



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

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## ABSTRACT

*Novel mixed ligand Co(II), Ni(II), Cu(II) and Zn(II) complexes of Schiff base derived through the condensation of N-(2-Pyrimidyl)-quinoline-2-thioamide have been synthesized. The investigated complexes have been characterized by elemental analysis, magnetic and conductance measurements, IR and UV-visible spectral studies. 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, Transition 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, S or N, S, 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 N-(2-Pyrimidyl)-quinoline-2-thioamide Schiff bases have various applications in antifungal, antibacterial and analgesic.

All these facts motivate our interest in the complex compound with these heterocyclic ligand. Here we report the synthesis, characterisation and antibacterial activities of Co(II), Ni(II), Cu(II) and Zn(II) with the N-(2-Pyrimidyl)-quinoline-2-thioamide, (L).

The structure of the N-(2-Pyrimidyl)-quinoline-2-thiocarboxamide (L) and the atom numbering scheme are given in fig.

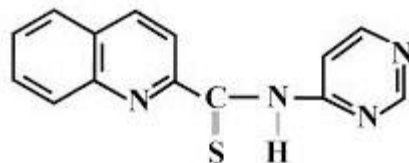


Figure - 1 : Structure of ligand (L)

## II EXPERIMENTAL

### Materials and Methods

All the chemical and solvents used for the synthesis were of analytical grade.

### Synthesis of ligand and metal complexes

The ligands N-(2-Pyrimidyl)-quinoline-2-thiocarboxamide were prepared using Willgerdot reaction. Quinaldine and 2-aminopyrimidine were distilled product and sulphur was of also pure from 2-amino- pyrimidine (distilled 50ml), quinaldine (distilled 25ml) and sulphur (powder 25 gm) were mixed in a 200 ml flask fitted with water reflux condenser. The mixture was refluxed at 160°C for 10 hours on a and bath maintained temperature between 165-175°C. The mixture was left over night.

## III RESULTS AND DISCUSSION

The complexes obtained are microcrystalline variously coloured powders whose melting points are higher than that of the free ligand. The results of elemental analysis (C,H,N and S) and the metal contents along with molecular formulae and melting points of the thiocarboxamide 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) and Zn(II) complexes are electrolytes.

Table-1

Complexes [Colour]	Yield (%)	MP(°C)	Analytical found (cal.)					$\lambda_M^*$ ( $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ )
			C	H	N	S	M	

								1)
Ligand (L) [C <sub>14</sub> H <sub>10</sub> N <sub>4</sub> S] Yellow	89	154	63.45 (63.39)	3.40 (3.39)	21.25 (21.13)	12.10 (12.07)	-	-
[CoL <sub>2</sub> Cl <sub>2</sub> ] Brown	87	197	50.95 (50.91)	2.74 (2.73)	16.95 (16.97)	9.72 (9.69)	9.10 (8.93)	15.08
[Ni L <sub>2</sub> Cl <sub>2</sub> ] Yellowish brown	85	193	50.95 (50.93)	2.80 (2.73)	16.98 (16.97)	9.72 (9.68)	8.92 (8.89)	14.65
[Cu L <sub>2</sub> Cl <sub>2</sub> ] Yellowish green	83	185	50.62 (50.56)	2.74 (2.71)	16.90 (16.85)	9.72 (9.63)	9.60 (9.56)	15.35
[Zn L <sub>2</sub> Cl <sub>2</sub> ] Yellowish	81	183	50.48 (50.42)	2.71 (2.69)	16.87 (16.80)	9.70 (9.61)	9.58 (9.53)	15.33

\*in 10<sup>-3</sup> 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 data of the IR spectra of investigated Schiff base ligands and their metal complexes are listed in Table-2 .The IR spectra of the complexes were compared with those of the free ligand in order to determine the involvement site in chelation.

**Table-2**

Ligand (L)	[CoL <sub>2</sub> Cl <sub>2</sub> ]	[NiL <sub>2</sub> Cl <sub>2</sub> ]	[CuL <sub>2</sub> Cl <sub>2</sub> ]	[ZnL <sub>2</sub> Cl <sub>2</sub> ]	Assignments
3456	3442	3442	3442	3442	ν NH
1625	1625	1625	1625	1625	ν C=Npy
1585	1574	1573	1575	1578	δ NH
1498	1512	1508	1510	1511	Thioamide I

					$\nu(\text{C-N}) + \delta(\text{C-H})$
1325	1330	1332	1333	1331	Thioamide II $\nu(\text{C-N}) + \delta(\text{CH}) + \nu(\text{C=S})$
1049	1064	1061	1063	1064	Thioamide III $\nu(\text{C-N}) + \nu(\text{C=S})$
788	812 720	811 714	792 703	791 704	Thiamide IV $\nu_{\text{S}}(\text{C-S}) + \nu_{\text{as}}(\text{C=C})$
678	662 637	662 633	657 636	659 638	$\delta(\text{C-S})$
535	510 525	515 530	515 520	516 519	$\pi(\text{C-S})$
-	425	415	425	426	M-N

### Magnetic moment and UV-Vis Spectra

#### 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 4.90 and 5.20, 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.

#### Antibacterial activity

**Table-3**

#### Anti-microbial activity of ligand (L) and their metal complexes (1-3)

Compounds	Zone of inhibition in (mm)			
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>	<i>Pseudomonas aeruginosa</i>	<i>Staphylococcus typhi</i>
L	19	20	19	20



[CoL <sub>2</sub> Cl <sub>2</sub> ]	21	20	19	20
[NiL <sub>2</sub> Cl <sub>2</sub> ]	22	21	20	21
[CuL <sub>2</sub> Cl <sub>2</sub> ]	22	22	20	21
Ciprofloxacin	23	23	21	22

#### IV CONCLUSIONS

The transition metal complexes with a thiocarboxamide ligand N-(2-pyrimidyl)-quinoline-2-thioamide (L) 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 sulphur 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 and Zn(II) complexes has a tetrahedral geometry.

#### REFERENCES

1. H.O.Desseyn,M.A.Herman,Spectrochim,Acta 23A,2457,(1967).
2. K.A.Petrov,L.N.Andreev,Usp.Khim.38,41,(1969)
3. S.p.McManus,K.Y.Lee,C.U.Pittman,J.Org.Chem.39,3041,(1974).
4. E. S. Raper, *Coord. Chem. Rev.* 61, 115, (1985).
5. P. J. Blower and J. R. Dilworth, *Coordination Chemistry Reviews* **76**, 121, (1987).
6. R. M. Oik, B. Oik, W. Dietzsch, R. Kirmse and E. Hoyer, *Coordination Chemistry Reviews*, 117, 99, (1992).
7. S.Florea,Rev.Roum.Chim,39,1138,(1994).
8. S. Yadav, O. P. Pandey and S. K. Sengupta, *Transition Metal Chemistry*, 20(2), 107, (1995).
9. M. Murrie *Chem. Soc. Rev.*, 2010, **39**, 1986 -1995
10. K. A. Jensen and P. H. Nielsen, *Acta Chem. Scand.* 20, 597, (1996).
11. P.Wipf,V.Venkatraman ,J.Org.Chem.61,8004,(1996).
12. A.Kriza,A,Reiss,V.Murecan,S.Florea,J.Indian,Chem,Soc,76(8),406,(1999).
13. A.Reiss,S.Florea,W.D.Rudorf,Polish J.Chem.Soc.74,589,(2000).
14. .Y.Hitotsuyanagi,T.Hasuda,Y.Matsumoto,K.Yamaguhi,H.Iokawa,K.Takeya,Chem.Commun.1633,(2000).
15. Y.Nakagawa,K.Irie,H.Ohigashi,.H.Hayashi,P.A.Wender,Bioorg.Med.Chem.Lett.10,2087,(2000).



16. Z.H.Chohan ,S.Kausar,Metal-Based Drugs 7(1).17.2000
17. .A.Kriza , A.Reiss,N,Stanica,Rev.Roum.de Chimie 46(5),503,(2001)
18. T. A. Vannelli, A. Dykman, P. R. O. de Montellano, *J. Biol. Chem.* **277**, 12824, (2002).
19. K.L.Yu,A.F.Torri,G.Luo,C.Cianci,K.GrantYoung,S.Danetz,L.Tiley,M.Krystal,N.A.Meanwell,Bioorg.Med.Chem.Lett.12,3379,(2002).
20. T.S. Jagodzinski, *Chem. Rev.* 103, 197, 2003
21. X.Hanouille,J.M.Wieruszkeski,P.RousselotPailley,I.Landieu,A.R.Baulard,G.Lippens,Biochem.Biophys.Res.Commun.331,452,(2005)
22. Q.L.Wel,S.S.Zhang,J.Geo,W.H.Li.L.Z,Xu,G.Z.Yu,Bioorg,Med,Chem,14,7146 (2006)
23. H. Prokopcova, C.O. Kappe, *J. Org. Chem.*, 72, 4440, 2007
24. O. V. Dolomanov , L. J. Bourhis , R. J. Gildea , J. A. K. Howard and H. Puschmann , *J. Appl. Crystallogr.*, 2009, **42** , 339
25. R. Bagai and G. Christou , *Chem. Soc. Rev.*, 2009, **38** , 1011 -1026
26. K. Graham , F. J. Douglas , J. S. Mathieson , S. A. Moggach , J. Schnack and M. Murrie , *Dalton Trans.*, 2011, **40** , 12271 -12276