with those of the free ligand in order to determine the coordination sites that may be involved in complexation. In the IR spectra of the Schiff base (**Table 5**), a strong peaks observed at 1595cm^{-1} in the spectra which was assigned to stretching vibration of azomethine ν (C = N) group. These relative shifts observed for ν (C = N) bonds in the complex clearly showed the participation of azomethine nitrogen in coordination to the metal ions.

Table 5. IR Spectral data for Ligand and its Metal(II) Complexes

S/N	Compounds	υ(C=N)	υ(C=C)	υ(M-N)	υ(C-H)
1	Ligand(L)	1595	1394	-	298
2	[Ni(L)] Cl_2	1592	1458	615	2936
3	[Co(L)] Cl_2	1693	1505	544	2901
4	[Cr(L)]Cl_2	1655	1599	482	2981
5	[Fe(L)] Cl_2	1587	1416	527	2974

L = Schiff Base [(C_{14} H_{20} BrN_3)(Cl_2)

3.6 U.V Analysis

The data for electronic spectra of the Schiff base, and its complexes are given in **Table 6.** Two essential absorption bands were observed at 204nm and 295nm, in the spectra of the Schiff base which were assigned to the π → π* transition of the conjugated electrons and n → π* transition of the lone pair electrons, respectively (Jabbi et al., 2021). These transitions existed also in the spectra of the complexes, but were shifted to different intensities n → π*(219, 230, 210,240) and π → π* (242, 292,243, and 258) for Ni(II),Co(II) Cr(II) and Fe(II) complexes respectively, confirming the coordination of the ligand to the metal ions.

Table 6. Absorption Spectral data(nm) of the Ligand and Metal (II) Complexes in DMSO.

S/N	Compounds	π-π*	n-π*
1	Ligand(L)	204	295
2	[Ni(L)] Cl_2	219	242
3	[Co(L)] Cl_2	230	292
4	[Cr(L)]Cl_2	210	243
5	[Fe(L)] Cl_2	240	258

L = Schiff Base [(C_{14} H_{20} BrN_3)(Cl_2)

3.7 Job's Method of Continuous Variation (Stoichiometries)

Job's method of continuous variation (stoichiometries) was used for the estimation of the ligand to metal ratio. The polt of absorption against mole fraction, ligand-metal ion mixture in each case at maximum absorbance corresponding to the ligand mole fraction suggest 1:1 metal – ligand ratio in the complexes as presented in **Table 7**. Similar result was reported by Javed *et al.*, (2022).

3.8 Antimicrobial and antifungal Activity

The synthesized Schiff base and its metal complexes of (Ni, Co, Cr, and Fe,) were tested against two gram-positive (Staphylococcus aureus, Streptococcus pneumoniae) and two gram-negative (Escharichia coli, Klebseilla pneumoniae,) bacterial strains by agar well diffusion method at concentrations of 1000 µg/mL, 500 µg/mL, 250 µg/mL, 125 µg/mL. The diameter of zone of inhibition (mm) was measured for each treatment (**Table 8a and b**). The activity was compared to that of a standard anti-fungal drug (ciprofloxacin). The synthesized compounds also showed good antifungal activity against the fungal isolates (**Table 9**).

Table 7. Stoichiometries (Job's Analysis)

Mole fractions, X		0.94	0.81	0.69	0.56	0.44	0.31	0.19	0.06
S/N	Compound	Absorbance at λmax							
1	$[Ni(L)Cl_2]$	0.18	0.398	0.724	0.92	1.238	0.748	0.462	0.056
2	$[Co(L)Cl_2]$	0.090	0.26	0.38	0.60	0.40	0.36	0.25	0.12
3	$[Cr(L)Cl_2]$	0.044	0.60	0.97	1.718	1.20	0.87	0.58	0.044
4	$[Fe(L)Cl_2]$	0.013	0.059	0.127	0.148	0.098	0.073	0.046	0.011

Table 8a. Antibacterial Activity of the Schiff base and its Metal (II) Complexes for gram positive Bacteria

Compounds	*Staphylococcus Aureus* Concentrations (µg/ml)				*Staphylococcus epidermidis* Concentrations (µg/ml)			
	1000	500	250	125	1000	500	250	125
LIGAND	11.2	9.6	7.7	ND	9.6	8.5	ND	ND
$[Ni(L)Cl_2]$	13.8	11.3	9.9	8.2	12.7	10.4	9.3	8.1
$[Co(L)Cl_2]$	15.1	10.9	10.1	8.8	14.9	11.6	10.1	8.4
$[Cr(L)Cl_2]$	11.9	9.2	7.1	ND	12.2	10	7	ND
$[Fe(L)Cl_2]$	8.9	8.3	7.2	ND	10.1	8.6	7.9	ND
Ciprofloxacin	25							32.6

Table 8b. Antibacterial Activity of the Schiff base and its Metal (II) Complexes for gram Negative bacteria

COMPOUNDS	*E.coli* Concentrations (µg/ml				Klebseilla pneumonia Concentrations (µg/ml			
LIGAND	1000	500	250	125	1000	500	250	125
$[Ni(L)Cl_2]$	10	8.3	7.1	ND	ND	ND	ND	ND
$[Co(L)Cl_2]$	14.1	10.4	8.4	7.6	13.7	10.7	9.2	8.3
$[Cr(L)Cl_2]$	22.5	18.1	15.8	15.2	14.3	11.7	10.1	9.4
$[Fe(L)Cl_2]$	13.2	11.3	8.9	8.3	8.5	8.1	ND	ND
Ciprofloxacin	28.1							28.1

The schiff base was found to be active against all the bacterial isolates at higher concentration, though was slightly higher and the activity increased with the increase in concentration (Jiang *et al.*, 2023). The complexes showed a wide range of activities (06 – 15) mm with the cupper (II) complex showing highest activity (15 mm) on Escharichia coli at the concentration of 1000µg/ml. On the contrary it was largely in active on Klebseilla pneumoniae. On the other hand Ni(II) complex though active on E.coli was completely without any activity Klebseilla pneumoniae at all concentration (Aminu *et al.*, 2021). Generally, the antifungal activities of most compounds were found to increase with increase in concentrations.

Table 9. Antifungal Activity of the Schiff base and its Metal (II) Complexes

COMPOUNDS	*C.albicans* Concentrations (μg/ml				*T.rubrum* Concentrations (μg/ml			
	1000	500	250	125	1000	500	250	125
LIGAND	9.1	7.0	ND	ND	11.3	9.7	8.1	ND
$[Ni(L)Cl_2]$	11.9	10.3	8.4	7.7	13.8	10.9	8.3	7.0
$[Co(L)Cl_2]$	15.2	12.9	10.4	8.1	11.6	9.2	7.3	ND
$[Cr(L)Cl_2]$	15.8	13.3	11.7	10.1	10.7	8.8	ND	ND
$[Fe(L)Cl_2]$	11.3	8.9	7.3	ND	9.1	7.8	ND	ND
Ciprofloxacin	24.6				22.7			

Conclusion

Schiff base derived from 2-acetyl-6-bromopyridine and 1-(2-aminoethyl)piperidine was successfully synthesized and structurally characterized. The coordination behavior of the ligand with Co(II), Cr(II), Fe(II), and Ni(II) ions was investigated, confirming the formation of stable octahedral complexes. Spectroscopic and analytical data supported the bidentate coordination mode of the ligand. Antimicrobial screening demonstrated that the metal complexes exhibited enhanced activity compared to the free Schiff base, with some complexes showing the most potent effects against tested microbial strains. These results suggest that Schiff base metal complexes hold potential for further development as antimicrobial agents, warranting future investigations into their biological mechanisms and broader pharmacological applications.

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Conflict of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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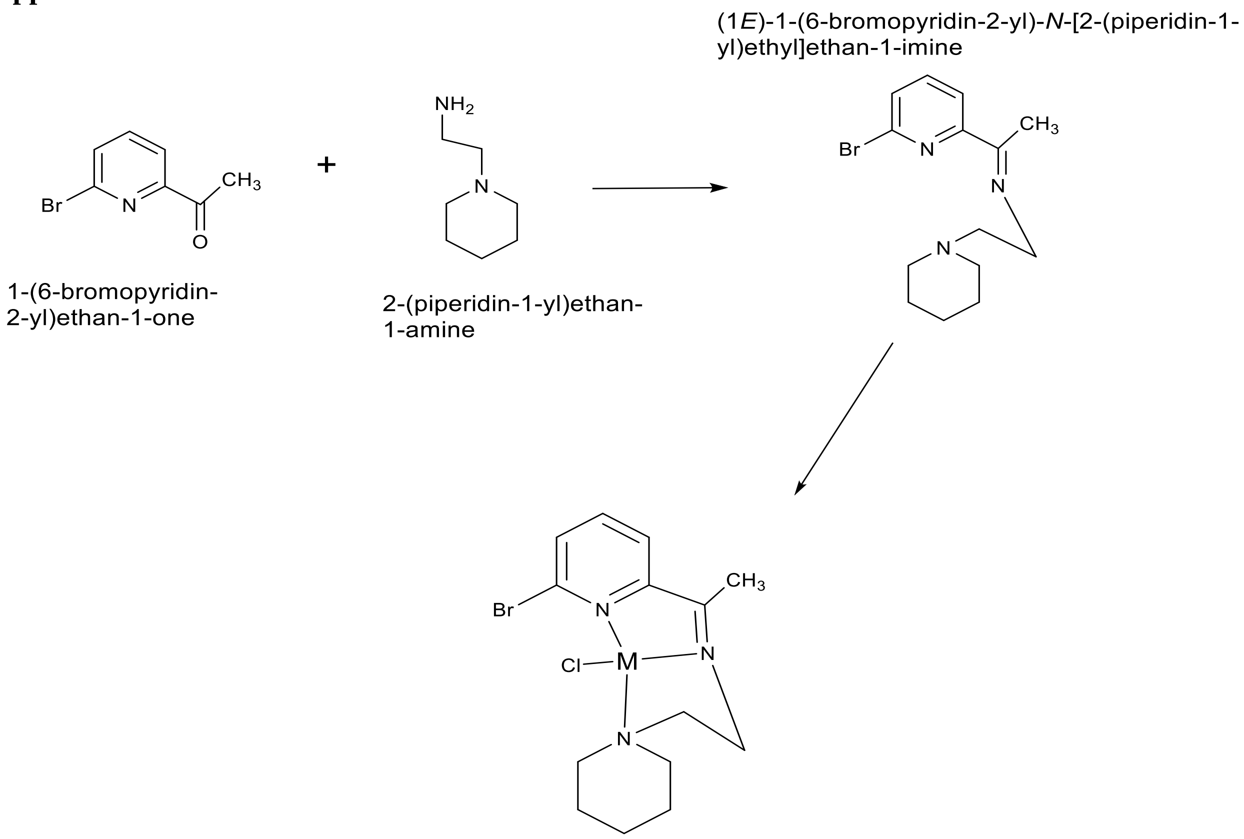
Supplemented Data



Where M=Ni(II),Co(II),Cr(II),Fe(II),Mn(II),Cu(II) and Zn(II).

Scheme 1. Synthesis of Schiff base metal Complexes

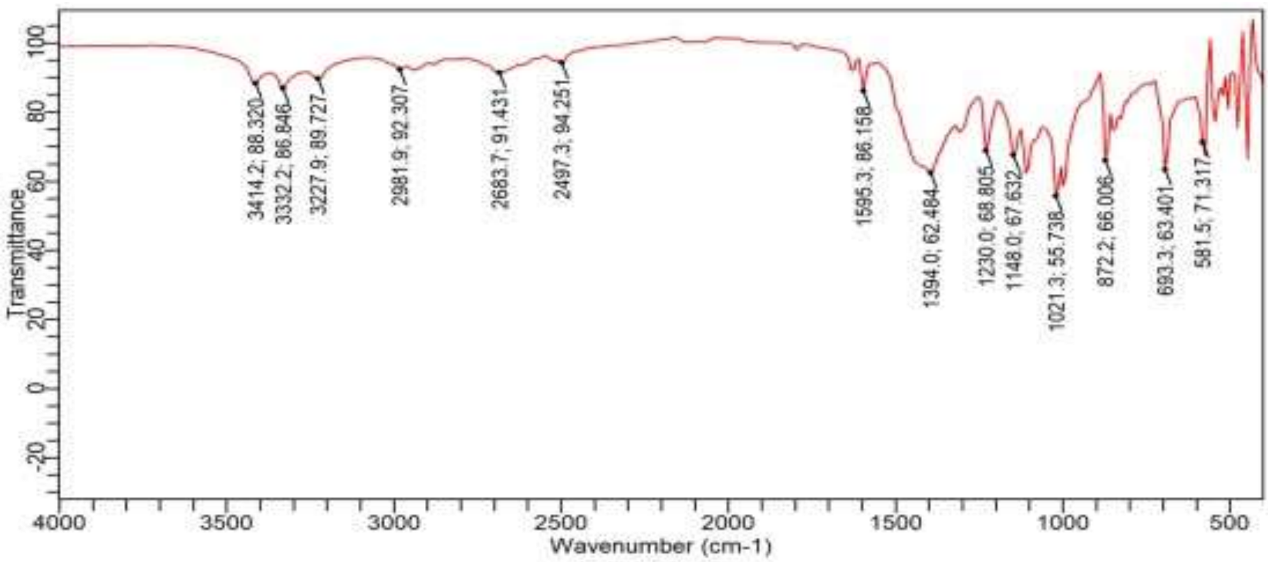


Figure 1. FTIR Spectrum of Ligand (L)

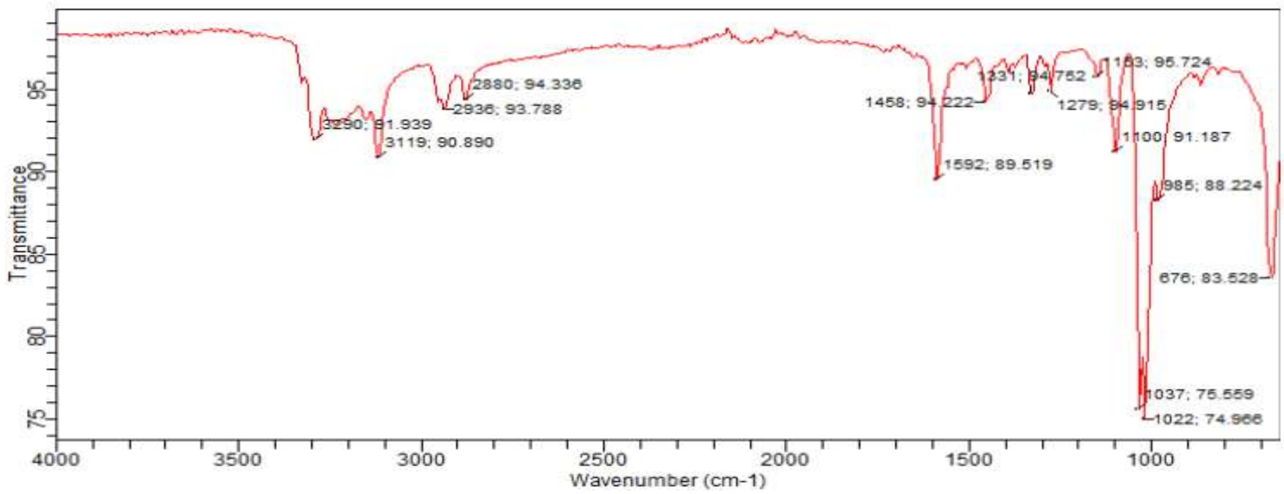


Figure 2. FTIR Spectrum of Nickel (II) Complex

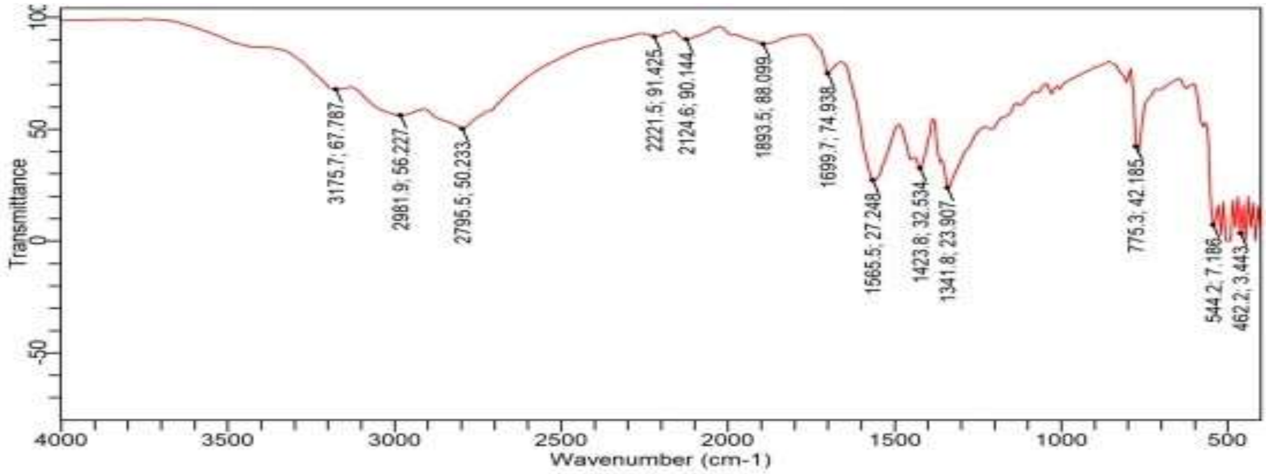


Figure 3. FTIR Spectrum of Cobalt (II) Complex

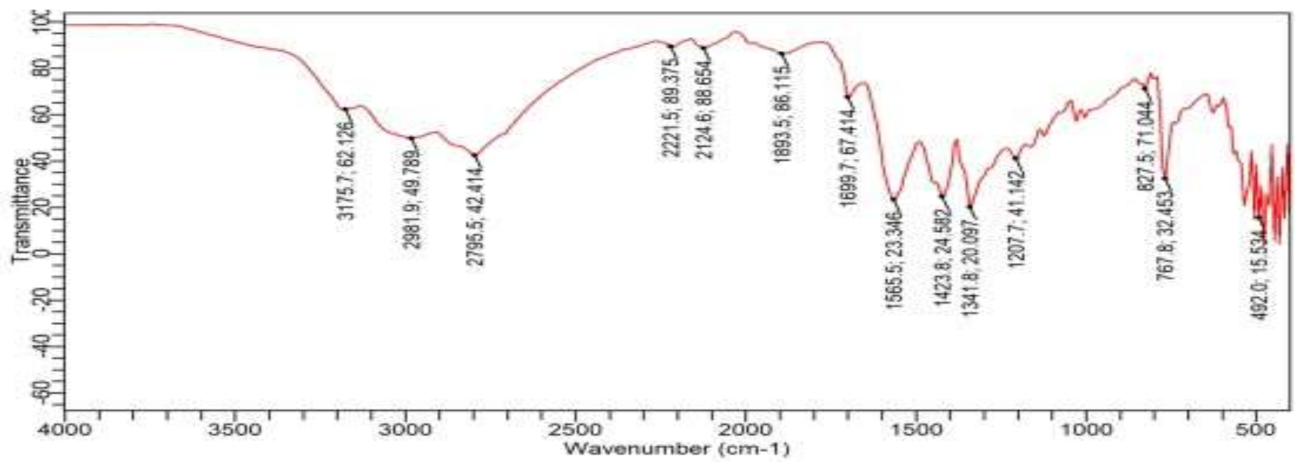


Figure 4. FTIR Spectrum of Chromium (II) Complex

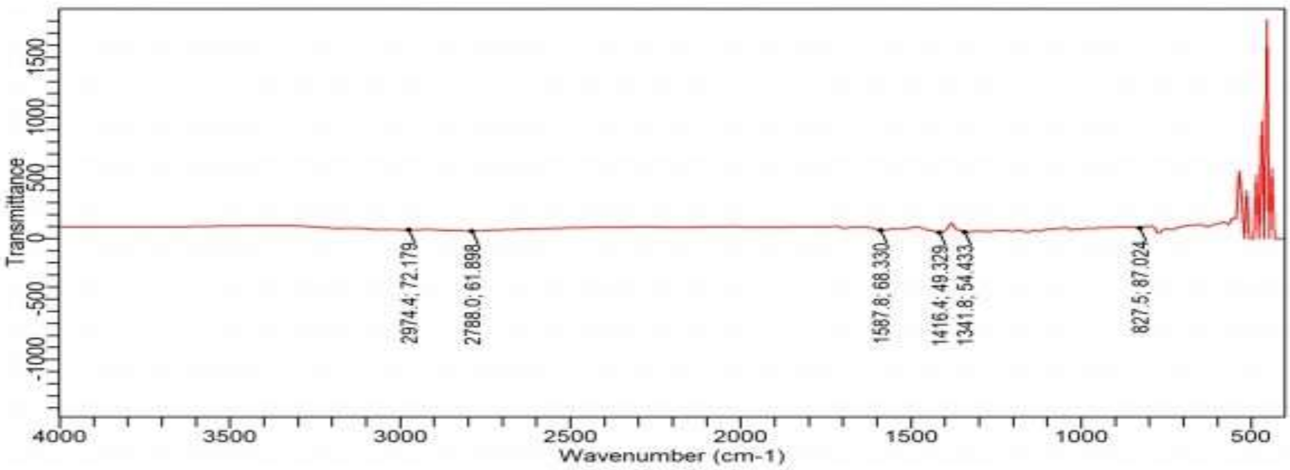


Figure 4. FTIR Spectrum of Iron (II) Complex

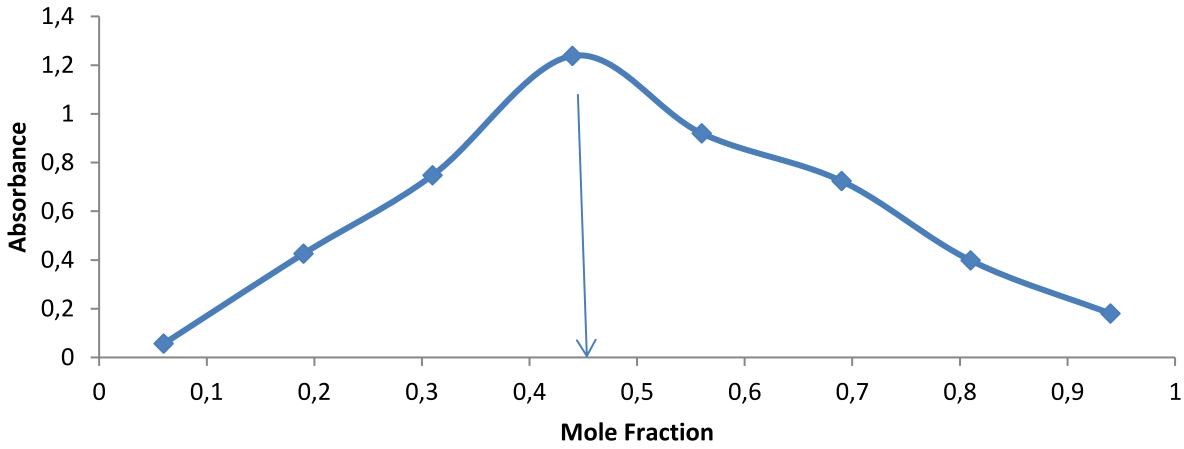


Figure 5. Job's Plot of Ni(L) Complex

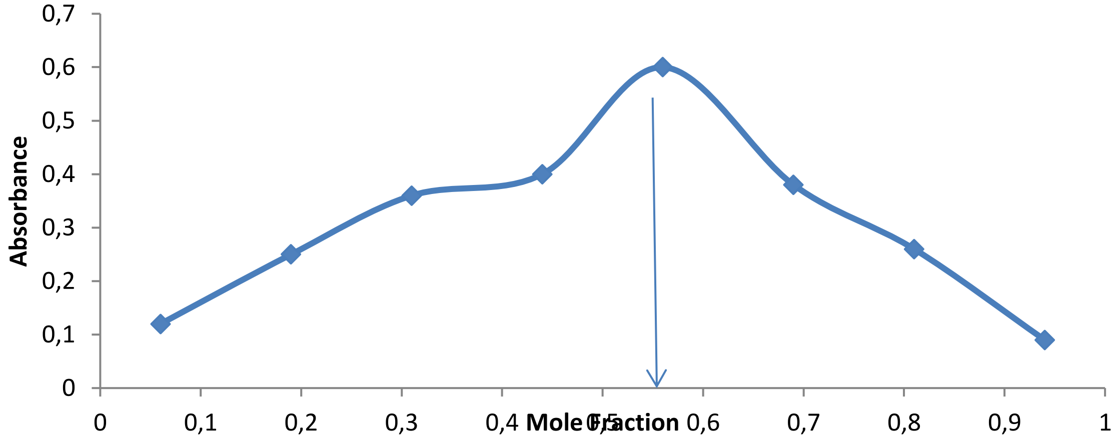


Figure 6. Job's Plot of Co(L) Complex

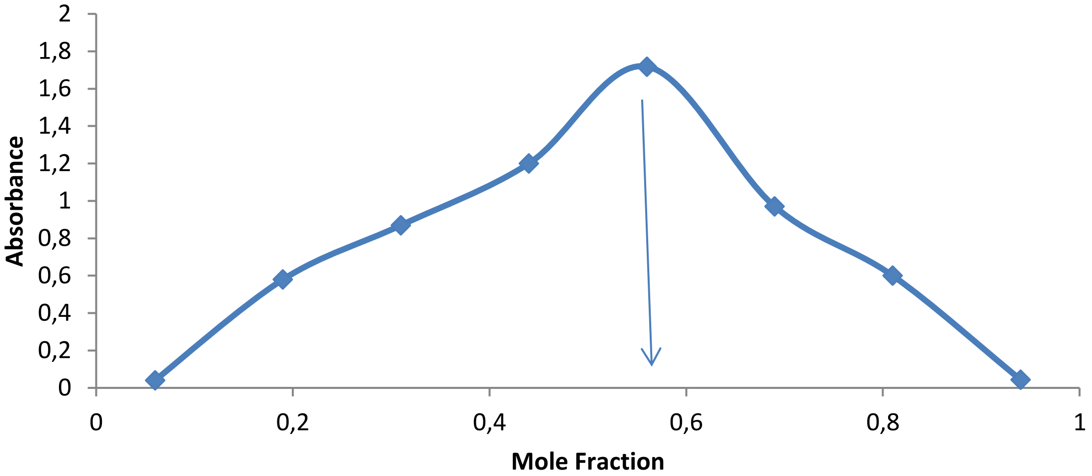


Figure 7. Job's Plot of Cr(L) Complex

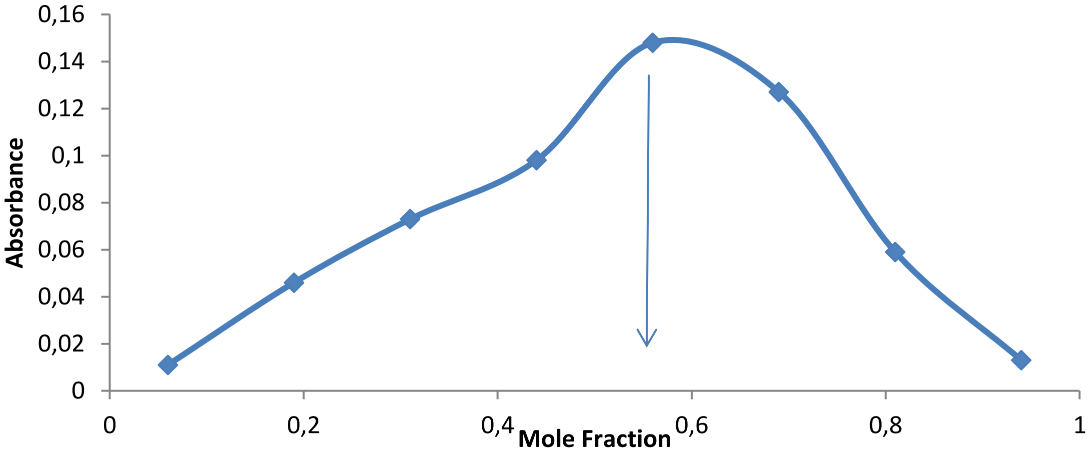


Figure 8. Job's Plot of Fe(L) Complex