

# SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL APPLICATIONS OF SCHIFF BASE COMPLEXES DERIVED FROM SOME NEW AMINO SUBSTITUTED 1, 2, 4-TRIAZOLE DERIVATIVES

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## ABSTRACT

A series of transition metal ion complexes with new amino triazole ligands such as 4-salicylidineamino-3-(4-chlorophenoxy methyl)-5-phenylamino-1,2,4-triazole **HL<sub>1</sub>**, 4-salicylidene amino-3-(4-bromo phenoxy methyl)-5-phenyl amino-1,2,4-triazole **HL<sub>2</sub>** and 4-(4-bromosalicylidene amino-3-(4-bromo phenoxy methyl)-5-phenyl amino-1,2,4-triazole **HL<sub>3</sub>** have been synthesized and characterized by analytical, magnetic, electronic, infrared, <sup>1</sup>H NMR and ESR spectral studies. The octahedral structure with coordination number six has been proposed for all the metal complexes of **HL<sub>1</sub>**, **HL<sub>2</sub>** and **HL<sub>3</sub>** ligands. Antibacterial and antifungal activities of all the synthesized ligands and their metal complexes have also been studied and it is found that the metal complexes of all the ligands show moderate to high activity against tested organisms than the corresponding ligand.

**Keywords:** Amino triazole, derivatives, octahedral, moderate, antibacterial and antifungal.

## INTRODUCTION

There is a growing interest in the studies on the metal complexes of Schiff bases derived from triazoles and its derivatives which are biologically important ligands. Schiff base metal complexes have been widely studied because of their industrial and biological applications. Several derivatives of these have been used as drugs. The triazole Schiff bases constitute one of the most important classes of O, N and S donor atoms. Triazoles and their derivatives have been proved effective bactericides, pesticides, fungicides and insecticides<sup>1-4</sup>.

Compounds containing triazoles have attracted much interest because of their biological applications<sup>5</sup>. Furthermore, triazoles appear frequently in the structures of various natural products<sup>6</sup>. Triazole containing compounds appear in many metabolic products of fungi and primitive marine animals. Many triazoles having different functionalities are used as dyes and as photographic chemicals<sup>7</sup>. The coordination chemistry of triazole and benzotriazole

derivatives is studied due to their importance in industry, agriculture and their biological activity.

The present work reports the synthesis and characterization of Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes with triazole ligands such as 4-salicylidineamino-3-(4-chlorophenoxy methyl)-5-phenylamino-1,2,4-triazole (**HL<sub>1</sub>**), 4-salicylidene amino-3-(4-bromophenoxy methyl)-5-phenyl amino-1,2,4-triazole (**HL<sub>2</sub>**) and 4-(4-bromosalicylidene amino-3-(4-bromophenoxy methyl)-5-phenyl amino-1,2,4-triazole (**HL<sub>3</sub>**).

## EXPERIMENTAL

The chemicals used were all of Analytical reagent grade or chemically pure grade. 4-Chlorophenol and 4-Bromophenol purchased were used as such. While Salicylaldehyde and hydrazine hydrate were distilled before use. The metal salts purchased from commercial sources were in their hydrated form and used as such.

### Synthesis of ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub>

The ligands, 4-Salicylideneamino-3-(4-substituted phenoxyethyl)-5-substituted amino-1, 2, 4-triazoles (HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub>) were synthesized by refluxing a suspension of p-substituted phenoxy acetyl thiosemicarbazide (0.1 mol) and hydrazine hydrate (10 ml, 99 %) for one hour. The solution was cooled and poured into crushed ice (100 gm) with stirring. The reaction mixture was acidified with dilute acetic acid and the precipitated solid was collected by filtration. The product was washed with ethanol, followed by cold water and recrystallised from aqueous ethanol. Further it was refluxed with salicylaldehyde or p-bromosalicylaldehyde in 150 ml absolute alcohol containing one drop of concentrated hydrochloric acid. The excess solvent was vaporized on water bath. The solid separated was filtered, dried and recrystallised.

### Synthesis of Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) Complexes

All Metal complexes were prepared by adding an alcoholic solution of metal (II) chloride (0.1 mol in 20 ml) to 0.1 mol (1.55/1.78 gm) ligand (HL<sub>1</sub>, HL<sub>2</sub> or HL<sub>3</sub>) in 30 ml alcohol. The reaction mixture was refluxed on water bath for about four hrs. on partial removal of the solvent (50 %) and cooling to room temperature, the

separated colored solid complexes were filtered out, washed with ethyl alcohol and dried in hot air oven.

### Analysis and spectral measurements

The elemental analysis of the complexes for metal, nitrogen and chloride were carried out according to the standard methods. The metal contents in the complexes were determined gravimetrically by using standard procedure<sup>8</sup>. The molar Conductance of the synthesized complexes in  $1 \times 10^{-3}$  mol dm<sup>-3</sup> DMF solution were measured using ELICO Digital Conductivity meter of CM-180 model. Magnetic susceptibilities of all the complexes were determined at room temperature with a Gouy balance<sup>9,10</sup>. Electronic spectra of the complexes in DMF Solution were recorded using EV 300, UV-Visible Spectrophotometer. The IR spectra of the ligands and complexes were obtained on a Nicolet impact 410, FTIR spectrophotometer in the region 4000-200 cm<sup>-1</sup> from Sipra Lab Hyderabad. The proton Nuclear magnetic resonance spectra of few ligands and their metal complexes were obtained from Sipra Lab Hyderabad and IISc. Bangalore. The electron spin resonance spectra of the synthesized copper complexes were obtained from Central University Hyderabad.

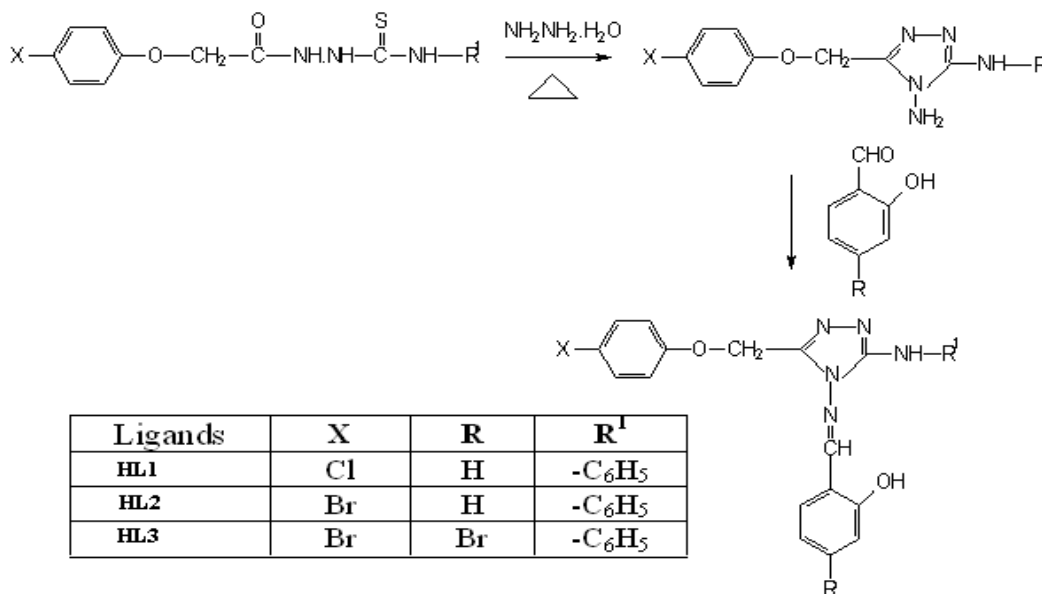


Fig. 1: Scheme of Synthesis of Schiff base ligands

## ANTIMICROBIAL ACTIVITY

The antimicrobial activity of all the ligands and their metal complexes were determined by agar cup-plate method<sup>11</sup>. The antibacterial activity against *Escherichia coli* and *P. Aerginosa* and antifungal activity against *Aspergillus niger* and *C. Albicans*, were screened by the ligands and their metal complexes. The medium was prepared as per the instructions of the manufacturer of dry Mueller Hinton agar powder (Hi-Media). The test ligands and their metal complexes were dissolved in DMSO at a concentration of 1 mg/ml. Ciprofloxacin (100µg/ml) in DMSO was used as a standard for antibacterial and Flucanazole (100µg/ml) in DMSO was used as reference standard for antifungal activity. The solvent control (only DMSO) was also maintained throughout the experiment. The zones of inhibition are reported in **Table-5.5**.

## RESULT AND DISCUSSION

### Nature and Stoichiometry

The elemental analysis of the complexes is given in the **Table-1(a-c)**. Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes are yellow to brown in color. All the complexes are insoluble in common organic solvents. However they are soluble in coordinating solvents like DMF and DMSO. Because of their insolubility in the common solvents molecular weights could not be determined. The results of elements analysis<sup>12</sup> [**Table-1(a-c)**] reveal that the Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes of the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> have 1:2 stoichiometry of the type M(L)<sub>2</sub>.

### Molar Conductance

The conductance measurements were carried out in DMF solution (1x10<sup>-3</sup> M) and the measured molar conductance values for the present complexes are too low to account for any dissociation of the complexes in DMF. Hence the complexes can be regarded as non-electrolytes<sup>13-15</sup> [**Table-5.1(a-c)**].

### Magnetic Property

The magnetic moments obtained at room temperature for the complexes are listed in **Table-5.1(a-c)**.

#### i) Cobalt (II) Complexes

In octahedral Co(II) complexes the ground state is <sup>4</sup>T<sub>1g</sub> and a large orbital contribution to the magnetic moment is expected. The mixing of the singlet states lowers the magnetic moments. The reported magnetic moment values for various Co(II) complexes are in the range of 4.12-4.7 B.M. and 4.7-5.2 for tetrahedral and octahedral respectively<sup>16-19</sup>. In the present investigation the

observed magnetic moment values for Co(II) complexes of HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> fall in the range of 4.46-5.33 B.M. which indicates octahedral geometry for these Co(II) complexes<sup>20-21</sup>.

#### ii) Nickel(II) Complexes

Nickel(II) is a d<sup>8</sup> ion having two unpaired electrons can form paramagnetic complexes having six coordinated octahedral geometry<sup>22</sup>. The magnetic moment reported for octahedral Ni(II) complexes range from 2.82-3.4 B.M., depending on the magnitude of the orbital contribution<sup>23-26</sup>. In the present study, the observed room temperature magnetic moments for the Ni(II) complexes of HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> are in the range of 3.10-3.24 B.M. This observation suggests that Ni(II) has an octahedral configuration in the complexes.

#### iii) Copper(II) Complexes

The Copper(II) complex with the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> exhibits magnetic moment values in the range of 1.76-1.91 B.M.<sup>27</sup>. These values suggest that there is no major coupling interaction in the complexes. These values are agreeable to spin only value. Hence observed magnetic moments for the Cu(II) complexes under study indicates octahedral configurations.

### Electronic Spectra

The electronic spectra of Co(II), Ni(II) and Cu(II) complexes with the ligand 4-salicylidineamino-3-(4-chlorophenoxy methyl)-5-phenylamino-1,2,4-triazole (HL<sub>1</sub>), 4-salicylidene amino-3-(4-bromo phenoxy methyl)-5-phenyl amino-1,2,4-triazole (HL<sub>2</sub>) and 4-(4-bromosalicylidene amino-3-(4-bromo phenoxy methyl)-5-phenyl amino-1,2,4-triazole (HL<sub>3</sub>)) have been studied to obtain more information on stereochemistry of the complexes and to produce more support for the conclusion deduced with the help of magnetic data. The electronic spectra are presented in the **Fig. 5.1(a-d)** and the spectral band maxima of the complexes under study are listed in **Table -5.2**.

#### i) Cobalt(II) Complexes

Cobalt(II) is a d<sup>7</sup> ion exists both in octahedral and tetrahedral geometry. In octahedral geometry Co(II) complexes, three spin allowed transitions are expected corresponding to the transitions, <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>2g</sub>(F), <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>2g</sub>(F) and <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>1g</sub>(P)

In the present investigation, the electronic spectra of Co(II) complexes of the ligands HL<sub>7</sub>, HL<sub>8</sub> and HL<sub>9</sub> exhibited bands in the region 9132-10214cm<sup>-1</sup>, 16528.92-17152 cm<sup>-1</sup> and 19531-21367 cm<sup>-1</sup> due to <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>2g</sub>(F) (ν<sub>1</sub>), <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>A<sub>2g</sub>(F) (ν<sub>2</sub>) and <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>1g</sub>(P)

( $\nu_3$ ), transitions respectively (**Fig. 5.1a**). These transitions suggest octahedral geometry for Co(II) complexes. The assigned transitions are further supported by earlier reports<sup>28-30</sup>.

### ii) Nickel (II) Complexes

Nickel(II) complexes are octahedral on the basis of magnetic data. The electronic spectra of octahedral Ni(II) complexes exhibit three spin allowed d-d transitions. They are designated as<sup>31-33</sup>.  ${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$  ( $\nu_1$ ),  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$  ( $\nu_2$ ) and  ${}^4A_{2g}(F) \rightarrow {}^3T_{1g}(P)$  ( $\nu_3$ ). The prepared paramagnetic Ni(II) complexes of the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> exhibit three bands in the region 10131-11148 cm<sup>-1</sup>, 12787.72-14025 cm<sup>-1</sup> and 21505-22727 cm<sup>-1</sup> respectively in DMSO solution. These bands 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. Moreover, the ratio  $\nu_2/\nu_1$  is in the range of 1.25-1.38 is indicative of octahedral stereochemistry for these Ni(II) complexes. Koji et al.<sup>34</sup> have reported a band around 950 nm for d-d component of the Ni(II) complex. In the present case band in the region 10135-111428 cm<sup>-1</sup> is attributed to a d-d component of the Ni(II) complexes. All these observations favor octahedral geometry for Ni(II) complexes.

### iii) Copper(II) Complexes

The band maxima with their assignments of Copper(II) Complex with the ligand HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> are tabulated in **Table-5.2**. The Cu(II) is a d<sup>9</sup> ion and provides a good example for John Teller effect. In the present investigation, three bands are observed in the range of 13800-14814.81 cm<sup>-1</sup>, 18181.81-20000 cm<sup>-1</sup> and 28000-32467.53 cm<sup>-1</sup> which may be assigned to the transitions of  ${}^2B_{1g} \rightarrow {}^2A_{1g}$ ,  ${}^2B_{1g} \rightarrow {}^2B_{2g}$  and  ${}^2B_{1g} \rightarrow {}^2E_{1g}$  respectively. These observations indicate that the Cu(II) Complex may possess probably six coordinated octahedral configuration.

### Infrared Spectra

The infrared spectra of the ligands (HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub>) and their complexes are expected to be rather complex and so unambiguous assignments and identification of the bands is difficult. This is due to the overlapping frequencies of the phenyl groups, -NH<sub>2</sub> group and the phenolic C-O bands. So semi quantitative studies in IR spectral analysis are needed to determine the gain or loss in intensity of bands so as to deduce shifts in positions of some of the bands due to complexation of the ligand units with metal ions.

But the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> contain secondary amine. The  $\nu(N-H)$  stretching frequencies of these ligands are observed

between 3450-3250 cm<sup>-1</sup>. These bands are shifted to higher energy in the complexes of the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub>, which indicates that the nitrogen of secondary amine is involved in the complex formation in these ligands

The IR spectra of the ligands show strong absorption between 3200-3000 cm<sup>-1</sup>, which can be attributed to hydrogen bonded -OH group<sup>35</sup>. The  $\nu(O-H)$  band of the ligand disappears in the complexes, suggesting that the -OH group is involved in the complex formation via deprotonation.

The strong absorption in the region 1615-1632 cm<sup>-1</sup> are assigned to  $\nu(C=N)$  of the azomethine group present in the Schiff bases<sup>36-37</sup>. This band shifts slightly to lower energy side in the region 1620-1581 cm<sup>-1</sup> indicating coordination of the nitrogen of this group with metal ions<sup>38</sup>.

The band in the region 1265-1292 cm<sup>-1</sup> observed in the spectrum of the ligands shifted slightly to higher frequencies indicating coordination through oxygen of the phenolic group.

### $\nu(M-N)$ and $\nu(M-O)$

In the present complexes, vibrations occurred in the region 679-503 cm<sup>-1</sup> and 500-400 cm<sup>-1</sup> have been assigned to  $\nu(M-N)$  and  $\nu(M-O)$  respectively.

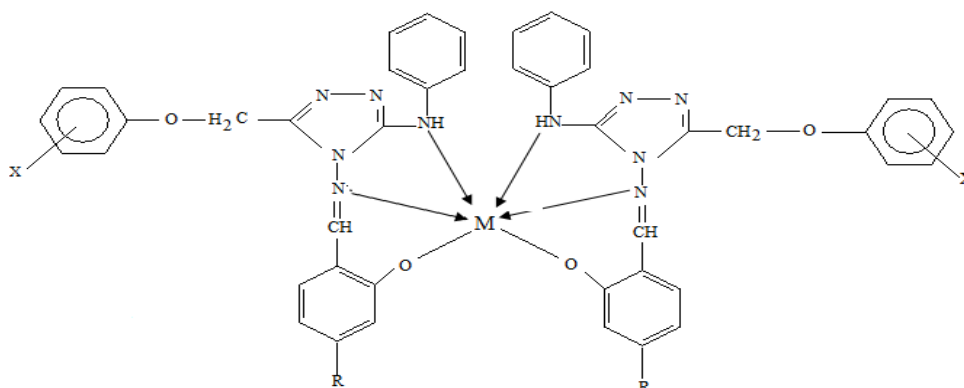
### <sup>1</sup>H NMR Spectra Studies

The <sup>1</sup>H NMR spectra of the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> and their Zn(II), Cd(II) and Hg(II) complexes are recorded and presented in the **Fig. 5.3(c-i)**. A comparison of the spectra of the ligands HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> and its complexes clearly indicates deprotonation of phenolic OH during complexation.

### Electron Spin Resonance Spectral Studies

ESR spectra of the copper complexes are recorded at room temperature and are presented in the **Fig. 5.4(a-b)**. When the monomeric species change into dimeric species having axial symmetry and identical sites, the 'g' values also change in the symmetry<sup>39-41</sup>. The width and shapes of the absorption lines offer a means of the substances. From the observed 'g' values  $g_{\parallel} > g_{\perp}$  it is evident that the unpaired electron lies predominantly in the  $d_{x^2-y^2}$  orbital of Copper(II) ion.

Kivelson and Neiman<sup>42</sup> have reported the 'g<sub>||</sub>' values less than 2.3 for covalent character of the metal ligand bond and greater than 2.3 for ionic character. In the present study the Cu(II) complex with the ligand HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> have 'g<sub>||</sub>' values 2.164-2.184. This indicates the metal ligand bonds are more covalent in character.



Ligands	X	R
HL <sub>7</sub>	Cl	H
HL <sub>8</sub>	Br	H
HL <sub>9</sub>	Br	Br

M= Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II)

**Table 5.1 (a): Elemental analysis, Melting point, Color and Molar Conductance data of the Ligand HL<sub>7</sub> and its metal complexes**

S. No	Ligand / Complexes	Color M.P(°C)/ Yield(%)	Analysis % Found (Calculated)					Magnetic Moments $\mu_{\text{eff}}$ B.M.	Molar Conductance $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$
			C	H	N	Cl	M		
1	HL <sub>1</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Cl	Yellow 188(62)	62.86 (62.93)	4.26 (4.29)	16.63 (16.68)	8.45 (8.48)	--	--	--
2	Co(L <sub>1</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	Brown >300(65)	58.87 (58.93)	3.76 (3.79)	15.59 (15.62)	7.86 (7.92)	6.55 (6.57)	4.46	44.66
3	Ni(L <sub>1</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	Brown >300(68)	58.90 (58.94)	3.75 (3.79)	15.60 (15.63)	7.83 (7.93)	6.50 (6.55)	3.10	44.32
4	Cu(L <sub>1</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	Green >300(70)	58.56 (58.63)	3.75 (3.77)	15.51 (15.54)	7.82 (7.88)	7.02 (7.05)	1.76	32.23
5	Zn(L <sub>1</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	yellow >300(67)	50.74 (50.78)	4.01 (4.03)	13.40 (13.46)	6.77 (6.82)	12.50 (12.55)	Diamagnet ic	43.34
6	Cd(L <sub>1</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	Yellow >300 (60)	46.50 (46.56)	3.65 (3.70)	12.30 (12.34)	6.20 (6.26)	19.81 (19.82)	Diamagnet ic	49.57
7	Hg(L <sub>1</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	Brown >300 (66)	40.25 (40.29)	3.17 (3.20)	10.63 (10.68)	5.39 (5.41)	30.57 (30.62)	Diamagnet ic	44.28

\* The values shown in the parenthesis are calculated ones.

**Table 5.1 (b): Elemental analysis, Melting point, Color and Molar Conductance data of the Ligand HL<sub>8</sub> and its metal complexes**

S.No	Ligand /Complexes	Color M.P(°C)/ Yield(%)	Analysis % Found (Calculated)					Magnetic Moments $\mu_{\text{eff}}$ B.M.	Molar Conductance $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$
			C	H	N	Br	M		
1	HL <sub>2</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Br	yellow 176(60)	56.89 (56.90)	3.85 (3.88)	15.05 (15.09)	17.20 (17.22)	--	--	--
2	Co(L <sub>2</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	Brown >300(67)	53.55 (53.61)	3.20 (3.24)	14.15 (14.21)	16.19 (16.22)	5.95 (5.98)	4.65	42.85
3	Ni(L <sub>2</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	Brown >300(63)	53.59 (53.63)	3.18 (3.25)	14.20 (14.22)	16.18 (16.23)	5.93 (5.96)	3.24	48.44
4	Cu(L <sub>2</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	Green >300(75)	53.30 (53.36)	3.20 (3.23)	14.13 (14.15)	16.12 (16.15)	6.41 (6.42)	1.89	35.27
5	Zn(L <sub>2</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	yellow >300(63)	46.67 (46.70)	3.68 (3.71)	12.34 (12.38)	14.10 (14.13)	11.50 (11.56)	diamagnet ic	43.35
6	Cd(L <sub>2</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	Yellow >300 (59)	43.20 (43.18)	3.40 (3.43)	11.40 (11.45)	13.03 (13.07)	18.34 (18.38)	diamagnet ic	45.59
7	Hg(L <sub>2</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	Brown >300 (60)	37.70 (37.74)	2.97 (3.00)	09.97 (10.00)	11.40 (11.42)	28.63 (28.67)	diamagnet ic	43.29

\* The values shown in the parenthesis are calculated ones

**Table 5.1(c): Elemental analysis, Melting point, Color and Conductance data of the Ligand HL<sub>9</sub> and its metal complexes**

S.No	Ligand /Complexes	Color M.P(°C)/ Yield(%)	Analysis % Found (Calculated)					Magnetic Moments $\mu_{\text{eff}}$ B.M.	Molar Conductance $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$
			C	H	N	Br	M		
1	<b>HL3</b> C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub>	Yellow 187(64)	48.61 (48.63)	3.08 (3.13)	12.85 (12.89)	29.39 (29.43)	--	--	--
2	Co(L <sub>3</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	Brown >300(68)	46.15 (46.21)	2.75 (2.80)	12.20 (12.25)	27.89 (27.97)	5.10 (5.15)	5.33	42.87
3	Ni(L <sub>3</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	Yellow >300(64)	46.17 (46.22)	2.77 (2.80)	12.22 (12.25)	27.92 (27.97)	5.05 (5.13)	3.17	46.45
4	Cu(L <sub>3</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	Pale green >300(66)	46.00 (46.02)	2.70 (2.79)	12.15 (12.20)	27.83 (27.87)	5.50 (5.53)	1.91	33.26
5	Zn(L <sub>3</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	yellow >300(65)	40.98 (41.04)	3.06 (3.10)	10.82 (10.88)	24.80 (24.84)	10.10 (10.16)	diamagnetic	44.35
6	Cd(L <sub>3</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	Yellow >300 (55)	38.18 (38.24)	2.84 (2.89)	10.12 (10.14)	23.10 (23.15)	16.12 (16.28)	diamagnetic	46.62
7	Hg(L <sub>3</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	Brown >300 (58)	33.87 (33.91)	2.57 (2.56)	8.93 (8.99)	20.47 (20.52)	25.60 (25.76)	diamagnetic	33.45

\* The values shown in the parenthesis are calculated ones

**Table 5.2: Electronic spectral and Magnetic susceptibility Measurement data of representative Co(II), Ni and Cu(II) Complexes of the ligands HL<sub>7</sub>, HL<sub>8</sub> and HL<sub>9</sub>**

S. No.	Complexes/ Abbreviations	Bands		Assignments
		Nm	cm <sup>-1</sup>	
1	Co(HL <sub>1</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	986	10204	<sup>2</sup> A <sub>1g</sub> → <sup>2</sup> B <sub>1g</sub>
		593	16863	<sup>2</sup> A <sub>1g</sub> → <sup>4</sup> E <sub>1g</sub>
		465	21505	L→M
2	Ni(HL <sub>1</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	987	10131	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		728	13736	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		452	22123	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
3	Cu(HL <sub>1</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	697	14347	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		537	18622	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
		356	28089	M→L
4	Co(HL <sub>2</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	979	10152	<sup>2</sup> A <sub>1g</sub> → <sup>2</sup> B <sub>1g</sub>
		592	16891	<sup>2</sup> A <sub>1g</sub> → <sup>4</sup> E <sub>1g</sub>
		473	21141	L→M
5	Ni(HL <sub>2</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	915	10928	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		725	13793	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		462	21645	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
6	Cu(HL <sub>2</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	713	14005	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		526	19011	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
		348	28735	M→L
7	Co(HL <sub>3</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	996	10040	<sup>2</sup> A <sub>1g</sub> → <sup>2</sup> B <sub>1g</sub>
		597	17152	<sup>2</sup> A <sub>1g</sub> → <sup>4</sup> E <sub>1g</sub>
		468	21367	L→M
8	Ni(HL <sub>3</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	897	11148	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		713	14025	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		451	22172	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
9	Cu(HL <sub>3</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub> ) <sub>2</sub>	723	13831	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> A <sub>2g</sub>
		514	19455	<sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub>
		337	29673	M→L

**Table 5.3(b): Some important IR stretching frequencies of ligand HL<sub>7</sub>, HL<sub>8</sub> & HL<sub>9</sub> and its metal complexes (in cm<sup>-1</sup>)**

S. No.	Ligands / complexes	$\nu$ N-H	$\nu$ OH	$\nu$ C = N	$\nu$ C - N	$\nu$ M - N	$\nu$ M - O
1	<b>HL1</b> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Cl	3188	3065	1621	1264	-	-
2	Co(L <sub>1</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3426	--	1602	1272	598	503
3	Ni(L <sub>1</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3421	--	1608	1281	508	421
4	Cu(L <sub>1</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3240	--	1610	1258	510	419
5	Zn(L <sub>1</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3214	--	1603	1278	587	469
6	Cd(L <sub>1</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3286	--	1617	1285	596	503
7	Hg(L <sub>1</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	3260	--	1606	1276	521	418
1	<b>HL2</b> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Br	3436	3188	1620	1292	-	-
2	Co(L <sub>2</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3419	-	1581	1312	613	504
3	Ni(L <sub>2</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3394	-	1608	1302	613	505

4	Cu(L <sub>2</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3435	-	1619	1323	628	522
5	Zn(L <sub>2</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3441	-	1618	1317	679	503
6	Cd(L <sub>2</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3446	-	1610	1309	593	503
7	Hg(L <sub>2</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3445	-	1612	1317	592	480
1	HL <sub>3</sub> C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub>	3426	3187	1632	1279	-	-
2	Co(L <sub>3</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3412	-	1610	1298	615	513
3	Ni(L <sub>3</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3396	-	1605	1295	597	460
4	Cu(L <sub>3</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3435	-	1606	1298	589	496
5	Zn(L <sub>3</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3435	-	1610	1289	603	486
6	Cd(L <sub>3</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3444	-	1615	1284	587	490
7	Hg(L <sub>3</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	3440	-	1620	1292	582	469

Table 5.4(b): <sup>1</sup>H NMR spectra data of ligands HL<sub>7</sub>, HL<sub>8</sub> and HL<sub>9</sub> their meta complexes

Ligands /Complexes abbreviations	Assignments				
	OH Protons	N-H Protons	CH=N Protons	Aromatic Pprotons	CH Protons
HL <sub>1</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Cl	12.55	10.20	8.15	7.25-7.55	1.62
Zn(L <sub>1</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	---	9.95	8.30	7.45-7.62	1.65
Cd(L <sub>1</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	---	9.93	8.40	7.33-7.66	1.64
Hg(L <sub>1</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	---	9.98	8.35	7.32-7.67	1.68
HL <sub>2</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Br	12.45	9.92	8.25	7.22-7.50	1.69
Zn(L <sub>2</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	10.02	8.45	7.35-7.61	1.68
Cd(L <sub>2</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	10.05	8.35	7.33-7.68	1.70
Hg(L <sub>2</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	9.98	8.35	7.30-7.62	1.68
HL <sub>3</sub> C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub>	12.50	9.93	8.10	7.22-7.50	1.65
Zn(L <sub>3</sub> ) <sub>2</sub> Zn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	9.96	8.44	7.35-7.61	1.66
Cd(L <sub>3</sub> ) <sub>2</sub> Cd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	10.05	8.48	7.33-7.68	1.69
Hg(L <sub>3</sub> ) <sub>2</sub> Hg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	---	10.02	8.50	7.30-7.62	1.65

Table 5.5: Antimicrobial activity of ligands and their complexes (Zone of inhibition in mm)

S. No.	Complex/ligand abbreviation	Antibacterial Activities		Antifungal Activities	
		<i>E.coli</i>	<i>P. Aerginosa</i>	<i>A.niger</i>	<i>C. albicans</i>
	HL <sub>1</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Cl	10	11	10	09
1.	Co(L <sub>1</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	12	11	10	11
2.	Ni(L <sub>1</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	10	09	11	09
3.	Cu(L <sub>1</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) <sub>2</sub>	16	16	15	16
4.	Zn(L <sub>1</sub> ).2H <sub>2</sub> OZn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) 2H <sub>2</sub> O	15	16	17	15
5.	Cd(L <sub>1</sub> ).2H <sub>2</sub> Ocd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) 2H <sub>2</sub> O	11	12	10	11
6.	Hg(L <sub>1</sub> ).2H <sub>2</sub> OHg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Cl) 2H <sub>2</sub> O	10	10	11	10
	HL <sub>2</sub> C <sub>22</sub> H <sub>18</sub> N <sub>5</sub> O <sub>2</sub> Br	10	11	09	10
7.	Co(L <sub>2</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	12	11	12	10
8.	Ni(L <sub>2</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	12	10	11	11
9.	Cu(L <sub>2</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	19	18	16	17
10.	Zn(L <sub>2</sub> ).2H <sub>2</sub> OZn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	16	17	15	16
11.	Cd(L <sub>2</sub> ).2H <sub>2</sub> Ocd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	11	10	10	11
12.	Hg(L <sub>2</sub> ).2H <sub>2</sub> OHg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	10	12	10	13
	HL <sub>3</sub> C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br <sub>2</sub>	13	15	10	11
13.	Co(L <sub>3</sub> ) <sub>2</sub> Co(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	12	16	11	13
14.	Ni(L <sub>3</sub> ) <sub>2</sub> Ni(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	14	13	16	14
15.	Cu(L <sub>3</sub> ) <sub>2</sub> Cu(C <sub>22</sub> H <sub>16</sub> N <sub>5</sub> O <sub>2</sub> Br) <sub>2</sub>	15	18	15	17
16.	Zn(L <sub>3</sub> ).2H <sub>2</sub> OZn(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	17	19	16	18
17.	Cd(L <sub>3</sub> ).2H <sub>2</sub> Ocd(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	13	15	14	14
18.	Hg(L <sub>3</sub> ).2H <sub>2</sub> OHg(C <sub>22</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> Br) 2H <sub>2</sub> O	16	15	13	13
	Ciprofloxacin	27	30		
	Flucanazole			24	23

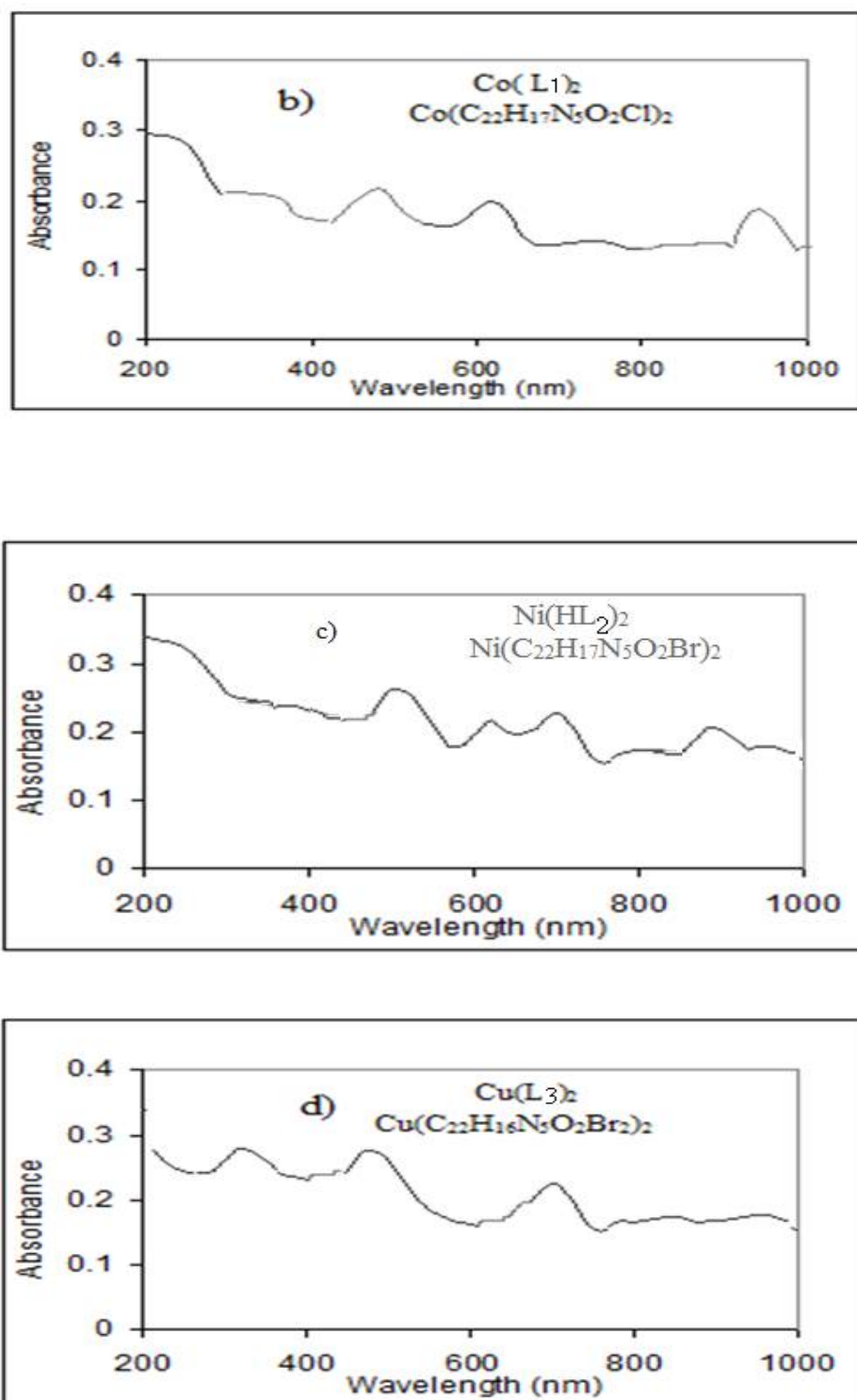
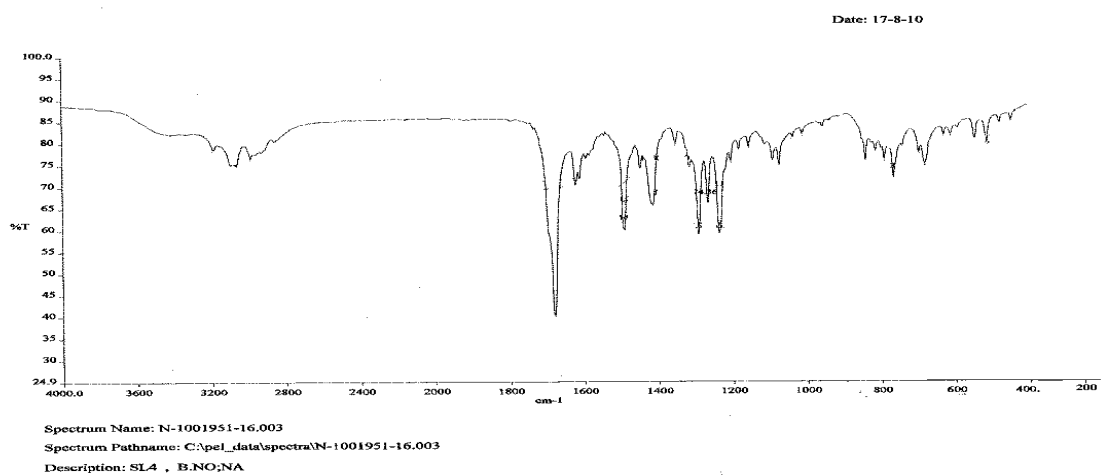
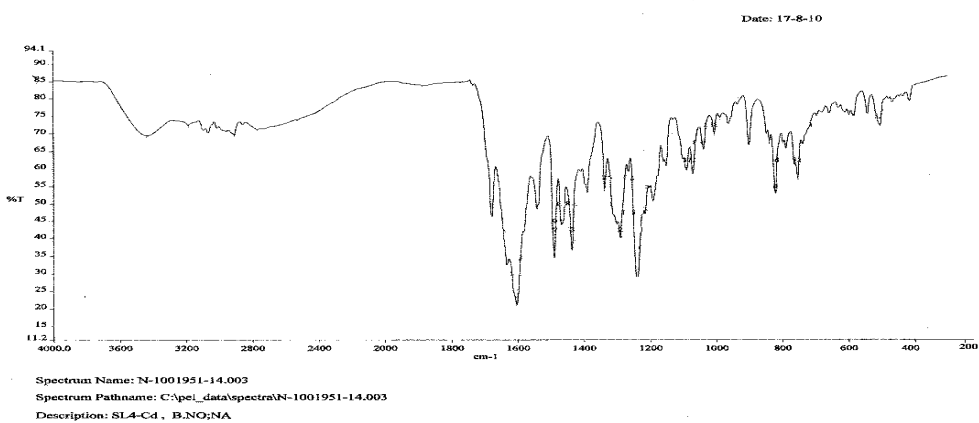
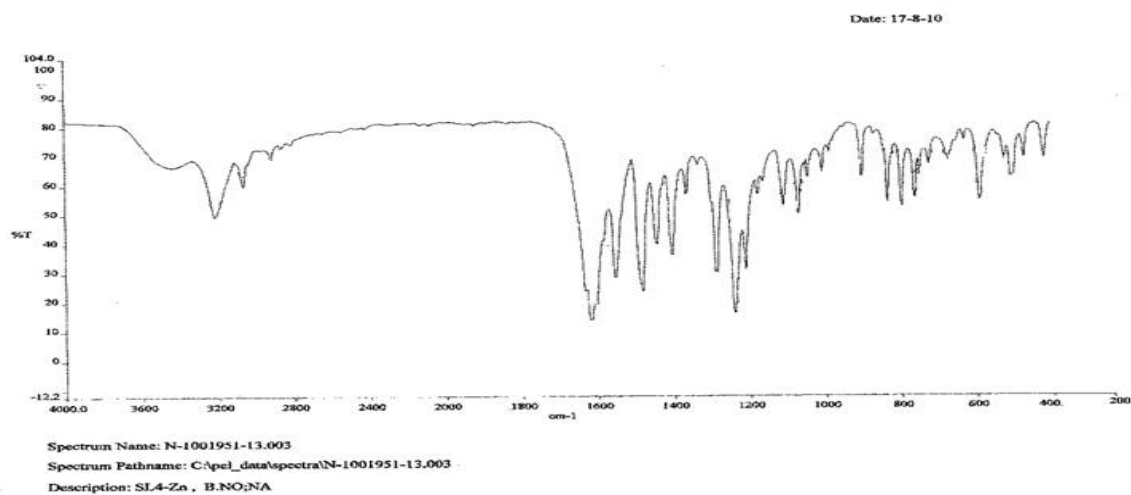


Fig. 5.1: Representative electronics spectra of  
b)  $\text{Co(HL}_1)_2$  c)  $\text{Ni(HL}_2)_2$  d)  $\text{Cu(HL}_3)_2$  complexes



Fig. 5.2(c): IR Spectrum of HL<sub>1</sub>ligandFig. 5.2(d): IR Spectrum of the Complex Cd(HL<sub>1</sub>)<sub>2</sub>Fig. 5.2(e): IR Spectrum of the Complex Zn(HL<sub>1</sub>)<sub>2</sub>

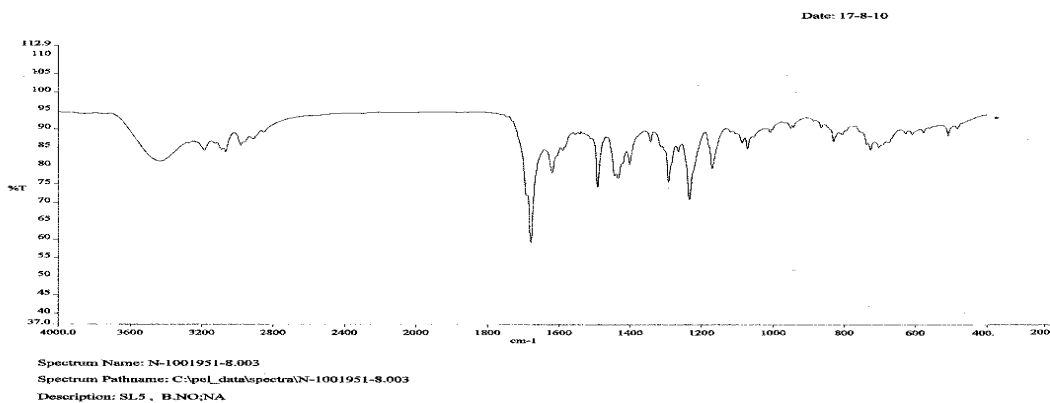


Fig. 5.2(f): IR spectra of the HL<sub>2</sub> ligand

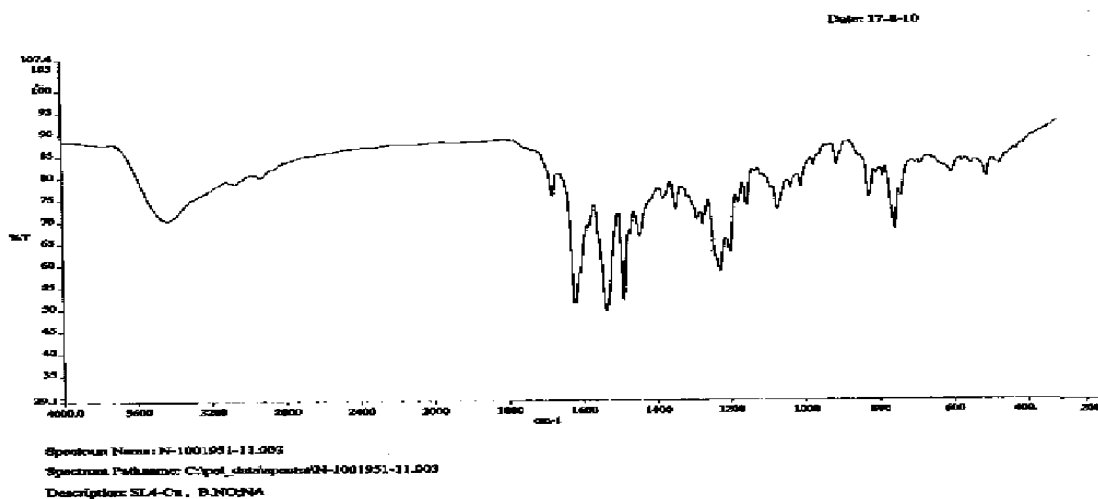


Fig. 5.2(h): IR spectra of the Cu(HL<sub>2</sub>)<sub>2</sub> complex

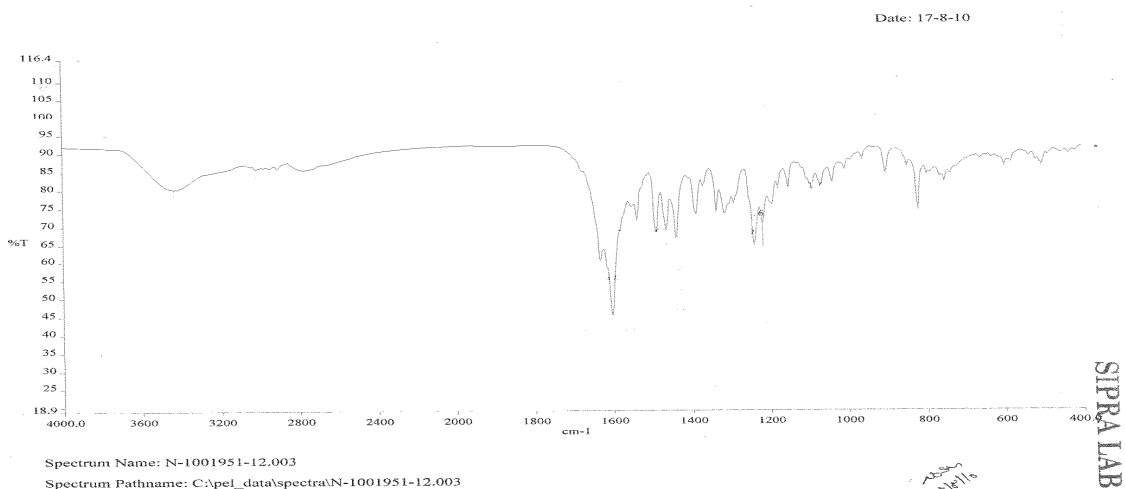


Fig. 5.2(i): IR Spectrum of Co complex with HL<sub>2</sub> ligand

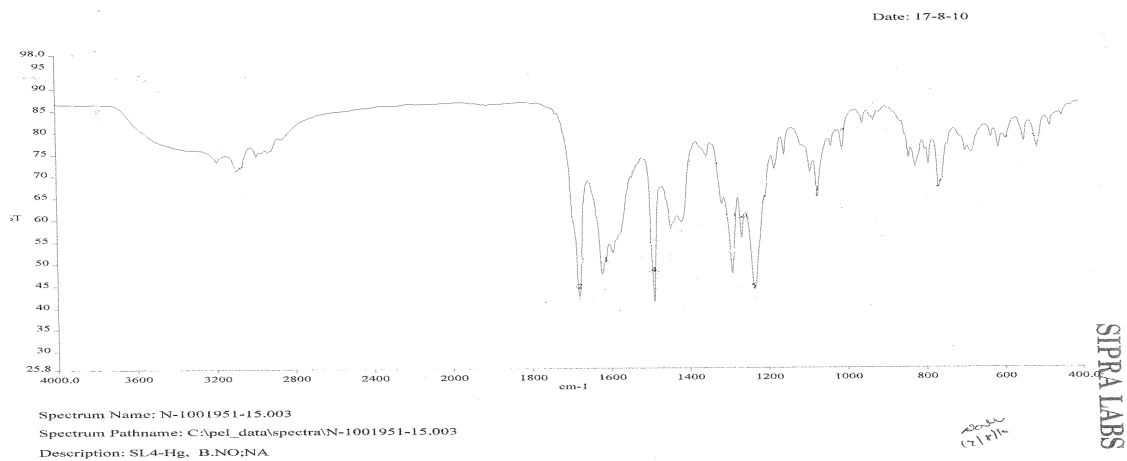


Fig. 5.2(g): IR spectra of the complex  $Hg(HL_3)_2$  ligand

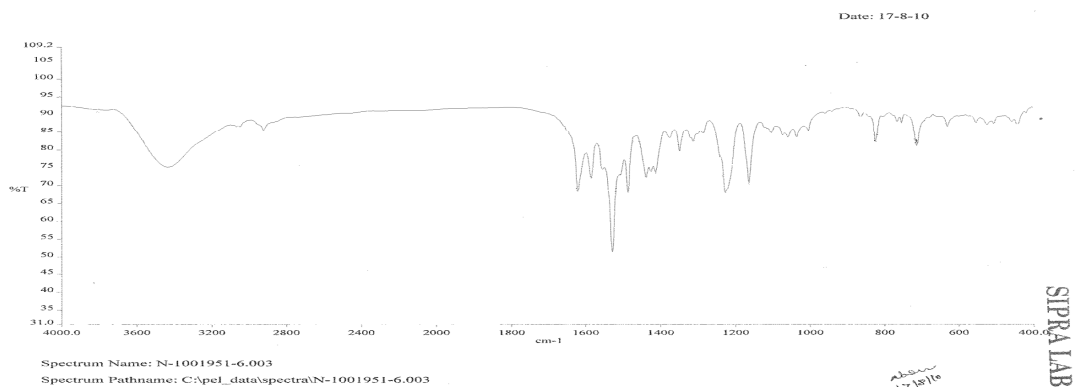


Fig. 5.2(j): IR Spectrum of Cu complex with  $HL_3$  ligand

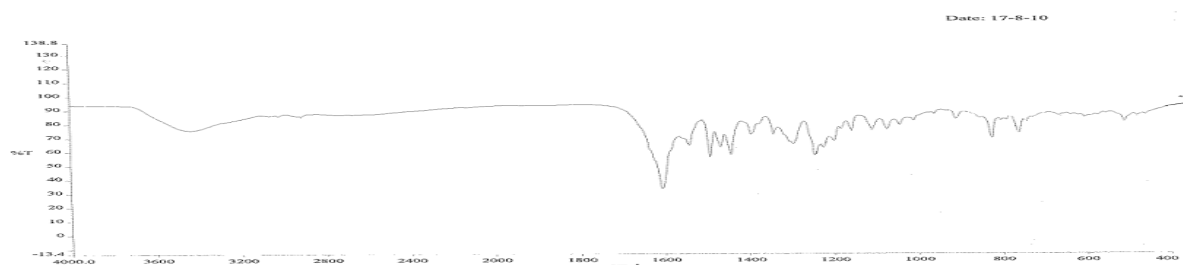
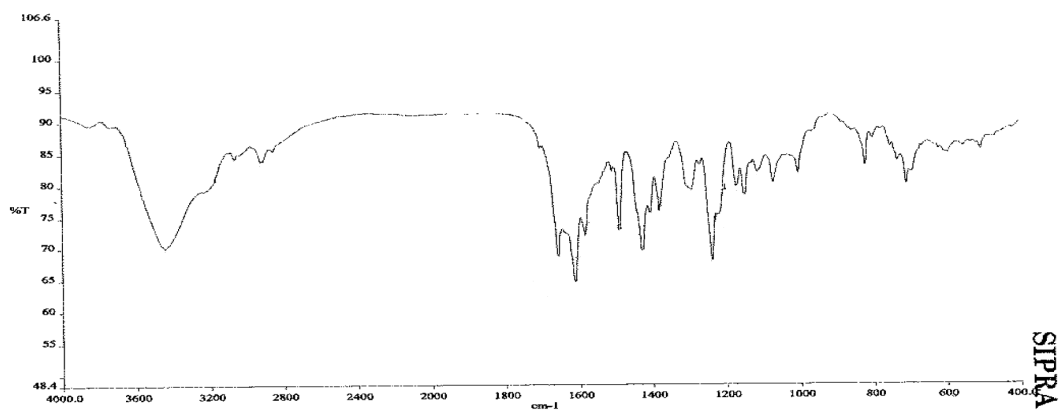


Fig. 5.2(k): IR Spectrum of Ni complex with  $HL_3$  ligand

Date: 17-8-10



Spectrum Name: N-1001951-4.003  
 Spectrum Pathname: C:\pel\_data\spectra\N-1001951-4.003  
 Description: SL5-Cd,B.NO:NA

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Fig. 5.2(l): IR Spectrum of Cd complex with HL<sub>3</sub> ligand

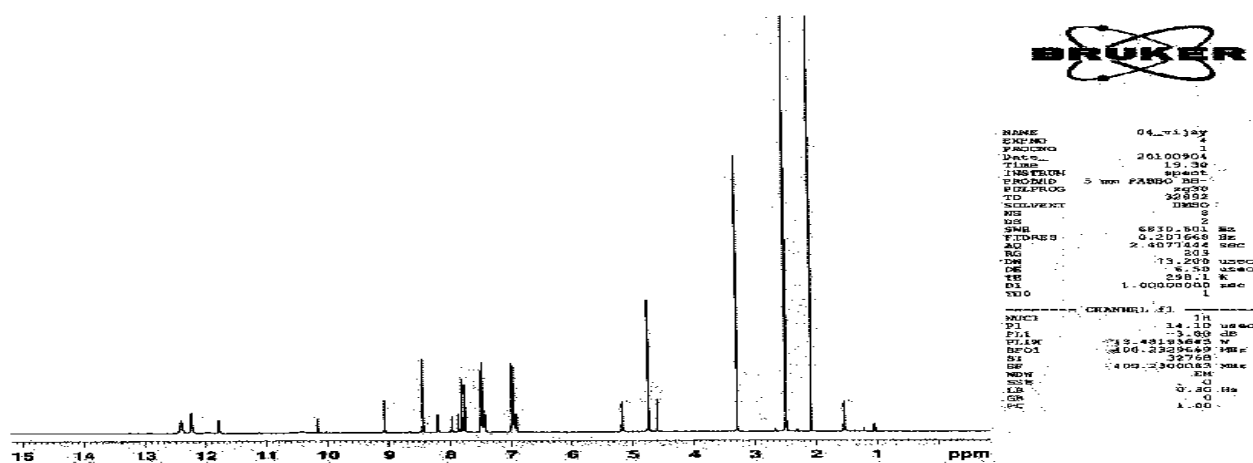


Fig. 5.3(c): <sup>1</sup>H NMR Spectrum of the ligand HL<sub>1</sub>

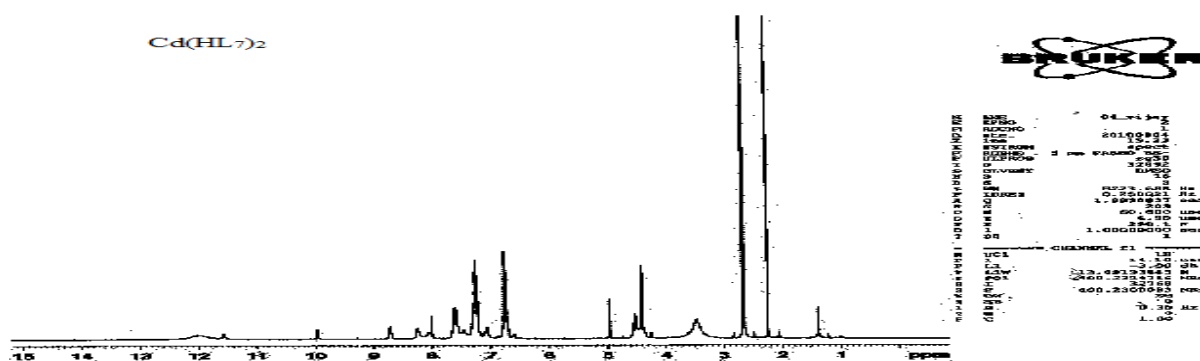
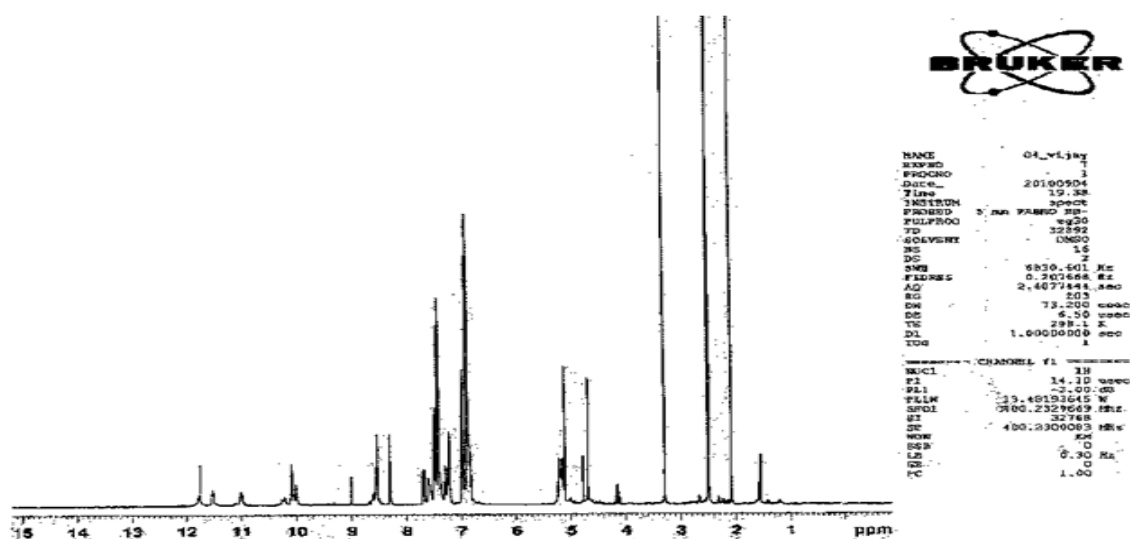
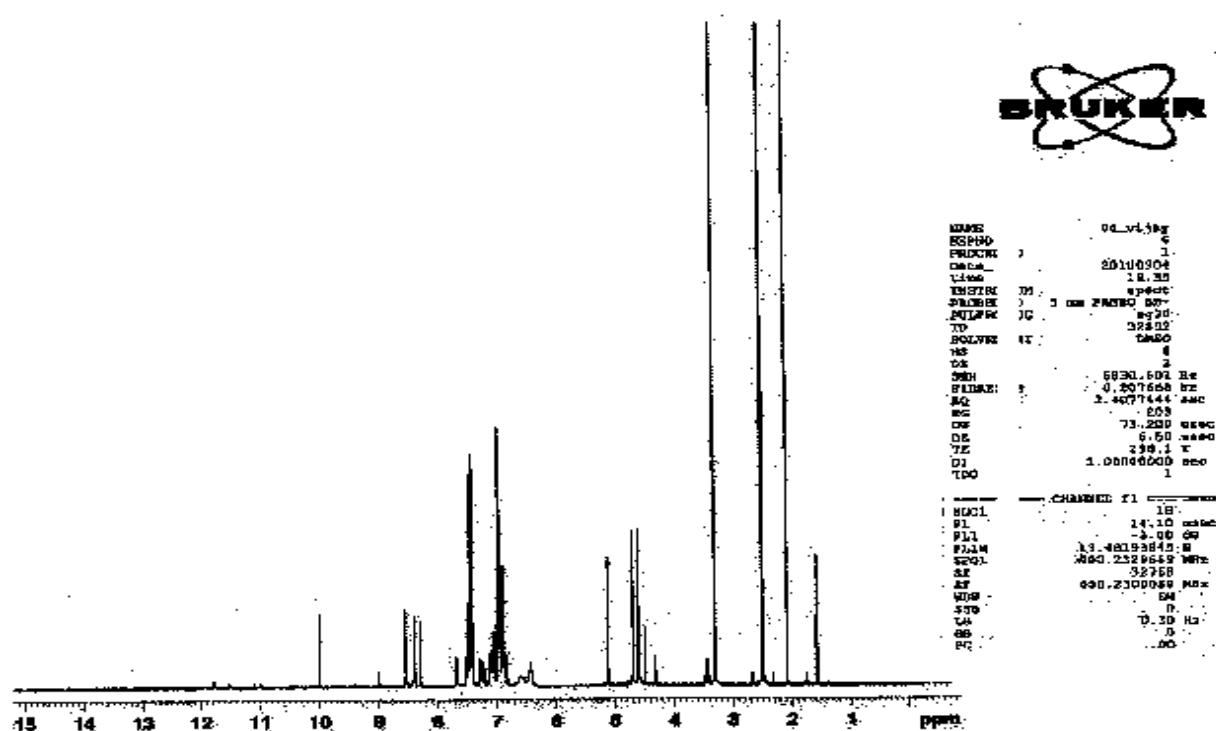


Fig. 5.3(d): <sup>1</sup>H NMR Spectrum of Cd(II) complex with the ligand HL<sub>1</sub>

Fig. 5.3(e):  $^1\text{H}$  NMR Spectrum of the ligand  $\text{HL}_2$ Fig. 5.3(f):  $^1\text{H}$  NMR Spectrum of  $\text{Zn(II)}$  complex with the ligand  $\text{HL}_2$

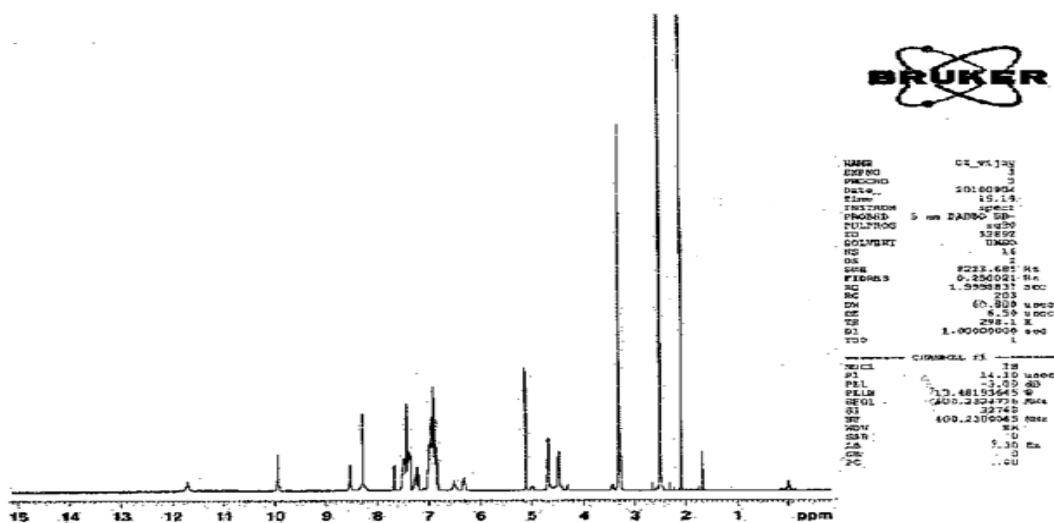


Fig. 5.3(g): <sup>1</sup>H NMR Spectrum of Cd(II) complex with the ligand HL<sub>2</sub>

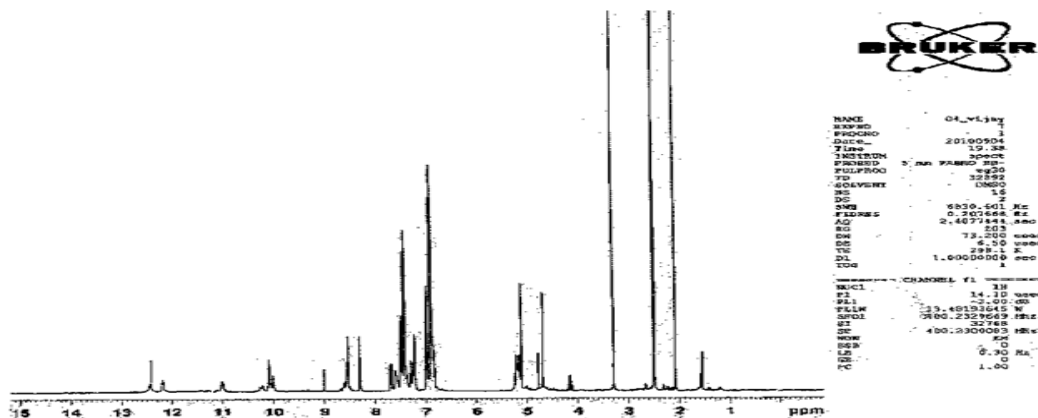


Fig. 5.3(h): <sup>1</sup>H NMR Spectrum of the ligand HL<sub>3</sub>

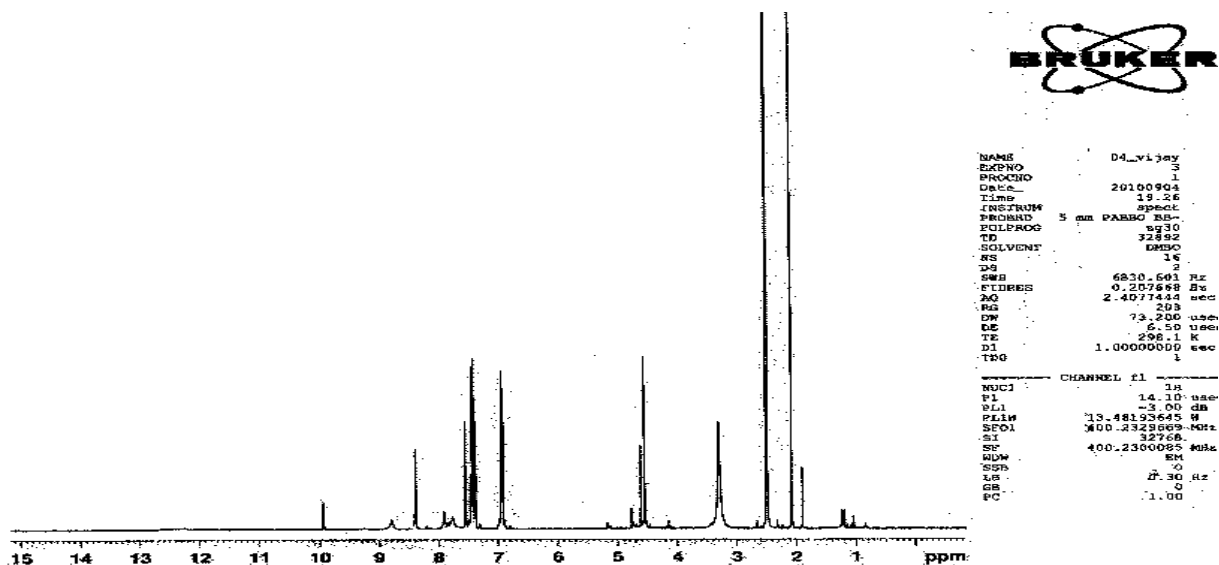


Fig. 5.3(i): <sup>1</sup>H NMR Spectrum of Zn(II) complex with the ligand HL<sub>3</sub>

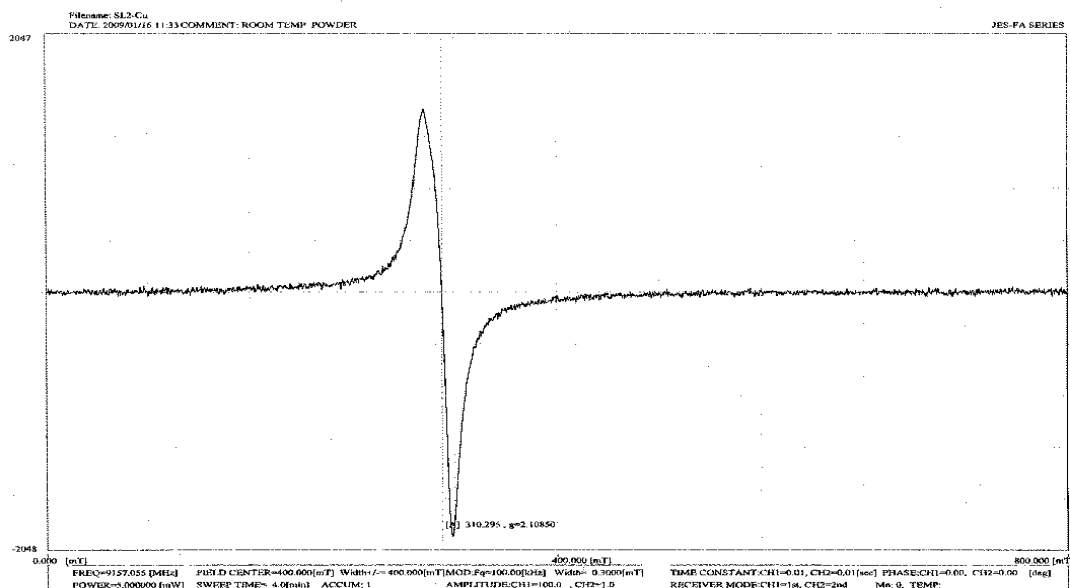


Fig. 5.4(b): ESR Spectrum of Cu(II) complex with the ligand HL<sub>1</sub>

## CONCLUSION

On the basis of analytical, magnetic, electronic, infrared, <sup>1</sup>H NMR and ESR spectral studies, the Octahedral structure with coordination number six has been proposed for all the complexes of HL<sub>1</sub>, HL<sub>2</sub> and HL<sub>3</sub> ligands.

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