

To Study Synthesis and Antimicrobial Activity of Bidentate Schiff's Base Ligand Derived from 2-Benzoylpyridine Thiosemicarbazide and Their Ni (II) Complexes

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Abstract—The complexes of nickel (II) derived from 2-benzoylpyridine and thiosemicarbazone ligand having general composition $[Ni(L)^2X_2]$ ($X = NO_3^-, Cl^-$ and $OAc^-, \frac{1}{2}SO_4^{2-}$) have been synthesised. The complexes and ligand under study are characterised by elemental analysis, IR spectroscopy, UV-Vis spectroscopy, mass spectra, conductivity measurement, magnetic susceptibility, EPR spectral studies and NMR Spectroscopy. IR spectral study indicates that ligands behaves as bidentate and coordinated to metal ion through nitrogen and sulphur donor atoms. The molar conductance was measured by using DMSO as solvent. The lower value of molar conductance suggests that the complexes are non electrolytic in nature. The value of magnetic susceptibility indicates that complexes are of low spin type. On the basis of above spectral studies an octahedral geometry may be proposed for all the 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. The synthesized ligand was bidentate and their complexes were found more active toward antimicrobial activity than ligands.

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

I. INTRODUCTION

Schiff bases are fascinating and significant ligands find extensive applications in the field of coordination chemistry [1-8]. Schiff bases also called as privileged ligands act as chelating ligands for many metal ions. In recent years, there has been enhanced interest in the synthesis and characterization of transition metal complexes containing Schiff bases as ligands due to their importance as catalysts in many reactions [9-13]. Also Schiff base complexes derived from heterocyclic compounds have acquired more attention in the field of bioinorganic chemistry because of their biological activities. Some heterocyclic ketone derived Schiff bases show antibacterial activity and some others can act as antibacterial agent [13- 17]. The present investigation deals with synthesis of complexes of Ni(II) by Schiff base ligand (L) derived from 2-benzoylpyridine and thiosemicarbazide and to study their antimicrobial activity.

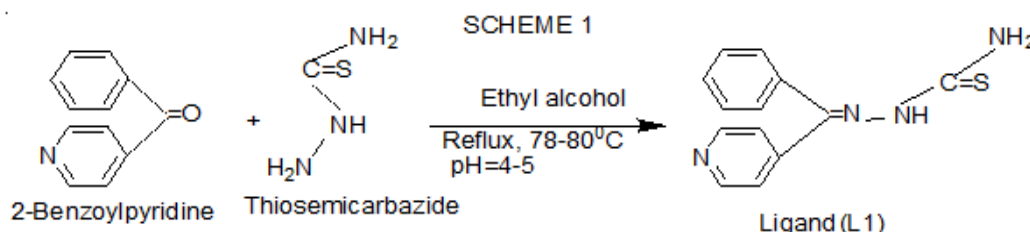
Experimental Section: All reagents were commercially available and used without further purification purchased from sigma Aldrich and metal salts were purchased from E. Merck.

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

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.

Preparation of ligand(L): Thiosemicarbazide (0.091g, 0.01mol) was dissolved in minimum quantity of ethanol. To this hot ethanolic solution of (0.18g, 0.01mol) 2-benzoylpyridine was added very slowly with constant stirring. The resulting solution was refluxed at 78-80°C for 8hrs. and the pH was adjusted to approximately 4-5 by 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 is highly soluble in water.



Preparation of Ni (II) complexes with ligand (L): Hot aqueous solution (20 ml) of corresponding metal salts (0.01mol) were mixed with hot aqueous solution of the ligand (0.02mol) with constant stirring and refluxed for 7-8 hrs. at 65-70°C. On cooling the contents, the coloured precipitates were separated out in each case. It was filtered, washed with ethanol and ether. The resultant mixture was dried over P4O10.

III. RESULT AND DISCUSSION

On the basis of elemental analyses, the complexes were found to have general composition Ni(L)²X₂ (where X = Cl⁻, NO₃⁻, OAc⁻, and ½SO₄²⁻). The molar conductance values lie between 11 and 19 (Ω⁻¹cm² mol⁻¹), indicating that all the copper complexes are non - electrolytic in nature and have composition [Ni(L)²X₂]. 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.

IV. ANTIMICROBIAL ACTIVITY

All the synthesized Ni (II) complexes were screened for antibacterial and antifungal activity in vitro by broth dilution method [17-25] 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 ± 29°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.

TABLE 1. Antibacterial screening results of ligand (L¹) and its Ni (II) 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
II				
Ni				
1.	L ¹ NiCl ₂	0.005	NA	NA
		0.050	NA	NA
2	L ¹ NiSO ₄	0.005	12	28
		0.050	22	42
3.	L ¹ NiBr	0.050	18	20
4.	L ¹ Ni(OAc)	0.005	NA	NA
		0.050	10	12

TABLE 2. Antifungal screening results of ligands and its Ni (II) complexes

S. No.	NAME	CONCENTRATIO NS (µg)	KRUSEI DIAM (mm)	PARAPSILOSIS (DIA (mm))
1.	Ligand L ¹	0.005	10	R
		0.050	15	10
		0.500	17	17
II				
NICKEL				
1.	L ¹ NiCl ₂	0.005	NA	NA
		0.050	10	16
2	L ¹ NiSO ₄	0.005	NA	NA
		0.050	10	12
3.	L ¹ NiNO ₃	0.005	NA	NA
		0.050	11	10
4.	L ¹ NiOAc	0.005	NA	NA
		0.050	12	13
5.	L ¹ NiBr	0.050	12	12

Antibacterial Activity of Prepared Schiff's Base Ligand and Their Ni(II) Complexes



V. CONCLUSION

The synthesised ligands and complexes were characterised by elemental analysis, IR spectroscopy, mass spectroscopy,

UV-Vis, ¹H NMR spectroscopy and EPR spectral studies. The proposed study of complexes indicates octahedral geometry for all the nickel (II) complexes. Ligands were found to be bidentate which coordinates to metal through azomethine

group as $\nu(\text{C}=\text{N})$ and through sulphur as $\nu(\text{C}=\text{S})$ group respectively. 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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