

**\*Synthesis, spectroscopic and antimicrobial studies of Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes with N-aminoquinoline derivatives.**

Received : 4\10\2014

Accepted : 9\2\2014

**I.A.Hassan****Chem. Dept. College of Science, Mosul University****ABSTRACT**

Novel transition metal (II) complexes,  $[M(L_1)_2]Cl_2$  and  $[M(L_2)_2]Cl_2$ , were synthesized from the reaction of  $MCl_2 \cdot nH_2O$  ( $M = Mn, Fe, Co, Ni, Cu, Zn$ ) with N-[N-(1,2-diphenyl)ethanol amino] cumarine ( $L_1$ ), N-[4-( $\alpha$ -methyl)pyridino] cumarine ( $L_2$ ).

The ligands were obtained from cumarine. N-aminoquinoline-2-one has been synthesized by the reflux of cumarine with hydrazine hydrate in ethanol for 12 hrs. the azomethanes ( $L_1, L_2$ ) were prepared from the corresponding benzoin and 4-acetylpyridine. The synthesized compounds were characterized by microanalysis (M), molar conductance, magnetic measurement, infrared and electronic spectra. The IR spectral data suggest the involvement of carbonyl, hydroxyl and azo methane nitrogen in coordination to the central metal ion. On the bases of the spectral studies an octahedral geometry has been for all complexes. The free ligands and its metal complexes have been tested in vitro against number of microorganisms in order to assess their antimicrobial properties.

**Introduction**

Schiff bases derived from N-amines quinolone and their complexes were studied extensively due to synthetic flexibility of there compounds and their selectivity as well as sensitivity towards the central metal atom<sup>(1)</sup>. They are designed for the substitution of certain bases in enzymes<sup>(2-6)</sup>. They are also important due to their biological activities as anti – inflammatory<sup>(7)</sup>, anticancer<sup>(8)</sup> and antifungal<sup>(9)</sup>. Some Schiff bases bearing heterocyclic studies possess biological activities such as analgesic antiviral<sup>(10)</sup>. The serious medical problem of bacterial and fungal resistance and the rate at which it develops has led to increasing levels of resistance to classical antibiotics<sup>(11-13)</sup>. The discovery and development of effective antibacterial and antifungal drugs with novel mechanism of action have become an urgent task for infectious diseases research programs<sup>(14)</sup>. Many investigations have proved that binding of drug to metallic element enhances its activity and in some cases, the complexes possesses even more heating properties that the parent drug<sup>(15)</sup>, coumarins derivatives are known to posses antibacterial<sup>(16,17)</sup>. Al-Amiery<sup>(18)</sup> reported the synthesis characterization and biological properties of Co(II), Ni(II), and Cu(II) complexes with N-amino quinolone derived from 2-hydroxybenzaldehyde (for  $L_1$ ) and (1-(1H indol -3-yl) ethanone for  $L_2$ ).

The present research reports the preparation, characterization and antimicrobial of some new complexes formed by the reaction of manganese (II), iron (II), cobalt (II), nickel (II), copper (II) and zinc(II)salt with Schiff bases derived from reaction of N-amino quinolone with benzoin and 4-acetylpyriden.

### Physical Measurement

Infrared spectra were recorded on a FTIR Burcker Tensor 27 spectrophotometer in 400 - 4000  $\text{cm}^{-1}$  range using KBr discs. The molar conductance of  $10^{-3}$  M solution of metal complexes in dimethylformamide (DMF) was measured at 25°C using Jenway 4070 conductivity meter and dip-cell with platinised electrodes. The U.V/VIS spectra were recorded on Shimadzu UV-160 spectrophotometer for  $10^{-3}$  M solution of complexes in DMF using 1cm quartz cell. Melting points were measured using an electro thermal digital melting point apparatus- Magnetic susceptibility was measured on the solid state by Faraday's method using Burker BM6 instrument. All magnetic susceptibility values were corrected for diamagnetic using Pascal's constant. Analysis of complexes carried out using atomic absorption spectrophotometer

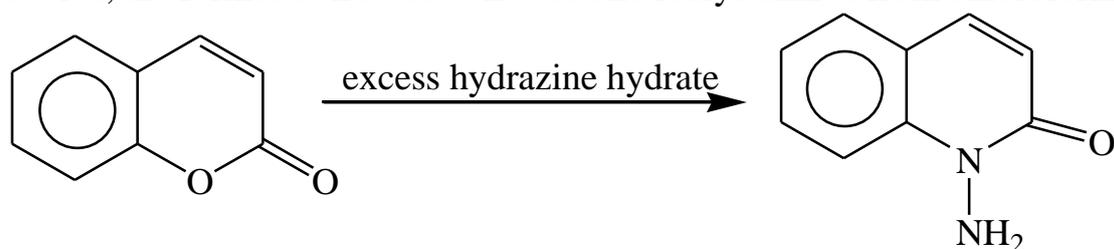
### Experimental

The ligands ( $L_1$ ) has been synthesized according to the reported procedure <sup>(19)</sup>.

### Synthesis of the ligands ( $L_1$ and $L_2$ ):

#### Synthesis of N-aminoquinoline-2-one (1):

Refluxing of coumarin (1.46 g, 0.01 mol) with excess hydrazine hydrate 99% (3.2 g, 0.1 mol) in absolute ethanol 25 ml for 12 hrs, then cooled, the formed solid was collected and recrystallized from chloroform



#### Synthesis of Schiff bases:

A mixture of compound 1 (1.58 g, 0.01 mol) and (2.13 g, 0.01 mol) of benzoin for ( $L_1$ ) or (2.62 g, 0.01 mol) of 4- acetylpyridine for ( $L_2$ ). The mixture was refluxed in absolute ethanol 20 ml for 7-8 hrs. Then was cooled and the product obtained was recrystallized from ethanol fig.(a,b).

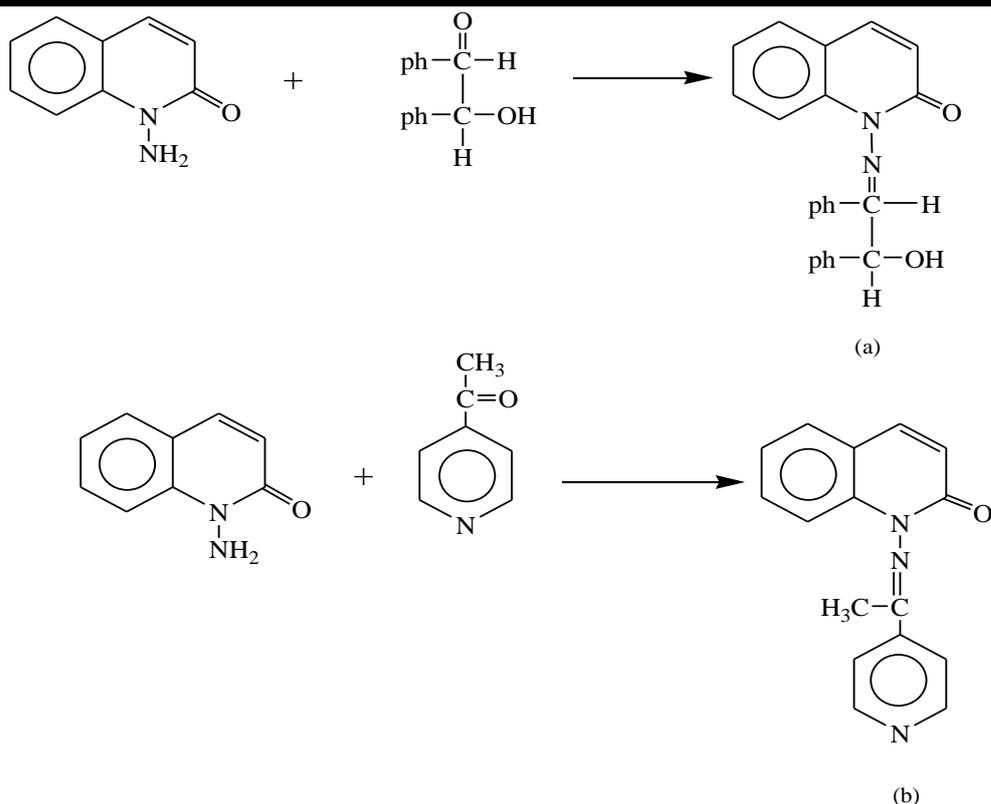


Fig. (a,b)

### Preparation of metal complexes

The hot ethanolic solution of ligands (0.002 mol) in 20 ml and hot ethanolic solution of corresponding metal salt (0.001 mol) in (20 ml) were mixed together with stirring. The mixture was refluxed for 2 hrs at 60-80°C. The product was filtered and washed with cold ethanol and dried under vacuum, all the required weight needed to prepare the complexes were listed in Table (1).

### Antibacterial activity:

The antibacterial activity estimated against *staphylococcus aureus*, as gram positive and *E. Coli* and *Pseudomonas aersginosa* as gram negative, and evaluated by using of agar disc diffusion method on the basis of the size of inhibition zone formed around the paper discs. For each concentration, the mean diameter (mm) of inhibition zone developed was calculated. The test compounds (200 mg 1 ml) in measured quantities were dissolved in DMSO to get different concentrations. Twenty five milliliter nutrient agar media was poured in each Petri plates. After solidification, 0.1mL of test bacteria spread over the medium using a spreader. The discs of Whatmann no.1 filter paper having the diameter 5.00 mm, were placed at four equidistant places at a distance of 2 cm from he centre in the inoculated Petri plates. Filter paper discs treated with clindamycin as a standard drug. These Petri plates were kept in refrigerator for 24 h for pre diffusion. Finally, Petri plates were incubated for 24 h 37°C the zone of inhibition was calculated in millimeters carefully.

**Results And Discussion**

The IR spectrum (Table 2) of N-[N-(1,2-diphenyl)ethanol amino] cumarine L<sub>1</sub> and N-[4- $\alpha$ -methyl pyrilidino]cumarine L<sub>2</sub>.

The spectrum of L<sub>1</sub> show moderately strongly bands at 3200 and 1621 cm<sup>-1</sup> assigned as  $\nu$  O-H and  $\nu$  C=N groups respectively. On complexes formation the C=N band shifts to the lower energy frequency while the phenolic O-H group disappears in the prepared complexes this supports the deprotonation and linkage of O atom to the central metal ion<sup>(19)</sup>. A strongly band appearing at 1681 cm<sup>-1</sup> can be assigned to carbonyl group. The spectra band of complexes at (1623-1658) cm<sup>-1</sup> were characterized for the carbonyl group which indicates that the oxygen atom of the carbonyl group was coordinated to the metal ion. The spectrum of ligand (L<sub>2</sub>) exhibited strong band at (1666) belongs to carbonyl and the other strong bands due to  $\nu$  (C=N) ,  $\nu$  (C-N) at 1558, 1245 respectively, while the infrared spectra of the prepared complexes of (L<sub>2</sub>) exhibited  $\nu$  (C=N) in the range of (1558-1512) cm<sup>-1</sup> which indicated the coordination of (L<sub>2</sub>) with metal ions through the nitrogen of azo methane group. The spectra band of complexes at (1637-1614) cm<sup>-1</sup> were characterized for carbonyl group which indicates that the oxygen atom of carbonyl group was coordinated to the metal ion.

The molar conductance of chelate complexes no.(1-6) were (157-173) ohm<sup>-1</sup> mol<sup>-1</sup> cm<sup>2</sup> which are corresponding conductivity complexes indicating 1:2 electronic nature. Were the complexes no.(8-12) gives low values (14-31) ohm<sup>-1</sup> mol<sup>-1</sup> cm<sup>2</sup> indicating non-electrolyte.

**Electronic spectra and magnetic behavior of the complexes**

Iron complexes: The magnetic moment values at room temperature (2.97-3.89) B.M for the Fe(II) complexes. These value for these complexes were in accord to the literature<sup>(20)</sup> .

The electronic spectra showed bands at (91910-10128) cm<sup>-1</sup> which attributed to the <sup>5</sup>T<sub>2g</sub> <sup>5</sup>E<sub>g</sub> transition and could be assigned to distorted octahedral structure.

Manganese (II) complexes: The magnetic moments values of these complexes were (5.97-6.02) B.M corresponding to five unpaired electrons <sup>(21)</sup>. The electronic spectra of these complexes exhibit three absorption band at (20584, 23038 and 25906)

cm<sup>-1</sup> with L<sub>1</sub> and (22322,31005)cm<sup>-1</sup> with L<sub>2</sub>. These values due to the <sup>6</sup>A<sub>1g</sub> <sup>4</sup>T<sub>1g</sub>(4G),

<sup>6</sup>A<sub>1g</sub>  $\rightarrow$  <sup>4</sup>T<sub>2g</sub>(4G), <sup>6</sup>A<sub>1g</sub>  $\rightarrow$  <sup>4</sup>E<sub>g</sub>(4G) transitions respectively. Because above result we can suggest octahedral geometry for the Mn(II) complexes<sup>(22)</sup>.

Cobalt (II) complexes: Co(II) chelate complexes showed magnetic moments in the range (4.69-4.92 B.M) at room temperature corresponding to three unpaired electrons. The electronic spectra of these complexes, recorded in DMF solution

exhibit absorption band in the region (10060-12951), (15050-16722), 25000 and 28770cm<sup>-1</sup>.

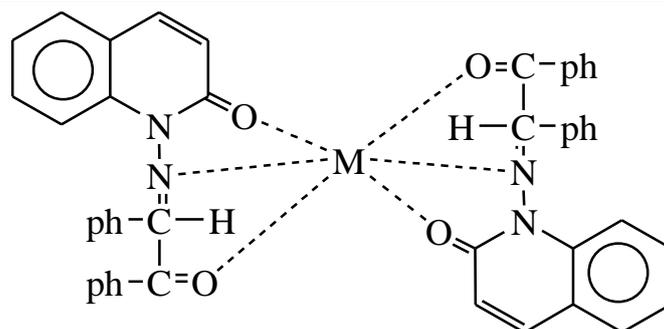
These bands may be assigned to  ${}^4T_{2g(F)} \rightarrow {}^4T_{2g(F)} \nu_1$ ,  ${}^4T_{2g(F)} \rightarrow {}^4A_{2g(F)} \nu_2$ , and  ${}^4T_{2g(F)} \rightarrow {}^4T_{1g(P)} \nu_3$ , transition respectively. The later band may be due to charge transfer. The position of these bands suggest an octahedral environment around to Co(II) ion<sup>(23)</sup>.

Nickel (II) complexes: At room temperature these complexes show magnetic moments in the range (3.24-3.09) B.M these values are in turn with a high- spin  $t_{2g}^6e_g^2$  configuration and shows the presence of an octahedral environment around Ni(II) ions in the complexes.

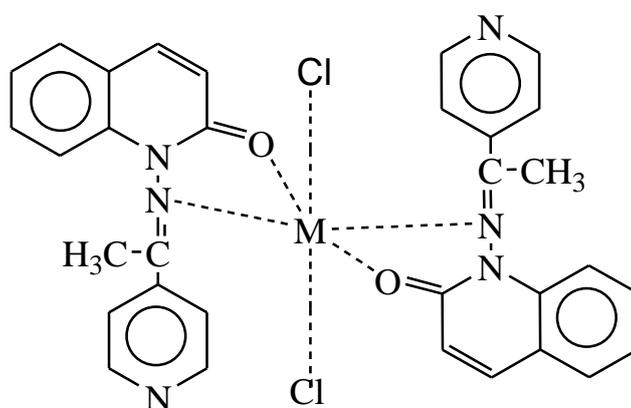
The electronic spectra of Ni(II) complexes shows three bands at 12112, 14817, 16129 cm<sup>-1</sup> corresponding to the three spin allowed transition  ${}^3A_{2g(F)} \rightarrow {}^3T_{2g(F)} \nu_1$ ,  ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(F)} \nu_2$  and  ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(P)} \nu_3$  respectively. These bands indicates that the complexes have octahedral geometry<sup>(24)</sup>.

Copper complexes: The magnetic moments of the two Cu(II) complexes at room temperature have the values (1.77- 2.22) B.M corresponding to one unpaired electron, electronic spectra of copper (II) complexes display bands at 10729, 20415 and 23255 cm<sup>-1</sup> assigned to the  ${}^2E_g \rightarrow {}^2T_{2g}$  transition in distorted octahedral structure around the Cu(II) ions<sup>(25)</sup>.

The magnetic susceptibility showed that all Zn(II) complexes<sup>(6,12)</sup> were diamagnetic and the electronic spectra of these complexes display bands at the rang (25906-30976) cm<sup>-1</sup> due to charge transfer .



Complexes (1-6)



Complexes (7-12)

Where M: Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II).

**Fig.(1): The suggested of structure complexes**

### Antibacterial activity

The Schiff base ligand, was found to be biologically active. It is known that chelation tends to make ligands act as more powerful and potent bactericidal agent. The value indicate that the metal complexes had a higher antibacterial activity than the free ligand by increasing activity of the metal complexes can be explained on the bases of the overtone concept and chelation theory<sup>(19)</sup>. According to the overtone concept of the cell permeability, the lipid membrane that surround favors the passage of only lipid soluble materials, due to which lipo solubility is an important factor controlling the antimicrobial activity On chelation the polarity of the metal ion is reduced to a great extent due to the overlap of the ligand orbital and the partial sharing of the positive charge of the metal ion with donor groups. Furthermore, it increases the delocalization of electrons over whole chelate ring and hances the lipo philicity of the complex. This increased lipo philicity enhances the penetration of the complex into the lipid membrane and blocks the metal binding sites on the enzymes of the microorganism antibacterial activity (Table 4-5) was leads to the following conclusion the metal complexes show more activity than the

ligands against tested bacteria antibacterial activity of Cu(II) complexes have higher activity than the other complexes, in *staphylococcus Aureus* while in Zn(II) complexes have higher activity against tested bacteria *Klebsiella pneumonia* , *pseudomonas*

*Aeruginosa* (Table -5-).

**Table 1: Physical properties of ligands and their complexes**

No.	complexes	Colour	Wt of Metal	M.P	Yield %	Molar conductivity $\text{ohm}^{-1}, \text{cm}^2, \text{mol}^{-1}$	Elemental analysis found (calculated)
	L <sub>1</sub>	Dark yellow	0.72	176	81		
1.	[Mn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Ridsh	0.2	243	89	163	7.068(7.18)
2.	[Fe(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Drak green	0.18	207	85	157	7.28(7.97)
3.	[Co(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Gray	0.23	189	76	167	7.55(8.14)
4.	[Ni(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Green	0.23	166	91	161	7.62(8.65)
5.	[Cu(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Yellow	0.12	194	88	159	8.25(8.90)
6.	[Zn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Pale yellow	0.14	232	68	173	8.49(9.05)
	L <sub>2</sub>	Brown	0.53	196	57		
7.	[Mn(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Dark yellow	0.2	290	72	20	7.91(9.01)
8.	[Fe(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Dark brown	0.18	203	85	14	8.05(8.89)
9.	[Co(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Brown	0.23	211	89	15	8.45(9.90)
10.	[Ni(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Dark gray	0.23	280	79	27	8.42(9.98)
11.	[Cu(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Green	0.12	222	92	31	9.11(10.86)
12.	[Zn(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	Pale yellow	0.14	241	80	22	9.37(11.30)

Table 2: Magnetic moment and electronic spectra data of the complexes

No.	Complexes	$U_{eff}$	$\lambda_{max} \text{ cm}^{-1}$
1	$[\text{Mn}(\text{L}_1)_2]\text{Cl}_2$	5.97	20584, 23038, 25906
2	$[\text{Fe}(\text{L}_1)_2]\text{Cl}_2$	2.97	91910
3	$[\text{Co}(\text{L}_1)_2]\text{Cl}_2$	4.69	10060, 15576, 16393, 25000
4	$[\text{Ni}(\text{L}_1)_2]\text{Cl}_2$	3.24	14409, 16129, 25641
5	$[\text{Cu}(\text{L}_1)_2]\text{Cl}_2$	2.22	12853, 121929, 23902
6	$[\text{Zn}(\text{L}_1)_2]\text{Cl}_2$	Dia	25906
7	$[\text{Mn}(\text{L}_2)_2]\text{Cl}_2$	6.02	22322, 31005
8	$[\text{Fe}(\text{L}_2)_2]\text{Cl}_2$	3.89	10128, 23923, 32495
9	$[\text{Co}(\text{L}_2)_2]\text{Cl}_2$	4.92	15050, 16722, 25773
10	$[\text{Ni}(\text{L}_2)_2]\text{Cl}_2$	3.09	24752, 31021
11	$[\text{Cu}(\text{L}_2)_2]\text{Cl}_2$	1.77	10724, 23255
12	$[\text{Zn}(\text{L}_2)_2]\text{Cl}_2$	Dia	26315, 30976

Table 3: Infrared spectral data of the ligands and their metal complexes.

No.	Complexes	$\nu\text{C=O}$	M-N	M-O	C=N	C-N	C-H
	L <sub>1</sub>	1681			1577	1207	2988
1	[Mn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1647	546	459	1500	1221	2965
2	[Fe(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1624	589	472	1570	1213	2967
3	[Co(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1624	540	434	1508	1213	2965
4	[Ni(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1658	509	438	1579	1211	2963
5	[Cu(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1658	526	453	1591	1211	2963
6	[Zn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1623	549	463	1531	1202	2956
	L <sub>2</sub>	1666			1558	1245	3012
7	[Mn(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1637	597	486	1515	1198	2989
8	[Fe(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1614	567	484	1558	1196	2987
9	[Co(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1635	581	418	1512	1196	2991
10	[Ni(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1616	570	472	1520	1196	2994
11	[Cu(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1616	574	424	1525	1200	2978
12	[Zn(L <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	1616	589	464	1517	1196	2990

Table-4- the antibacterial activity of ligands and metal complexes against the tested bacteria.

No .	Complexes	<i>Proteus Mirabilis</i>	<i>Klebsiella Pneumonia</i>	<i>Staphylococcus Aureus</i>	<i>Pseudomonas Aeruginosa</i>
	L <sub>1</sub>	----	----	----	----
1	[Mn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	----	----	----	----
2	[Fe(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	----	----	----	----
3	[Co(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>		----	----	15
4	[Ni(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	6	6	6	----
5	[Cu(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	6	8	8	15
6	[Zn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	7	15	7	20

the antibacterial activity of ligands and metal complexes the zone of inhibition was measured in mm(concentration in ppm).

Table-5- The minimum inhibitory concentration (MIC) for the compounds against tested bacteria.

No .	Complexes	<i>Proteus Mirabilis</i>	<i>Klebsiella Pneumonia</i>	<i>Staphylococcus Aureus</i>	<i>Pseudomonas Aeruginosa</i>
	L <sub>1</sub>	----	----	----	----
1	[Mn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	----	----	----	1.5
2	[Fe(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	----	----	----	----
3	[Co(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	----	----	----	12.5
4	[Ni(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	100	100	100	----
5	[Cu(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	100	3.125	25	1.5
6	[Zn(L <sub>1</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	100	1.5	100	1.5

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تحضير ودراسة طيفية وبأبولوجية لمعقدات المنغنيز ، الحديد ، الكوبلت ، النيكل ،  
النحاس والزنك الثنائية مع مشتقات N امينو كوينوتين

تاريخ القبول: 2014\2\9

تاريخ الاستلام: 2014\10\4

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الخلاصة :

تم تحضير عدد من معقدات جديدة لبعض العناصر الانتقالية مثل (Mn(II)، Fe(II)، Co(II)، Ni(II)، Cu(II) فضلا عن Zn(II) مع قواعد شيف الناتجة من التفاعل التكتيفي بين البنزوين و اسيتيل بريدين مع n-امينوكوينولين-2-ون شخضت المعقدات المحضرة بواسطة التحليل الدقيق للعناصر (M) و قياسات التوصيلية المولارية و القياسات المغناطيسية و الأشعة تحت الحمراء والأطياف الالكترونية، وقد أتضح من هذه الدراسات أن الليكاندين يتناسقان من خلال ذرتي النيتروجين مجموعة الازو ميثان وذرتي الأوكسجين مجموعة الكربونيل اضافة الى مجموعة الهيدروكسيل بالنسبة لليكاند الاول وقد تبين ان جميع المعقدات المحضرة ثمانية السطوح غير الكتروليتية، كما تم قياس الفاعلية الحيوية لليكاندين مع المعقدات المحضرة.