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“Metal complexes of 3- acetyl 4- hydroxyquinoline and
amino pyridine schiff base moiety: Synthesis,
Characterization and Antimicrobial activity”

S. Anjanikar¹, S. Chandole^{*2}

¹ Department of Chemistry, Sharadchandra College, Naigaon, District- Nanded
MS- 431709, India,

^{*2} Department of Chemistry, S.G.B. College, Purna Jn., MS- 431511, India.

*Corresponding Author: schandole@reddifmail.com

Abstract

Metals complexes of Ni (II) and Hg (II) were synthesised with Schiff bases prepared by condensation of 3-acetyl 4-hydroxyquinolin- 2-one with substituted amino pyridine. Newly synthesised metal complexes were characterized by their elemental analysis, magnetic moment, molar conductance along with electronic, thermal, infrared spectral analysis. Ni (II) and Hg (II) complexes of ligand (L₃) were subjected for their XRD Study. On the basis of magnetic, XRD and spectral studies octahedral geometry is assigned to Ni (II) Complexes while Hg (II) Complexes possess tetrahedral geometry. All the synthesized complexes have been evaluated for their antibacterial as well as antifungal activities which has show significant activity for both properties. The Ni (II) and Hg (II) complexes with ligand L₄ have shown excellent activity as compared to standard drug used.

Keywords: Schiff's bases, Metal Complexes, Spectral and Biological Study

Introduction

Among a large variety of N-containing heterocyclic compounds, quinoline derivatives have attracted wide attention because of their pharmacological properties¹⁻³ and clinical applications⁴⁻⁶. They are widespread in nature and their applications to biologically active pharmaceuticals, functional materials

agrochemical.⁷⁻⁸ They have been in the focus of interest of medicinal chemists in the past decades because of the outstanding biological activities exhibited by several derivatives incorporating the heterocyclic moiety. Applications for both the Schiff base ligands and their transition metal complexes include biological research,⁹ analytical chemistry,¹⁰ catalysis,¹¹ clinical use,¹² the dyes industry,¹³ and the food business.¹⁴

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Accordingly, the present work is aimed at the design and synthesis of new Ni (II) and Hg(II) metal complexes of quinolone derived Schiff bases by condensation of 3-acetyl 4-hydroxyl quinolin-2-one with amino pyridine. Moreover, the study includes testing of target compounds for their expected antimicrobial and anti-fungal effect.

Materials and Methods

Melting points were determined by open capillary method and were uncorrected. IR spectra were carried out as KBr discs on a Shimadzu, IR-470, Spectrometer. The elemental analysis was

determined using the Thermo Fisher Scientific CHN/S/O analyzer instrument. The magnetic susceptibilities of complexes were measured by the Gouy's method at room temperature using the Sherwood scientific magnetic susceptibility balance and using distilled water as the calibrant. Molar conductance of the metal complexes was measured using the Fisher Scientific conductometer. The molar conductance measurements were performed using 0.001 M solutions of the complexes in DMSO at room temperature. Thermal analysis measurements were performed on Shimadzu thermo-analyzer.

Reaction scheme:

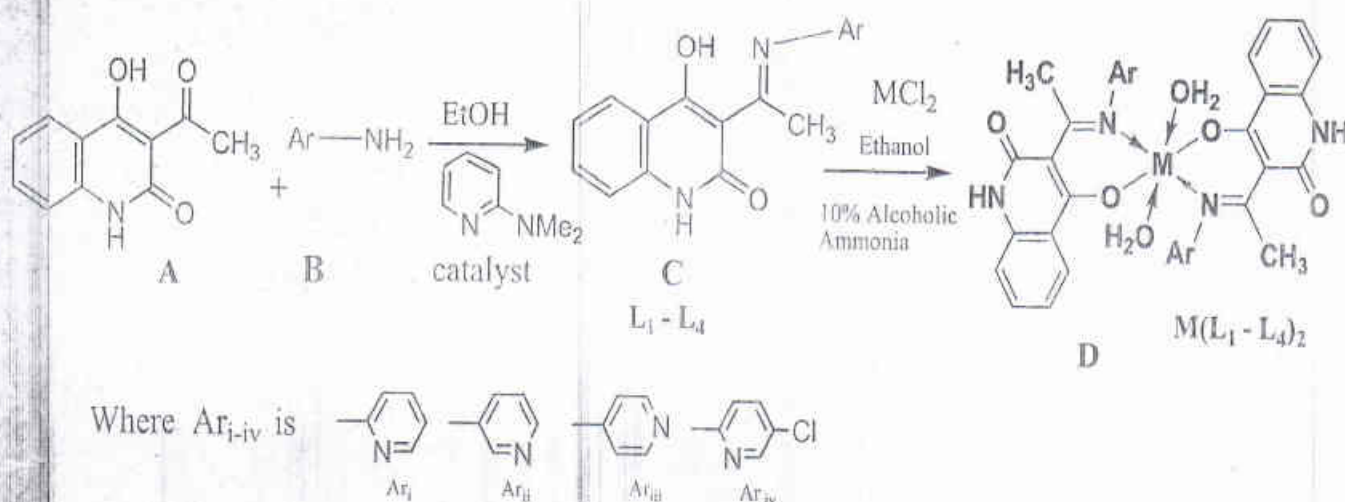


Fig. 1 Synthesis of Metal Complexes of Ni(II) and Hg (II)

Experimental:

General Procedure for synthesis of Metal Complexes:

The synthesis of each complex is processed by taking 0.02 moles of ligand (L₁-L₄) in a round-bottomed flask containing 50ml of ethanol. The contents are heated for a few minutes followed by gradual addition of 0.01 moles of solution of metal salt dissolved in 20ml of ethanol and added gradually in a hot solution of ligand. The contents are refluxed for two hours and cooled. A freshly

prepared 10% alcoholic ammonia solution is progressively added in a cold container with refluxed contents with constant stirring. At a particular pH, precipitation appears.

The precipitate of complex is digested for one hour. Any change in pH, if observed, is adjusted and further digested for an hour. The digested precipitate of a complex is filtered in hot, washed with alcohol (hot), followed by petroleum ether (40–60°C) and dried in vacuum desiccators over calcium chloride.

Antimicrobial study

Anti bacterial activity:

The agar well diffusion method was used to test the antibacterial activity.¹⁵ Mueller Hinton Agar for bacteria was used for all tests for antibacterial activity. For positive control of bacteria Ampicillin was used. The solvent and positive control used was DMSO. Antibiotics and dehydrated media powder were brought from Hi-Media, India. Using sterile wire-loop, test organisms were aseptically added to sterile MH broth before being incubated at 37°C for 18 hours. This suspension was utilized as an inoculant. Wells in the media plates with a 10mm diameter were made using a sterile cork borer for the addition of compound solutions and controls. With the aid of a micropipette, 100 µl of the compound solution was aseptically added to the wells to reach a final concentration of 10 g of compound in each well. As controls, the same quantity of DMSO and ampicillin solution were introduced. The plates were cooled for 30 minutes to allow solutions to diffuse through the agar substrate. Plates were then incubated for 24 hours at 37°C. *Bacillus subtilis* and *Salmonella typhi* were gram positive bacteria that were utilized as test organisms, whereas *Staphylococcus aureus* and *Escherichia coli* were gram negative microorganisms. The zone margin should be regarded as the region that does not clearly display any expansion that the unaided eye can see. With a measuring scale in millimetres, the clean zone was measured

Antifungal activity

The poison plate approach was used to provide antifungal activity.¹⁶ For the evaluation of antifungal activities, *Aspergillus niger*, *Aspergillus flavus*, *Fusarium moneliforme*, and *Penicillium chrysogenum* species were selected. Potato Dextrose Agar (PDA) media was utilized

as a culture. To sterilize the medium, it was autoclaved at 121°C for 25 minutes under 15 psi of pressure. 20 ml of sterilized, melted PDA was added to sterilized petri plates with 2 ml of each component, and the mixture was then gently stirred in a circular motion to get homogenized. With positive Neomycin and negative DMSO controls, the identical process was followed. The fungal spores from the slant culture were transferred to a test tube containing sterile saline and thoroughly mixed with a sterile wire loop. As an inoculant, this spore solution was employed. The plates were incubated for four days at room temperature. After incubation, the growth of the infected fungi was monitored on the plates. The outcomes were noted.

Results and Discussion

All the Nickel complexes prepared in the present work are greenish-yellow to yellow. They are appreciably soluble in dimethyl formamide (DMF) and dimethyl sulphoxide (DMSO). The low solution conductivity values of the complexes in DMSO indicate their non-electrolytic nature.¹⁷ The elemental analysis data of metal complexes confirm the ligand to the metal ratio for Nickel complexes as 2:1, proposing a monomeric complex. Mercury complexes are found colourless. They are stable in air and moisture. The Hg(II) complexes decompose at high temperature (above 300°C). They are insoluble in non-polar and common polar solvents. At a very low concentration, solutions can be prepared in methanol and DMSO. The molar conductance value of the complexes in DMSO (10^{-3} M) is very low (11.20 -15.88 $\text{mhos}^{-1} \text{cm}^2 \text{mol}^{-1}$), indicating the non-electrolytic nature of complexes. The metal to ligand ratio was shown as 1:2, predicting a monomeric structure. The colour, melting/decomposition temperature, conductivity measurement and elemental analysis of Ni (II), Hg (II) complexes synthesized from ligand L₁ - L₄ are presented in the table - 1.


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Table No. 1 Analysis Ni (II) and Hg(II) Complexes

S. No.	Ni (II) Complexes	Molecular formula	Colour	M.P./Decom. °C	Mol. Wt.	Soln cond. μ_v	Elemental analysis Found(Calculated)					
							%C	%H	%N	%Cl	%Ni	
(1)	[Ni(L ₁) ₂]	[Ni(C ₁₆ H ₁₂ N ₃ O ₂) ₂ (H ₂ O) ₂]	Yellowish Green	246	651.31	15.84	59.93 (59.01)	4.26 (4.33)	12.84 (12.90)	---	8.96 (9.01)	
(2)	[Ni(L ₂) ₂]	[Ni(C ₁₆ H ₁₂ N ₃ O ₂) ₂ (H ₂ O) ₂]	Yellowish Green	256	651.31	15.88	59.92 (59.01)	4.22 (4.33)	12.80 (12.90)	---	8.94 (9.01)	
(3)	[Ni(L ₃) ₂]	[Ni(C ₁₆ H ₁₂ N ₃ O ₂) ₂ (H ₂ O) ₂]	Yellowish Green	238	651.31	15.81	59.94 (59.01)	4.25 (4.33)	12.82 (12.90)	---	8.88 (9.01)	
(4)	[Ni(L ₄) ₂]	[Ni(C ₁₆ H ₁₁ ClN ₃ O ₂) ₂ (H ₂ O) ₂]	Greenish Yellow	255	720.19	12.40	53.24 (53.37)	3.60 (3.64)	11.59 (11.67)	9.68 (9.84)	8.08 (8.15)	
(5)	[Hg(L ₁) ₂]	[Hg(C ₁₇ H ₁₃ N ₂ O ₂) ₂]	White	>300	757.17	14.19	50.64 (50.76)	3.14 (3.20)	11.02 (11.10)	---	24.40 (26.49)	
(6)	[Hg(L ₂) ₂]	[Hg(C ₁₇ H ₁₃ N ₂ O ₂) ₂]	White	>300	757.17	11.20	50.68 (50.76)	3.21 (3.20)	11.04 (11.10)	---	26.43 (26.49)	
(7)	[Hg(L ₃) ₂]	[Hg(C ₁₇ H ₁₃ N ₂ O ₂) ₂]	White	>300	757.17	13.83	50.72 (50.76)	3.22 (3.20)	11.14 (11.10)	---	26.37 (26.49)	
(8)	[Hg(L ₇) ₂]	[Hg(C ₁₆ H ₁₁ ClN ₃ O ₂) ₂]	White	>300	815.24	12.96	46.51 (46.53)	2.64 (2.68)	10.11 (10.17)	---	24.20 (24.28)	

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Table No. 4XRD Data of Ni (II) Complex of ligand L₃

Crystal system: Monoclinic Lattice Type: P
 Lattice Parameter: a= 4.9168 b= 4.8866 c= 5.4089
 Lattice Parameter: Alpha= 90 Beta= 94.6 Gama=90
 Radiation: Cu WaveLength: 1.540598
 2Theta Start= 10 2Theta End= 60

H	K	L	D	2Theta	SinT	SinT ²
0	0	1	5.39148	16.428	0.142873	0.020413
-1	0	0	4.90096	18.086	0.157173	0.024703
1	0	0	4.90096	18.086	0.157173	0.024703
0	1	0	4.8866	18.139	0.157635	0.024849
-1	0	1	3.7806	23.513	0.203751	0.041514
0	1	1	3.62071	24.567	0.212748	0.045262
1	0	1	3.48991	25.503	0.220722	0.048718
-1	1	0	3.46041	25.724	0.222603	0.049552
1	1	0	3.46041	25.724	0.222603	0.049552
-1	1	1	2.99017	29.857	0.25761	0.066363
1	1	1	2.84	31.475	0.271232	0.073567
0	0	2	2.69574	33.207	0.285747	0.081651
-2	0	0	2.45048	36.643	0.314346	0.098813
2	0	0	2.45048	36.643	0.314346	0.098813
-1	0	2	2.44631	36.707	0.314882	0.099151
0	2	0	2.4433	36.754	0.31527	0.099395
0	1	2	2.36039	38.094	0.326344	0.1065
-2	0	1	2.30148	39.108	0.334698	0.112023
1	0	2	2.28586	39.387	0.336984	0.113558
0	2	1	2.22544	40.502	0.346133	0.119808
-2	1	0	2.19049	41.177	0.351656	0.123662
2	1	0	2.19049	41.177	0.351656	0.123662
-1	1	2	2.1875	41.236	0.352136	0.124
-1	2	0	2.18663	41.253	0.352276	0.124098
1	2	0	2.18663	41.253	0.352276	0.124098
2	0	1	2.16638	41.657	0.35557	0.12643
-2	1	1	2.08211	43.426	0.369961	0.136871
1	1	2	2.07052	43.682	0.372031	0.138407
-1	2	1	2.05206	44.096	0.375379	0.140909
1	2	1	2.00153	45.269	0.384855	0.148113
2	1	1	1.98048	45.778	0.388946	0.151279
-2	0	2	1.8903	48.096	0.407501	0.166057
0	2	2	1.81036	50.364	0.425496	0.181046
0	0	3	1.79716	50.76	0.42862	0.183715
-2	1	2	1.76299	51.816	0.436928	0.190906
2	0	2	1.74496	52.392	0.441443	0.194872

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The electronic spectra of Ni(II) complexes observed in the present work show absorption bands in three regions. $V_1 = 11495 \text{ cm}^{-1}$, $V_2 = 16530 - 16130 \text{ cm}^{-1}$ and $V_3 = 26315-25640 \text{ cm}^{-1}$. These observed bands may be assigned to three spin allowed transition ${}^3A_{2g} \rightarrow {}^3T_{2g}$, ${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$

and ${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$ charge transfer band respectively, which are characteristic of the distorted octahedral field.^{18,19} The electronic spectra of Ni(II) complex with L_3 and L_4 ligand are given in Fig. 2 & 3, and their spectral data are presented in Table - 2.

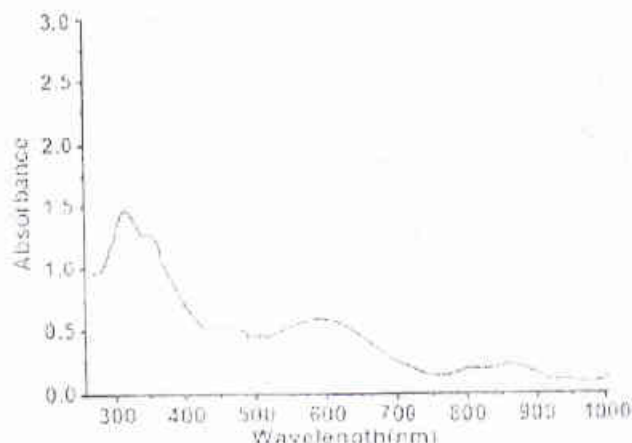


Fig.2 Electronic Spectrum of Ni (II) with L_3

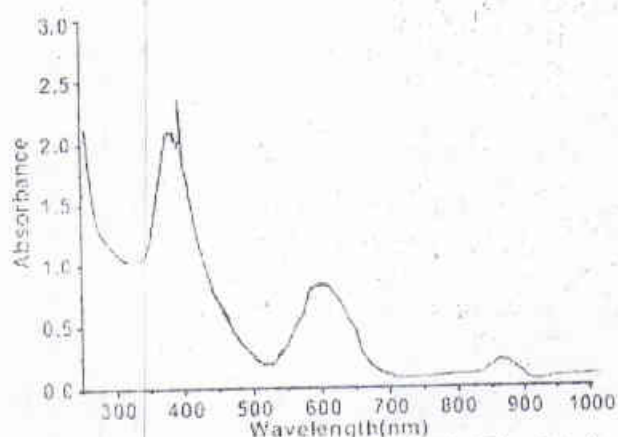


Fig.3 Electronic Spectrum of Ni (II) with L_4

Table No. 2
Electronic Absorption Spectral Data of Ni(II) complexes

Ni (II) Complexes	Absorption Maxima cm^{-1} (nm)		
	V_1	V_2	V_3
4-hydroxy-3-(1-((4-methylpyridin-2-yl)imino)ethyl) quinolin-2(1H)-one (L_3)	11495 (870)	16530 (605)	26315 (380)
3-(1-((5-chloropyridin-2-yl)imino)ethyl)-4-hydroxy quinolin-2(1H)-one (L_4)	11495 (870)	16130 (620)	25640 (390)

The observed magnetic moment values (μ_{eff}) of all the Nickel(II) complexes in the present investigation were found to be in the range 3.11-

3.21 B.M., agreeing to two unpaired electrons.²⁰ Their spectral data are presented in Table -3 while All Hg (II) complexes are diamagnetic.²¹

Table No. 3
Magnetic Susceptibility Data of Ni (II) Complexes at Room Temperature

Ni (II) Complexes of ligand	$\chi_M \times 10^6$ (CGS)	$\chi_A \times 10^6$ (CGS)	μ_{eff} (B.M.)
4-hydroxy-3-(1-(pyridin-2-ylimino)ethyl)quinolin-2(1H)-one(L_1)	3747.52	4052.58	3.11
4-hydroxy-3-(1-(pyridin-3-ylimino)ethyl)quinolin-2(1H)-one (L_2)	3802.36	4131.14	3.14
4-hydroxy-3-(1-(pyridin-4-ylimino)ethyl)quinolin-2(1H)-one(L_3)	3884.56	4237.06	3.18
3-(1-((5-chloropyridin-2-yl)imino)ethyl)-4-hydroxy quinolin-2(1H)-	3979.39	4317.39	3.21

The crystal lattice parameters of the Ni(II), Hg(II) complexes with ligand L_3 were determined by the X-ray diffraction powder method. The X-ray diffraction patterns of complexes were recorded in the 2θ range from 10° to 80° . The data obtained

and reciprocal lattice (h, k, l) are listed in the Table - 4 & 5. Based on the results and the literature support, the complexes with ligands are crystalline due to sharp reflexes shown, Fig.-4 & and have been assigned monoclinic.^{22,23}



Table No. 5XRD Data of Hg (II) Complex of ligand L₃

Crystal system: Monoclinic Lattice Type: P
 Lattice Parameter: a= 5.8990 b= 5.2336 c= 5.7888
 Lattice Parameter: Alpha= 90 Beta= 102 Gama=90
 Radiation: Cu Wave Length: 1.540598
 2Theta Start= 10 2Theta End= 60

H	K	L	d	2Theta	SinT	SinT ²
0	1	1	3.84335	23.124	0.200424	0.04017
1	0	1	3.67725	24.183	0.209477	0.043881
-1	1	1	3.42983	25.957	0.224588	0.05044
1	1	1	3.00881	29.667	0.256015	0.065544
-2	0	0	2.88505	30.971	0.266997	0.071287
-3	1	1	1.82624	49.896	0.421795	0.177911
-3	1	0	1.80531	50.515	0.426685	0.18206
3	1	0	1.80531	50.515	0.426685	0.18206
-1	1	3	1.79875	50.712	0.428242	0.183391
0	1	3	1.7755	51.424	0.433849	0.188225
-3	0	2	1.77133	51.554	0.434872	0.189113
2	2	1	1.76002	51.91	0.437664	0.19155
-2	0	3	1.7555	52.054	0.438792	0.192538
1	2	2	1.75066	52.208	0.440005	0.193605
0	3	0	1.74453	52.406	0.44155	0.194967
2	1	2	1.73469	52.726	0.444055	0.197185
3	0	1	1.71577	53.353	0.448953	0.201559
-2	2	2	1.71491	53.382	0.449176	0.201759
1	0	3	1.69291	54.132	0.455016	0.207039
-3	1	2	1.67783	54.659	0.459104	0.210776
1	3	0	1.66988	54.941	0.46129	0.212788
-1	3	0	1.66988	54.941	0.46129	0.212788
0	3	1	1.6672	55.037	0.462032	0.213473
-2	1	3	1.66436	55.138	0.462819	0.214201
3	1	1	1.63039	56.389	0.472464	0.223222
-1	3	1	1.62849	56.46	0.473015	0.223743
1	1	3	1.61074	57.139	0.478228	0.228702
1	3	1	1.57616	58.513	0.48872	0.238847
-3	2	1	1.56295	59.056	0.492849	0.2429
-3	2	0	1.54977	59.609	0.49704	0.247049
3	2	0	1.54977	59.609	0.49704	0.247049
-1	2	3	1.54561	59.785	0.498377	0.24838

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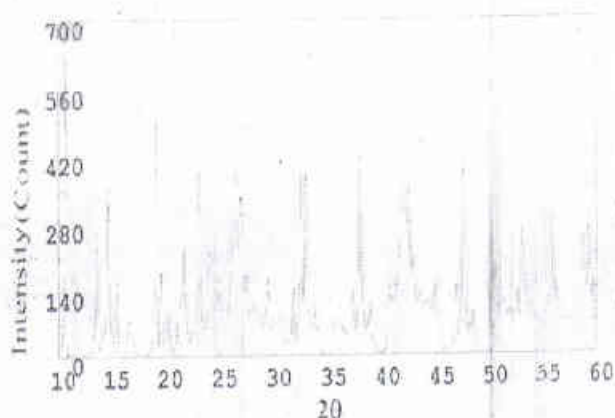


Fig.4 XRD Spectrum of Ni (II) with L₃

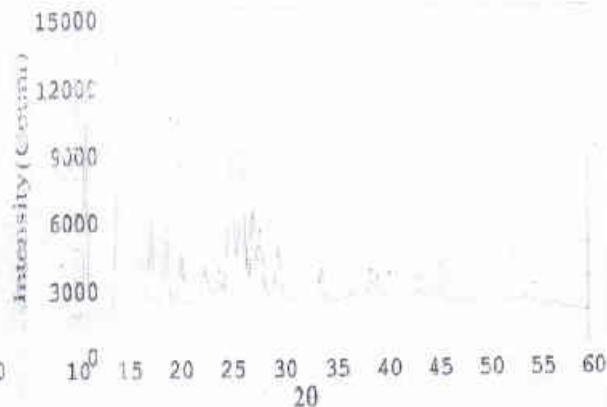


Fig.5 XRD Spectrum of Hg (II) with L₃

in the IR spectra of the corresponding metal complexes are reported in table - 6, medium to weak bands appeared in the region 1595-1586 cm⁻¹ were assigned to C=N stretching vibration mode.²⁴ The medium intensity absorption bands in the region 1278-1240 cm⁻¹ in the spectra of metal complexes were predictable to enolic C-O stretching frequency.²⁵ The band assigned to the lactam C=O stretching frequencies in the

corresponding complexes were observed in the 1666-1652 cm⁻¹. The metal complexes of Ni(II) in the present research show a broad band in the region 3500-2600 cm⁻¹ which is attributed to the coordinated water present in these complexes.²⁶ Such bands are not observed in IR spectra of Hg(II) complexes. However, a peak observed in all the spectra in the range 3431-3249 cm⁻¹ is assigned to -N-H stretching. (Fig. 6-9)

Table No. 6 Infrared Absorption Frequencies (cm⁻¹) of complexes

Sr. No.	Ligand / Complex	Bond vibrational modes (stretching - ν)				New Peaks	
		Lactam (C=O)	Pyridine (C=N)	Azo-methine (C=N)↓	Enolic (C-O)↑	M-O	M-N
1	Ni(L ₁) ₂	1655	1600	1588	1240	483	452
2	Ni(L ₂) ₂	1652	1602	1586	1242	480	454
3	Ni(L ₃) ₂	1656	1601	1587	1243	488	453
4	Ni(L ₄) ₂	1665	1605	1595	1278	498	454
5	Hg(L ₁) ₂	1654	1600	1588	1244	482	447
6	Hg(L ₂) ₂	1652	1600	1589	1245	488	443
7	Hg(L ₃) ₂	1658	1602	1586	1245	482	442
8	Hg(L ₄) ₂	1666	1602	1590	1250	496	442

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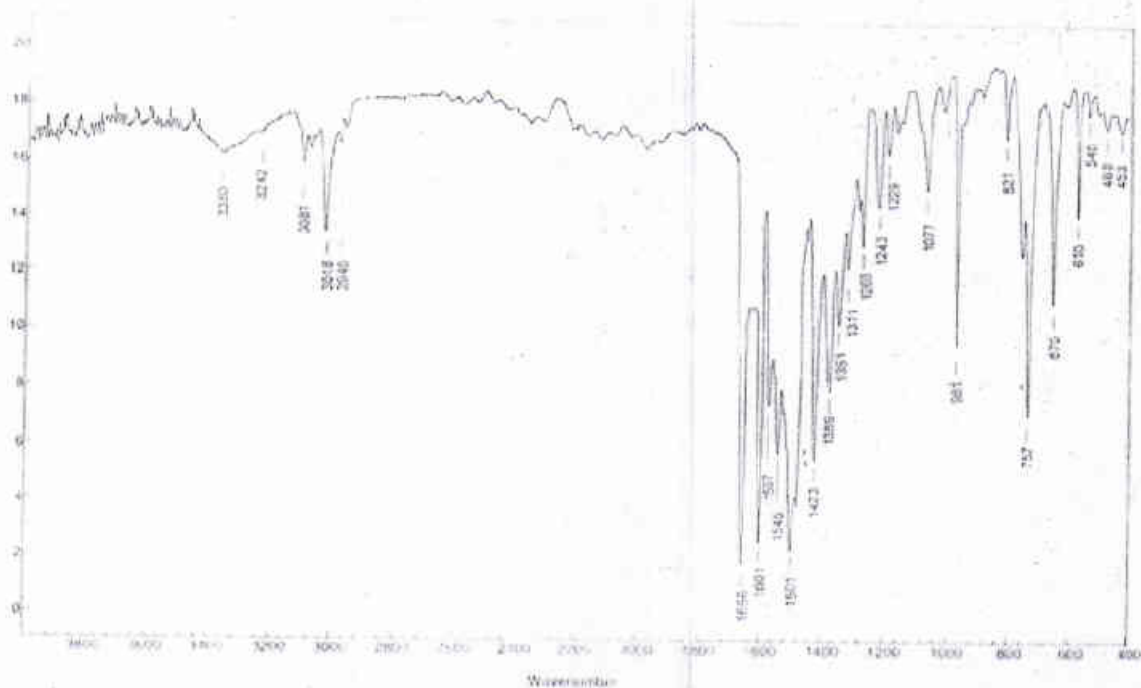


Fig. 6 IR Spectrum of Ni (II) complex with Ligand L₃

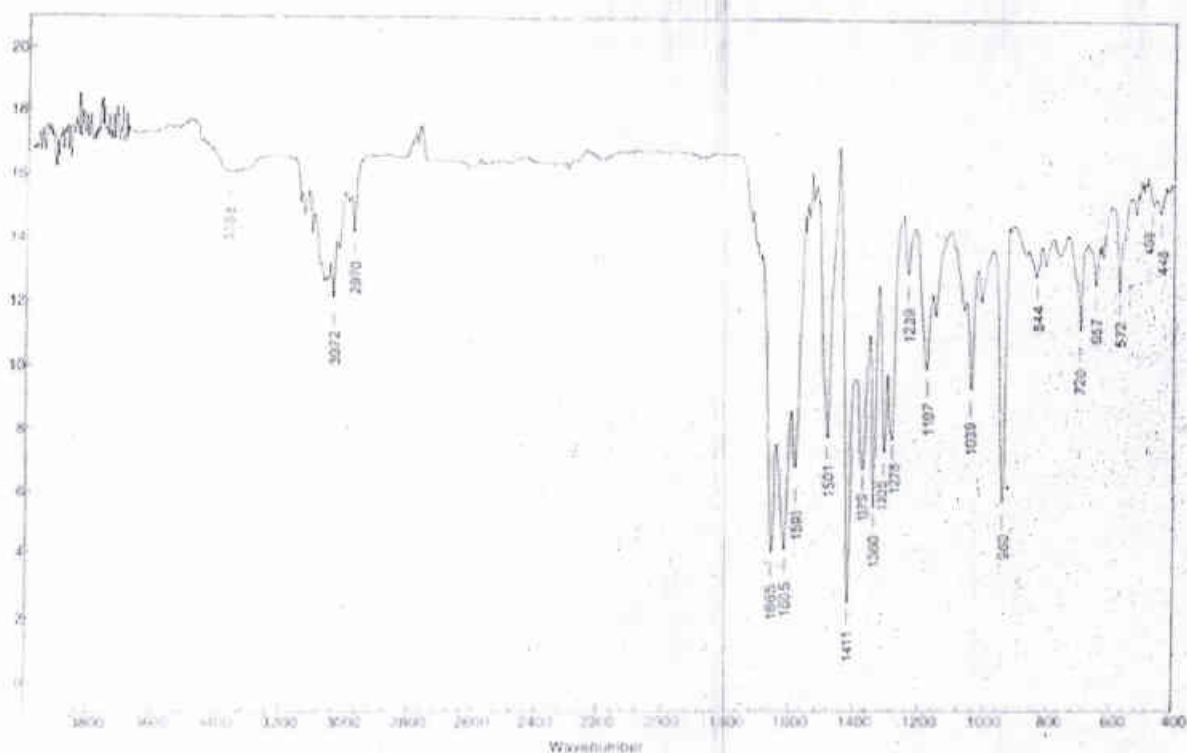


Fig. 7 IR Spectrum of Ni (II) complex with Ligand L₄


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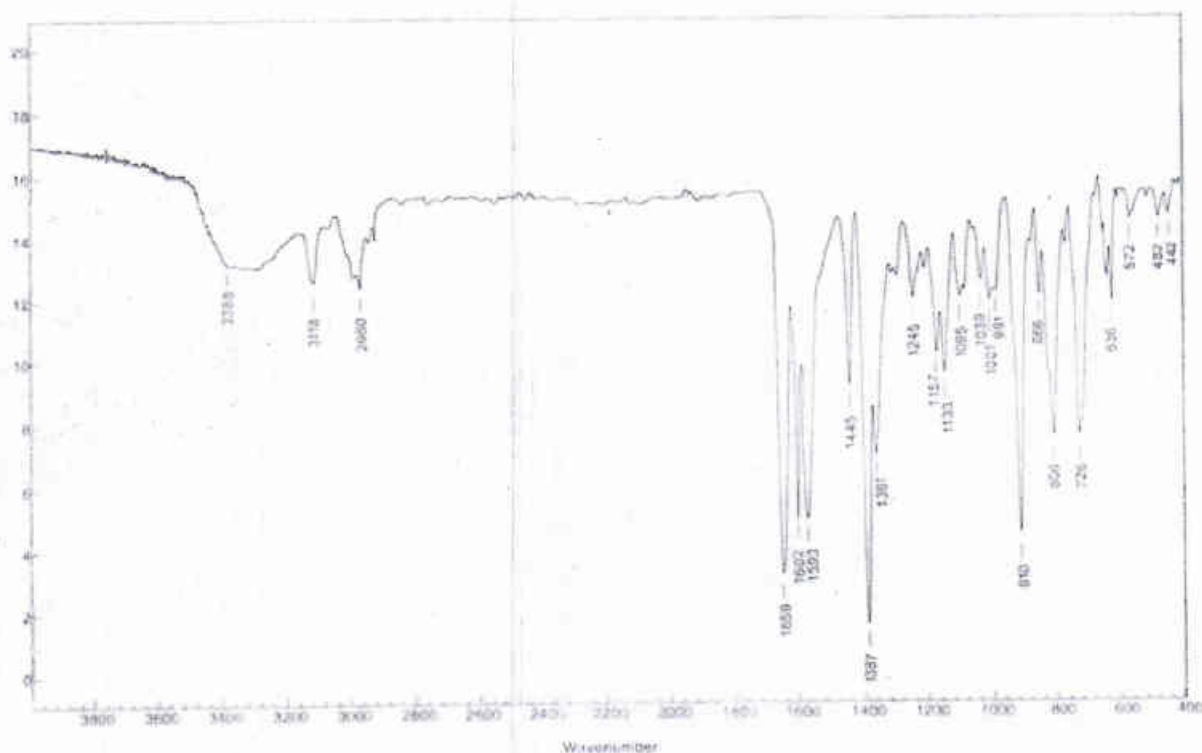


Fig. 8 IR Spectrum of Hg (II) complex with Ligand L₃

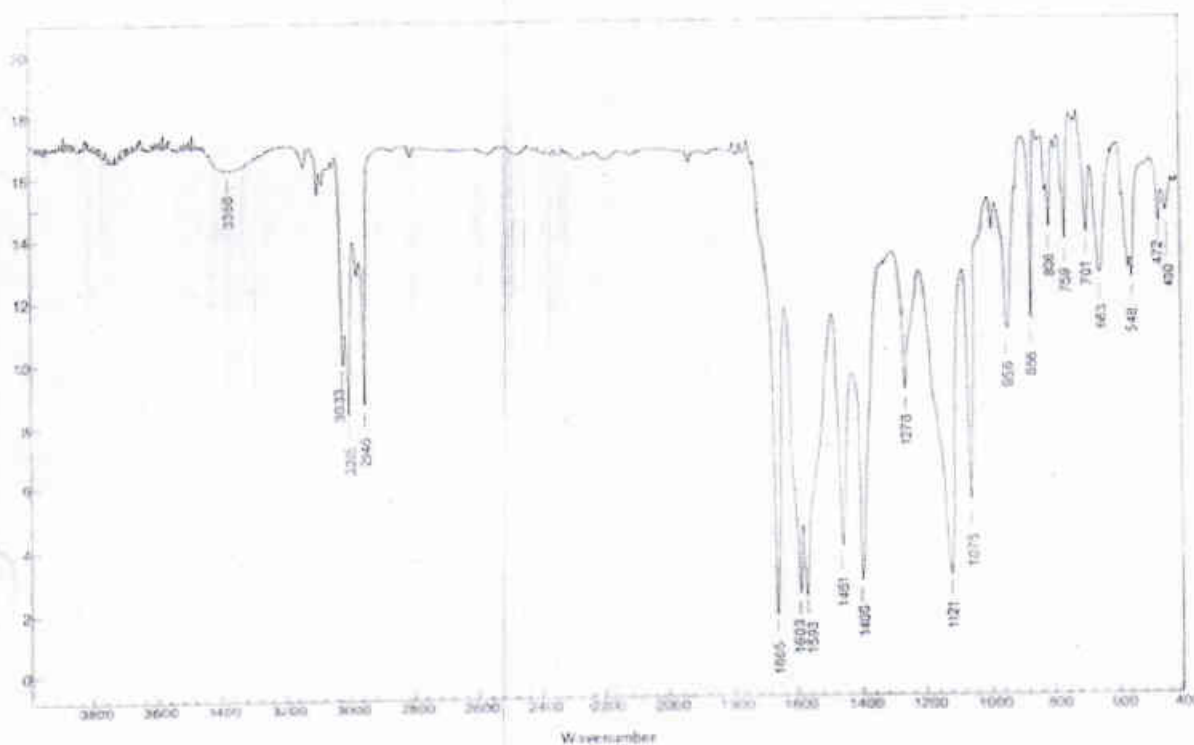


Fig. 9 IR Spectrum of Hg (II) complex with Ligand L₄

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Based on conductivity, elemental and metal analysis, magnetic moment and electronic spectral data of the complexes, it is proposed that the Ni(II) complexes in the present investigation are having an octahedral configuration while Mercury complexes have tetrahedral geometry.

The synthesized complexes of Ni (II) and Hg (II) were subjected for evaluation of antimicrobial activity. The findings are reported in Table 7.

Metal complexes of Ni (II) and Hg (II) have demonstrated good antibacterial activity with all bacterial species in the range of 21-17 mm diameter of zone of inhibition. The antifungal test of metal complexes with all ligands revealed that they exhibit significant activity. The antibacterial and antifungal activity observed in complexes of Ni (II) and Hg (II) with ligands (L₄) is higher which might be due to presence of chlorine atom in the moiety.²⁷

Table No.7 Anti-Bacterial and Anti-Fungal Activity

Synthesized Schiff base ligands	Antibacterial Study				Antifungal Study			
	Zone of Inhibition(diameter in mm)				Growth of Fungi			
	Gram Positive		Gram Negative		<i>A. niger</i>	<i>A. flavus</i>	<i>F. moniliforme</i>	<i>P. chrysogenum</i>
<i>S. typhi</i>	<i>B. subtilis</i>	<i>E. coli</i>	<i>S. aureus</i>					
Ampicillin (Reference)	19	16	18	17	Neomycin (Reference)	-	-	-
Ni(L ₁) ₂	18	18	18	17	++	++	+	++
Ni(L ₂) ₂	19	19	18	17	+	+	++	++
Ni(L ₃) ₂	18	18	17	18	++	+	+	-
Ni(L ₄) ₂	20	20	21	20	-	-	-	-
Hg(L ₁) ₂	17	18	19	18	++	++	++	++
Hg(L ₂) ₂	18	18	18	19	+++	+++	+	+
Hg(L ₃) ₂	18	19	19	20	+	+	+	+
Hg(L ₄) ₂	19	20	20	19	-	+	+	-

Moderate growth (++) , Reduced growth (+) and No growth (-) of fungi

Conclusion

In summary, with the objective to have contribution in coordination chemistry, we demonstrated synthesis, Characterization and antimicrobial activity of metal complexes of Ni (II) and Hg (II) with Schiff bases derived from heterocyclic moieties such as pyridine and quinoline derivatives. The synthesized metal complexes of Ni (II) are monomeric and have an octahedral configuration while Zn (II) are observed to be monomeric with tetrahedral geometry. Antimicrobial study of all complexes revealed that they possess potent antibacterial as well as antifungal activity.

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Co-ordinator
IQAC

Shri Guru Buddhiswami Mahavidyalaya
Purna (Jn) Dist. Parbhani - 431511 (M.S.)



Principal
Shri Guru Buddhiswami Mahavidyalaya
Purna (Jn.) Dist. Parbhani