Metal (II) Complexes with Tridentate N, N,O Ligand: Synthesis, Characterization and Biological Studies

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Abstract

The preparation of some new coordination compounds for nikel (II), manganese (II), copper (II), cobalt (II)and mercury (II), with ligand obtained from Benzoinand2-amino pyridine.The ligand[6-(2-hydroxy-1,2-diphenylethylideneamino)pyridin-3-ylium)](L) was made from reactin ethanol with metal salts in (1:1)(metal : ligand)ratio.[MLCl] was the inclusive formula of the complexes where M= Mn(II),Co(II),Ni(II),Cu(II) and Hg(II). Metal analysis by electronic spectra, atomic absorption ,infrared spectra, ^{1}H&^{13}C-NMR(only ligand)spectral studies, magnetic moment and molar conductance measurements used to describe the compounds.The determinations indicated that the ligand coordinates with the metal (II) ion in neutral tridentate manner through the azomethine nitrogen atom, nitrogen atom for pyridine and oxygen atom of the benzoin, all the studies reveal coordination four for the metals in all the complexes. Tetrahedral and square planar structures were suggested for metal complexes.

Key words: Schiff base, 2-amino pyridine, Characterization, Metal chelate complexes.

Introduction

Amino compounds as heterocyclic containing one or more possibilitygiver centers took asignificant assignment in the study of reaction of atridentate Schiff base system [1]. They are recognized to act ligands as bidentate or tridentate when chelated to metalion [2]. Schiff bases are a grade of significant compounds due to their broad domain of enforcements and qualities [3].Schiff base and its complexes have presented with a lot notice because of their use as models for biological arrangement and applications as i.e. in the areas of catalysis clinical application[4], pharmacologically and photochemical[5].Schiff bases are reported to possess antibacterial[6],antifungal[7],anticancer[8],herbicidal anticonvulsant [9] and diuretic activities[10].These ligands have ONO giver atom group were attracted the interest of many authors are well recognized to coordinate with various metal ions and this has [11].

In this study, Schiff base of 2-aminopyridine with benzoin has been synthesized,characterized and antibacterial activity study of the new complexes were prepared.Further more, the structures of prepared compounds were definite by spectral studies.The arrangement of the compounds is displayed in scheme(1) and(2).

Experimental

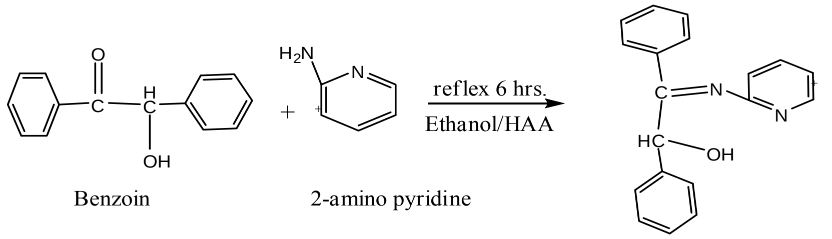
2-amino pyridine and Benzoin were purchased from Sigma Chemical Co. (USA). Glacial acetic acidreagent from" (Aldrich and Sigma)".The all metal salts used were gave from (BDH).

Instrumentation

Melting point were determined on "Gallenkamp melting point Apparatus ".Elemental microanalysis C.H.N. were carried out using "Euro Vector EA 3000 A Analysis".FT-IR measurements were recorded on "Shimadzu- 8300 Spectrophotometer". Electronic spectra were recorded using "U.V-Vis. spectrophotometer type CECIL" .^{1}H and^{13}C-NMR spectra were recorded by using a Bruker 300 MHZ (Switzerland),Chemical shift . Conductivity measurements were obtained from WTW conductivity meter .The chloride content determined using potentiometric titration method on "686–Titro Processor –665 Dosim A–Metrohm/Swiss".rature. Atomic Absorptionof complexeswere obtained from a shimadzu PR-5.ORAPHIC PRINTER atomic spectrophotometer".

Synthesis of [6-(2-hydroxy-1,2-diphenylethylideneamino)pyridin-3-ylium)] (L)

The [L]was prepared according tothe method published in literature [1]scheme (1)An ethanolic solution of (30 mL) 2-aminopyridine (0.094g, 0.001 mmole) was added to an ethanolic solution of benzoin (0.21, 0.01 mmole)and three drops from glacial acetic acid. The resultant mixture was refluxed for *ca.* 6 h. The solid product formed was filtered and recrystallized from ethanol [2]



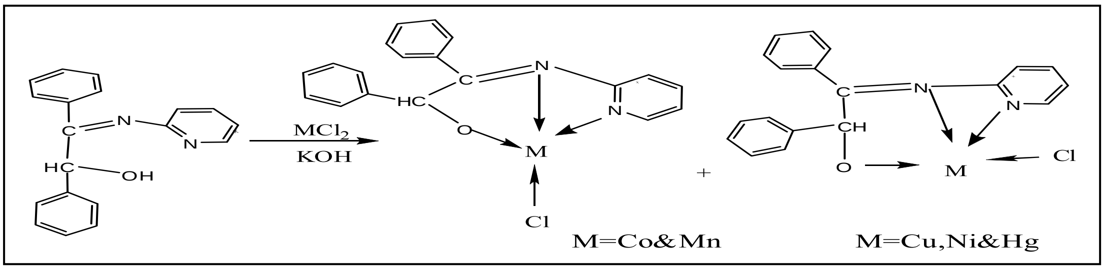
Benzoin

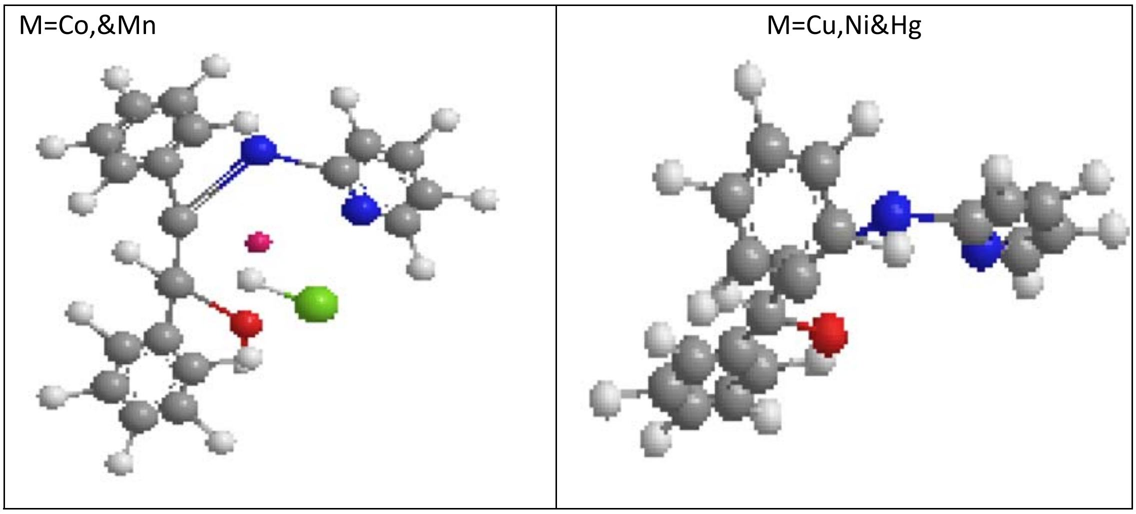
2-amino pyridine

(*Z*)-6-(2-hydroxy-1,2-diphenylethylideneamino)pyridin-3-ylium

Synthesis of M(II) Complexes

A solution of metal (II) chlorides in ethanol (2mmole) was stirred with ethanolic solution of the Schiff base (2mmole)and three drops from KOH solution, for ca. 2 h on a magnetic stirrer at 50°C. The solid complex precipitated was filtered off and washed thoroughly with ethanol and dried in vacuous, recrystallized from a hot of (10mL) ethanol, a coloured precipitate was formed [5, 6].The physical properties of complexes were listed in Table (1).





Results and Discussion

NMR spectrum

The ^{1}H NMR spectrum shows signals at: 1.15 ppm singlet for 1H benzoin H atome; singlet at 2.50 ppm due to DMSO solvent ;singlet signal at 3.385 for 1H hydroxylic proton ,the remaining [bands multiplied] from 5.6 to 8.2 ppm belong to the three aromatic ring protons that overlap with each other [10].

The ^{13}C NMR spectrum: in the spectrum the 40.8 ppm signal is due to the solvent C atoms of DMSO; 73.2 attributed to C-OHmoiety; the aromatic carbon peaks appeared in the range from 124.43 to 149.88 ppm ,finely the two peaks above 160 ppm, returned to the two sp^2 Carbone atom that linked with Nitrogen , the firs are for Schiff base band and the other one returns to α –imine pyridine [12].

The IR spectra for the ligand and its complexes

The important IR absorption bands for the synthesized Schiff bases are listed. In the L, the band shows at 1604 cm^{-1}that it was assigned to the *v*(C=N) stretching mode. In complexes this band was shifted to lower regions, 1598cm^{-1}, 1593cm^{-1},1591cm^{-1}1595cm^{-1}, and 1596cm^{-1} for Mn(II),Co(II),Ni(II),Cu(II) and Hg(II)complexes respectively, suggesting the coordination of azomethine nitrogen to metal atom in complexation [13].The (IR) spectra of the complexes showed band at the range(1576-1581) cm^{-1}were assigned to υ(C=N) groups for the pyridine ring in 2-amino pyridine,which shifted the frequency to a lower in comparison with that of Schiff base was showed at (1588)cm^{-1},when coordination with the metal ion referring to share of two nitrogen's*v*(-C=N).The bands show at 1531 cm^{-1} and 1498 cm^{-1}were assigned to the *v*(C=C) stretching mode in the ligand.These bands prove the presence of benzene. IR spectrum of the ligand showed a board band at 3344 cm^{-1} due to *v* (OH) stretching mode in the ligand [14]. This band was absent in the spectra of complexes, they indicate the disappearing of the alcoholic proton on complexation and involvement of alcoholic anionic oxygen in coordination [15]. Moreover, the strong band at1187 cm^{-1}is due to *v*(C-O) (alcoholic) in the ligand. Amedium band corresponding to pyridine nitrogen *v*(C-N) was observed at 1253 cm^{-1}[16,17].The bands arising due to vibration *v*(C-O) mode at 1187 cm^{-1}and the *v*(C-N) frequency in the free ligand at 1253 cm^{-1}were observed to be shifted to higher or lower frequencies, at 1240–1257 cm^{-1} and 1183–1198 cm^{-1}.Alsothe bands show at(582-547) ,(460-482) due to *v*(M-O) and *v*(M-N), respectively.In the metal complexes indicating the involvement of nitrogen of pyridine ring and oxygen of the alcoholic group in the complex formation [18-19].

The UVSpectral Studies

The Cu(II), Co(II), Ni(II), Mn(II) and Hg(II) complexes of ligand L were subjected to their UV-Visible spectral studies at room temperature in the range of 200–1100nm by using DMF as a solvent, and the data of the ligand (L) were recording Table (5).

The U.V-Vis spectrum of Schiff base, Fig.(3) in Table (5)displayed two absorptions. The first absorption at (280)nm (35714cm^{-1}) may be assigned to π–σ* transition. The second peak at (312) nm (32051cm^{-1}) was attributed to n–π* electronic transition [20].

The electronic spectrum of the Co (II)complex showed two peaks, the first peak (331)nm (30211cm^{-1})which was assigned to C.T transition.The second peak at (611) nm (16366) cm^{1} was allocated to(d-d) electronic transition type$^{4}A_{2\,(F)} \rightarrow {}^{4}T_{1\,(P)}$.(5.71) B.M. is a magnetic sensibility of this complex. [21] It has been a tetrahedral environment [22].

The U.V-Vis spectrum of the Ni (II)complex displayed two peaks, the first peak (332) nm (30120cm^{-1}) which was assigned to C Ttransition.The second peak at (770) nm (12987cm^{-1})was allocated to(d-d) electronic transition type$^{1}A_{1}g_{(F)} \rightarrow {}^{2}Eg$ .It has been asquare planar configuration. Diamagnetic is amagnetic sensibility of this complex[23].

The U.V-Vis spectrum of the Copper (II)complex showed two peaks, the first peak(327)nm (30581cm^{-1})which was assigned to C.T. transition.The second peak at(543)nm(18416cm^{-1})was referred to (d-d) transition type$^{1}B_{1}g \rightarrow {}^{2}A_{1g}$. 1.76B.M is a magnetic moment.It has been asquare planar configuration of this complex.[24].

While the absorptions of manganese (II)complex showed three bands,the first bond (335)nm (29650cm^{-1})which was assigned to C Ttransition.The second and third bonds at 453nm (12903cm^{-1}) and 586 nm (22075cm^{-1}) which have been assigned to$^{6}A_{1} \rightarrow {}^{4}A_{1\,(G)}$ and $^{6}A_{1} \rightarrow {}^{4}E_{(G)}$ transitions respectively. 5.75B.M. is amagnetic sensibility of this complex.It has been a tetrahedral configuration [25].

The complex mercury (II) is diamagnetic giving two peaks at 334 nm (29940cm^{-1}) and 410 nm (24390cm^{-1}) were allocated to C T transitions. The peaks disappear in the visible region. It has been a tetrahedral configuration[26].

Biological Activities

Tridentate Schiff base ligand derived by the condensation2-amino pyridine with benzoin and its complexes showed biological activities against the type of bacterial(*Bacillus*) except[Cu(L)Cl].[27 and 28].The results indicate that the complexes showed more activity than the ligand under similar experimental conditions, table (6) DMF (which was used as a solvent and Controller) was a good solvent to prepare 1ppm of each tested sample .All compounds ligand and its complexes have good biological activity against the type of bacterial (*Escherichia coli*), (*Staphylococcusaureu*) and (*Escherichia coli*) in Fig (5).Table (6) Diameter of zone of inhibition.

Molar conductivity

The conductance values in DMFof the complexes show in the ambit10 to 25 ohm^{-1}.cm^{2}mol^{-1} which is quite lower than that expected for an electrolyte and appear their non-electrolytic nature as in Table(5) .

Conclusion

The new ligand (L) and all complexes were synthesis. The metal (II) ions are coordinated by one amine (H-C=N) atom ,N of pyridine and O atom of benzoin . Spectroscopic and structural data display(4)-chelate in metal complexes obtaining to the bind of tridentates ligand groups with one chelated chloride in schemes (1&2).

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Table (1) Some properties of prepared compounds

Empirical Formula	Chemical Formula	Molecular Weight	Yield %	M.Pº C	Colour	Calc.)%(Found				
						C	H	N	Cl	Metal
L	$C_{19}H_1N_2O$	358.43	60	140	yellow	(76.74) 76.50	(5.81) 5.40	(8.13) 8.42	-	-
[Co(L)Cl]	$C_{19}H_{14}ClCoN_2O$	380.01	67	220	Blue	(59.94) 60.76	(3.71) 3.65	(7.36)	(9.31) 9.63	(15.48) 11.20
[[Ni(L)Cl]	$C_{19}H_{14}ClN_2NiO$	380.47	76	254	Pule brown	(59.98) 60.16	(3.71) 3.78	(7.36)7. 05	(9.32) 9.24	(15.43) 11.30
[Mn(L)Cl]	$C_{19}H_{14}ClMnN_2O$	382.32	82	208	Greenth yellow	(60.58) 59.86	(3.75) 3.83	(7.44)7. 25	(9.41) 9.15	(14.58) 10.35
[Cu(L)Cl]	$C_{19}H_{14}ClCuN_2O$	385.33	73	219	brown	(59.23) 59.17	(3.66) 3.09	(7.27) 6.76	(9.20) 9.80	(16.49) 15.88
[Hg(L)Cl]	$C_{19}H_{14}ClHgN_2O$	522.37	84	241	Pule brown	(43.69) 43.43	(2.70) 3.34	(5.36)5. 41	(6.79) 6.20	(38.40) 38.52

Table (2)^{1}H-NMR Shifts for Schiff base (in DMSO-d_6)

CH-O$\underline{H}$	DMSO	OH-C$\underline{H}$	Py-$\underline{H}$	Ar-$\underline{H}$
1.15	2.5	3.82	5.39-6.18	6.83-8.12

Table (3) ^{13}C-NMR shifts for Schiff base (in DMSO-d_6)

DMSO	$\underline{C}$-OH	Py=$\underline{C}$	Ar=$\underline{C}$	Py-$\underline{C}$=N	H$\underline{C}$=N
40.80	73.29	124.70-129.61	130.20-141.56	160.39	164.47

Table (4) FT-IR data for Schiff base and the complexes

Compound	υ(O-H)	$\upsilon(H - C)_{aromatic}$	$\upsilon(H - C)_{aliphatic}$	$\upsilon(N=C)_{imine}$ υ(N=C)-py	υ(O-C)	υ(N – M	υ(O –M)
L	3344	3059	2937	1604 1588	1240	-	-
[Co(L)Cl]	-	3068	2937	1595 1579	1242	547	476
[Ni(L)Cl]	-	3056	2924	1593 1581	1243	547	474
[Cu(L)Cl]	-	3076	2926	1591 1578	1241	549	460
[Mn(L)Cl]	-	3026	2924	1598 1576	1243	582	482
[Hg(L)Cl]	-	3040	2927	1596 1578	1242	545	464

Table (5) UV-Vis data of Schiff base and the complexes

Comp.	μ_{eff}	$\Lambda_m$ohm.cm^{2}mole^{-1}	λnm	υ'wave number cm^{-1}	Assignments	Proposed structure
L	-	-	280	35714	$\pi\rightarrow\pi^*$	-
			312	32051	$n\rightarrow\pi^*$	
[Co(L)Cl]	5.71	10	331	30211	C.T	tetrahedral
			611	16366	$^4A_{2\ (F)}\rightarrow{}^4T_{1\ (P)}$	
[Ni(L)Cl]	Dai.	20	332	30120	C.T	square planar
			770	12987	$^1A_1g_{(F)}\rightarrow{}^2Eg$	
[Cu(L)Cl]	1.76	15	327	30581	C.T	square planar
			543	18416	$^2B_{1g}\rightarrow{}^2A_{1g}$	
[Mn(L)Cl]	5.75	19	335	29850	C.T	tetrahedral
			453	12903	$^6A_1\rightarrow{}^4A_{1\ (G)}$	
			586	22075	$^6A_1\rightarrow{}^4E_{\ (G)}$	
[Hg(L)Cl]	-	25	334	29940	C.T	square planar
			410	24390	C.T	

Table (6) Diameter of zone of inhibition (mm)

Compound.	DMF control	L	Co complex	Mncom plex	Nicompl ex	Cucomplex	Hgcomplex
Escherichia. Coli	1	3	10	10	6	7	10
Staphylococcus aureus	-	5	12	14	9	8	13
Bacllus	-	7	9	10	-	7	11
Pseudmonas	1	6	8	13	10	14	12

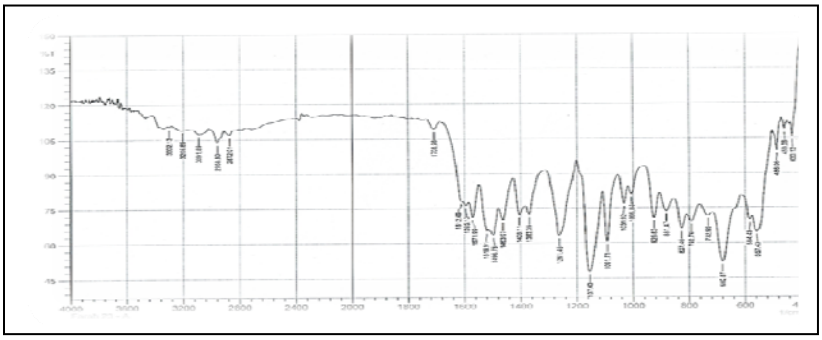

Figure (1) The IR spectrum of Schiff base

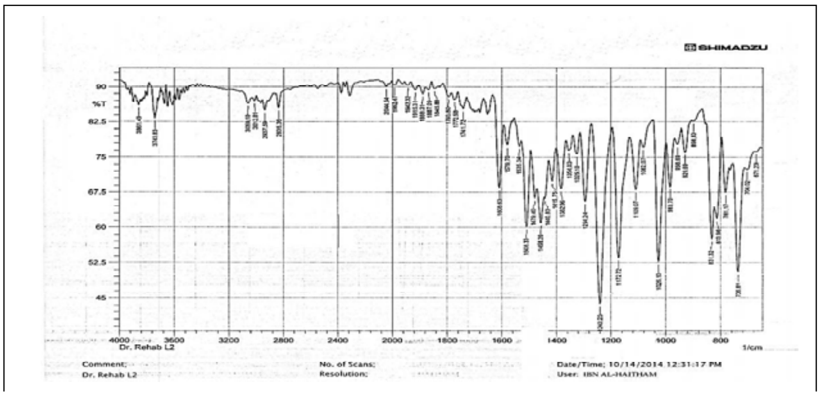

Figure (2) The IR spectrum of Ni complex

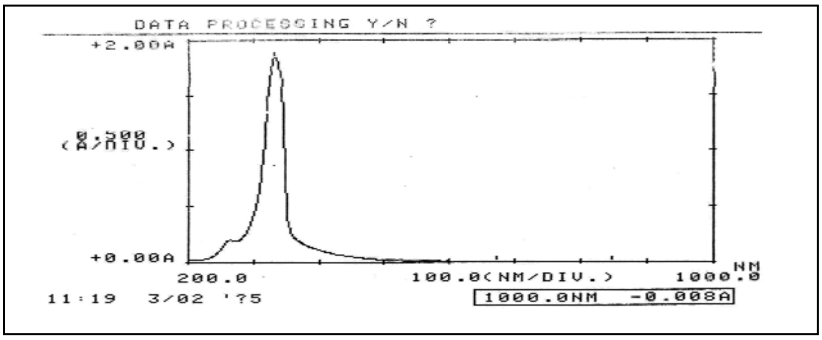

Figure (3) The UV spectrum of Schiff base

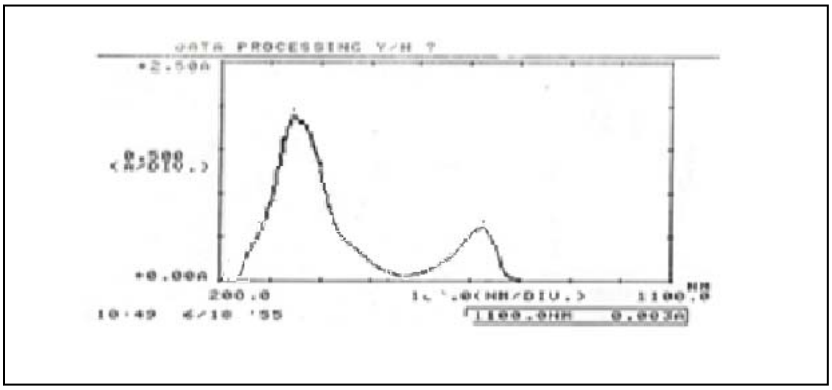

Figure (4) The UV spectrum of Ni complex

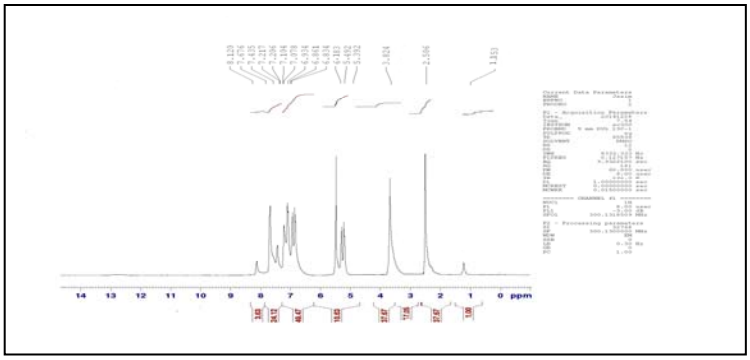

Figure (5) The1H-NMR spectrum of Schiff base

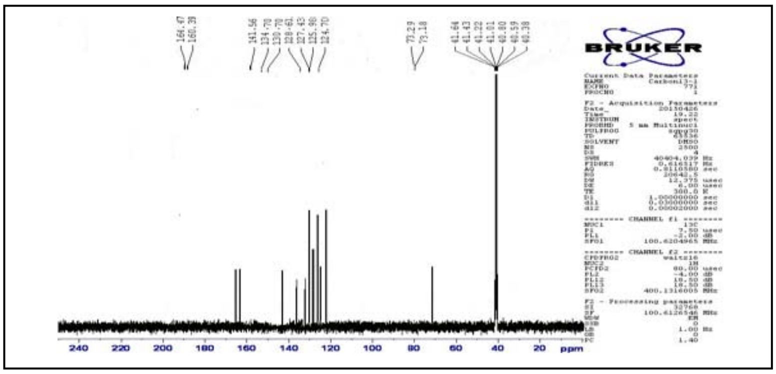

Figure (6) The13C-NMR spectrum of Schiff base

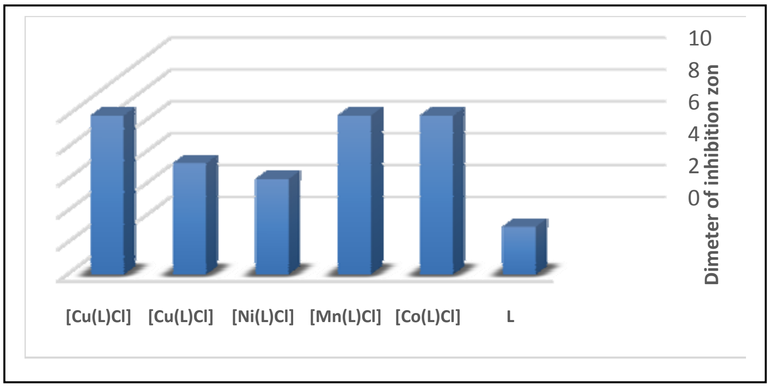

Figure (7) Effect of *Escherichia .Coli* gram negative

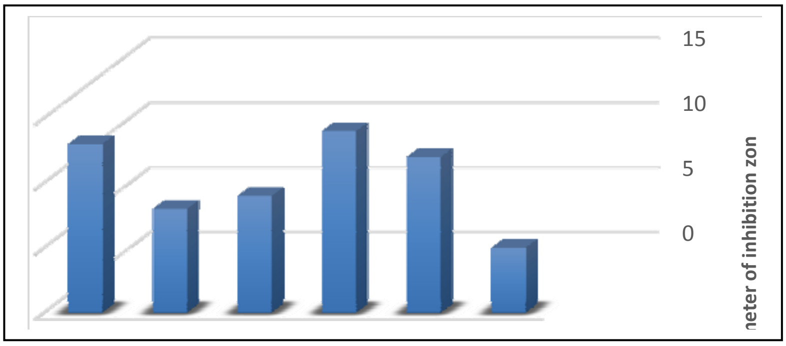

Figure (8) Effect of *Staphylococcusaureus*

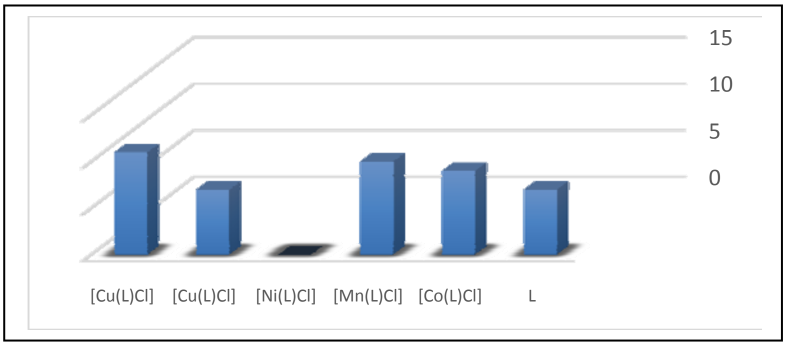

Figure (9) Effect of *Bacillus* gram

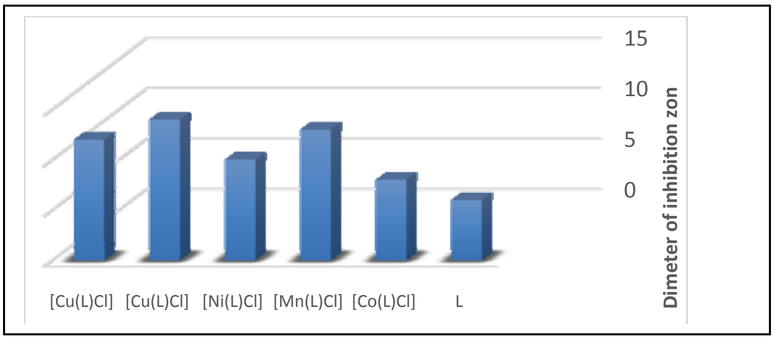

Figure (10) Effect of *pseudmonas* gram

معقدات الفلزات الثنائية مع ليكاند ثلاثي السن:N,N,Oدراسة تحضير وتشخيص والفعالية البايولوجية

علي نبيل نصيف

قسم الكيمياء/ كلية التربية ابن الهيثم للعلوم الصرفة(ابن الهيثم) / جامعة بغداد

استلم في:22/كانون الأول/2016، قبل في:28/حزيران/2016

الخلاصة

تحضير بعض المركبات التناسيقة الجديدة للكوبالت (II)، النيكل (II) ، النحاس (II) ، المنغنيز (II) والزئبق (II) مع الليكاند قاعدة شيف المستمدة من 2- امينو البيريدين والبنزوين . حيث يتفاعل مع الأملاح الفلزية في الإيثانول كمادة مذيبة في 1:1 نسبة (فلز: ليكاند). المعقدات التي لها الصيغة العامة [MLCl] حيث M = المنغنيز(II) ، النيكل(II) ، الكوبلت (II)، النحاس (II) والزئبق (II) .تم تشخيص الليكاند والمعقدات الفلزية وذلك باستخدام تحليل الفلزات عن طريق الامتصاص الذري والرنين النووي والمغناطيسي البروتوني والكربوني (لليكاند فقط)،أطياف الأشعة تحت الحمراء، الأطياف الإلكترونية، التوصيلية المولية وقياسات العزم المغناطيسي. وأشارت القياسات أن الليكاند تناسيق مع أيون الفلز (II)بطريقة ثلاثي السن من خلال ذرة النيتروجين للازموثين ، ذرة النيتروجين من البيريدين و ذرة الأوكسجين من البنزوين جميع الدراسات تكشف أن التناسق رباعي للفلزات في جميع المعقدات. واقترحت بنية رباعي السطوح ومربع مستوي لمعقدات الفلزية.

الكلمات المفتاحية: قاعدة شيف، 2-امينو بيريدين ، تشخيص ومعقدات مخلبية فلزية