



# Synthesis, Characterization and Antimicrobial Activity of Cu(II), Co(II) and Ni(II) Complexes with O, N, and S Donor Ligands

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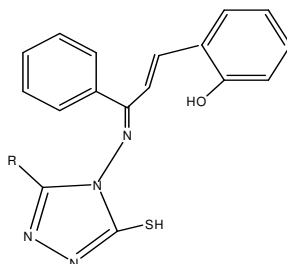
**Abstract:** The complexes of the type  $ML_2$  [where M = Cu(II), Co(II), and Ni(II) ] L = 1-phenyl-1-ene-3-(2-hydroxyphenyl)-prop-2-ene with 3- substituted-5-mercapto-4-amino-1,2,4-triazoles. Schiff base ligands have been prepared by reacting 3-(2-hydroxyphenyl)-1-phenylprop-2-en-1-one and 3-phenyl/pyridyl-4-amino-5-mercapto-1,2,4-triazoles in an alcoholic medium. The complexes are non-electrolytes in DMF. The resulting complexes were characterized by elemental analysis, magnetic measurements, conductivity measurements and spectral studies. The Schiff base acts as a tridentate dibasic and coordinating through the deprotonated oxygen, thioenolic sulphur and azomethine nitrogen atoms. It is found that Cu(II), Co(II), and Ni(II) complexes exhibited octahedral geometry. The antimicrobial activities of ligands and its complexes were screened by cup plate method.

**Keywords:** Chalcones, Triazoles, Metal complexes, Spectral, Magnetic moment and Antimicrobial activity

## Introduction

There is great interest in synthesis and characterization of ligands which contain O, N, S-sequence and their metal complexes. Chalcones were synthesized by the condensation of acetophenones with aromatic aldehyde in the presence of acidic<sup>1</sup> and basic<sup>2</sup> media. Chalcones were found to be pharmacologically and physiologically active<sup>3</sup>. Chalcone derivatives are associated with some important biological activities such as antitubercular, anthelmintic<sup>4</sup>, fungicidal<sup>5</sup>, antitumor<sup>6</sup> and antibacterial activity<sup>7</sup>. The presence of a reactive  $\alpha,\beta$  unsaturated keto in chalcones is found to be responsible for their antimicrobial activity. The tridentate Schiff bases with heterocyclic amines containing O, N, S donor sequence have been tried for complexation with transition metals<sup>8</sup>. The search for a new sequence has resulted by the condensation of chalcone and triazoles. The neutral tridentates are shown to form octahedral complexes with transition metal, where two ligands encompass the metal ion in an octahedral array<sup>9</sup>. In this paper we wish to report the synthesis and characterization of

Cu(II), Co(II) and Ni(II) complexes with the Schiff bases obtained from the condensation of 1-phenyl-1-ene-3-(2-hydroxyphenyl)-prop-2-ene with 3-Substituted-5-mercapto-4-amino-1,2,4-triazoles resulted as shown in Figure 1.



R = Phenyl , pyridine

**Figure 1.** 2-{(1*E*,3*Z*)-3-[(5-mercapto-3-Phenyl/Pyridyl-4*H*-1,2,4-triazol-4-yl)imino]-3-phenylprop-1-en-1-yl}phenol.

## Experimental

All the chemicals are reagent grade. Solvents were dried and distilled before use according to standard procedures<sup>10</sup>. The metal salts used were in their hydrated form.

### *Synthesis of ligands*

One mole of chalcone in 30 mL of ethanol was taken in round bottom flask, to this one mole of 3-phenyl/pyridyl- 5-mercapto-4-amino 1, 2, 4 triazoles were added. The reaction mixture was refluxed for 6-7 hours, evaporated the solvent on water bath and solid separated was collected, the product was washed with absolute alcohol, the product was crystallized from rectified spirit to obtain yellow coloured crystals.

### *Preparation of complex*

Warm ethanolic solutions of metal(II)chloride (0.01M) were added to ethanolic solution of ligands  $H_2L^1$  and  $H_2L^2$ (0.02M) in about 30 mL of ethanol. The resulting solutions were refluxed for about 6 hours. The complex thus formed was filtered and washed with alcohol and dried in vacuum over fused  $CaCl_2$ . The metal estimation was carried out by standard methods, nitrogen by Kjeldahl method and sulphur<sup>11</sup> in the complexes estimated as  $BaSO_4$ . The CHN analyses were carried out by STIC Cochin. The conductance was measured in DMF and DMSO solvent on an Elico CM-82 conductivity bridge. The magnetic susceptibility measurements at room temperature were made on Gouy balance at room temperature using  $HgCo(NCS)_4$  as calibrant. The IR spectra of ligand and its complexes were recorded on a Perkin-Elmer instrument in KBr pellets in the range of  $4000-350\text{ cm}^{-1}$ . UV-Visible spectra were recorded on an Elico SL 164 double beam UV-Visible spectrometer in the range 200-1200nm. <sup>1</sup>HNMR spectra were recorded on an AMX-400 NMR spectrometer using TMS as internal standard and DMSO as a solvent. Electron spin resonance spectra complexes in polycrystalline state were recorded on Varian E-4x-band ESR spectrometer using DPPH free radical as 'g' marker ( $g=2.0027$ ) at room temperature.

## Results and Discussion

The elemental analysis shown in Table 1 indicates that, all the metal complexes have 1:2 stoichiometry and are dark colored amorphous substances, soluble in DMF and DMSO.

The molar conductance values obtained for these complexes at the concentration of  $10^{-3}$  M are in the range of 20-30  $\text{ohm}^{-1} \text{mol}^{-1} \text{cm}^2$ . These values are too low to account for any dissociation of the complexes in DMF. Hence these complexes can be regarded as non-electrolytes.

**Table 1.** Physical, analytical, magnetic susceptibility and molar conductance data of the ligand  $\text{H}_2\text{L}^1$  and  $\text{H}_2\text{L}^2$  and its complexes

Ligand / Complex	Empirical Formula	Mol. Wt.	M.P. $^{\circ}\text{C}$	Yield %	M	C	H	N	S	$\mu_{\text{eff}}$ -M.	Molar Conductance $\wedge_{\text{m}} \text{Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$
$\text{H}_2\text{L}^1$	$\text{C}_{23}\text{H}_{18}\text{N}_4\text{OS}$	398.12	180	70		69.31 (69.32)	4.15 (4.55)	14.01 (14.00)	8.06 (8.05)	-	-
$\text{Cu}(\text{L}^1)_2$	$\text{C}_{46}\text{H}_{32}\text{N}_8\text{O}_2\text{CuS}_2$	855.14	295	70	7.41 (7.42)	64.50 (64.51)	3.41 (3.77)	13.10 (13.08)	7.44 (7.41)	1.6	17.15
$\text{Co}(\text{L}^1)_2$	$\text{C}_{46}\text{H}_{32}\text{N}_8\text{O}_2\text{CoS}_2$	851.14	280	65	7.00 (6.98)	64.84 (64.86)	3.42 (3.79)	13.17 (13.15)	7.54 (7.53)	5.05	17.38
$\text{Ni}(\text{L}^1)_2$	$\text{C}_{46}\text{H}_{32}\text{N}_8\text{O}_2\text{NiS}_2$	850.14	275	68	6.90 (6.89)	64.85 (64.88)	3.72 (3.79)	13.15 (13.16)	7.51 (7.53)	2.8	19.89
$\text{H}_2\text{L}^2$	$\text{C}_{22}\text{H}_{17}\text{N}_4\text{O}_2\text{S}$	399.46	242	60	-	67.52 (67.54)	5.92 (5.89)	15.73 (15.75)	7.25 (7.21)	-	15.13
$\text{Cu}(\text{L}^2)_2$	$\text{C}_{44}\text{H}_{30}\text{N}_{10}\text{O}_2\text{CuS}_2$	857.17	265	65	7.41 (7.42)	53.41 (61.56)	3.56 (3.52)	16.35 (16.32)	6.49 (7.47)	1.85	18.26
$\text{Co}(\text{L}^2)_2$	$\text{C}_{44}\text{H}_{30}\text{N}_{10}\text{O}_2\text{CoS}_2$	853.10	272	75	6.93 (6.92)	61.90 (61.89)	3.53 (3.54)	16.41 (16.40)	7.52 (7.51)	5.14	19.00
$\text{Ni}(\text{L}^2)_2$	$\text{C}_{44}\text{H}_{30}\text{N}_{10}\text{O}_2\text{NiS}_2$	852.10	293	70	6.866 (6.88)	61.90 (61.92)	3.52 (3.54)	16.43 (16.41)	14.01 (7.51)	3.20	11.10

### Magnetic moment

The magnetic moment values for Cu(II), Co(II) and Ni(II) complexes of the ligands  $\text{H}_2\text{L}^1$  and  $\text{H}_2\text{L}^2$  are shown in Table 1. Co(II) complexes are in the range of 5.05-5.14 BM indicating that the Co(II) complexes are typically high spin complexes and having octahedral structure. The Ni(II) complexes exhibit the magnetic moment values in the range 2.8-3.2 BM, indicating octahedral co-ordination of the ligands around Ni(II) ion. The Cu(II) complexes exhibit magnetic moment in the range of 1.61-1.85 BM suggestive of distorted octahedral nature for these complexes<sup>12</sup>.

### Electronic spectra

The electronic spectral data of Cu(II), Co(II) and Ni(II) complexes of the ligands  $\text{H}_2\text{L}^1$  and  $\text{H}_2\text{L}^2$  were recorded in DMF as shown in Table 2. The electronic spectra of Co(II) complexes exhibit bands in the region of 8895 -21000  $\text{cm}^{-1}$  and 15000 -18000  $\text{cm}^{-1}$  are attributed to  ${}^4\text{T}_{1g} \rightarrow {}^4\text{A}_{2g}(\text{F})$  ( $\nu_1$ ) and  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{p})$  ( $\nu_3$ ) transitions respectively. The bands due to the  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$  ( $\nu_2$ ) transition could not be observed because of its very low intensity. However the position of the  $\nu_2$  band has been computed (15549  $\text{cm}^{-1}$ ) by the equation  $\nu_2 = \nu_1 + 10Dq$ . The Co(II) complex under present investigation possesses interelectronic repulsion parameter (B) 749  $\text{cm}^{-1}$ . The Racah parameter (B) is less than free ion value (971) suggesting a considerable orbital overlap and delocalization of electrons on the metal ion. The nephelauxetic ratio ( $\beta$ ) for the present Co(II) complex (0.77). This is less than one, suggesting partial covalency in the metal ligand bond. The values Dq,  $\beta$  %, LFSE and  $\nu_2/\nu_1$  (Table 2) suggest the octahedral geometry for Co(II) complex.<sup>13,14</sup> These observations suggest that Co(II) complexes have octahedral geometry. Ni(II) complexes exhibit three bands at 8990-13810  $\text{cm}^{-1}$ , 16088-16335  $\text{cm}^{-1}$  and 24964-25210  $\text{cm}^{-1}$  are

assigned to  ${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$  ( $\nu_1$ ),  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$  ( $\nu_2$ ) and  ${}^4A_{2g}(F) \rightarrow {}^3T_{1g}(p)$  ( $\nu_3$ ) transitions respectively are in confirmatory with the octahedral geometry for the Ni(II) ion. The Table 2 shows the ligand field parameter such as Dq, B,  $\beta$ ,  $\beta\%$  and LFSE have been calculated by using Band-fitting equation given by Underhill and Billing<sup>15</sup>. Racah parameter B is less than the free ion value of 1040  $\text{cm}^{-1}$  indicating the covalent character of the complex. The ratio  $\nu_2/\nu_1$  and  $\beta\%$  are further support the octahedral geometry around the Ni(II) ion<sup>16</sup>. The Cu(II) complexes exhibits three bands in the region 11330 $\text{cm}^{-1}$  ( $\nu_1$ ), 17300  $\text{cm}^{-1}$  ( $\nu_2$ ) and 19500-21000  $\text{cm}^{-1}$  ( $\nu_3$ ) are of equal energy and giving rise to single broad band which may be assigned to the transitions  ${}^2B_{1g}v \rightarrow {}^2A_{1g}(\nu_1)$ ,  ${}^2B_{1g} \rightarrow {}^2B_{2g}(\nu_2)$ ,  ${}^2B_{1g} \rightarrow {}^2E_g(\nu_3)$  respectively. The broadness of the band is due to the ligand field and the John-Teller effect<sup>17</sup>. These observations favor the octahedral geometry for the Cu(II) ion.

**Table 2.** Electronic spectral data and ligand field parameters of Cu(II), Co(II) and Ni(II) complexes

Complexes	$\nu_1$	$\nu_2$	$\nu_3$	Dq ( $\text{cm}^{-1}$ )	B <sup>1</sup>	$\beta$	$\beta\%$	$\nu_2/\nu_1$	$\nu_3/\nu_2$	LFSE kcal $\text{mol}^{-1}$
Cu ( $L^1$ ) <sub>2</sub>	-	17300	21000	1413	-	-	-	-	-	24.50
Co ( $L^1$ ) <sub>2</sub>	8892	15439	20085	1002	749	0.778	23.03	1.7	2.1	17.17
Ni ( $L^1$ ) <sub>2</sub>	13810	15151	25316	899	865	0.820	18.00	1.4	1.8	30.82

### Infrared spectra

The important infrared frequencies exhibited by the ligands  $H_2L^1$  and  $H_2L^2$  and its complexes are given in Table 3. The ligand molecule exists in both thiol and thione forms due to tautomerism. A medium intensity band around 2560 $\text{cm}^{-1}$  due to  $\nu(S-H)$  indicates the thiol form of the ligand. A comparison of IR spectra of ligand with those of complexes is based on earlier studies of similar ligand<sup>18</sup>. The ligand molecule shows a band at 700-820 due to  $\nu(C=S)$ . The coordination *via* thioketo sulphur atom causes the decrease in frequency of the  $\nu(C=S)$ . The complexes show a new band at 650-700 $\text{cm}^{-1}$  due to conversion of  $\nu C=S$  into C-S- band indicates the thione  $\leftrightarrow$  thiole tautomerism followed by deprotonation of thiol group and consequent coordination of sulphur atom<sup>19, 20</sup> and is indicated by absence of band at 2560 (due to SH) in the spectra of complexes. The infrared spectra of ligands exhibit high intensity band around 1612  $\text{cm}^{-1}$  is due to  $\nu(C=N)$  and the band around 3450  $\text{cm}^{-1}$  due to phenolic OH. In the complexes, the low frequency shift (15-20 $\text{cm}^{-1}$ ) are observed around 1594 $\text{cm}^{-1}$  are due to  $\nu(C=N)$  is suggestive of coordination through nitrogen of the azomethine group<sup>21</sup> to the metal(II) Chlorides. The band around 3450  $\text{cm}^{-1}$  due to phenolic OH, which is observed in ligands, disappears in complexes, this indicates the ligands co-ordinate to the metal ion through phenolic oxygen atom<sup>21</sup> of OH group *via* deprotonation. The appearance of three new bands 550-520 $\text{cm}^{-1}$ , 450-420 $\text{cm}^{-1}$ , 380-415 $\text{cm}^{-1}$  are due to M-N, M-S and M-O bonds respectively.

### <sup>1</sup>H NMR spectra

The <sup>1</sup>H NMR spectrums of ligands  $H_2L^1$  and  $H_2L^2$  and its complexes were recorded in DMSO  $d_6$  in the range 0-15 $\delta$  (ppm). The multiplets were observed around  $\delta$ 6.9-8.6 (m, 14, Ar-H) are due to phenyl protons. The protons due to CH=CH are observed around  $\delta$  7.4-7.8 (d 2H). In the ligand  $H_2L^2$  Signals due to pyridine ring protons occurs as multiplets between  $\delta$ 8.03-9.10 (m, 4H Pyridine-H). A proton due to -OH group at 2-position of salicylaldehyde ring has resonated as a singlet at  $\delta$ 11.20 and  $\delta$ 11.34 (S, 1H, OH) respectively. Signals in the range of  $\delta$  9.6 and 9.5 (S 1H, SH) are due to SH protons<sup>18</sup>. In the <sup>1</sup>H NMR spectrum of Cd ( $L^1$ ) and Zn ( $L^2$ ) complexes, the OH moiety of salicylaldehyde and SH which was observed in the ligands disappears in case of complexes indicating the involvement of phenolic oxygen<sup>18</sup> and sulphur in the coordination *via* deprotonation.

**Table 3.** Characteristic IR bands ( $\text{cm}^{-1}$ ) of the ligand  $\text{H}_2\text{L}^1$  &  $\text{H}_2\text{L}^2$  and its complexes

Ligand/ Complexes	$\nu_{\text{OH}}$	$\nu_{\text{SH}}$	$\nu_{\text{C=S}}$	$\nu_{\text{C=N}}$	$\nu_{\text{C-O}}$	$\nu_{\text{M-O}}$	$\nu_{\text{M-N}}$	$\nu_{\text{M-S}}$
$\text{H}_2\text{L}^1$	3450	2560	780	1612	1255	-	-	-
$\text{Cu}(\text{L}^1)_2$	-	-	720	1560	1258	380	527	420
$\text{Co}(\text{L}^1)_2$	-	-	732	1594	1262	385	523	427
$\text{Ni}(\text{L}^1)_2$	-	-	738	1583	1267	389	529	432
$\text{H}_2\text{L}^2$	3360	2570	770	1610	1260	-	-	-
$\text{Cu}(\text{L}^2)_2$	-	-	726	1570	1272	392	535	439
$\text{Co}(\text{L}^2)_2$	-	-	729	1597	1278	395	542	445
$\text{Ni}(\text{L}^2)_2$	-	-	720	1567	1285	415	555	450

### ESR spectra

The ESR spectra of the polycrystalline sample of the complexes were recorded at room temperature (Table 4). The observed value for the Cu(II) complex for the ligand  $\text{H}_1\text{L}^1$  are  $g_{\parallel} = 2.22$ ,  $g_{\perp} = 2.05$  and  $G = 4.4$ , while for  $\text{H}_2\text{L}^2$   $g_{\parallel} = 2.23$ ,  $g_{\perp} = 2.05$  and  $G = 4.6$ . The anisotropic G values have been calculated by Kneubuhl's method<sup>22</sup>.  $G = (g_{\parallel} - 2) / (g_{\perp} - 2)$  which measures the exchange interaction between copper(II) centers. According to Hathaway<sup>23</sup> if the G value is greater than 4, the exchange interaction is negligible, while a value of less than 4 indicates a considerable exchange interaction in the complexes. As  $G = 4.4$  and  $4.6$  for the present complexes indicates that there is no spin exchange interaction in the copper complexes and hence distorted octahedral geometry proposed for the Cu(II) complex.

**Table 4.** ESR data of the Cu (II) complex of the ligands  $\text{H}_2\text{L}^1$  and  $\text{H}_2\text{L}^2$ 

Complexes	ESR spectral data			
	$g_{\perp}$	$g_{\parallel}$	$g_{\text{av}}$	G
$\text{Cu}(\text{L}^1)_2$	2.22	2.05	2.053	4.4
$\text{Cu}(\text{L}^2)_2$	2.23	2.04	2.054	4.6

### Antimicrobial activity

The biological and medicinal potency of coordination compounds has been established by their antitumor, antiviral and antimalarial activities. This characteristic property has been related to the ability of the metal ion to form complexes<sup>24</sup> with ligand containing sulfur, nitrogen and oxygen donor atoms. The synthesized ligands and its complexes were screened for their antibacterial activity<sup>25</sup> against *E.coli* and *S.auriosus* and antifungal activity against<sup>26</sup> *A.niger* and *A.flavous* at  $100 \mu\text{g}/0.1 \text{ cm}^3$  concentration. The standard drugs streptomycin and chlotrimazole were also tested for their antibacterial and antifungal activity at the same concentration under the conditions similar to that of the test compounds concentration. The zones of inhibitions of the antimicrobial activity have been presented in Table 5. The results of antibacterial activity of ligands and its complexes Cu(II), Co(II) and Ni(II) shows weak activity against *E.coli* and *S.auriosus* when compared with standard streptomycin. The antifungal activity results revealed that the ligands and its Cu(II), Co(II) and Ni(II), complexes have exhibited weak to good activity against *A.niger* and *A.flavous*. The ligand and its Cu(II) and Co(II) complexes shows weak activity when compared to the standard drug chlotrimazole.

**Table 5.** Antimicrobial activity of the ligands H<sub>2</sub>L<sup>1</sup> & H<sub>2</sub>L<sup>2</sup> and its complexes

Compound	Antibacterial Activity of zone of inhibition, mm		Antifungal Activity of zone of inhibition, mm	
	<i>E. coli</i>	<i>S. aureus</i>	<i>A. niger</i>	<i>A. falvours</i>
Ligand H <sub>2</sub> L <sup>1</sup>	14	14	12	13
Cu (L <sup>1</sup> ) <sub>2</sub>	17	13	13	12
Co (L <sup>1</sup> ) <sub>2</sub>	15	12	11	14
Ni (L <sup>1</sup> ) <sub>2</sub>	16	11	18	17
Ligand H <sub>2</sub> L <sup>2</sup>	15	12	13	15
Cu (L <sup>1</sup> ) <sub>2</sub>	18	16	16	14
Co (L <sup>1</sup> ) <sub>2</sub>	17	12	12	18
Ni (L <sup>1</sup> ) <sub>2</sub>	12	13	15	16
Streptomycin	24	21	-	-
Chlorometazole	-	-	24	23
DMF (Control)	0	0	0	0
Bore size	08	08	08	08

## Conclusion

The elemental analysis, magnetic susceptibility, electronic, IR, <sup>1</sup>H NMR and ESR spectral observations suggest the octahedral geometry for the Cu(II), Co(II) and Ni(II) complexes and exhibit coordination number six.

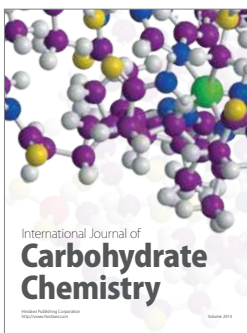
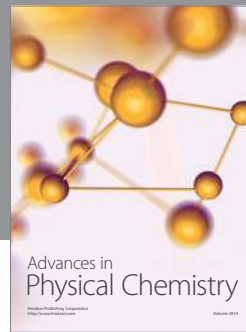
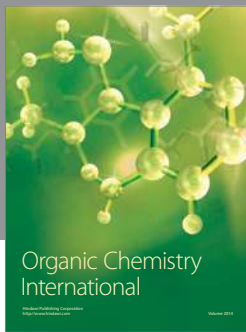
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