Synthesis, Spectroscopic and Biological Studies of a New Some Complexes with N-Pyridin-2-Ylmethyl-Benzene-1,2-Diamine

Shaymaa H. Naji
Lekaa K. Abdul Karim
Falih H. Mousa
Dept. of Chemistry/College of Education For Pure Science(Ibn Al- Haitham)/
University of Baghdad

Received in :23 November 2011 , Accepted in 12 February 2012

Abstract

N-Pyridin-2-ylmethyl-benzene-1,2-diamine (L) was prepared from the reaction of ortho amino phenyl thiol with 2 – amino methyl pyridine in mole ratio (1:1) . It was characterized by elemental analysis (C.H.N) , FT-IR , Uv – Vis , ^{1}H , ^{13}C-N.M.R . The complexes of the bivalent ions (Co , Ni , Cu ,Pd , Cd , Hg and Pb) and the trivalent (Cr) have been prepared and characterized too . The structural was established by elemental analysis (C.H.N) , FT-IR , Uv – Vis spectra , conductivity measurements , atomic absorption and magnetic susceptibility . The complexes showed characteristic behavior of octahedral geometry around the metal ions and the (N,N,N) ligand coordinated in tridentat mode except with Pd complexes showed sequare planer . α ,Kf,εmax for the complexes were estimated too. β for Co – complex was calculated . The study of biological activity of the ligand (L) and its complexes showed various activity toward *staphylococcus aureus* and *E.coli*.

Key words : Synthesis ,Phenyl diamine , Complexes , Biological study , Bivalent ions.

Introduction

The macrocyclic compounds types N3 are considered to be a good coordinated ligands because involved hard nitrogen atoms as well as Co^{II} , Cu^{II}[1,2] ,more over a greatest importance of tridentate ligands type N3 with variety substituents on the molecule and their complexes with metals , Mg^{II} and Ca^{II} in the metallic enzyme, blood protein[3] which contains cobalt ion and in the chlorophyll which contains Mg^{II}[4]. Recently complexes containing macrocyclic ligand type N3 donor atoms play a very important role in biological system such as Ca^{II}, Fe^{II} and Pd^{II} complexes[5] and high stable complexes of this type ligand with Tc, Re are used for radiopharmaceuticals applications[6,7] and in magnetic resonance imaging[8] ,also with Ni^{II} used as a catalyst for division DNA molecules and Fe^{II} complex with porphyrin as a model for biological proteins such as hemoglobin in blood[9].

This paper reports the synthesis and characterization of new ligand derived from the reaction of ortho amino phenyl thiol with 2 – amino methyl pyridine, the new ligand complexes with (Cr^{III},Co^{II},Ni^{II},Cu^{II},Pd^{II},Cd^{II},Hg^{II} and Pb^{II}) were prepared too.

Experimental

a. **Chemicals ;**

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

b. **Materials** : ortho amino phenyl thiol (C6H7NS),2 – amino methyl pyridine ($C_6N_2H_8$) , cobalt chloride hexa hydrate (CoCl2.6H2O), Nickel chloride hexa hydrate (NiCl2.6H2O), copper chloride dihydrate (CuCl2.2H2O), Paladium chloride (PdCl2) ,Cadimum chloride dihydrate (CdCl2.2H2O) , Mercury chloride (HgCl2), Lead nitrate Pb(NO3)2 , Chromium trichloride hexa hydrate (CrCl3.6H2O) , Ethanol 99% (CH3CH2OH) , Dimethyl sulphoxide 99.5% (DMSO) , carbon tetra chloride 99.5% (CCl4) , chloroform 99% (CHCl3) and Toluene 99% (C7H8) .

c. **Instruments :**

-Elemental analysis for the new ligand (L) and complexes were determined by calibration type ;
Linear Regression Euro EA Elemental Analysis were made in AL-Mustansiriyah University and Ministry of Science and Technology .
-Melting points were determined by Gallen –Kamp apparatus .
-'H,^{13}C-NMR spectra were recorded in DMSO using BurKer 300 MHZ spectro meter at the AL-Albeit University Amman –Jordan .
- FT-IR spectra were recorded as KBr discs in the range (4000 - 400)cm^{-1} using Shimadzu-FT-IR .
-UV-Visible spectra were recorded by Shimad Zu-UV-Vis -160 A Ultra Violet spectrophotometer at $25c^{o}$,using 1 cm quartz cell and examined at the range of (200 -1100)nm at 10^{-3} M in Dimethyl sulphoxide.
-Atomic Absorption (A .A) technique have been measured using ashimad Zu AA 680 G atomic absorption spectro photo meter in center of Ibn –Cina .
-Molar conductivity of the complexes were measured on pw 9526 digital conductivity in Dimethyl sulphoxide at 10^{-3}M .
-Magnetic susceptibility were recoded by Magnetic susceptibility blance ,model ,MsB-MK1 in AL–Nahrain University .

Preparation

Synthesis of N-Pyridin-2-ylmethyl-benzene-1,2-diamine (L)

Orthoaminophenyl thiol (0.125gm , 1mmol) and 2-amino methyl pyridine (0.10 gm , 1 mmol)in ethanol (10 cm^3) was stirred for 3 hours . The precipitate was filtered and recrystallized from hot absolute ethanol , fine yellow crystals were obtained ,m.p 83c^{o} ,yield 85% and it is soluble in most organic solvents as indicated in Table (1) .

Synthesis of the metal complexes

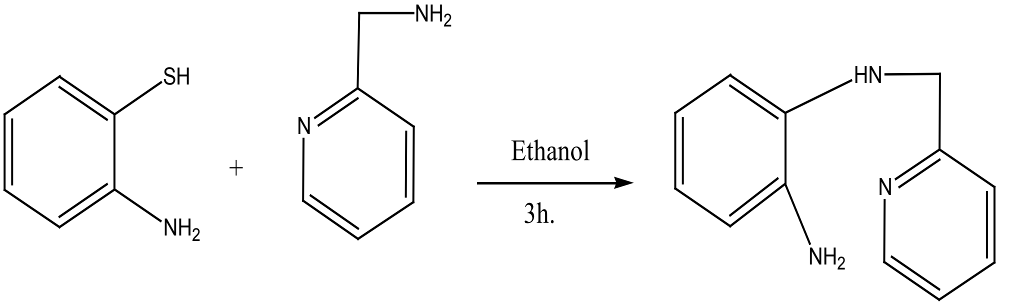
An ethanolic solution of the following metal salts $Crcl_3.6H_2O$(0.26gm,1mmol), $CoCl_2.6H_2O$ (0.24gm,1mmol), $NiCl_2.6H_2O$ (0.24gm,1mmol), $CuCl_2.2H_2O$ (0.1gm,1mmol), $PdCl_2$(0.17gm,1mmol), $HgCl_2$(0.27gm,1mmol) and $Pb(NO_3)_2$(0.33gm,1mmol) were added to a solution of the ligand (L) (0.302 gm,1mmol) in ethanol .

The mixture was stirred for 30min. The precipitate formed was filtered , washed with distilled water and dried under vacuum. Physical properties were given in (Table1) .

Results and Discussion

Synthesis and Physical properties of the ligand N-Pyridin-2-ylmethyl-benzene-1,2-diamine (L) .

The ligand was obtained by the reaction of one mole of ortho amino phenyl thiol with one mole of 2 – amino methyl pyridine ;



Orthoaminophenyl thiol   2-(amino methyl) pyridine   *N*-Pyridin-2-ylmethyl-benzene-1,2-diamine

Elemental analysis (C.H.N) confirmed the purity of the ligand(L) with the formula C12H13N3 .

The ^{1}H,^{13}C – NMR spectrum of the new ligand

The ^{1}H-NMR spectrum of the new ligand in DMSO(d^{6}) at ambient temperature exhibit peak at 5.471ppm indicate the existence of amino group[10]. Other peaks around 6.42 – 7.11 ppm are related to aromatic hydrogens[11,12]. The^{13}C – NMR spectrum showed two singlets at δ 133 and δ153 correspond to the carbons of the benzene ring that do not bear hydrogens .This leaves the signals δ117 and δ137 for carbon have one hydrogen.Singlet at δ 40 due to(– CH2 –) . Fig (1,2) showed chemical shifts δ(PPm) of the ligand (L) **.**

The infrared spectrum of the ligand(L)

The infrared spectrum of the ligand(L) in the solid state does not contain the(S-H) which appears in the starting material ortho amino phenyl thiol at (2600– 2500) cm^{-1} region .This indicates the displacement of SH in ortho amino phenyl thiol by means of (– NH – CH2 –) . Bands in the (3380,3290)cm^{-1}, (1585) cm^{-1} and (1303)cm^{-1} are diagnostic of the primary aromatic amine[13,14].Fig(3)The infrared spectra of all complexes showed ,the multi bands in the range (3460 – 3282)cm^{-1} and split or broader band at (1604 – 1558) cm^{-1}and (1303)cm^{-1} with the formation of a new absorption bands for the coordination through nitrogen atom[15,16] .(M – N) stretching vibration have been assigned in Table(2) . The bands characteristic of coordinated water and (M – O) are seen in complexes in the region (837 -

817)$cm^{-1}$and (563-545)cm^{-1}[17].(Fig4). The pb complex spectrum exhibited bands at (933)cm^{-1},(663)cm^{-1} due to coordinate nitrate ion[18,19].(Table 2) describes characteristic stretching vibration frequencies (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 (267nm,37453cm^{-1}) and (313nm,31948cm^{-1}), (353nm ,28328cm^{-1}).Which are due to the $\pi - \pi^*$ and $n - \pi^*$ transition[20].Fig(5)

The UV visible spectrum of Cr^{+3} complex showed one band in the region (580nm ,17241 cm^{-1})is due to $^4A2\,g \longrightarrow {}^4T2\,g$[21] .

The UV –visible spectrum of Co^{+2} complex showed bands in the region (800nm ,12500 cm^{-1})due to $^4T1\,g \longrightarrow {}^4T_{2g(F)}$,(660 nm ,15151 cm^{-1} due to $^4T1\,g \longrightarrow {}^4A2\,g$ and (520nm ,19230 cm^{-1}) due to $^4T1\,g(F) \longrightarrow {}^4T1\,g(p)$[22,23] .

The ratio of 19230cm^{-1} to 12500cm^{-1} is 1.54 which fits with Tanaba –Sugano d^7 Curve (Fig6) for E/B=26 and $\Delta o/B'=16$. $\Delta o=12500cm^{-1}$

B' complex = 12500/16 = 781cm^{-1}

B for free Co^{+2}=971cm^{-1}

β(nephelavetic effect) = B' complex / B Co^{+2} = 781/971 = 0.804

which indicates of d – electron delocalization on the ligand hence a significant covalent character in the complex[24] .

The UV–Visible spectrum of Ni^{+2} showed one band at (825nm,12121cm^{-1}) which is due to $^3A2g \longrightarrow {}^3T1g$ .Fig(7)

Cu^{+2} complex appeared band at (660nm,15151cm^{-1}) is due to $^2Eg \longrightarrow {}^2T2g$. The brown palladium complex exhibited a strong band at (400nm ,25000 cm^{-1}) due to $^1A_{1g} \longrightarrow {}^1B_{1g}$ transition[25] . The UV – Visible spectra of Cd^{+2},Hg^{+2} and Pb^{+2} complexes showed shifted bands compared with free ligand(L) are due to charge transfer (Table3) .

The molar conductance of all complexes in DMSO were found to be low which suggested coordination of anion to the metal[21] .

The μeff value of Cr,Co, Ni , Cu complexes are within the range (3.77,4.82 ,3.01,1.98) respectively expected for spin–free octahedral structures[26,27] . Electronic spectra , conductance in (DMSO) magnetic moment (B.M) of the ligand complexes are given in Table(3) .

Study of Cr^{+3},Co^{+2},Ni^{+2},Cu^{+2}complexes formation in solution

The complexes of the ligand(L) with selected metal ions (Cr^{+3},Co^{+2},Ni^{+2},Cu^{+2}) were studied in solution using ethanol as solvent , in order to determine (M:L) ratio in the prepared complexes , following molar ratio method[28] . A series of solution were prepared having a constant concentration (C) 10^{-3}M of the hydrated metal salts and the ligand(L) . The (M:L) ratio was determined from the relation ship between the absorption of the observed light and the mole ratio (M:L) found to be (1:1) for all complexes .

The results of complexes formation in solution are shown in (Table4,5,6,7)

Vm = volume of metal in ml
VL = volume of ligand in ml

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

$Kf = 1- \alpha / \alpha^2 c$ …… (1)

$\alpha = Am-As /Am$………(2)

(α) is the degree of the dissociation , (c) is the concentration of the complex (10^{-3}M) . (As) and (Am) are the absorbance value of the partially and fully formed complex respectively (Table8) .

The absorbance of the solutions were measured at (λmax) of the maximum absorption . The molar absorptivity (εmax)(eq.3) has been calculated using equation;

$A = \varepsilon max . b . c$ ……. (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 the molar ratio calculation of [metal : ligand](M:L) for the synthesis of complexes as well . The results showed a ratio M:L (1:1) for all complexes (Table 1)

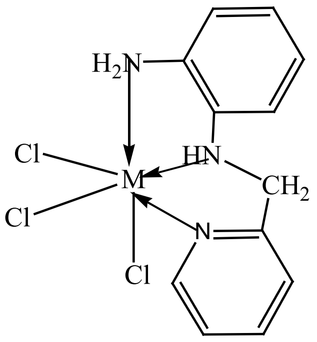
Biological activity study

The biological activity of the prepared new ligand and its complexes were studied against selected types of micro organisums which include gram positive bacteria like staphylococcus aureues and gram negative bacteria like E.coli in agar diffusion method , which is used (DMSO) as asolvent . Agar diffusion method involves the exposure of the zone of inhibition toward the diffusion of micro organisms on agar plate . The plates were in cubated for (24) hrs. at ($37c^o$) The zone of inhibition of bacterial grouth around the disc was observed (Table9).

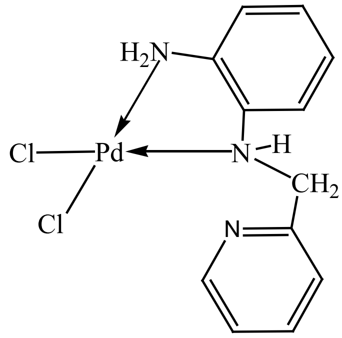
Conclusion

A series of complexes of Cr^{+3} ,Co^{+2}, Ni^{+2} ,Cu^{+2} ,Pd^{+2} ,Cd^{+2} ,Hg^{+2} ,Pb^{+2} with N-Pyridin-2-ylmethyl-benzene-1,2-diamine (L)have been prepared and characterized .

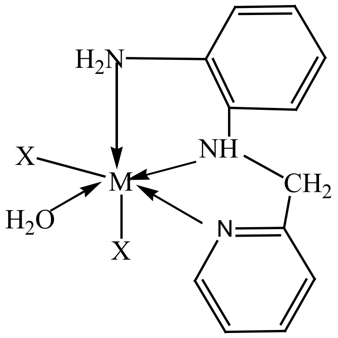
The tridentate ligand (L)(N,N,N)is binding metal ion forming octahedral structure except with pd is forming square planer as follows;



M^{+3} =Cr



M^{+2} =Pd



M^{+2}=Co,Ni,Cu,Cd,Hg
$X=Cl^{-1}$
M^{+2}=Pb
$X=NO3^{-1}$

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 .

Refrences

1. Zolotov , Y.A.(1979)Macrocyclic compounds in Analytical chemistry , ed.2nd, New York.
2. Handel , I.L.; Muller ,R,N. and Gnglielmeti ,R.N. (1983)metal ions , Inorg chimi.,Acta 66(8):511-514.
3. Brow ,D.G.(1973)structure and spectroscopic study ,prig – Inorg. Chem. 13(8):177-180 .

4. Warren , M.J. and Shah,H.N. (2000) biological study , Biolog. Chem. 221(21):421-430

5. Kimura , E.; Koike,T. ;Watanabe ,T.; Aoki , S. and Shiro , M.(1996) The modern structural theory of organic chemistry , J. Am.chem. 14(112):210-217 .

6. Naji, S.H.; Fathel ,H.A. and Musa ,F.H. (2008) Synthesis characterization of some metal salts with 1,1' bis – (ortho amino phenyl thio) – methane , J.Al-Mustansiriya Sci.19(12):59-62 .

7. Naji,S.H.(2008)synthesis and spectral studies of transition metals (CoII,NiII,CuII,CdII,HgII,PbII) with aniline – 2 – thio methylene chloride complexes ,J.Al - Mustansiriya Sci.19(11):53-56 .

8. Naji , S.H. and Musa ,F. H. (2010)synthesis ,spectroscopic and Biological studies of some metal complexes with 1,2 bis – (ortho amino phenyl thio) ethane , J. Al – Mustansiriya Sci.21(3):35-50 .

9. Naji , S.H. (2011) Synthesis , Spectroscopic and biological studies of some metal complexes with ortho amino hydrazo benzene , J. Baghdad . Sci . 8(2):375-387 .

10. Manhel , R.A. (2010) synthesis of pheyl alanine Schiff Base ligand and its complexes with some metal (II) ions and Evalulation of their Anti bacterial Activity , J.Al – Mustansiriya Sci. 21(3):65 – 78 .

11. Bellamy , L.J. (1975)The infrared spectra of complex molecules , Hasted press , Division of John Willey and sons , Inc.,ed. 3rd . New York .

12. Cooper , J.W. (1980) spectroscopic Techniques for organic chemistry , John Willey and sons , ed. 2nd ., New York .

13. Keeton , M. N. ; and Lever . A.B.P. (1971) Four , Five ,and Six coordinate metalcomplexes of di(2 pyridyl) disulphide and 1, 2 –di (2` – pyridyl) ethane , Inorg. Chem. 10 (5):49 -52.

14. AL–Obadi , N. J.; Abid ,K. K. and AL–Naimi , Y. J. (2006) Transition metal complexes of 2 – (thiosemicarbazino) – 5 – (o – hydroxyl phenyl) – 1 , 3 , 4 –oxadiazole , J. Tikrit . Sci. 11(24):164–167.

15. Saha , B. G. and Banerji , S. K. (1982) Synthesis and characterization of complexes , J. Indian Chem., Soc, 1(5): 928 – 932 .

16. Bahel , S.C. (1982) Synthesis and structural studies of complexes of Zn(II),Ni(II),and Co(II) with 3 –Aryloxymethyl 4 – aryl – 5 mercapto – 1 , 2 , 4 – triazoles , J. Indian .1(3):1127 – 1135 .

17. Iskender , M. F.; El –Sayed , L.; Hefny ,A. F. and Zayan . S. E. (1976) structural studies of some metal ions ; J. Inorg Chem, 38 (1):13 – 19 .

18. Greay ,W. G. (1970)Coordination Chemistry reviews , Elsevier publishing company , ed. 3rd ., Amsterdam .

19. Clark , R. J. H. and Erring ton . W. R. (1966)Sulpher donor complexes , Inorg . Chem., 5(2):650–660 .

20. Kadeoka , W. N. (1976) Crystal and Molecular structure of Dichloro bis (2 – pyridyl) disulphide cobalt(II) , Inorg Chem. 15 (12):812 – 820 .

21. Foresman , J. C. J. and Frisch, C . A. (1996) Exploring chem , With Elecronic structure methods , Gaussian – Inc , ed. 2nd ., Pittsuburgh .

22. Rudolph , W. K. (1965) Mixed chelates from thio picolinamides and β – diketones , Inorg Chem. 4 (11):1047 – 1055 .

23. El – Asmy , A. A. (1990) Structural studies on Cd(II) , Co (II) , Cu (II) , Ni (II) and Zn (II) complexes of 1 – malonyl – bis (4-phenyl thio semicarbazide) ,Transition Met. Chem. 4 (5): 12

24. Sutton , D.L. (1968) Electronic Spectra of Transition metal complexes , Mc Graw – Hill , ed.1st ., London .

25. Gajendragad , M.R. and Agarwala. U.A. (1975) Synthesis and characterization of complexes , J. Inorg Nucl. Chem. 37(11):29 – 35 .

26. Figgis , B. N. (1966)Introduction to ligand Fields , John – Wiley and sons , Inc., ed. 2nd ., New York .

27. Figgis , B. N. and Lewis. J.N(1967) Modern Coordination Chemistry,eddited by Lewis and Wilkins R. G. ; Interscience , ed. 3rd. , New York .

28. Dubey , S. N. and Kaushik . B. N.(1985) Triazoles copmplexing Agent , Indian J.Chem. 24(8):950-959.

Table (1):Color , melting point , yield , metal analysis and solubility for the ligand(L) and its complexes

No.	compound	no. of mole and gm	Color	m. p. c° or (dec)	Yield %	Metal analysis found (calculated)				Solubility
						C %	H %	N %	M %	
1	(L) $C_{12}H_{13}N_3$	1.00 0.19	yellow	83	85	72.29 (72.361)	6.531 (6.532)	21.230 (21.105)	—	ETOH , CCl4 , CH Cl3 ,C7H8, DMF,DMSO
2	L-CrC$_{l3}$	1.00 0.26	green	152	72	40.253 (40.279)	3.549 (3.636)	11.725 (11.748)	14.923 (14.545)	DMF , DMSO
3	L-CoC$_{l2}$.H_2O	1.00 0.24	Dark blue	204 (dec)	63	41.508 (41.498)	4.213 (4.322)	12.110 (12.103)	—	DMF , DMSO
4	L-NiC$_{l2}$.H_2O	1.00 0.24	Light green	184	74	41.483 (41.534)	4.421 (4.326)	12.108 (12.114)	—	DMF , DMSO
5	L-CuC$_{l2}$.H_2O	1.00 0.17	blue	174	81	—	—	—	18.678 (18.088)	DMF , DMSO
6	L-PdC$_{l2}$	1.00 0.17	Brown	210 (dec)	92	38.241 (38.257)	3.395 (3.453)	11.098 (11.158)	28.023 (28.267)	DMF , DMSO
7	L-CdC$_{l2}$.H_2O	1.00 0.27	Yellow	202 (dec)	88	—	—	—	27.987 (28.071)	DMF , DMSO
8	L-HgC$_{l2}$.H_2O	1.00 0.33	Violet	192	87	—	—	—	42.012 (41.044)	DMF , DMSO
9	L-Pb$(NO_3)_2$.H_2O	1.00 0.33	Light yellow	222	78	26.301 (26.277)	2.715 (2.737)	12.751 (12.773)	—	DMF , DMSO

dec. = Decomposition

Table(2) : Characteristic stretching vibrational frequencies (cm^{-1}) located in the FT-IR of the ligand(L) and its complexes

No.	compound	ט (N – H) cm^{-1}	ט (C-H)cm^{-1} Ar.	ט (C-H)cm^{-1} Ali.	ט (M-N) cm^{-1}	ט (M-X) cm^{-1}	ט (M-O) ט (OH2)aq. cm^{-1}	NO_3 cm^{-1}
1	(L) C12H13N3	3380(asym)(m) 3298(sym)(m) 1612(s),1585(m) 1303(s),1246(s)	3186(m) 3066(m)	2930(asym)(w) 2877(sym)(m) 1469(s),1442(s)	—	—	—	—
2	L-CrCl3	3414(asym)(br) 3356(sym)(br) 1608(s),1546(s) 1315(s),1234(m)	3190(m) 3074(m)	2930(br) 2823(m) 1473(s),1435(s)	528(s)	410(s)	—	—
3	L-CoCl2.H2O	3414(asym)(br) 3340(sym)(br) 1604(s),1573(m) 1303(s),1249(s)	3190(w) 3066(m)	2920(m) 2880(m) 1477(s),1442(s)	515(s)	412(s)	551(s) 830(s)	—
4	L-NiCl2.H2O	3410(asym)(br) 3282(sym)(s) 1612(sh), 1593(s) 1315(m),1226(s)	3120(s) 3082(s) 3062(s)	2924(m) 2850(m) 1473(s),1442(s)	520(s)	412(s)	550(s) 829(s)	—
5	L-CuCl2.H2O	3452(asym)(br) 3352(sym)(br) 1600(s),1558(w) 1307(s),1253(s)	3190(w) 3086(m)	2940(m) 2890(w) 1492(s),1427(s)	536(s)	416(s)	551(s) 817(s)	—
6	L-PdCl2	3414(asym)(br) 3390(sym)(br) 1604(br),1558(br) 1307(m),1265(br)	3182(br) 3055(br)	2935(w) 2885(w) 1473(s),1442(s)	515(s)	415(s)	—	—
7	L-CdCl2.H2O	3379(asym)(m) 3305(sym)(m) 1608(s),1562(sh) 1307(s),1246(s)	3180(m) 3062(s)	2927(m) 2890(m)	513(s)	408(s)	545(m) 817(m)	—
8	L-HgCl2.H2O	3417(asym)(br) 3317(sym)(br) 1604(s),1570(sh) 1319(s),1249(s)	3190(m) 3059(m)	2990(w) 2893(br) 1442(w),1473(m) ,1492(m)	520(s)	410(s)	563(m) 825(m)	—
9	L-Pb(NO3)2.H2O	3460(asym)(s) 3371,3302(sym)(s) 1608(s),1566(s) 1307(s),1246(s)	3186(m) 3066(m)	2966(m) 2876(m) 1473(s),1442(s)	516(s)	405(s)	540(m) 837(m)	933(s) 663(s)

Table (3) : Electronic spectra DMSO, conductance in DMSO magnetic moment (B.M) of the ligand (L) and its complexes.

No.	compound	λ (nm) Cm $^{-1}$	Assignment Bands	Λ s. cm -1 DMSO (10^{-3}M)	μ eff (B.M)
1	(L) C12H13N3	37453(267) 31948(313) 28328(353)	$\pi - \pi^*$ $n - \pi$	——	——
2	L-CrCl3	17241(580)	$^4A2g \rightarrow {}^4T2g$	20.30	3.77
3	L-CoCl2.H2O	19230(520) 15151(660) 12500(800)	$^4T1g \rightarrow {}^4T2g(F)$ $^4T1g \rightarrow {}^4A2g$ $^4T1g \rightarrow {}^2Eg$	9.34	4.82
4	L-NiCl2.H2O	12121(825)	$^3A2g \rightarrow {}^3T1g$	11.23	3.01
5	L-CuCl2.H2O	15151(660)	$^2Eg \rightarrow {}^2T2g$	18.80	1.98
6	L-PdCl2	25000(400)	$^1A_{1g} \rightarrow {}^1B_{1g}$	8.20	——
7	L-CdCl2.H2O	27173(368)	Charge- transfer	6.15	——
8	L-HgCl2.H2O	32258(310)	Charge-transfer	9.26	——
9	L-Pb(NO3)2.H2O	28571(350)	Charge-Transfer	9.23	——

B.M = Bohr Magneton

Table (4):continuous variation slop for Cr^{+3}ion λ(368mm)

$L - Cr\ Cl_3$		
V M	VL	Abs
1 ml	0.25	0.15
1	0.50	0.21
1	0.75	0.43
1	1	0.60
1	1.25	0.58
1	1.50	0.61
1	1.75	0.61
1	2	0.62
1	2.25	0.63
1	2.5	0.61

Table (5):continuous variation slop for Co^{+2}ionλ(350mm)

$L - CoCl_2.H_2O$		
V M	VL	Abs
1 ml	0.25	0.41
1	0.50	0.74
1	0.75	1.11
1	1	1.50
1	1.25	1.48
1	1.50	1.51
1	1.75	1.49
1	2	1.46
1	2.25	1.52
1	2.5	1.51

Table (6):continuous variation slop for Ni^{+2}ion λ(348mm)

$L - NiCl_2.H_2O$		
V M	VL	Abs
1 ml	0.25	0.22
1	0.50	0.44
1	0.75	0.68
1	1	0.90
1	1.25	0.89
1	1.50	0.91
1	1.75	0.92
1	2	0.93
1	2.25	0.91
1	2.5	0.91

Table (7):continuous variation slop for Cu^{+2}ionλ(336mm)

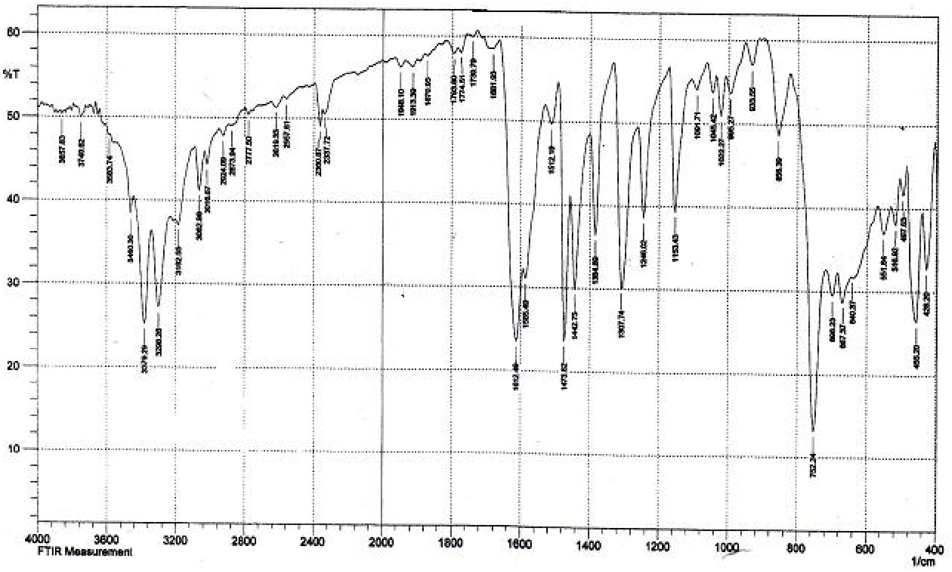
$L - Cu\ Cl_2.H_2O$		
V M	VL	Abs
1 ml	0.25	0.21
1	0.50	0.41
1	0.75	0.62
1	1	0.80
1	1.25	0.81
1	1.50	0.79
1	1.75	0.78
1	2	0.82
1	2.25	0.83
1	2.5	0.79

Table(8) : As,Am,Kf,Єmax and λmax of the Cr^{+3},Co^{+2},Ni^{+2},Cu^{+2} complexes

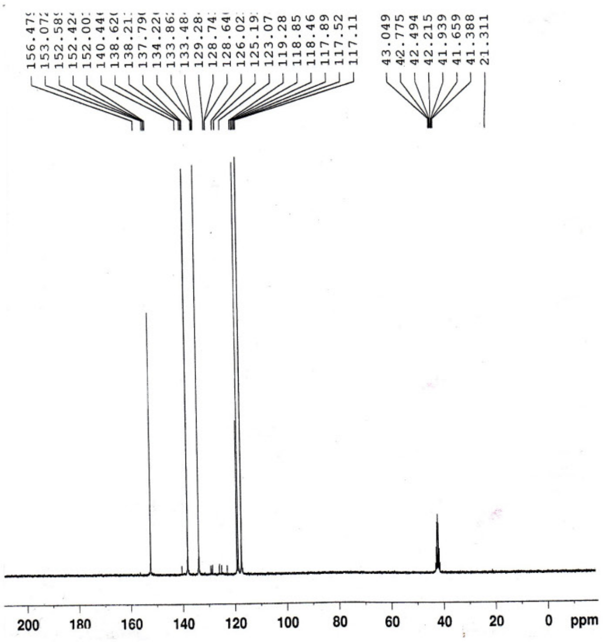
NO.	Compound	As	Am	α	Formation Constant (Kf)	Molar Absorpitivity εmax $L.mol^{-1}.cm^{-1}$	Maxג (nm)
1	Cr-complex	0.60	0.63	0.043	$4.314x\ 10^{5}$	2983	368
2	Co-complex	1.50	1.52	0.013	$5.840x10^{6}$	4702	350
3	Ni-complex	0.90	0.93	0.051	$9.462x10^{5}$	1048	348
4	Cu-complex	0.80	0.83	0.0361	$7.378x10^{5}$	2105	336

Table(9): Effect of the ligand and its complexes on gram positive and gram negative bacteria

No.	compound	Diameter of inhibition zone (mm) at concentration 1mg/ml	Diameter of inhibition zone (mm) at concentration 5mg/ml	Diameter of inhibition zone (mm) at concentration 1mg/ml	Diameter of inhibition zone (mm) at concentration 5mg/ml
		Staphylococcus aureus		E.coli	
1	(L) C12H13N3	40	50	80	35
2	L-CrCl3	30	32	10	15
3	L-CoCl2.H2O	18	20	25	26
4	L-CuCl2.H2O	14	24	30	40
5	L-PdCl2	30	40	22	25
6	L-HgCl2.H2O	22	26	18	20



(Fig 1) :^{1}H NMR spectrum of the ligand (L) .



(Fig 2) :^{13}C- NMR spectrum of the ligand (L) .

Fig. (3): Infrared spectrum of the lignd (L).

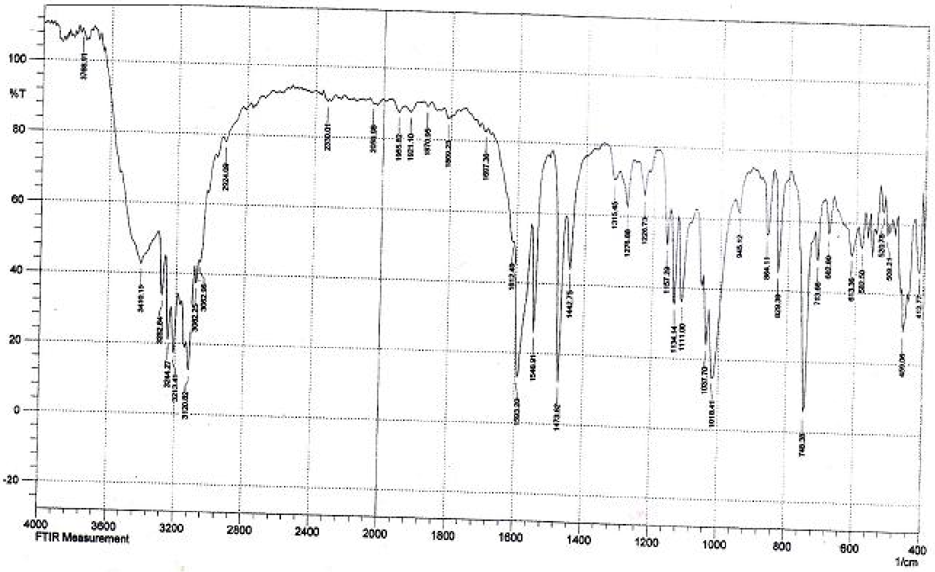


Fig. (4): Infrared spectrum of L-$NiCl_2.H_2O$.

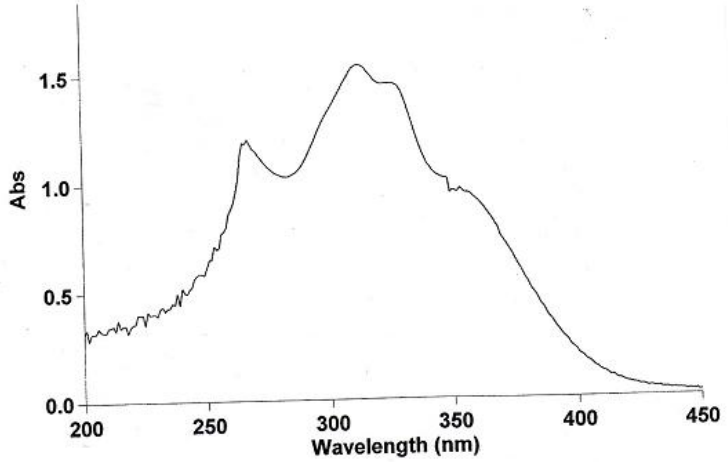


Fig. (5): UV-visible spectrum of the ligand (L).

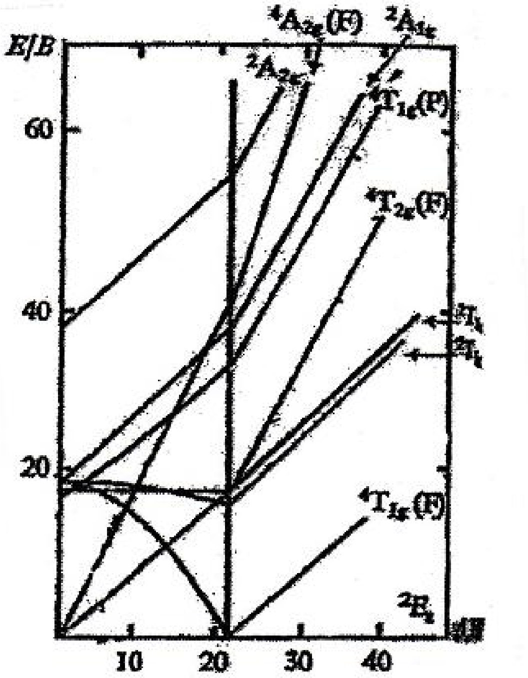


Fig. (6): Tanabe-Sugano diagram of the Co^{+2} ion in Octahedral complexes.

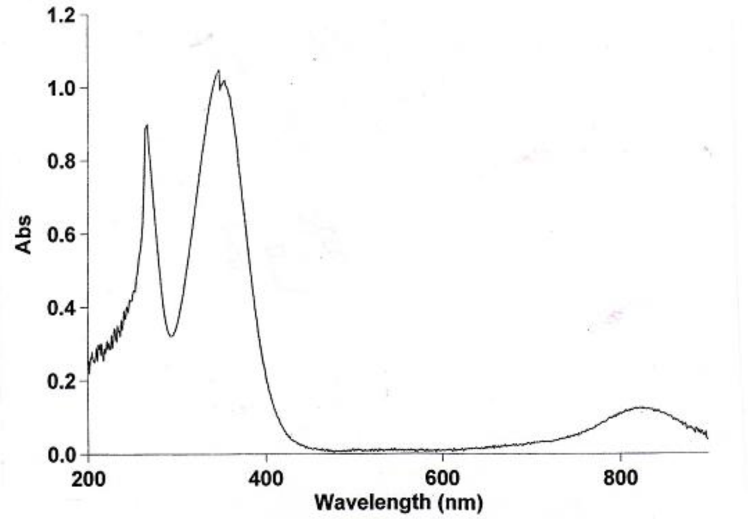


Fig. (7): UV-visible spectrum of L-$NiCl_2.H_2O$.

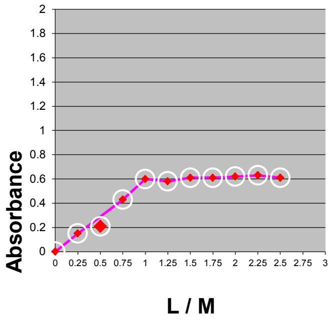


Fig.(8):The molar-ratio curve
The complex L-$CrCl_3$

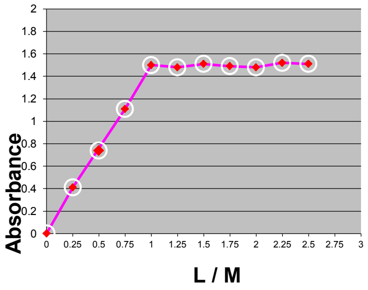


Fig.(9):The molar ratio curve
The complex L- $CoCl_2.H_2O$

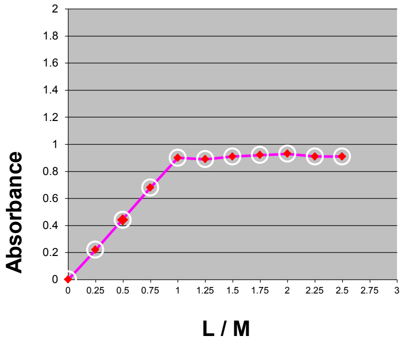


Fig.(10):The molar-ratio curve
the complex L-$NiCl_2.H_2O$

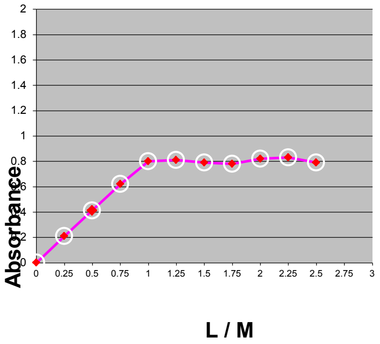


Fig.(11):The molar-ratio curve
the complex L-$CuCl_2.H_2O$

تحضير و دراسة طيفية وبايولوجية لبعض المعقدات الفلزية الجديدة مع N – بريدين -2- يل مثيل – بنزين -2,1- ثنائي امين

شيماء هادي ناجي
لقاء خالد عبدالكريم
فالح حسن موسى
قسم علوم الكيمياء / كلية التربية للعلوم الصرفة (ابن الهيثم) / جامعة بغداد

استلم البحث في : 23 تشرين الثاني 2011 ، قبل البحث في: 12 شباط 2012

الخلاصة

حضر الليكاند الجديد(L) N – بريدين -2- يل مثيل – بنزين -2,1- ثنائي امين من تفاعل اورثو أمينو فنيل ثايول مع 2 - أمينو مثيل بريدين وبنسبة 1:1 وشخص بوساطة التحليل الدقيق للعناصر (N.H.C) الأشعة تحت الحمراء ، الأشعة فوق البنفسجية –المرئية وطيف الرنين النووي المغناطيسي كما حضرت وشخصت معقدات أملاح بعض ايونات العناصر الثنائية التكافؤ (Pb,Hg,Cd, Pd , Cu , Ni ,Co) و الايون الثلاثي التكافؤ (Cr) مع الليكاند (L). استعملت تقنيات تحليل العناصر (N.H.C) ، الأشعة تحت الحمراء والأشعه فوق البنفسجية-المرئية و التوصيلية الكهربائية و الامتصاصية الذرية والحساسية المغناطيسية وأستنتج من التحاليل ان المعقدات لها شكل ثماني السطوح حول ايون الفلز مع الليكاند (L) ثلاثي السن (N,N,N) ماعدا Pd معطياً الشكل مربع مستو وقد حسبت قيم Єmax,Kf, α للمعقدات وحساب β لمعقد الكوبلت . كما درست الفعالية البايولوجية لليكاند ومعقداته وقد اظهرت النتائج امتلاكها فعالية متفاوته اتجاه انواع البكتريا *E.coli , Staphylococcus aureus* .

الكلمات المفتاحية : تحليل ، فنيل ثايول ،معقدات ، دراسة بايولوجية ،ايونات ثنائية التكافؤ