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STRUCTURAL ELUCIDATION AND THERMAL STUDIES OF SOME NOVEL MIXED LIGAND SCHIFF BASE METAL (II) COMPLEXES

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ABSTRACT

Novel mixed ligand Co(II), Ni(II) and Cu(II) complexes of Schiff base derived through the condensation of Bis (2-Hydroxy Phenyl) Ethane -1,2-Diamide have been synthesized . The investigated complexes have been characterized by elemental analysis, magnetic and conductance measurements, IR and UV-visible spectral studies . The spectral results suggests octahedral geometry for Co(II) and Ni(II) complexes. Distorted octahedral geometry has been assigned for Cu(II) complex.. All ligands and their metal complexes were screened for antimicrobial activity. The results of antimicrobial activity indicated that metal complexes have significantly higher activity than corresponding ligands.

Key Words: Antimicrobial activity, Bivalent ligand, Ttransition metal, Schiff Base

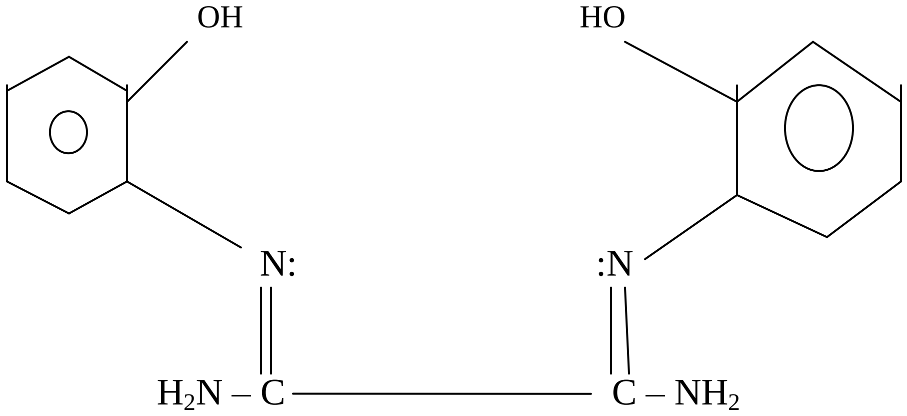
I INTRODUCTION

Schiff base ligands have significant importance in chemistry; especially in the development of Schiff base complexes, because Schiff base ligands are potentially capable of forming stable complexes with metal ions Mixed ligand complexes of transition metals containing ligands with N, O donors are known to exhibit interesting stereochemical, electrochemical and electronic properties .In recent decades, a great deal of interest in the metal complexes of nitrogen-oxygen chelating agents derived from ligand Schiff bases have various applications in antifungal, antibacterial and analgesic.

The synthesis and structural investigations of the transition metal complexes of various ligands are of considerable centre of attention becase of their potentially beneficial pharmacological properties and a wide variation in their mode of binding . The transition metal with heterocyclic ligands, especially those containing N and O have diverse application in various fields and get more attraction recently.

All these facts motivate our interest in the complex complound with these heterocyclic ligand. Here we report the synthesis, characterisation and antibacterial activities of Co(II), Ni(II) and Cu(II) with the Bis (2-Hydroxy Phenyl)Ethane -1,2-Diamide (L).

The structure of the Bis (2-Hydroxy Phenyl)Ethane -1,2-Diamide (L) is given in fig.



Experimental

Materials and Methods

All the chemical and solvents used for the synthesis of ligand and complexes under investigation were of analytical grade.

Synthesis of the ligand

0.9 gram (0.01 mole) of ethane -1,2-diamide was completely dissolved in minimum volume of aqueous alcoholic solution , 22 grams (0.01 mole) of o-hydroxy aniline was also dissolved in minimum volume of ethyl alcohol .Both the solutions were mixed together gradually with regular shaking. The resulting solution was refluxed under condenser fitted with water tap for three and half an hour. Then the solution was cooled under ice-bath while a dirty yellowish solid was obtained. The solid was separated by filtration and washed with cold water and a little alcohol. The compound was easily soluble in acetone,less soluble in cold ethanol but more soluble in hot ethanol. The solid was re-crystallized with ether and dried over KOH pellets. The melting point of the compound was recorded to be 154℃.

Synthesis of the Metal Complexes

2.7 grams (0.001 mole) of the ligands –bis(o-hydroxy phenyl) ethane -1,2-diamide was dissolved in a minimum volume of aqueous ethanolic solution . It was then treated with an ethanolic solution of 0.23 gram (0.001 mole) $Co^{(II)}$ choride hexahydrade . The resulting solution wes heated under reflux on bath for 2 hours when a light brown crystalline solid was obtained which was separated by filteration and washed with small quantity of alcohol followed by ether and dried over KOH pellets placed in desiccators . It was further analysed and found to contain .

III RESULTS AND DISCUSSION

The complexes obtained are crystalline variously coloured powders whose melting points are higher than that of the free ligand. The results of elemental analysis (C,H and N) and the metal contents along with molecular formulae and melting points of the and its complexes are presented in Table-1. The molar electric conductivities showed that Co(II) and Ni(II) complexes are non-electrolytes while Cu(II) are electrolytes.

Table-1

Complexes [Colour]	Yield (%)	MP(℃)	Analytical found (cal.)				λ_{M*} ($\Omega^{-1}cm^2mol^{-1}$)
			C	H	N	M	
Ligand (L) $[C_{14}H_{14}N_4O_2]$ Yellowish	83	149	63.80 (63.22)	5.20 (5.18)	20.78 (20.74)	-	-
$[Co(L)(B)_2]$ Brownish	81	182	61.14 (61.50)	5.10 (5.06)	16.44 (16.37)	8.08 (7.91)	14.01
$[Ni (L)(B)_2]$ Green	76	174	60.54 (60.85)	5.06 (5.07)	16.46 (16.38)	7.92 (7.81)	13.89
$[Cu(L)(B)_2]$ Green	72	171	62.20 (62.51)	4.10 (4.09)	15.70 (15.62)	8.68 (9.52)	14.25

*in 10^{-3} DMF solution at room temperature

The observed molar conductance value are too low to account for any dissociation of the complexes in DMF at room temperature ,indicating non-electrolytic nature of the complexes.

IR Spectral Studies

The IR spectra provide information about any functional group present in the molecules that support the successful formation of Schiff bases and metal complexes. The comparative analysis of IR spectra of the Schiff bases and metal complexes. Strong bands corresponding to aldehyde v(C=O) disappeared in the infrared spectra of compounds were successfully formed through condensation. The stretching vibration of v(N-H) ,at 3430 and 3445 cm^{-1} were not present in the metal complexes. This is due to the deprotonation of the nitrogen atom upon complexation. Compounds displayed sharp peaks at 1580 and 1599 cm^{-1} ,which corresponded to v(C=N) ,attributable to the quinoline and pyridine group. This stretching band schifted to lower wavenumber upon complexation to the metal ions.In this work, the v(C=N) decreased upon complexation ,indicating the coordination of compounds to the metal ions via azomethine nitrogen. The hifting of hydrazinic v(N-N) band to bath higher and lower wavenumbers in the infrared spectra of complexes showed evidence of metal coordination through the quinoline and pyridine nitrogen atom,as this was related to the reduction in the repulsion between lone pairs of electrons on the nitrogen atoms upon complexation .

The data of the IR spectra of investigated Schiff base ligands and their metal complexes are listed in following table. The IRspectra of the complexes were compared with those of the free ligand in order o determine the involvement site in chelation.

Infrared,agnetic susceptibility and electronic spectral data of compounds 1-8.

Compound	IR Bands (cm^{-1})			μeffBM	Elactronic Spectra(IN DMSO) ʎ max
	v(NH)	v (C=N)	v (N-N)		
1	3420	1560	1210	-	352(4.86)
2	3430	1580	1228	-	341(4.63)
3	-	1530	1207	1.60	352(4.93) 601(2.57)
4	-	1510	1211	Paramagnetic	354(4.90) 609(2.08)
5	-	1590	970	Paramagnetic	354(5.21)
6	-	1570	979	1.67	624(2.23) 422(3.92)
7	-	1540	965	Paramagnetic	342(4.88) 444(3.55)
8	-	1583	940	Paramagnetic	609(2.07) 341(5.08)

Magnetic moment and UV-Vis Spectra

Electronic spectra and magnetic measurements were performed in order to obtain information about the geometry of the complexes.

The values of Co(II) complexes were found to be 4.86 and 5.10, indicating an octahedral geometry around the metal ion and 1.96 B.M. for Cu(II) complex respectively, indicating a square-planar geometry. Ni(II) complex is diamagnetic. The first band placed in the UV region is due to a $\pi \rightarrow \pi^*$ type electronic transition from the aromatic cycles. The remainder absorption bands are due to v $\rightarrow \pi^*$ electronic transitions.

The electronic spectrum of Co(II) complex showed bands at 14800 and 15600 cm^{-1} assignable to $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, and $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ transitions, respectively, which are characteristic to the octahedral configuration.

Ni(II) complex exhibited two transitions at 15500 and 16400 cm^{-1} assignable to $^3A_{2g}$ (F)$\rightarrow$ $^3T_{2g}$(F) and $^3A_{2g}$ (F) $\rightarrow$ $^3T_{2g}$ (P) transitions, respectively, which correspond to a square-planar geometry.

The UV-vis spectrum of Cu(II) complex displays a broad band at 10000 cm^{-1} to 145000 cm^{-1} attributable to $^2B_{1g} \rightarrow {}^2A_{1g}$ transition, which favours the square-planar geometry around the central metallic ion.

APPLICATIONS

Antibacterial activity

The difference in antibacterial activities of the investigated liand and its transition metal(II) complexes with the standard were studied exclusively and the obtained . The experimental results indicate that the investigated complexes are found to be more active in comparison to the free ligand against the same micro-organisms under the similar experimental conditions. The antibacterial activity of the ligand and its metal complexes follow the order.

Standard > Co-complex > Ni-complex > Cu-complex > Ligand

CONCLUSIONS

The transition metal complexes with a ligand Bis (2-Hydroxy Phenyl) Ethane -1,2-Diamide were prepared and characterized. The IR spectra indicate ion that the ligand acts in bidentate fashion by bonding to the central metal through the nitrogen and oxygen atoms. Electronic spectra and magnetic measurements gave information about the octahedral geometry of the Co(II) and Ni(II) complexes and Cu(II) complex have a distorted octahedral geometry . This means that the metal chelation significantly affects the antimicrobial behaviour of the organic ligand.

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