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Synthesis and antibacterial study of the ligand type Schiff base derived from amino acid [L- Phenylalanine] and its complexes with Co (II), Ni (II), Cu (II) and Zn (II) ions

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Abstract

L-Phenylalanine amino acid was condensed with 2-hydroxybezaldehyde to give the Schiff base sodium 2-(2-hydroxybenzylideneamino)-3-phenylpropanoate, which was used as a precursor [NaHL]. The precursor was reacted with 1,2-dichloroethane to give the Schiff base sodium 2,2'-(2,2'-(ethane-1,2diylbis(oxy))bis(2,1-phenylene))bis(methan-1-yl-1-ylidene)bis(azan-1-yl-1-ylidene)bis(3-phenyl propanoate), which was used as a ligand [Na_2L], in complexation with some metal (II) chloride MCl_2, where [M= Co(II), Ni(II), Cu(II) and Zn(II)], to give [M(L)] complexes. The [Na_2L] ligand and All complexes were characterized by spectroscopic methods, [FTIR, UV-Vis, atomic absorption], melting point, chloride content, conductivity and magnetic susceptibility measurements, as well as the Na_2L ligand characterized by ^1H NMR. The data, of these studies and measurements were important to suggest an octahedral geometry around Co(II), Ni(II), Cu(II) and Zn(II) ions. The synthesized ligand, along with its metal complexes, was screened for in *vitro* antibacterial activity against gram-positive bacteria like *Staphylococcus aureus*, *and Acinetobacter baumannii.* This study showed an effective inhibition of gram-positive bacteria.

Introduction

Schiff bases have been used as chelating ligands in the field of coordination chemistry and their metal complexes are of great interest for many years [1]. Chemists have reported on the chemical, structural and biological properties of Schiff bases, Schiff bases are characterized by the -N=CH- (imine) group which is important in elucidating the mechanism of transamination and rasemination reaction in biological system [2, 3]. Schiff base metal complexes have been widely studied because they have industrial, antifungal, antibacterial, anticancer and herbicidal applications [4, 5]. They serve as models for biologically important species and find applications in bio mimetic catalytic reactions, Schiff bases are active against a wide range of organisms, for example; Candida Albicans, Escherichia coli Staphylococcus aureus, Bacillus polymxa, the Trychophyton gypseum. Antibacterial activity has been studied more than antifungal activity, because bacterium can achieve resistance to antibiotics through biochemical and morphological modifications [6, 7]. Chelating ligands containing N, S and O donor atoms showed a broad biological activity and are of special interest because of the variety of ways in which they are bonded to metal ions. It is known that the existence of metal ions bonded to biologically active compounds may enhance their activities [8-10]. The aim of the present study is to synthesis, characterize and evaluate the metal complexes as antibacterial agent as promising addition of new class of complexes as metal based drugs.

Experimental

The used chemicals were of analytical grades; metals were used as chloride salts. The complexes were identified by atomic absorption technique, using Schimadzu (A.A) 680 G atomic absorption spectrophotometer. I.R data were recorded as (KBr) disc using Schimadzu 4800S FTIR spectrophotometer in the range (4000-400) cm^{-1}. ^1H NMR spectra were recorded in DMSO-d_6 using Brucker 300 MHz spectrometer. (UV-Vis.) spectra were obtained in (MeOH) on a CECIL, CE 2700 spectrophotometer in the range (200-900) nm using quartz cell. The Magnetic measurements were carried out on solid compounds using 6 Bruker B.M instrumental. Melting points were recorded on an electro thermal Stuart apparatus and are uncorrected. Electrical conductivity measurements of the complexes were recorded at 25C° for 10^{-3}M solutions in (MeOH) as a solvent using a Wissenchaftilich tecchnich werksttaten, D1820 bweilheimI.F 42 conductivity meter. Chloride contents for complexes were determined potentiometerically by using (686-titro processor-665), Dosinat-metrom Swiss. Antibacterial screening was used agar diffusion technique [11,12].

Preparation of the ligand [Na_2L]

The ligand was prepared by two **steps**

Step (1): L-phenylalanine (1 g, 6.0 mmol), was added to (10 ml), (0.24g, 6.0 mmol) sodium hydroxide-methanol solution, the mixture was stirred until a clear solution was obtained. A solution of (0.74 g, 6.0 mmol) of 2-hydroxy bezaldehyde in (10ml) methanol was added to reaction mixture, which was catalyzed with (5drops) of glacial acetic acid and stirred for (3 hours). Yellow precipitate was formed, filtered, washed with ethanol, and re-crystallized from hot methanol to give yellow crystals of the precursor [NaHL], Yield (67%), mp (225-232°C).

Step (2): A solution of the precursor [NaHL] (2.35g, 8.08mmole) in (10 ml) methanol was added to (10ml), (0.45g, 8.08mmol) potassium hydroxide-methanol solution, then a solution of (0.4g, 4.04mmole) of 1,2-dichloroethane in (10ml) methanol was added to reaction mixture was refluxed with stirring for (3 hours). Orange precipitate was formed, filtered, washed with ethanol, and re-crystallized from hot methanol to give orange crystals of the ligand [Na_2L], Yield (72%), mp (260-266°C).

Preparation of [Na_2L] complexes with metal ions

A solution of (0.08g, 0.33mmole) of [Na_2L] ligand in methanol (5ml) was added to stirred clear solution of a solution of (0.2g, 0.33mmole) of $CoCl_2.6H_2O$ in (5ml) methanol. Reaction mixture was refluxed for (4 hours); dark-brown precipitate was formed, filtered and washed by water and re-crystallized with ethanol. The complexes [Ni(L)], [Cu(L)] and[Zn(L)], were obtained in a similar method to that mentioned in the preparation of [Co(L)] complex described above. Physical properties of the prepared complexes were given in (Table 1).

Results and Discussion

Synthesis of the ligand

The ligand [Na_2L] was prepared according to the general method shown in (scheme 1). The I.R spectral analysis of the precursor [NaHL], (Table 2), showed phenolic (–OH) band at (3433) cm^{-1}, and υ(C=N) bands at (1638) cm^{-1}, also the spectrum showed bands at (1519) cm^{-1}, and (1404) cm^{-1} which assigned to $\upsilon_{as}(COO^-)$ and $\upsilon_s(COO^-)$ respectively and showed band at (1149) cm^{-1} which can be attributed to υ(C-O)phenolic group [13-17], by comparing with the I.R spectral analysis of the ligand [Na_2L], Fig(1), (Table 2), showed bands at (1635) cm^{-1} which assigned to υ(C=N) and band at (1219) cm^{-1} which assigned to ether group (C-O-C), also the spectrum showed bands at (1539) cm^{-1}, (1404) cm^{-1} which assigned to $\upsilon_{as}(COO^-)$, and $\upsilon_s(COO^-)$ respectively [13-17]. Electronic spectral analysis of the precursor [NaHL], (Table 3), showed four absorption peaks at (231.5) nm ε_{max} (2426) $molar^{-1}cm^{-1}$, (248.5) nm ε_{max} (2503) $molar^{-1}cm^{-1}$, (316.1) nm ε_{max} (1149) $molar^{-1}cm^{-1}$ and (404.3) nm ε_{max} (777) $molar^{-1}cm^{-1}$, which assigned to ($\pi\rightarrow\pi^*$), ($\pi\rightarrow\pi^*$), ($n\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transition respectively [18,19]. by comparing with electronic spectral analysis the ligand [Na_2L], Fig(2), (Table 3), showed four absorption peaks at (236.0) nm ε_{max} (2395) $molar^{-1}cm^{-1}$, (259.0) nm ε_{max} (2501) $molar^{-1}cm^{-1}$, (319.2) nm ε_{max} (1129) $molar^{-1}cm^{-1}$, and (407.5) nm ε_{max} (697) $molar^{-1}cm^{-1}$, which assigned to ($\pi\rightarrow\pi^*$), ($\pi\rightarrow\pi^*$), ($n\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transition respectively [18,19]. ^{1}H NMR spectral analysis of the ligand [Na_2L], Fig(3), showed chemical shifts δ (ppm) of Schiff base proton (-CH=N-) at (7.9) as a singlet peak. A multiple signals at rings (6-7 and 7-7.5)ppm for phenylalanine proton and phenyl-ether proton. (-CH) proton of (-C=N-CH-) group at [3.7, 1H, t], (-CH-) methelene group at [3.4, 2H, d], and triplet peak at (4.4)ppm, which assigned to ($-CH_2-$) in ($Ph-O-CH_2-$) group. A singlet high peak at (4.9)ppm for the trace MeOH solvent [14,15,20,21] .

Synthesis of the complexes

Complexation of ligand [Na_2L] with Co(II), Ni(II), Cu(II) and Zn(II) were carried out in MeOH. These complexes are stable in solution. The analytical and physical data (Table 1), and spectral data (Table 2, and 3), are compatible with the suggested structure (Scheme 1). The I.R spectra of the complexes [Co(L)](1), Fig(4), [Ni(L)](2), ([Cu(L)](3)) and [Zn(L)](4), (Table 2), exhibited the band at (1635) cm^{-1} in the free ligand spectrum which assigned to υ(C=N) imine group Shifted to lower frequency and appeared at (1619) cm^{-1}, (1614) cm^{-1}, (1617) cm^{-1} and (1614) cm^{-1} for the complexes (1),(2),(3), and (4) respectively [15-18]. These bands were assigned the υ(C=N) stretches of reduced bond order, this can be attributed to the delocalization of metal-electron density into the ligand π-system (HOMO→LUMO) [22, 23], (HOMO=Highest occupied molecular orbital, LUMO= Lowest unoccupied molecular orbital). The ether (C-O-C) stretching vibration appeared at (1219) cm^{-1} in the free ligand was Shifted to higher frequency and appeared at (1311) cm^{-1}, (1300) cm^{-1}, (1300) cm^{-1} and (1292) cm^{-1} for the complexes (1),(2),(3), and (4) respectively, all that indicated a linkage between oxygen of ether group and the metal [14, 22], also the spectrum showed bands at (1539) cm^{-1}, (1404) cm^{-1} which assigned to $\upsilon_{as}(COO^-)$, and $\upsilon_s(COO^-)$ respectively in the free ligand spectrum, was shifted to lower frequency and appeared at [(1496), (1350)], [(1486), (1377)], [(1450), (1369)] and [(1480), (1373)] cm^{-1} for the complexes (1),(2),(3), and (4) respectively, all that indicated a linkage between oxygen of carboxylate group and the metal [24,25]. The spectra showed the appearance of bands at (460) cm^{-1}, (482) cm^{-1}, (478) cm^{-1} and (474) cm^{-1} refer to υ(M-N) for complexes (1),(2),(3), and (4), these bands confirm the coordination of the nitrogen atom to the metal center, while the bands at (576) cm^{-1}, (563) cm^{-1}, (574) cm^{-1} and (568) cm^{-1} assigned to υ(M-O) of complexes (1), (2), (3) and (4) respectively, bands confirm the coordination of the oxygen atom of carboxylate group of the ligand to metal center, and the bands at (547) cm^{-1}, (532) cm^{-1}, (543) cm^{-1} and (536) cm^{-1} assigned to υ(M-O) of complexes (1),(2),(3), and (4) respectively, theses bands indicating that the etheric oxygen in the ligand is involved the coordination with metal ions in complexes [22-23, 26,27].

Electronic spectral analysis of the complexes, (Table 3):

Complex[Co(L)]: showed two high intense peaks at (245.5) nm ε_{max} (2642) $molar^{-1}cm^{-1}$ and (268.6) nm ε_{max} (2532) $molar^{-1}cm^{-1}$ were assigned to the ligand field, while a medium peak at (382.0) nm ε_{max} (943) $molar^{-1}cm^{-1}$ was assigned to (C.T), a weak broad peaks at (491.2) nm ε_{max} (62) $molar^{-1}cm^{-1}$,(531) nm ε_{max} (56) $molar^{-1}cm^{-1}$, and (622.0) nm ε_{max} (16) $molar^{-1}cm^{-1}$, were assigned to (d-d) electronic transition ($^4T_{1g(F)}\rightarrow{}^4T_{2g(F)}$), ($^4A_{2(g)}\rightarrow{}^4T_{1g}$), and ($^4T_{1(g)}\rightarrow{}^4T_{1g(p)}$) respectively, suggesting octahedral geometry [19].

Complex [Ni(L)]: showed one high intense absorption at (245.5) nm ε_{max} (2613) molar^{-1}cm^{-1} are due to the ligand field, another high intense peak at (364.5) nm ε_{max} (1825) molar^{-1}cm^{-1} was assigned to (C.T), while a weak broad peaks at (450.6) nm ε_{max} (49) molar^{-1}cm^{-1}, (510.4) nm ε_{max} (22) molar^{-1}cm^{-1}, and (642.5) nm ε_{max} (17) molar^{-1}cm^{-} were assigned to (d-d) electronic transition ($^3A_{2g}\rightarrow{}^3T_{2g}$), ($^3A_{2g}\rightarrow{}^3T_{1g}$), and ($^3A_{2g}\rightarrow{}^3T_{1g(p)}$) respectively, suggesting octahedral geometry [19].

Complex [Cu(L)]: Fig(5), showed two high intense absorption peaks at (243.0) nm ε_{max} (2624) molar^{-1}cm^{-1}, and (272.1) nm ε_{max} (2543) molar^{-1}cm^{-1} are due to the ligand field, a high intense absorption peak at (369.0) nm ε_{max} (1831) molar^{-1}cm^{-1} was assigned to (C.T), while a weak broad peak at (660.5) nm ε_{max} (41) molar^{-1}cm^{-1} was assigned to (d-d) electronic transition ($^2E\rightarrow{}^2T_2$) suggesting octahedral geometry [19].

Complex [Zn(L)]: showed two peaks at (242.0) nm ε_{max} (2537) molar^{-1}cm^{-1} and (272.0) nm ε_{max} (2315) molar^{-1}cm^{-1} are due to the ligand field. While a high intense peak at (361.0) nm ε_{max} (1763) molar^{-1}cm^{-1} was assigned to (C.T), the d^{10} configuration of Zn^{II} ion along with the data obtained confirms a octahedral structure around the ion [19].

The molar conductance of the complexes in methanol lie in the range (5.1-12.9 Ohm^{-1}cm^{-2}mol^{-1}), (Table 3), indicting their non-electrolyte having mole ratio of metal:ligand as 1:1 [28]. At the room temperature magnetic moments of the all complexes showed normal magnetic moment in (Table 3), the magnetic moment data were ca. (4.13), (3.25) and (1.65) B.M for Co(II) in [Co(L)], Ni(II) in [Ni(L)], Cu(II) in [Cu(L)], respectively and (0.00) B.M to Zn(II) in [Zn(L)]. The magnetic moment studies showed that these complexes were octahedral geometry [29].

Biological activity

The antibacterial activity of the synthesized ligand [Na_2L] and its complexes [Co(L)](1) , [Ni(L)](2), [Cu(L)](3), and [Zn(L)](4) (Table 4 and 5), were tested utilizing the agar diffusion technique [30]. The organism tested was *staphylococcus aureus*, and *Acinetobacter baumannii.* The agar media were inoculated with test organisms and a solution of the tested compound (100µg/ml) was placed separately in cups (6 mm diameter) in the agar medium. The inhibition zones were measured after 24 hours incubation. Separate studies were carried out with the solution alone of DMSO and the showed no activity against any bacterial strains [31]. The results of these studies revealed that the ligand and metals complexes showed an effective in the inhibition of *Acinetobacter baumannii* and *Staphylococcus aureus*. Biological activity of the previous compounds in inhibition of bacterial growth could be attributed to one of the following mechanisms, the first mechanism is by the inhibition of the bacterial cell wall synthesis by bounding to the precursor of the cell wall, and second mechanism revealed that some antibodies have similar stereo structure to substrate (D-alanyl D-alanine). So it will act competitive inhibitions for the enzymes (transpeptidase and /or carboxpeptidase) which are the main enzymes catalyzed the end step in the biosynthesis of peptidoglycans of the bacterial cell wall, other mechanisms could contributed to the results found in the study which include the inhibition of biosynthesis of bacterial proteins by linking to the ribosome by doing so, the ribosome will not be in contact with tRNA (transfer ribonucleic acid) , so the bacteria will not survive, an other mechanisms were postulated that some antibodies inhibit the denovo synthesis of bacterial DNA by splitting DNA in DNA-enzyme complexes by inhibition DNA ligase [32-34].

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Table (1): The microanalysis results and some physical properties for the ligand [Na_2L] and its complexes

Compounds	**Formula**	**Colour**	**M.p (° C)**	**Yield%**	**Chloride content**	**Metal**	**M.wt**
Na_2L	$C_{34}H_{30}N_2Na_2O_6$	Orange	260-266	72	Nil	-----	608.59
[Co(L)](1)	$C_{34}H_{30}N_2O_6Co$	Dark brown	285 dec.	81	Nil	8.83 (9.48)	621.54
[Ni(L)](2)	$C_{34}H_{30}N_2O_6Ni$	Light green	279 dec.	79	Nil	8.80 (9.45)	621.31
[Cu(L)](3)	$C_{34}H_{30}N_2O_6Cu$	Dark green	291 dec.	84	Nil	9.45 (10.15)	626.16
[Zn(L)](4)	$C_{34}H_{30}N_2O_6Zn$	Yellowish Brown	274 dec.	68	Nil	9.70 (10.42)	628.02

dec. = decompose

Table (2): Infrared spectral data for the ligand [Na_2L] and its metal complexes cm^{-1}

Compound	**υ(C=N)**	**υ(O–H)**	**υ $_{as}$(COO) υ $_a$(COO)**	**υ(C=C) Aromatic**	**υ(C–H) Aromatic**	**υ(C–H) Aliphatic**	**υ(C–O–C) etheric**	**υ(M–O) etheric**	**υ(M-O) Carboxylate ion**	**υ(M–N)**
NaHL	1638	3433	1519 1404	1585	3028	2939	-----	----	----	----
Na_2L	1635	-----	1539 1404	1589	3028	2947	1219	----	-----	----
[Co(L)](1)	1619	-----	1496 1350	1590	3028	2924	1311	547	576	460
[Ni(L)](2)	1614	-----	1486 1377	1587	3028	2931	1300	532	563	482
[Cu(L)](3)	1617	-----	1450 1369	1587	3028	2924	1300	543	574	478
[Zn(L)](4)	1614	-----	1480 1373	1590	3024	2924	1292	536	568	474

Table (3): Electronic spectral data for the ligand [Na_2L] and its metal complexes

Compound	λ nm	ε_{max} molar^{-1}cm^{-1}	Assignment	Ratio	Molar conductivity S.cm^2.mol^{-1}	Magnetic susceptibility B.M	coordination
NaHL	231.5	2426	π→π*	-----	-------	------	------
	248.5	2503	π→π*				
	316.1	1149	n→π*				
	404.3	777	n→π*				
Na_2L	236.0	2395	π→π*	-----	-------	------	------
	259.0	2501	π→π*				
	319.0	1129	n→π*				
	407.5	697	n→π*				
[Co(L)](1)	245.5	2642	Ligand field	neutral	8.7	3.87 (4.13)	Octahedral
	268.6	2532	Ligand field				
	382.0	943	C.T				
	491.2	62	$^4T_{1g(}\rightarrow{}^4T_{2g}$				
	531.0	56	$^4A_{2g}\rightarrow{}^4T_{1g}$				
	622.3	16	$^4T_{1g}\rightarrow{}^4T_{1g\,(p)}$				
[Ni(L)](2)	245.5	2613	Ligand field	neutral	5.1	2.83 (3.25)	Octahedral
	364.5	1825	C.T				
	450.4	49	$^3A_{2g}\rightarrow{}^3T_{2g}$				
	510.4	22	$^3A_{2g}\rightarrow{}^3T_{1g}$				
	642.5	17	$^3A_{2g}\rightarrow{}^3T_{1g(p)}$				
[Cu(L)](3)	243.0	2624	Ligand field	neutral	5.6	1.7 (1.65)	Octahedral
	272.1	2543	Ligand field				
	369.0	1831	C.T				
	660.5	41	$^2E\rightarrow{}^2T_2$				
[Zn(L)](4)	242.0	2537	Ligand field	neutral	12.9	Diamagnetic	Octahedral
	272.0	2315	Ligand field				
	361.0	1763	C.T				

Table (4): Biological activity of the ligand [Na_2L] and its metal complexes against *Acinetobacter baumannii* bacteria

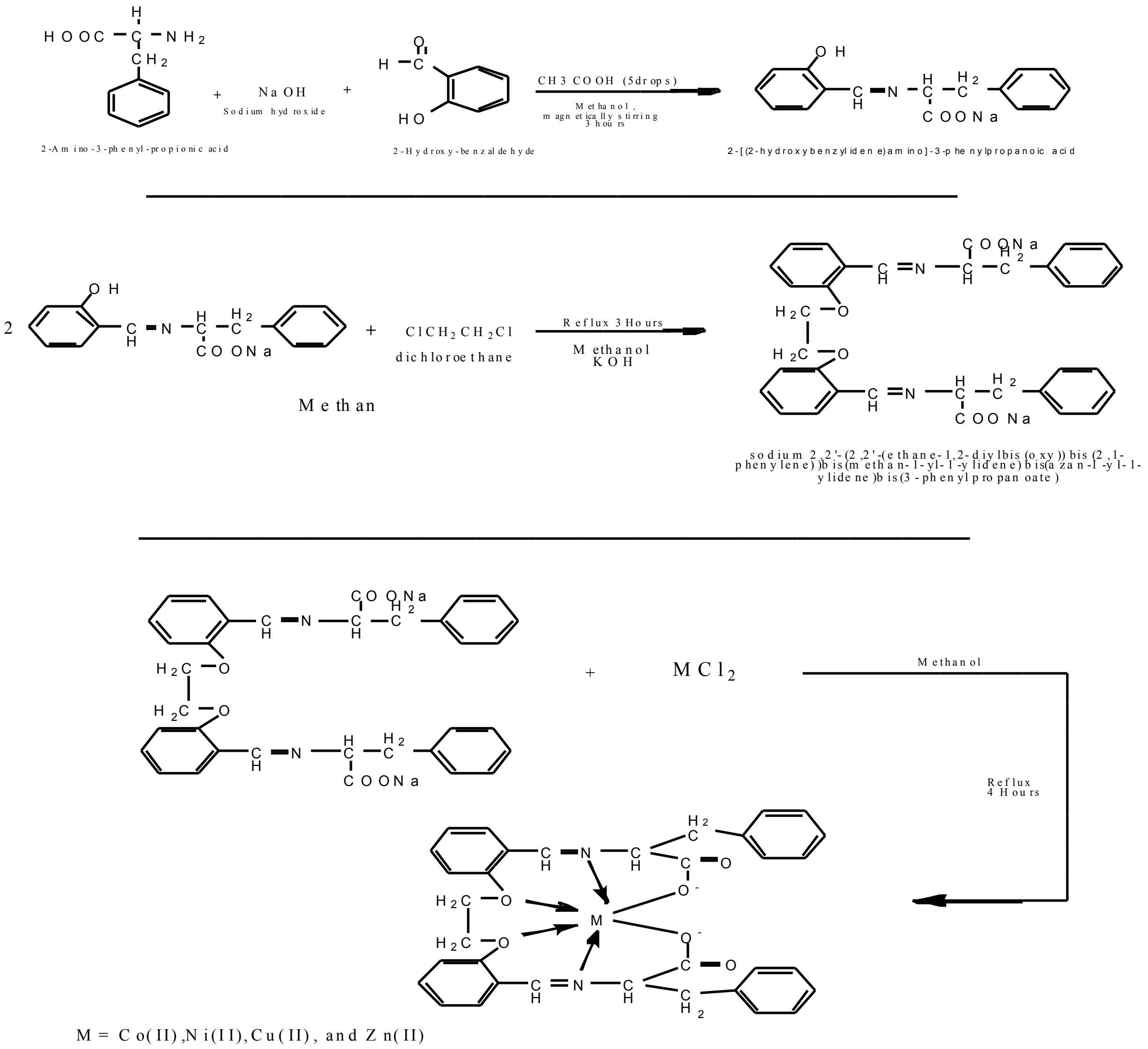
Compound	Bacteria (zone of inhibition (diameter mm))									
	A_1	A_2	A_3	A_4	A_5	A_6	A_7	A_8	A_9	A_{10}
Na_2L	13	10	14	15	14	15	13	14	14	14
[Co(L)](1)	13	9	8	16	16	16	13	13	11	15
[Ni(L)](2)	15	9	14	16	16	17	13	Nil	13	17
[Cu(L)](3)	Nil	9	14	14	13	14	12	Nil	Nil	15
[Zn(L)](4)	10	10	12	13	13	17	Nil	Nil	12	12

A= *Acinetobacter baumannii* bacteria

Table (5): Biological activity of the ligand [Na_2L] and its metal complexes against *Staphylococcus aureus* bacteria

Compound	Bacteria (zone of inhibition (diameter mm))									
	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8	S_9	S_{10}
Na_2L	Nil	9	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
[Co(L)](1)	Nil	Nil	Nil	Nil	Nil	Nil	12	Nil	Nil	11
[Ni(L)](2)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
[Cu(L)](3)	Nil	Nil	14	Nil	Nil	Nil	Nil	8	Nil	Nil
[Zn(L)](4)	Nil	Nil	15	16	Nil	Nil	Nil	Nil	Nil	Nil

S= *Staphylococcus aureus* bacteria



Scheme (1): Preparation of the ligand [Na$_2$L] and its metal complexes

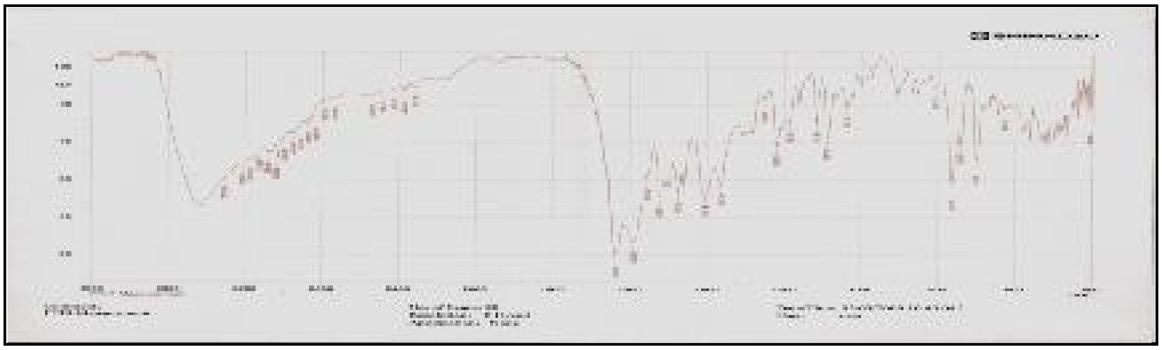
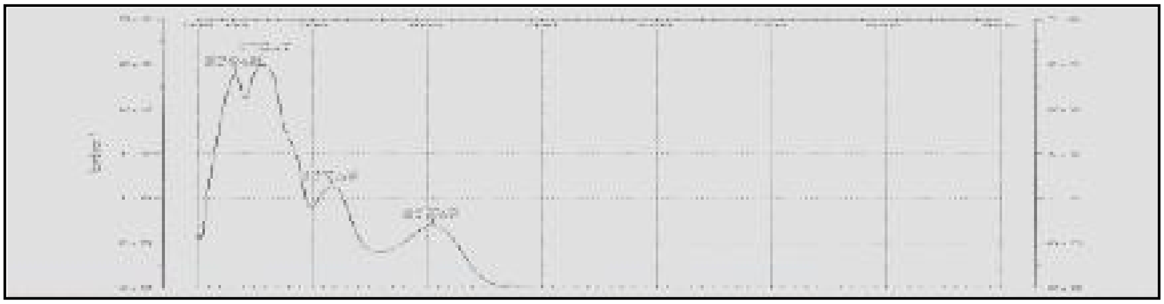


Fig (1): Infrared spectrum of the ligand (Na_2L)



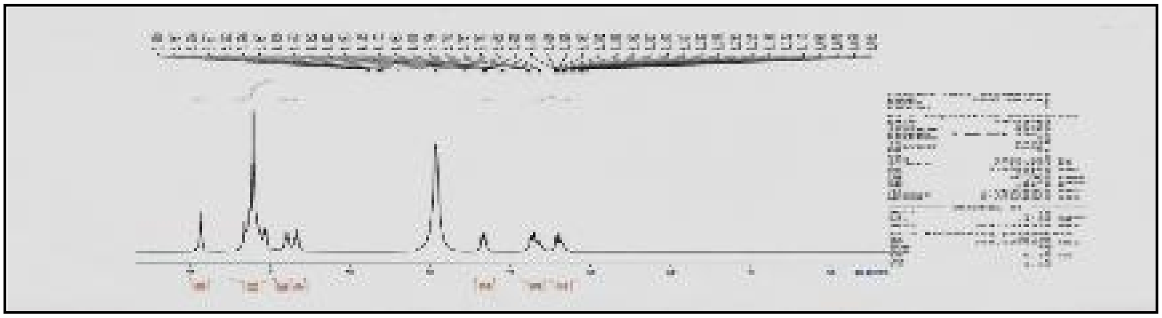


Fig. (2): Electronic spectrum of the precursor (Na_2L)

Fig. (3) ^{1}H NMR spectrum of the ligand (Na_2L)

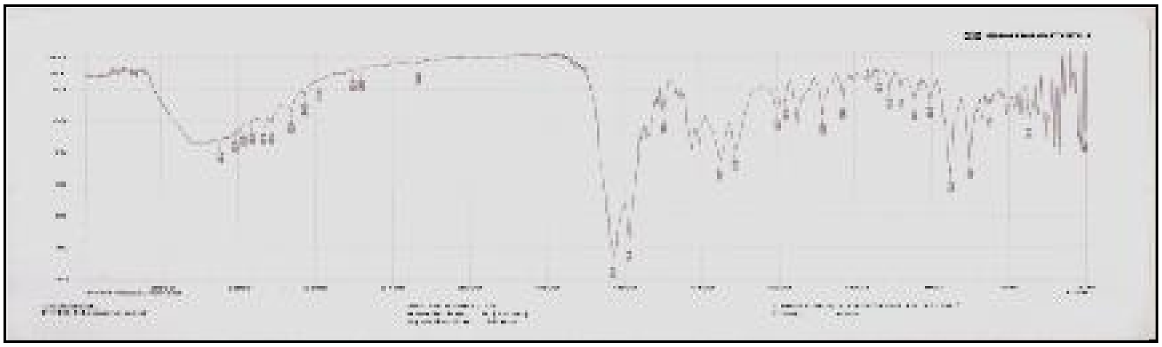


Fig .(4): Infrared spectrum of the complex [Co(L)]

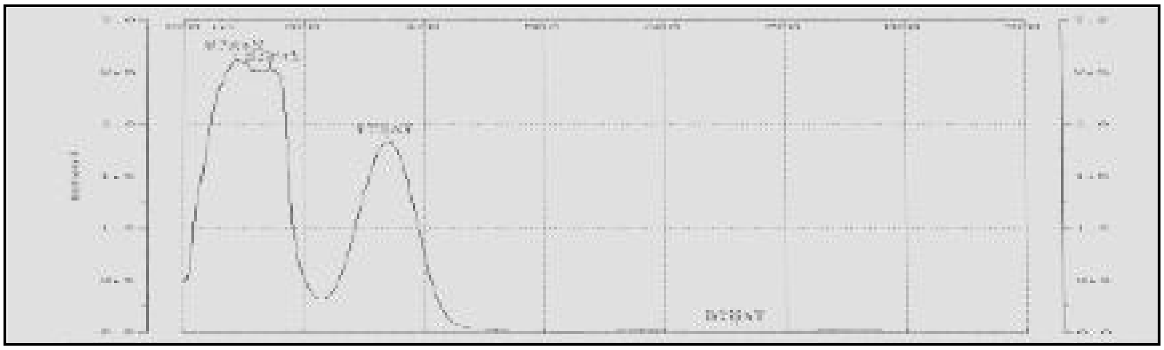


Fig.(5): Electronic spectrum of the complex [Cu(L)]

مجلة ابن الهيثم للعلوم الصرفة والتطبيقية المجلد 23 (2) 2010

تخليق ودراسة الفاعلية البايولوجية لليكاند من نوع قاعدة شيف المشتقة من حامض أميني (ل–فنيل الألنين) ومعقداته مع الأيونات

Co(II), Ni(II), Cu(II) and Zn(II)

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الخلاصة

تضمن البحث مفاعلة الحامض الأميني ل– فنيل الألنين مع 2–هيدروكسي بنزالديهايد للحصول على قاعدة شيف Sodium2-(2-hydroxybenzylideneamino)-3-phenylpropanoate التي أستعملت مادة وسطية [NaHL] ثم مفاعلتها مع 1,2–داي كلورو أيثان أذ أعطى التفاعل قاعدة شيف Sodium2,2'-(2,2'-(ethane-1,2'diylbis(oxy))bis(2,1-phenylene))bis(methan-1-yl-1-ylidene)bis(azan-1-yl-1-ylidene)bis (3-phenyl propanoate). والتي أستعملت كليكاند [Na_2L] ثم مفاعلة الليكاند مع أيونات بعض العناصر الفلزية ، اذ تكونت معقدات ذوات الصيغة [M(L)] ، M= Co(II), Ni(II), Cu(II), and Zn(II).

شخصت جميع المركبات بالطرائق الطيفية ،الاشعة تحت الحمراء والاشعة فوق البنفسجية –المرئية والتحليل الكمي الدقيق للعناصر و التوصيلية المولارية الكهربائية ومحتوى الكلور ودرجة الانصهار والحساسية المغناطيسية ، و كذلك شخص الليكاند بواسطة ^1H NMR .فأن من نتائج البحث كان الشكل الفراغي المقترح للمعقدات المحضرة ثماني السطوح .

درست الفعالية البايولوجية خارج الخلية الحية لليكاند ومعقداته ضد بكتريا موجبة لصبغة كرام ال *Staphylococcus aureus* و *Acinetobacter baumannii* ، و أظهرت وجود تأثير مانع للبكتريا الموجبة لصبغة كرام.