

To Study the Synthesis and Biological Activity of Thiosemicarbazone-2-Benzoylpyridine Schiff's Base ligand and Its Cu (II) Transition Metal Complexes

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Abstract—

Objective: The objective of this study is to synthesize Schiff's base ligands of thiosemicarbazone-2-benzoylpyridine and their copper complexes and study their antimicrobial activity.

Materials and Method: Thiosemicarbazone-2-benzoylpyridine Schiff's base ligand was prepared by reflux method by using ethyl alcohol as solvent for 7–8 hrs. Ligand under study was found to be highly soluble in water, and its complexes were prepared in water. P_H was adjusted as per the requirement in each case (approximately 6–7). Round bottom flask (100 mL), condenser, and heating mantle were used as apparatus.

Result: The complexes of Cu(II) having the general composition CuL_2X_2 (where $L = 2$ -benzoylpyridine thiosemicarbazone and $X = Cl^-, NO_3^-, \frac{1}{2}SO_4^{2-}$) have been synthesized. The complexes of Cu(II) under study were characterized by IR, elemental analysis, magnetic susceptibility, UV, molar conductivity, mass spectrometry, and electron EPR spectral studies. Molar conductance value was calculated using DMSO as solvent. The magnetic moment indicates that all the complexes were of high spin type. On the basis of spectral studies, an octahedral geometry has been proposed for Cu (II) complexes. The complexes under study were screened for antifungal (i.e., *Candida krusei* and *Candida parapsilosis*) and antibacterial (i.e., *Escherichia coli* and *Staphylococcus*) activities.

Conclusion: The synthesized ligand was bidentate and their complexes were found more active toward antimicrobial activity than ligands.

Keywords— Mass, IR, NMR, EPR, Bidentate, Cu (II), Thiosemicarbazone, Magnetic Moment.

I. INTRODUCTION

Coordination chemistry is the most interesting and important branch of inorganic chemistry. The discovery of Coordination compounds brought the revolution in inorganic chemistry because of its wide range of application such as analytical chemistry [1-4], biological activities pharmaceuticals [5-10], catalyst and potentiometric sensor. More recently these ligands and their metal complexes have been found to possess antiviral [11-13], antitumour [14-18], antioxidant [19], DNA binding and DNA cleavage, antifungal and antibacterial activities. Due to these applications it is highly desirable to synthesize transition metal complexes with such ligands. In present paper we report the synthesis of complexes derived from 2-benzoylpyridine thiosemicarbazone with corresponding copper metal salt.

II. EXPERIMENTAL SECTION

All reagents were commercially available and used without further purification purchased from Sigma Aldrich and metal salts were purchased from E. Merck. Solvents were

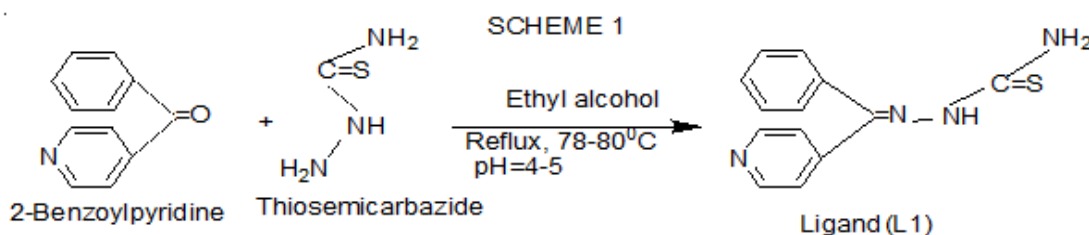
spectroscopic pure from SRL/BDH or purified by conventional methods.

III. PREPARATION OF LIGAND (L^1)

In minimum quantity of ethanol thiosemicarbazide (0.091 g, 0.01 mol) was dissolved. In this solution hot ethanolic solution (0.18 g, 0.01M) of 2-benzoylpyridine was added very slowly with constant swirling. This resulting solution was refluxed at 78 – 80°C for 8 hrs. and the pH of this solution was adjusted to approximately 4 – 5 using acetic acid (according to Scheme 1). On cooling, bright yellow-coloured crystals were separated out. These crystals were washed out several times with cold ethanol. The ligand was found to be highly soluble in water.

IV. SYNTHESIS OF Cu(II) COMPLEXES

The complexes of Cu (II) were synthesized from ligands (L^1) by condensation reaction between ligand and corresponding metal salts in 1 : 2 ratio at pH range approximately 6–7.



V. RESULT AND DISCUSSION

On the basis of elemental analyses, the complexes were found to have general composition $Cu(L)^2X_2$ (where $X = Cl^-, NO_3^-, OAc^-$, and $\frac{1}{2}SO_4^{2-}$). The molar conductance values lie between 11 and 19 ($\Omega\text{-}1\text{cm}^2\text{ mol}^{-1}$), indicating that all the copper complexes are non - electrolytic in nature and have composition $(Cu(L)^2X_2)$. All complexes and ligands were found to be biologically active toward test fungi and bacteria. Complexes were found to be more biologically active than ligands.

VI. ANTIMICROBIAL ACTIVITY

All the synthesized $Cu(II)$ complexes were screened for antibacterial and antifungal activity in vitro by broth dilution method [20-24] with two Gram-positive bacteria *Staphylococcus aureus*, one Gram-negative bacteria *Escherichia coli*, and two fungal strains *Candida parapsilosis*

and *Candida krusei*. Serial dilutions of the test compounds and biological activities of ligands and their metal complexes were prepared in Mueller-Hinton agar. Drugs (0.005, 0.050, and 0.500 mg) were dissolved in DMSO, 1 mL. 0.5 McFarland solution of *E. coli*, *S. aureus*, and *C. parapsilosis* and *C. krusei* was prepared and applied on Mueller-Hinton agar contained in a Petri plate with the help of sterilized swab. Then, 10 μL solution of concentration (0.005, 0.050, and 0.500 mg) in 1 mL DMSO was dropped on it with the help of micropipette. This Petri plate was incubated for 24 h at $22 \pm 29^\circ\text{C}$. The growth of fungi and bacteria was measured diametrically. The values are listed in Tables 1-2.

To make sure that the DMSO had no effect on the bacterial growth, a control test was performed with the test medium supplemented with DMSO at the same dilutions as used in the experiments and it was observed that solvent had no effect on the microorganisms in the concentrations studied.

Biological Activity of Prepared Schiff's Base Ligand and Their $Cu(II)$ Complexes



TABLE 1. Antibacterial screening results of ligand (L^1) and its complexes

S. No.	NAME	CONC. (μg)	E.COLI(Dim) (mm)	STAPH Diam.(mm)
1.	Ligand 1	0.005	NA	10
		0.050	10	14
		0.500	14	17
I	Cu			
1.	L^1CuAc	0.005	NA	12
		0.050	19	19
		0.500	20	23
2.	L^1CuSO_4	0.005	NA	22
		0.050	27	28
3.	L^1CuNO_3	0.005	19	28
		0.050	27	23
4.	L^1CuNO_3	0.050	19	18
		0.025	17	17
5.	L^1CuSO_4	0.050	24	20
		0.025	15	14
6.	L^1CuCl_2	.005	14	13
		0.050	18	17

TABLE 2. Antifungal screening results of ligand 1 and its complexes

S. No.	NAME	CONCENTRATIONS (μg)	KRUSEI DIAM (mm)	PARAPSILOSIS DIA (mm)
1.	Ligand L ¹	0.005	10	R
		0.050	15	10
		0.500	17	17
I	Cu			
1.	L ¹ CuAc	0.005	12	12
		0.050	19	16
		0.500	21	18
2.	L ¹ CuSO ₄	0.005	10	11
		0.050	12	15
3.	L ¹ CuNO ₃	0.005	12	11
		0.050	20	18
4.	L ¹ CuCl ₂	.005	14	13
		0.050	18	17

VII. CONCLUSION

The antimicrobial screening of all investigated compounds provided information about the biological activity of ligand and its complexes, and it was found that complexes are more biologically active than ligands.

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