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STRUCTURAL STUDIES OF COPPER(II) COMPLEXES WITH TETRADENTATE SCHIFF BASE LIGAND

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ABSTRACT

A noble series of Cu(II) complexes with tetradentate Schiff base ligand have been prepared from the condensation reaction of 2,6-diacetyl pyridine and α-amino acetohydroxamic acid in aqueous alcoholic medium at room temperature. The synthesized Schiff base and its copper complexes were characterized by using elemental analysis, molar conductivity measurements, magnetic moment measurements, IR and UV-Vis spectral data investigations. The all complexes of Cu(II) are stable in air and coloured. The molar conductance value of synthesized copper(II) complexes in dimethyl formamide solution indicates they are non-electrolytic nature. The presence of unpaired electrons in the d-orbital makes all copper (II) complexes are paramagnetic in nature. The infrared spectral data of investigated Schiff base show that ligand acts as tetradentate and coordinate through two oxyimino nitrogen atoms and two azomethine nitrogen atoms to the metal ions. On the basis of elemental analysis, magnetic behavior and different spectral analysis distorted octahedral geometry for Cu (II) complexes were proposed.

KEYWORDS: Copper(II) complexes, Schiff base, octahedral geometry, α-amino acetohydroxamic acid.

1. INTRODUCTION

Many interesting features in the field of coordination chemistry have resulted from the initial investigation of co-ordination compounds with inorganic ligands, followed by organic ligands. Schiff bases are a versatile class of organic compounds obtained through the condensation reaction of a primary amine with active carbonyl compounds.[1-4] The Hugo Schiff was first to synthesized them in 1864. These compounds have a general formula RCH=N−R^1 where R and R^1 represent alkyl, aryl, cycloalkyl, or heterocyclic groups.[5-7] They are also commonly referred to as anils, imines, or azomethines. Schiff bases are a significant type of ligand that connect with metal ions through the azomethine (>C=N-) nitrogen.[8-10] Schiff bases formed from aliphatic aldehydes tend to be quite unstable and can easily undergo polymerization.[11-14] Schiff bases derived from aromatic aldehydes are more stable due to effective conjugation.[15-17]

Aldehydes react more quickly than ketones in condensation reactions because the reaction centers of aldehydes are less sterically hindered compared to those of ketones.[18] The azomethine group plays an important role in determining the stability, reactivity, and biological activity of Schiff bases and their metal complexes.[19] Schiff base containing nitrogen and oxygen atoms play a significant role in coordination chemistry and bioinorganic compounds because of their flexibility and denticity. The denticity of the ligand also played a significant role in the chelation process between the ligand and metal ions. The chelation ability of Schiff bases results in the formation of complexes with enhanced stability and contributes to the various geometries of such complexes. Coordination chemistry is now attracting the interest of inorganic chemists due to its prospective applications in organic, inorganic, analytical, and medicinal areas.[20-21] The coordination compounds are very useful because they can make coordinate bonds between the core metal ion and the ligands. Schiff base transition metal complexes shows various biological activities like antibacterial, antimalarial, anticancer, antidiabeticetcs. In the present study, we have synthesized and characterized Cu(II) complexes derived from tetradentate Schiff base ligand.

2. EXPERIMENTAL

Materials and Methods: All the chemicals used for the synthesis of Schiff base ligand and its copper(II) complexes were of analytical grade. All the solvents were used without any further purification. The melting points of the synthesized complexes were recorded on digital 9100 melting point apparatus The C, H and N analysis for Schiff base and complexes was carried out using Perkin-Elmer 2400 elemental analyzer. The molar conductivity of the synthesized complexes in DMF solution of 10^{-3}M concentration on Elico conductivity cell. The infrared spectra of the ligand and synthesized complexes were recorded on Shimadzu infrared spectrophotometer using KBr discs in the range of 4000-400 cm^{-1}. The electronic spectra of the investigated complexes were recorded on Perkin-Elmer UV-Visible spectrophotometer. The magnetic susceptibility of the complexes was carried out on Gouy balance using $Hg[Co(SCN)_4]$ as a standard.

Synthesis of Schiff base

The Schiff base ligand was prepared by condensation of 0.01 mole of 2,6-diacetyl pyridine with 0.02 mol of α-amino acetohydroxamic acid in ethanol(25 ml). The resulting mixture was refluxed under condenser fitted with a water tap for about 3-4 hours. The reaction mixture was monitored by TLC.The reaction mixture was cooled till yellowish solid was obtained. The resultant solid was separated by filtration and washed with ethanol and recrystallized with acetone and dried over anhydrous $CaCl_2$ in a desiccator.The melting point of the solid compound was found to be 192^0C.

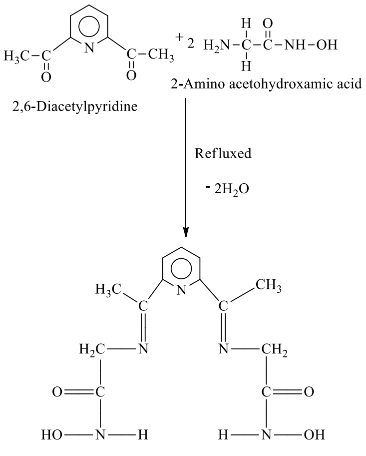

Fig. 1: Schiff base Ligand.

Synthesis of the copper complexes

The investigated Cu (II) complexes were obtained by refluxing ethanolic solution of Schiff base ligand 2,6-bis(α-imino acetohydroxamic acid)di-acetyl pyridine and Copper(II)chloride hydrate in a molar ratio 1:1 (ligand: metal). The resulting solution was filtered and the filtrate was refluxed for 1-2 hours on water bath. The resultant coloured solid product was collected by filtration and washed with cold ethanol, and finally dried over anhydrous $CaCl_2$ in a desiccator. The complexes of copper(II) with Schiff base ligand 2,6-bis(α-imino acetohydroxamic acid)di-acetyl pyridine were prepared separately in the presence of different bases like water, ammonia, pyridine, quinolone, phenyl-isocyanide and α-picoline, keeping the metal –ligand ratio in each cases as 1:1. The colour of complexes formed with different bases varied with time of reflux.

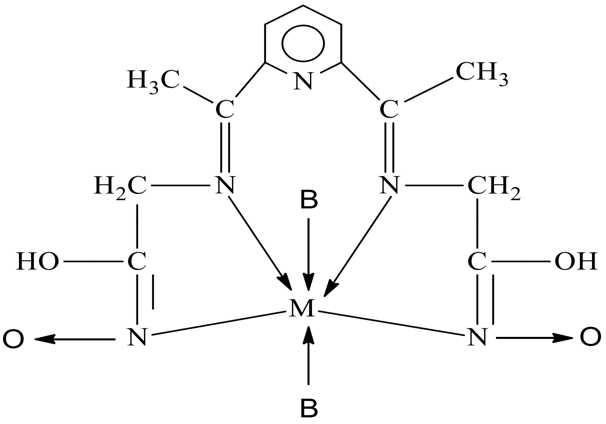

Figure 2: Structure of copper(II) complexes.

M = Cu(II) and B = water, ammonia, pyridine, quinolone, phenyl-isocyanide and α-picoline.

3. RESULTS AND DISCUSSION

All the synthesized Cu(II) complexes are coloured, solid and stable towards atmospheric air at room temperature.They are soluble in some common organic solvents like dimethylsulphoxide (DMSO), dimethylformamide (DMF), methanol, acetone, ethanol, and chloroform. The analytical data of Schiff base and its Cu(II) complexes are listed in Table- 1. The molar conductivity values and magnetic moment data are listed in Table-2. The observed molar conductance of investigated Cu(II) complexes in DMF solution ranges from 10 to 24 Ω^{-1} cm^2 mol^{-1}, suggesting non-electrolytic nature of complexes. The metal: ligand ratio is 1:1 in all synthesized complexes. The micro-analytical and spectral results suggested the synthesized complexes correspond to $[Cu(L)(B)_2]$ stoichiometry, where B = water, ammonia, pyridine, quinolone, phenyl-isocyanide and α-picoline.

Magnetic behaviour and Electronic absorption spectra

The magnetic moment values of investigated copper(II) complexes have been found in the range of 1.83 – 1.98 B.M., suggested distorted octahedral geometry of Cu (II) complexes. The electronic spectral data of metal complexes are listed in Table-3. The electronic absorption spectra of the Schiff base and its copper(II) complexes were recorded in DMSO solution at room temperature. The electronic absorption spectrum of ligand shows two bands at 285 nm and 340 nm corresponding to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions respectively. These transitions arise due to the presence of lone pair of electrons on nitrogen atom of the Schiff base and due to double bonds containing π – electrons. The disappearance of $n \rightarrow \pi^*$ transition in investigated copper(II) complexes, suggested the azomethine nitrogen atom coordinating with copper(II) ion. The appearance of one broad and unsymmetrical band in the range of 610-650 nm also suggested distorted octahedral geometry of copper(II) complexes.

Table 1: Micro- analytical data of Cu(II) complexes.

Compounds	M.P (^{0}C)	Colour	Found (Calculated) %			
			C	H	N	Cu
Ligand(L)	192	Yellow	50.81	5.58	22.79	-
$[Cu(L)(H_2O)_2]$	225.4	Blue	38.57(38.52)	4.73(4.68)	17.30(17.23)	15.70(15.64)
$[Cu(L)(NH_3)_2]$	218.6	Blue	38.75(38.72)	5.25(5.19)	24.34(24.30)	15.77(15.72)
$[Cu(L)(C_5H_5N)_2]$	232.3	Deep blue	52.42(52.39)	4.78(4.72)	18.60(18.52)	12.06(12.01)
$[Cu(L)(C_6H_5NC)_2]$	228.6	Light blue	56.39(56.32)	4.38(4.29)	17.05(17.01)	11.05(10.98)
$[Cu(L)(C_9H_7N)_2]$	234.7	Blue	59.37(59.34)	4.66(4.59)	15.63(15.57)	10.13(10.02)
$[Cu(L)(C_5H_4NCH_3)$	241.3	Light blue	54.09(53.89)	5.27(5.23)	17.66(17.69)	11.45(11.38)

Table 2: Molar conductivity and magnetic moment data of Cu(II) complexes.

Compounds	Solvent	Λ_m (Ω^{-1} cm^{2}mol^{-1})	$\mu_{eff(BM)}$	Magnetic behaviour
$[Cu(L)(H_2O)_2]$	DMF	18	1.96	Paramagnetic
$[Cu(L)(NH_3)_2]$	DMF	16	1.92	Paramagnetic
$[Cu(L)(C_5H_5N)_2]$	DMF	10	1.98	Paramagnetic
$[Cu(L)(C_6H_5NC)_2]$	DMF	20	1.83	Paramagnetic
$[Cu(L)(C_9H_7N)_2]$	DMF	24	1.94	Paramagnetic
$[Cu(L)(C_5H_4NCH_3)$	DMF	16	1.86	Paramagnetic

Table 3: Electronic spectral data of Cu(II) complexes.

Compounds	Ligand bands(nm)	d-d bands(nm)	Geometry
Ligand	285, 340	-	-
$[Cu(L)(H_2O)_2]$	285	610	Distorted octahedral
$[Cu(L)(NH_3)_2]$	285	618	Distorted octahedral
$[Cu(L)(C_5H_5N)_2]$	285	632	Distorted octahedral
$[Cu(L)(C_6H_5NC)_2]$	285	640	Distorted octahedral
$[Cu(L)(C_9H_7N)_2]$	285	650	Distorted octahedral
$[Cu(L)(C_5H_4NCH_3)$	285	645	Distorted octahedral

Infrared spectra

The Schiff base ligand 2,6-bis(α-imino acetohydroxamic acid)di-acetyl pyridine is highly sensitive into the enol form to form the complexes. This is expected that two hydrogen atoms of two Oxime group (-N=O-H) have been deprotonated producing negative charges on each of nitrogen atom. Two azomethine groups are capable to participate in bond formation with the metal cations. Thus, the ligand behaves as bi anionic tetradentate molecule. Thus, on the basis of infra-red spectral data two nitrogen atoms of oxime group and two nitrogen atoms of two azomethine groups are the coordinating sites of the ligand. In almost all the complexes, (OH) band, azomethine band (C=N), oxime band (C=N) and (N-O) band of the ligand molecule are appreciably affected after the complex formation. The strong and sharp bands due to the vibrations of (O-H) and (N-H) bonds of the ligand obtained at 3260 cm^{-1} disappears in all the complexes of the ligand and a new broad band appears in the complexes at about 3430-3480 cm^{-1} confirming the presence of at least one free (-OH) group even in the complexes. The disappearance of the strong band at 1240 cm^{-1} further suggests the deprotonation of the hydroxy group (-OH) group i.e., (N - OH) proton located at hydroxamic acid moiety. The azomethine band located at 1640 cm^{-1} in the ligand molecule is also shifted to lower frequency in almost all the complexes by about 45-60cm^{-1}. This shift of (>C=N) band towards lower frequency in the complexes suggests the coordination of azomethine nitrogen of the ligand in the formation of the complexes. The appearance of one more band in the range of 740 - 860 cm^{-1} due to rocking and wagging mode of vibration suggests that H_2O is present in the coordination sphere. The pyridine ring breathing mode of vibration located at 990 cm^{-1} in the complexes has been taken to be characteristic vibrations of coordinated pyridine. The appearance of (M - N) band obtained between 420-460 cm^{-1} in all the complexes, which suggested the coordination through nitrogen atoms of azomethine, oximinio and bases such as Water, ammonia, quinolone, phenyl-isocyanide, α-picoline and pyridine of the ligand.

4. CONCLUSION

The Schiff base ligand 2,6-bis(α-imino acetohydroxamic acid)di-acetyl pyridine has been synthesized by the condensation of 2- amino acetohydroxamic acid and 2,6-diacetyl pyridine in an alcoholic medium at room temperature. The Schiff base and its metal complexes were characterized by micro elemental analysis and different spectroscopic techniques. All the metal complexes have 1:1 (metal: ligand) stoichiometry. The lower molar conductance values of investigated metal complexes in DMF at room temperature revealed their non- electrolytic nature. All the metal complexes have been found to be paramagnetic. The investigated ligand acts as tetradentate and coordinated through two azomethine nitrogen atoms and two oxyimino nitrogen atoms in the all complexes of copper. Thus, on the basis of elemental analysis and spectral studies distorted octahedral geometry for Cu (II) complexes were proposed.

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