

## Synthesis, Spectroscopic and Biological Studies of Some Metal Complexes with 2,2'-Diamino-N-Phenyl Hydrazo Benzene

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### Abstract

The ligand of 2,2'-diamino-N-phenyl hydrazo benzene (L) has been prepared from the reaction of ortho amino hydrazo benzene with ortho amino phenyl thiol in mole ratio 1:1 and characterized by elemental analysis (C, H, N), IR, UV-Vis. The complexes of bivalent (Co, Ni, Cu, Zn, Pd, Cd, Hg and Pb) ions and trivalent (Cr) ion have been prepared and characterized too. The structural diagnosis were established by IR, UV-Visible spectrum, Conductivities, atomic absorption and magnetic susceptibility. The complexes showed octahedral geometry around the metal ion with the (N,N,N) ligand coordinated in tridentate mode except pd complex showed trigonal bipyramidal.  $\alpha$ ,  $k_f$ ,  $\epsilon_{\max}$  for the complexes were estimated too.  $\beta$  for Co – complex was calculated. The study of biological activity of the ligand (L) and its complexes showed various activity toward *Streptococcus pyogenes*, *Staphylococcus aureus*, *E. coli*, *Pseudomonas aeruginosa* and *Candida albicans*.

Keywords: synthesis, hydrazo benzene, complexes.

### Introduction

The chemistry of hydrazides, hydrazones derived from hydrazine have been intensively investigated in recent years. The reasons are manifold; first is the coordinating ability of these compounds to chelate metal ions particularly transition and lanthanide ions<sup>(1-3)</sup>. The synthesis and characterization of many metal ions complexes that contain arrange of ancillary ligands such as macrocyclic ligands incorporating benzimidazol<sup>(4)</sup>, leucine-o-acetic acid<sup>(5)</sup>, oxadiazole derivatives<sup>(6)</sup>, Triazol derivatives<sup>(7, 8)</sup>, dioxadiazole and ditriazole<sup>(9)</sup>, ortho amino phenyl thiol derivatives<sup>(10, 11)</sup>, bis oxadiazole derivatives<sup>(12)</sup>. A considerable number of above compounds have been reported to demonstrate antibacterial and antifungal activities<sup>(13-18)</sup>. In this paper we report the synthesis, characterization and biological activities of 2, 2' - diamino- N - phenyl hydrazo benzene and its complexes with (Cr, Co, Ni, Cu, Zn, Pd, Cd, Hg and Pb).

### Experimental

#### a- Chemicals:

All reagents used were analar or chemically pure grade by British Drug Houses (BDH), Merk and Fluka.

-The chemical materials: ortho amino phenyl thiol ( $C_6H_7NS$ ), ortho amino hydrazo benzene ( $C_{12}H_{13}N_3$ ), Cobalt chloride hexahydrate ( $CoCl_2.6H_2O$ ), Nickel chloride hexa hydrate ( $NiCl_2.6H_2O$ ), Palladium chloride ( $Pd Cl_2$ ), Cadimium chloride dihydrate ( $Cd Cl_2.2 H_2O$ ), Mercury chloride ( $Hg Cl_2$ ), Copper Sulphate pentahydrate ( $Cu SO_4.5H_2O$ ), Zinc sulphate heptahydrate ( $Zn SO_4. 7 H_2O$ ), Lead nitrate ( $Pb(NO_3)_2$ ), Chromium trichloride hexahydrate ( $CrCl_3.6H_2O$ ) Ethanol 99% ( $CH_3CH_2OH$ ), Dimethyl formamide 99.5% (DMF), Dimethyl sulphoxide 99.5% (DMSO), Tetra chloro carbon 99.5% ( $CCl_4$ ), Chloroform 99% ( $CHCl_3$ ), Toluene 99% ( $C_7 H_8$ ).

#### b- Instruments:

- Elemental analyses C, H, N, were determined by calibration type: Linear Regression Euro EA Elemental Analysis.
- Melting points were determined by Gallen-Kamp apparatus.
- IR spectra were recorded as KBr discs in the range ( $4000 - 400$ )  $cm^{-1}$  using Shimadzu-FTIR.
- UV-Visible spectra were recorded by Shimadzu -UV-VIS -160A Ultra violet spectro photometer at  $25^\circ C$  using 1 cm

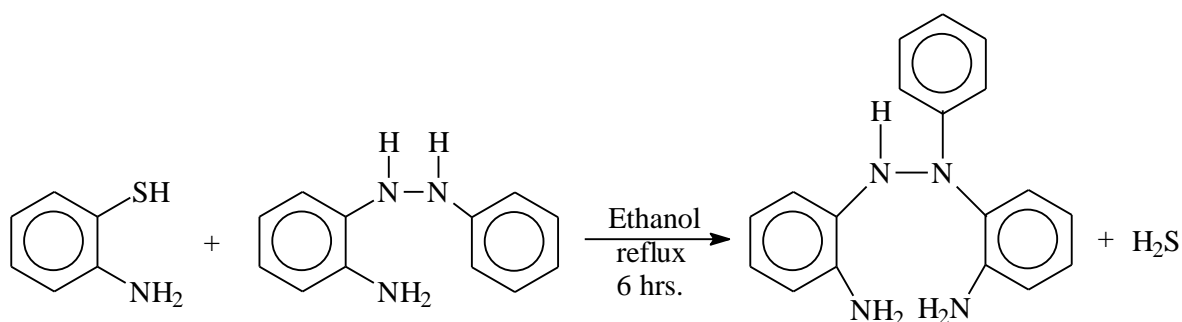
quartz cell and examined at the range of (200–1100)nm at  $10^{-3}$ M in DMSO.

- Atomic Absorption (A. A) technique using a shimadzu AA 680 G atomic absorption spectro photometer.
- Molar conductivity of the complexes were measured on pw 9526 digital conductivity in DMSO at  $10^{-3}$  M
- Magnetic susceptibility were recorded by Magnetic susceptibility Blance, model, MsB – MK<sub>1</sub>.

## Preparation

### Synthesis of 2,2'-diamino-N-phenyl hydrazo benzene (L)

Orthoaminophenyl thiol (0.125 gm, 0.001 mol) and ortho aminohyazo benzene (0.29 gm, 0.001 mol) in ethanol (20 cm<sup>3</sup>) was stirred under reflux for (6 hours). The precipitate was filtered and recrystallized from hot absolute ethanol, a fine yellow crystals were obtained m.p at 94 °C yield 88% and soluble in most organic solvents.



Elemental analysis (C, H, N) confirmed the purity of the ligand (L) with formula C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>.

The infrared spectrum of the ligand (L) in the solid state doesnot contain the  $\nu$  (S – H) which appears in the starting material orthoaminophenyl thiol at (2600 – 2500) cm<sup>-1</sup> region. This indicates the displacement of SH in orthoaminophenyl thiol. Bands observed at 3379 cm<sup>-1</sup>, 1581 cm<sup>-1</sup> and 1303 cm<sup>-1</sup> are diagnostic of the primary aromatic amine<sup>(19)</sup>. Bands around 3301 cm<sup>-1</sup> are due to  $\nu$ (N–H)<sup>(20)</sup> group. (Fig. (1)).

## Complexes

### Synthesis of the metal complexes

An ethanolic solution of metal salts one mole was added to one mole of the ligand (L) [CrCl<sub>3</sub>.6 H<sub>2</sub>O (0.26 gm. 1.00 mmol), CoCl<sub>2</sub>. 6 H<sub>2</sub>O(0.24gm. 1.00 mmol), NiCl<sub>2</sub>.6 H<sub>2</sub>O (0.24 gm. 1.00 mmol), CuSO<sub>4</sub>. 5H<sub>2</sub>O (0.24gm. 1.00 mmo)l, ZnSO<sub>4</sub>.7 H<sub>2</sub>O(0.28 gm. 1.00 mmol), PdCl<sub>2</sub>(0.17 gm.1.00 mmol), CdCl<sub>2</sub>. 2 H<sub>2</sub>O (0.22 gm.1.00 mmol), HgCl<sub>2</sub> (0.27 gm. 1.00 mmol) and Pb(NO<sub>3</sub>)<sub>2</sub> (0.33 gm. 1.00 mmol)] were added to (0.29gm.1.00 mmol) of the ligand (L). The mixture was stirred for 30 min. The product was filtered and washed with distilled water and dried under vacuum. Color, melting point, yield, metal analysis and solubility of the ligand and its complexes are given in (Table (1)).

## Results and Discussion

### Synthesis and characterization of the ligand 2,2'-diamino-N-phenyl hydrazo benzene (L)

The ligand was obtained by the reaction of one mole of orthoaminophenyl thiol and slightly excess one mole of ortho amino hydrazo benzene.

The infrared spectra of all complexes showed, the multi bands in the range (3460 – 3209) cm<sup>-1</sup> and split or broader band at (1604 – 1542) cm<sup>-1</sup> and at (1335) cm<sup>-1</sup> with the formation of a new absorption bands for the coordination bands (M – N) in the region (540 – 500) cm<sup>-1</sup> suggesting the coordination through nitrogen atom<sup>(21-22)</sup>. M – N stretching vibration have been assigned in Table (2). The bands characteristic of coordinated water and (M–O) are seen in complexe in the region (856 - 817) cm<sup>-1</sup> and (570 – 547) cm<sup>-1(23)</sup>. The Cu, Zn complexes showed bands at 1020 cm<sup>-1</sup>, 960 cm<sup>-1</sup>, 678 cm<sup>-1</sup> and was assigned to

coordinated sulphato group<sup>(24)</sup>. The Pb complex spectrum exhibited bands at  $925\text{cm}^{-1}$ ,  $680\text{cm}^{-1}$  due to coordinate nitrate ion<sup>(25, 26)</sup>. (Table (2)) describes characteristic stretching vibration frequencies ( $\text{cm}^{-1}$ ) located in the FT-IR of the ligand and its complexes.

### The UV – Visible spectra of the ligand (L) and complexes

The UV-Visible spectra of the ligand (L) in DMSO solution exhibited strong absorption bands at (266 nm,  $37593\text{cm}^{-1}$ ) and (351 nm,  $28490\text{cm}^{-1}$ ). This may attributed to the  $\pi - \pi^*$  transition<sup>(27)</sup>. (Fig.(2)).

The UV-Visible spectrum for  $\text{Cr}^{+3}$  complex showed one band in the region (575 nm,  $17391\text{cm}^{-1}$ ) is due to  $A_{2g} \longrightarrow T_{2g}$  (28).

The UV-Visible spectrum for  $\text{Co}^{+2}$  complex showed three bands in the region (800 nm,  $12500\text{cm}^{-1}$ ) due to  $T_{1g} \longrightarrow E_g$ , (660 nm,  $15151\text{cm}^{-1}$ ) due to  $T_{1g} \longrightarrow A_{2g}$  and (550 nm,  $18181\text{cm}^{-1}$ ) due to  $T_{1g}(F) \longrightarrow T_{1g}(p)$  (29,30).

The ratio of  $18181\text{cm}^{-1}$  to  $12500$  is 1.54 which fits with Tanabe-Sugano  $d^7$  curve (Fig.(3)) for  $E/B = 26$  and  $\Delta_0 / B = 17$ .  $\Delta_0 = 12500\text{cm}^{-1}$   
 $B \text{ complex} = 12500 / 17 = 735\text{cm}^{-1}$   
 $B \text{ for free } \text{Co}^{+2} = 971\text{cm}^{-1}$   
 $B (\text{nephelauxetic effect}) = B \text{ complex} / B \text{ Co}^{+2}$   
 $= 735 / 971 = 0.756$ .

Which indicates of d – electron delocalization on the ligand hence a significant covalent character in the complex<sup>(31)</sup>.

The UV-Visible spectrum for  $\text{Ni}^{+2}$  showed one band at (823 nm,  $12150\text{cm}^{-1}$ ) which is due to  $A_{2g} \longrightarrow T_{1g}$ .

The spectrum of the  $\text{Cu}^{+2}$  complex exhibited a band at (660 nm,  $15151\text{cm}^{-1}$ ) which is due to  $E_g \longrightarrow T_{2g}$ .

The brown palladium complex exhibited a strong band at (400 nm,  $25000\text{cm}^{-1}$ ) due to  $d - \pi^*$  transition<sup>(32)</sup>. The UV-Visible spectra of  $\text{Zn}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Hg}^{+2}$  and  $\text{Pb}^{+2}$  complexes showed shifted bands compared with free ligand (L) are due to charge transfer (Table (3)).

The molar conductance of all complexes in DMSO were found to be low which suggested coordination of anion to the metal.

The  $\mu_{\text{eff}}$  value of Cr,Co,Ni,Cu complexes are within the range (3.75,4.46,2.90,1.99) respectively expected for spin-free octahedral structures<sup>(33,34)</sup>. Electronic spectra, Conductance in (DMSO), Magnetic moment (B.M) of the ligand and its complexes are given in Table (3)

### Study of $\text{Cr}^{+3}$ , $\text{Ni}^{+2}$ complexes formation in solution

The complexes of the ligand (L) with selected metal ions ( $\text{Cr}^{+3}$ ,  $\text{Ni}^{+2}$ ) were studied in solution using ethanol as solvent in order to determine (M:L) ratio in the prepared complexes, following molar ratio method<sup>(35)</sup>. A series of solutions were prepared having a constant concentration (C)  $10^{-3}\text{M}$  of the hydrated metal salts and the ligand (L). The results of complexes formation in solution are given in (Table (4)).

The stability constant (kf) was evaluated using the following equations:

$$Kf = 1 - \alpha / \alpha^2 c \dots\dots\dots(1)$$

$$\alpha = A_m - A_s / A_m \dots\dots\dots(2)$$

( $\alpha$ ) is the degree of the dissociation, (c) is the concentration of the complex ( $10^{-3}\text{M}$ ). ( $A_s$ ) and ( $A_m$ ) are the absorbance value of the partially and fully formed complex respectively Table (5).

The absorbance of the solutions were measured at ( $\lambda_{\text{max}}$ ) of the maximum absorption. The molar absorptivity ( $\epsilon_{\text{max}}$ ) (eq.3) has been calculated using equation:

$$A = \epsilon_{\text{max}} \cdot b \cdot c \dots\dots\dots(3)$$

(A) is the average of three measurement of the absorption containing the same amount of metal ion and three fold excess of ligand, (b) is the depth of the quartz cell usually equal 1 cm.

The atomic absorption analysis was used to confirm our molar ratio calculation of [metal:ligand] (M:L) for Synthesis complex as well. The results showed a ratio M:L (1:1) for all complexes (Table (1)).

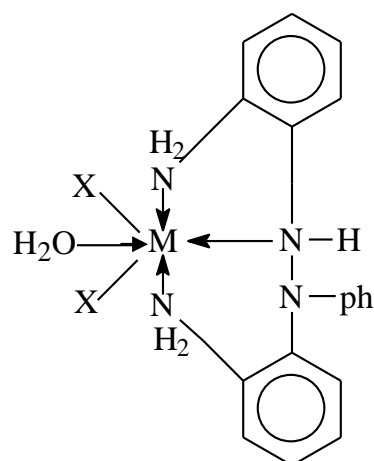
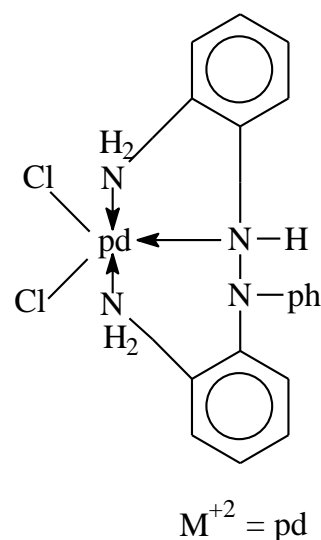
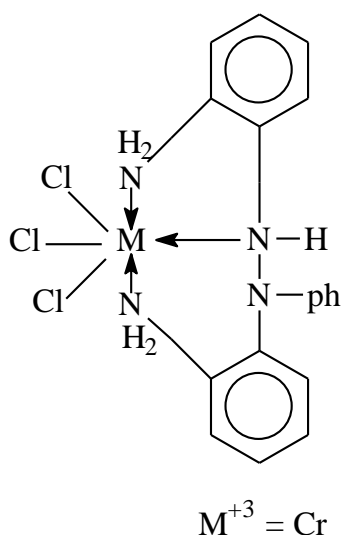
### Study of biological activity

The biological activity of the prepared new ligand and its complexes were studied against selected types of microorganisms which include gram positive bacteria like *Streptococcus pyogenes*, *Staphylococcus aureus* and gram negative bacteria like *E. coli*, *Pseudomonas aeruginosa* and yeast like *Candida albicans*, in agar diffusion method, which include Cephalosporin, Amoxicillin and Nystatin as control. Agar diffusion method involves the exposure of the inhibition zone toward the diffusion of microorganisms on agar plate. The plates were incubated for (24) hrs at (37 C°). The inhibition zone of bacterial growth around the disc was observed.

### Conclusion

A series of complexes of  $\text{Cr}^{+3}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Zn}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Hg}^{+2}$ ,  $\text{Pb}^{+2}$ , with 2, 2'-diamino-N-phenyl hydrazo benzene(L) have been prepared and characterized.

The tridentate ligand (L) (N,N,N) is binding metal ion forming octahedral structure except with  $\text{Pd}^{+2}$  is forming trigonal bipyramidal as follows.



$\text{M}^{+2} = \text{Co}, \text{Ni}, \text{Cd}, \text{Hg}$

$\text{X} = \text{Cl}^{-1}$

$\text{M}^{+2} = \text{Cu}, \text{Zn}$

$\text{X}_2 = \text{SO}_4^{-2}$

$\text{M}^{+2} = \text{pb}$

$\text{X} = \text{NO}_3^{-1}$

Biological effects of new ligand and its complexes indicated that the new ligand and its complexes exhibited antibacterial activity against both gram positive and gram negative bacteria specially zinc and mercury complexes, while the effects of new ligand and its complexes on *Candida albicans* showed less than effects except zinc and mercury complex. generally the results of the prepared complexes exhibited antimicrobial activity toward bacteria more than yeast *Candida albicans*.

Table (1)

*Color, melting point, yield, metal analysis and solubility of the ligand and its complexes.*

No.	Compound	Color	m. p. c° or (D)	Yield %	Metal analysis found (calculated)				Solubility
					C %	H %	N %	M %	
1	(L) C <sub>18</sub> H <sub>18</sub> N <sub>4</sub>	Yellow	94 c°	88	74.399 (74.482)	6.206 (6.206)	19.300 (19.310)	–	EtOH, CCl <sub>4</sub> , CHCl <sub>3</sub> C <sub>7</sub> H <sub>8</sub> , DMF, DMSO
2	L – Cr Cl <sub>3</sub>	Green	220 c°	82	48.159 (48.160)	4.012 (4.013)	12.484 (12.486)	11.56 (11.59)	DMF, DMSO
3	L – Co Cl <sub>2</sub> .H <sub>2</sub> O	Dark blue	180 c°	75	49.316 (49.315)	4.569 (4.566)	12.786 (12.788)	13.35 (13.47)	DMF, DMSO
4	L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	Light green	120 c°	91	49.349 (49.348)	4.570 (4.569)	12.795 (12.794)	13.40 (13.41)	DMF, DMSO
5	L – Cu SO <sub>4</sub> .H <sub>2</sub> O	blue	180 c°	87	46.202 (46.203)	4.277 (4.278)	11.979 (11.978)	13.59 (13.58)	DMF, DMSO
6	L – Zn SO <sub>4</sub> .H <sub>2</sub> O	yellow	220(D)	79	46.023 (46.025)	4.260 (4.261)	11.930 (11.932)	13.90 (13.91)	DMF, DMSO
7	L – Pd Cl <sub>2</sub>	brown	224 (D)	86	46.200 (46.213)	3.849 (3.851)	11.985 (11.981)	22.79 (22.76)	DMF, DMSO
8	L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	Light yellow	192 c°	92	43.954 (43.956)	4.06 (4.07)	11.397 (11.396)	22.86 (22.87)	DMF, DMSO
9	L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	violet	202 c°	71	37.270 (37.273)	3.450 (3.451)	9.665 (9.663)	34.57 (34.59)	DMF, DMSO
10	L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	Light yellow	134 c°	94	33.798 (33.797)	3.127 (3.129)	13.144 (13.143)	32.39 (32.40)	DMF, DMSO

*D = Decomposition*

**Table (2)**  
**Characteristic stretching vibrational frequencies ( $\text{cm}^{-1}$ ) located in the FT – IR of the ligand (L) and its complexes.**

No.	Compound	$\nu$ (N–H) $\text{cm}^{-1}$	$\nu$ (C–H) $\text{cm}^{-1}$ Ar.	$\nu$ (N–N) $\text{cm}^{-1}$	$\nu$ (M–N) $\text{cm}^{-1}$	$\nu$ (M–X) $\text{cm}^{-1}$	$\nu$ (M–O) + $\nu$ (OH <sub>2</sub> ) <sub>aq.</sub> $\text{cm}^{-1}$	$\nu$ SO <sub>4</sub> -2 $\nu$ NO <sub>3</sub> - $\text{cm}^{-1}$
1	(L) C <sub>18</sub> H <sub>18</sub> N <sub>4</sub>	3379(asym) (m.s) 3301(sym) (s.s) 1612 (s.s), 1581(m.s.) 1303 (s.s), 1242 (s.s)	3186 (m.s) 3062 (m.s) 856 (di-sub) 758 (mon - sub)	1442 (s.s)	–	–	–	–
2	L – Cr Cl <sub>3</sub>	3379,3301(asym)(sp)(m.br) 3209 (sym) (sh) 1612 (s.s),1590 (sh),1558(m.s) 1380 (sh), 1303 (s.s) 1242(s.s),1230(sh)	3120 (sh) 3030 (m.s)	1400 (sh) 1442(m.s)	547 (s.s)	462 (m. br)	–	–
3	L – Co Cl <sub>2</sub> .H <sub>2</sub> O	3417 (asym) (s. br) 3379 (sym) (s. br) 1618 (s.s), 1580 (m.s) 1300(sh), 1305(m.s),1245(m.s)	3120 (sh) 3040 (m.br)	1434(m.br)	520(m.br)	440 (m. br)	570 (m.br) 825 (m.br)	–
4	L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	3284 (asym)(m.s), 3247,3211(sym)(m.s) 1610(sh), 1593 (m.s) 1315 (m.s), 1278 (m.s)	3124 (m.s) 3085 (w.s)	1430 (sh) 1442 (m.s)	510 (w.s)	416 (m.s)	547 (m.s) 825 (s.s)	–
5	L –Cu SO <sub>4</sub> .H <sub>2</sub> O	3386,3301(asym) (m.s) 3200(sym) (sh) 1620 (s.s), 1575 (sh) 1303 (s.s), 1242 (m.s)	3120 (sh) 3062 (sh)	1435 (sh) 1442 (m.s)	520 (m.br)	420 (m.br)	555 (m.br) 856 (m.s)	1020(sh) 960 (sh) 675(m.br)
6	L –Zn SO <sub>4</sub> .H <sub>2</sub> O	3379(asym)(m.br) 3247,3209 (sym) (m.br) 1589 (s.s), 1550 (m.s), 1380 (m.br) 1310 (m.s), 1280 (s.s) 1226 (s.s)	3124 (m.br) 3085 (sh)	1440 (m.s)	510 (m.br)	416 (m.s)	547 (s.s) 825 (s.s)	1018(s.s) 948(m.s) 678(m.s)
7	L – Pd Cl <sub>2</sub>	3440-3332 (asym) (m.br) 3280 (sym) (m.br) 1604(s.s), 1560 (sh), 1388(m.br) 1303 (s.s), 1249 (s.s),1203 (sh) 1290 (sh)	3170 (m.br) 3055 (m. br)	1430 (sh) 1442 (m.s)	540 (m.br)	420 (m.br)	v	–
8	L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	3448-3355 (asym) (m.br) 3286 (sym) (m.br) 1604 (s.s), 1550 (sh) 1325 (sh), 1303 (s.s) 1270 (sh),1250 (sh)	3147 (m.br) 3055(m.br)	1419(m.s)	500 (sh)	415 (m.s)	555 (m.br) 817(s.s)	–
9	L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	3448-3325 (asym) (m.br) 3278 (sym) (m.br) 1604 (s.s), 1560 (sh) 1303 (s.s), 1250 (sh)	3147 (m.br) 3030 (m.br)	1400 (m.s)	500 (sh)	440 (s.s)	555 (s.s) 817 (s.s)	–
10	L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	3460,3380 (asym) (m.br) 3320 (sym) (sh) 1605 (s.s), 1570 (sh) 1380 (m.s), 1315 (s.s) 1240 (s.s), 1200 (m.s)	3180 (m.br) 3050 (m.s)	1450 (m.s)	535 (m.br)	440 (m.br)	560 (m.br) 820 (s.s)	925 (s.s) 680 (s.s)

Ar. =aromatic, Ali =aliphatic, asym =asymmetric, sym =symmetric, sh =shoulder  
 S =sharp, br =broad, m =medium, s =strong, w =weak

**Table (3)**

**Electronic spectra (DMSO), conductance in DMSO, Magnetic moment (B.M) of the ligand (L) and its complexes.**

No.	Compound	$\lambda$ (nm) $\text{cm}^{-1}$	Assignment Bands	$\Lambda$ s. $\text{cm}^{-1}$ DMSO ( $10^{-3}$ M)	$\mu_{\text{eff}}$ (B.M)
1	(L) C18 H18 N4	37593 (266) 28490 (351)	$\pi - \pi^*$	—	—
2	L – Cr Cl <sub>3</sub>	17391 (575)	$^4\text{A}_{2g} \longrightarrow ^4\text{T}_{2g}$	20.72	3.75
3	L – Co Cl <sub>2</sub> .H <sub>2</sub> O	18181 (550) 15151 (660) 12500 (800)	$^4\text{T}_{1g(\text{F})} \longrightarrow ^2\text{T}_{1g(\text{P})}$ $^4\text{T}_{1g} \longrightarrow ^4\text{A}_{2g}$ $^4\text{T}_{1g} \longrightarrow ^4\text{E}_g$	12.35	4.46
4	L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	12150 (823)	$^3\text{A}_{2g} \longrightarrow ^3\text{T}_{1g}$	10.59	2.90
5	L –Cu SO <sub>4</sub> .H <sub>2</sub> O	15151 (660)	$^2\text{E}_g \longrightarrow ^2\text{T}_{2g}$	11.53	1.99
6	L –Zn SO <sub>4</sub> .H <sub>2</sub> O	27777 (360)	Charge – Transfer	7.95	—
7	L – Pd Cl <sub>2</sub>	25000 (400)	$\text{d} - \pi^*$	5.32	—
8	L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	27777 (360)	Charge - Transfer	9.44	—
9	L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	31746 (315)	Charge - Transfer	10.39	—
10	L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	28169 (355)	Charge - Transfer	11.34	—

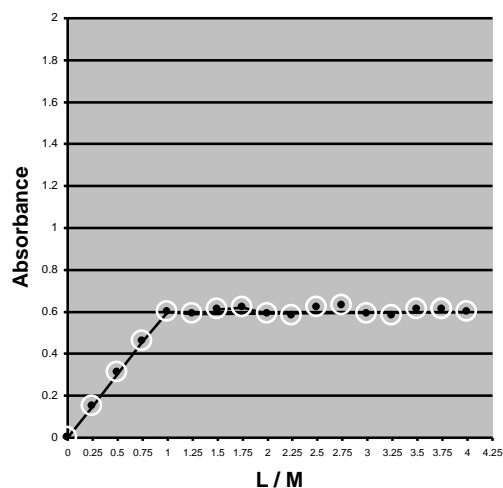
**B. M = Bohr Magnetron**

**V<sub>m</sub> = volume of metal in ml**

**V<sub>L</sub> = volume of ligand in ml**

**Table (4) Fig.(4) (a)**

L – Cr Cl <sub>3</sub>		
V <sub>M</sub>	V <sub>L</sub>	Abs
1 ml	0.25	0.15
1	0.50	0.31
1	0.75	0.46
1	1	0.60
1	1.25	0.59
1	1.50	0.61
1	1.75	0.62
1	2	0.59
1	2.25	0.58
1	2.50	0.62
1	2.75	0.63
1	3	0.59
1	3.25	0.58
1	3.50	0.61
1	3.75	0.61
1	4	0.60



**Fig. (4) (a) Continous variation slop for Cr<sup>+3</sup> ion  $\lambda$  (368 nm).**

Table (4) Fig. (4) (b)

<i>L – Ni Cl<sub>2</sub>.H<sub>2</sub>O</i>		
<i>V M</i>	<i>VL</i>	<i>Abs</i>
1 ml	0.25	0.28
1	0.50	0.59
1	0.75	0.91
1	1	1.20
1	1.25	1.19
1	1.50	1.21
1	1.75	1.22
1	2	1.19
1	2.25	1.18
1	2.50	1.21
1	2.75	1.19
1	3	1.22
1	3.25	1.21
1	3.50	1.21
1	3.75	1.19
1	4	1.22

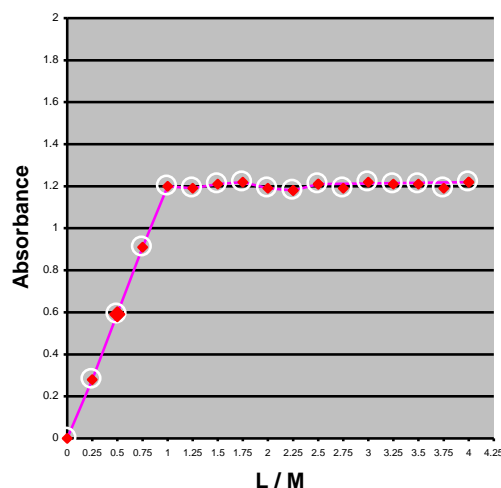
Fig. (4) (b) Continous variation slop for  $Ni^{+2}$  ion  $\lambda$  (390 nm).

Table (5)

*As, Am, Kf,  $\epsilon_{max}$  and  $\lambda_{max}$  of the  $Cr^{+3}$  and  $Ni^{+2}$  complexes.*

No.	Compound	As	Am	$\alpha$	Formation constant ( $K_f$ )	molar absorptivity $\epsilon_{max}$ $L. mol^{-1}.cm^{-1}$	$\lambda_{max}$ (nm)
1-	Cr – complex	0.60	0.63	0.047	$4.314 \times 10^5$	3276	368
2-	Ni – complex	1.20	1.22	0.016	$3.843 \times 10^6$	2925	390

Table (6)

*Effect of ligand and its complexes on gram positive bacteria.*

Compound	Diameter of inhibition zone (mm)at concentration 1mg/ml		Diameter of inhibition zone (mm)at concentration 5mg/ml	
	<i>E. coli</i>	<i>Pseudomonas aeruginosa</i>	<i>E. coli</i>	<i>Pseudomonas aeruginosa</i>
Cephalosporin	26.4	25.8	28.6	27.2
(L) C18 H18 N4	14.6	14	15.2	14.8
L – Cr Cl <sub>3</sub>	12.4	11.8	12.8	12
L – Co Cl <sub>2</sub> .H <sub>2</sub> O	13.2	13	13.4	13.4
L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	16.2	16	16.6	16
L –Cu SO <sub>4</sub> .H <sub>2</sub> O	15	15	15.4	15.2
L –Zn SO <sub>4</sub> .H <sub>2</sub> O	17.2	17	17.8	17
L – Pd Cl <sub>2</sub>	12.8	12.6	13	13
L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	14.4	13.6	14.8	13.8
L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	17.6	16.6	18	17.2
L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	17.2	16	17.4	16



Table (7)

*Effect of ligand and its complexes on gram negative bacteria.*

Compound	Diameter of inhibition zone (mm)at concentration 1mg/ml		Diameter of inhibition zone (mm)at concentration 5mg/ml	
	<i>Streptococcus pyogenes</i>	<i>Staphylococcus aureus</i>	<i>Streptococcus pyogenes</i>	<i>Staphylococcus aureus</i>
Amoxicilin	27.4	27	29.2	28.6
(L) C18 H18 N4	14.8	14.6	15.2	14.8
L – Cr Cl <sub>3</sub>	13.6	12.8	14	13
L – Co Cl <sub>2</sub> .H <sub>2</sub> O	12.6	12.4	13	12.6
L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	17.2	17	17.4	17
L –Cu SO <sub>4</sub> .H <sub>2</sub> O	14.4	14	14.6	14
L –Zn SO <sub>4</sub> .H <sub>2</sub> O	16.6	16	17.2	16.4
L – Pd Cl <sub>2</sub>	13.4	12.6	13.6	12.8
L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	13.8	13.6	13.8	13.8
L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	18.2	17.6	18.8	17.8
L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	18	17.4	18	17.6

Table (8)

*Effect of ligand and its complexes on Candida albicans.*

Compound	Diameter of inhibition zone (mm)at concentration 1mg/ml	Diameter of inhibition zone (mm)at concentration 5mg/ml
Nystatin	15.6	18.4
(L) C18 H18 N4	13.6	13.8
L – Cr Cl <sub>3</sub>	10.2	10.8
L – Co Cl <sub>2</sub> .H <sub>2</sub> O	9.8	10.2
L – Ni Cl <sub>2</sub> .H <sub>2</sub> O	10.4	10.4
L –Cu SO <sub>4</sub> .H <sub>2</sub> O	12.4	12.4
L –Zn SO <sub>4</sub> .H <sub>2</sub> O	11.8	12
L – Pd Cl <sub>2</sub>	10	10.4
L – Cd Cl <sub>2</sub> .H <sub>2</sub> O	10.2	10.2
L – Hg Cl <sub>2</sub> .H <sub>2</sub> O	14.8	15.6
L–Pb (NO <sub>3</sub> ) <sub>2</sub> .H <sub>2</sub> O	9.8	10

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#### الخلاصة

حضر الليكاند (L) 2 ،  $\frac{1}{2}$  – ثنائي امينو – N – فنيل هيدرازو بنزين (L) من تفاعل اورثو امينو هيدرازو بنزين مع اورثو امينو فنيل ثايول وبنسبة 1:1 وشخص بواسطة التقنيات تحليل العناصر (N, H,C)، الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية كما حضرت وشخصت معقدات أملاح بعض ايونات العناصر الثنائية التكافؤ Hg,Cd, Pd, (Pb ،Zn, Cu,Ni, Co) والثلاثي التكافؤ (Cr) استخدمت تقنيات طيف الأشعة تحت الحمراء،الأشعة فوق البنفسجية – المرئية، التوصيلية الكهربائية، الامتصاصية الذرية والخواص المغناطيسية واستنتج من التحليل ان المعقدات لها شكل ثنائي السطوح حول ايون الفلز مع الليكاند (L) ثلاثي السن (N, N, N) ما عدا Pd معطيا الشكل ثنائي الهرم مثلثي.وقد تم حساب قيم  $\alpha$ ،  $k_f$  ،  $\epsilon_{max}$  للمعقدات وحساب  $\beta$  لمعقد الكوبلت.كما تم دراسة الفعالية البايولوجية لليكاند ومعقداته وقد أظهرت النتائج امتلاكها فعالية متباينة اتجاه، Staphylococcus, Pseudomonas aeruginosa, E. coli, Streptococcus pyogenes, aureus, Candida albicans.

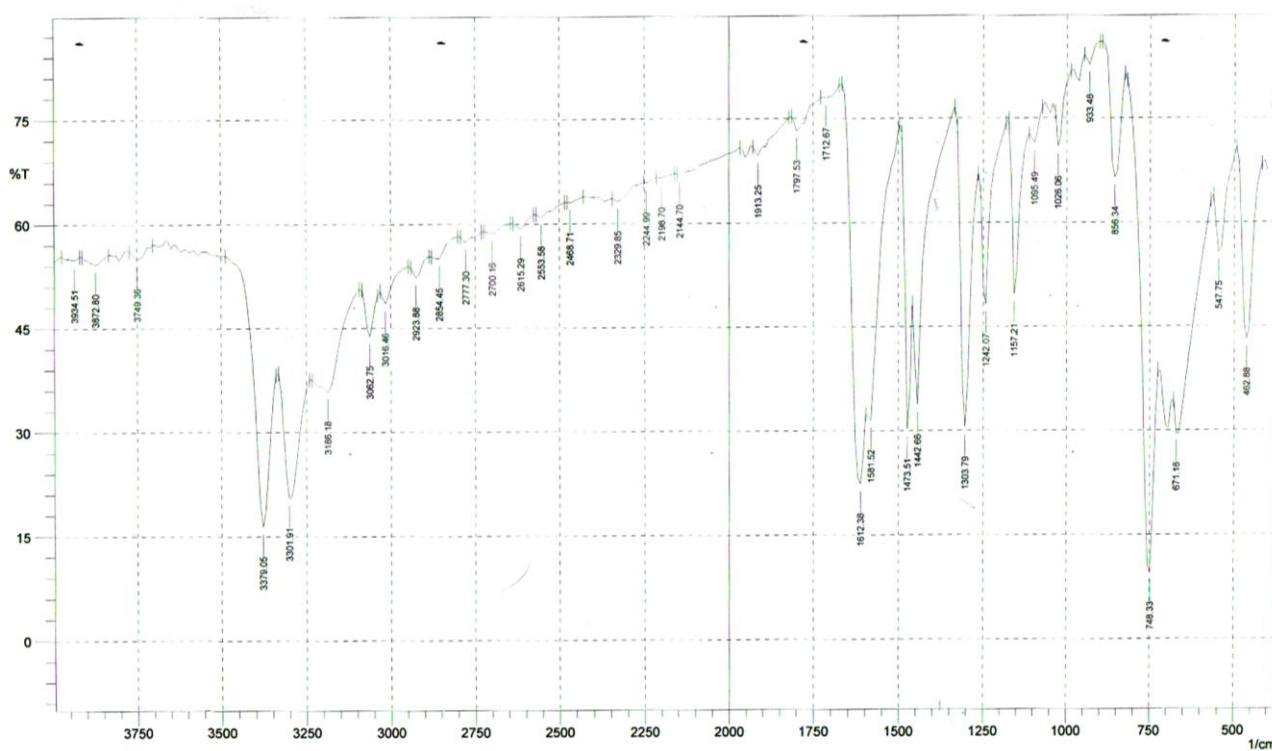


Fig.(1a) Infrared spectra of Ligand (L).

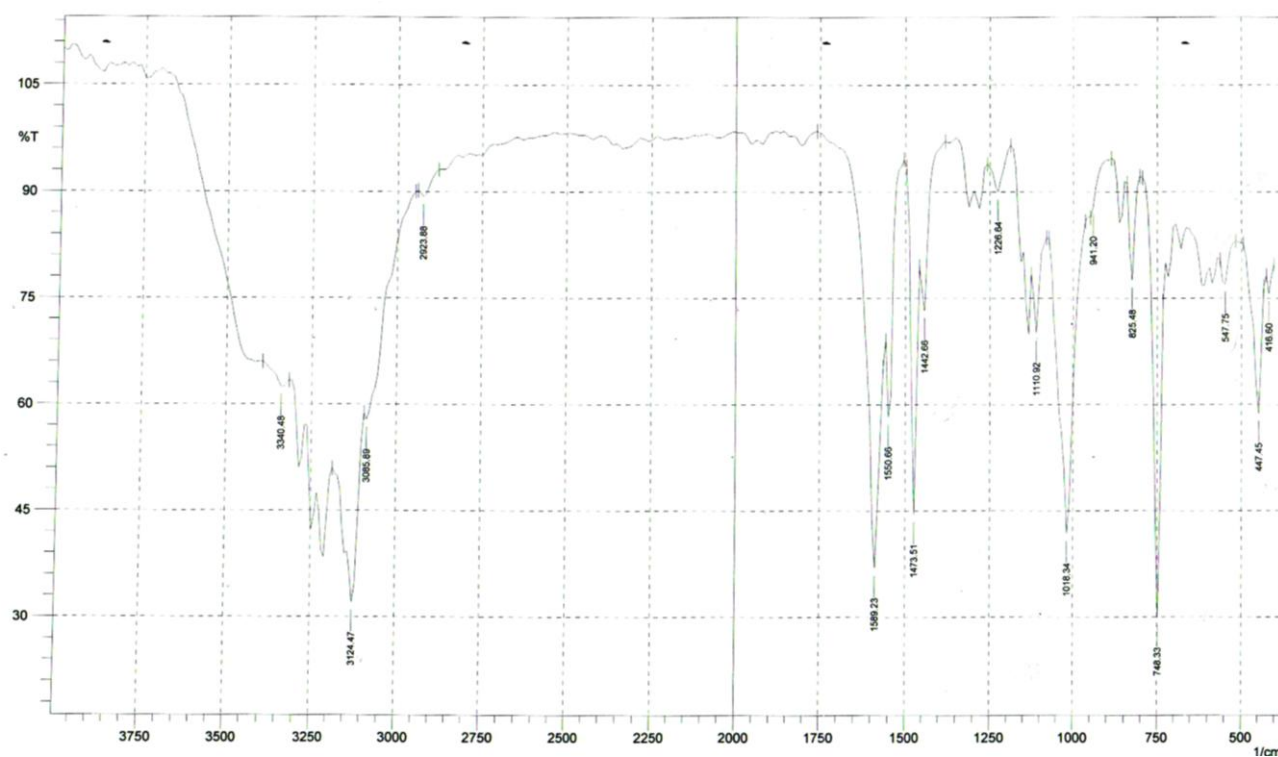
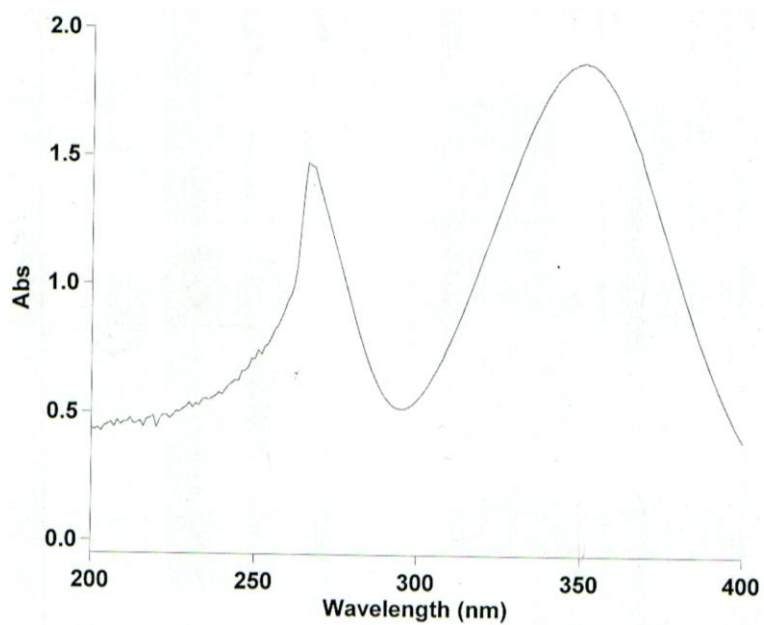
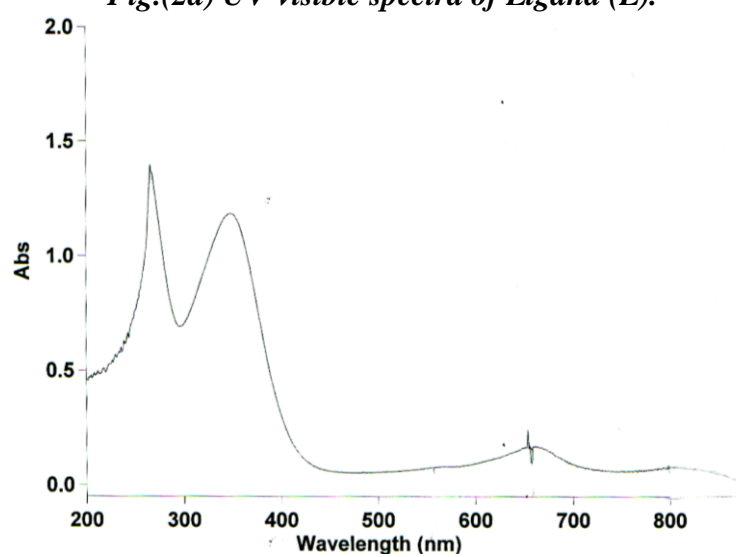


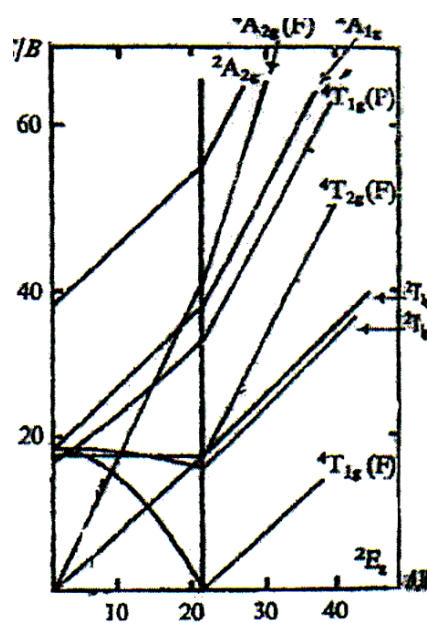
Fig.(1b) Infrared spectra of  $L.NiCl_2.H_2O$ .



*Fig.(2a) UV-visible spectra of Ligand (L).*



*Fig.(2b) UV-Visible spectra of  $L.CoCl_2.H_2O$ .*



*Fig.(3) Tanabe-Sugano diagram for the  $Co^{+2}$  ion in octahedral complexes.*