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FORWARD

With well- established ambitious steps on continuing success way, IJST is coming for you all today in its new volume, the seventh volume for year 2012.

Year after year, IJST proves its strength and faithful belief in developing our scientific communities among Arab World, especially in Iraq by giving an opportunity to all researchers to present their fruitful achievements in main vital fields to let all world knows that we are still the first leaders in civilized scientific life, despite all the unfortunate situations or constraints.

It is my pleasure to welcome you and present you a new issue of our Journal, Volume 7, No. 4 (2012), the fourth issue of this year, with diversity of researches and elite experts of the Editorial Board and Advisory Group. The members of Editorial Board, the ICAST and TSTC teamwork and I hope you will find this collection of research articles useful and informative.

IJST has owned a new ISSN registration number, that is: ***2305-9346*** *instead of the previous one, as the first volumes in 2006 issued by Ibn alhaythum, any change in the title needs a new ISSN according to the International Standardization Organization, and this step had been taken for ensuring the high quality and standards of our journal for being internationally recognized.*

The journal is one of the scientific contributions offered by ***the International Centre for Advancement of Sciences and Technology*** *in cooperation with* ***Treasure Est. for Scientific Training and Consultations*** *to the science and technology community (Arab region with specific focus on Iraq and International).*

Finally, on behalf of the International centre, I would like to express my gratitude and appreciation to the efforts of the Editorial Board, Advisory group with their valuable efforts in evaluating papers and the Editorial Board Secretary for managing the scientific, design, technical and administrative aspects of the Journal and for preparing this issue for final printing and publishing.

Editor-in-Chief

IJST

Abdul Jabbar Al- Shammari

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Synthesis, characterization and Antibacterial activities of some metal (II) heterocyclic polyamine complexes with 6,6'-(1,4-phenylenebis(azanediyl) bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol) ligand.

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ABSTRACT

A new heterocyclic polyamine compound as ligand(L) (6,6'-(1,4-phenylenebis(azanediyl) bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol) has been synthesized through a reaction of urea with compound (Dimethyl3,3'-(1,4phenylenebis(azanediyl))dibut-2-enoate) (1) in strong alkaline solution at low temperature which has been prepared from a reaction of 1,4- phenylenediamine with methyl acetoacetate in (1:2) mole ratio.
The prepared ligand was characterized by (FT IR,UV–Vis) , H^1NMR spectra , and melting point.
The ligand was reacted with some metal ions under reflux in ethanol with
(1 metal :1 ligand) mole ratio which gave complexes of the general formula:
$[M(L)_2]Cl_2$, M= Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) and Hg(II), L= $C_{16}H_{20}N_6O_4$.
Products were found to be solid crystalline complexes, which have been characterized through the following techniques:
Molar conductivity .Spectroscopic Method [FTIR and UV-Vis], additional measurement magnetic suspeliblity, Chloride content and Program [Chem. office–CS. Chem.–3D pro 2006]was used.
Our research includes studying the bio–activity of the complexes . The magnetic moment coupled with the electronic spectra suggested an octahedral geometry for all the complexes .

Key words: heterocyclic compound , polyamine complexes , Antibacterial activities , spectral studies.

الملخص باللغة العربية

تم تحضير المركب متعدد الامين الغير متجانس الحلقة

(L)= (6,6'-(1,4-phenylenebis(azanediyl) bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol)

كليكاند من تفاعل (1) Dimethyl3,3'-(1,4phenylenebis(azanediyl))dibut-2-enoate مع اليوريا وبدرجة حرارة منخفضة والناتج من خلال تفاعل 4–ثنائي امين الفنيل مع مثيل اسيتو اسيتيت بنسبة مولية (1:2), تم تشخيص الليكاند بمطيافيات (FT IR,UV–Vis) , H^1NMR)
كما تم مفاعلة محلول الليكاند المحضر في الايثانول مع محلول الايونات الفلزية بنفس المذيب بنسبة مولية تحت التصعيد لتعطي معقدات بالصيغة العامة (1فلز 1:ليكاند)

$[M(L)_2]Cl_2$, M= Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) and Hg(II),
L= $C_{16}H_{20}N_6O_4$

المعقدات المحضرة بلورات صلبة درست من النواحي الآتية: الاستقرارية الحرارية، التوصيلية الكهربائية المولارية، الذوبانية، تقدير النسبة المئوية للأيون الفلزي في المعقدات بوساطة مطيافية الامتصاص الذري، الدراسات الطيفية: وتضمنت أطياف (الأشعة تحت الحمراء، الأشعة فوق البنفسجية– المرئية، الخواص المغناطيسية ومحتوى الكلور) مع استعمال البرنامج . (Chem Office– Cs. chem– 3D pro 2006) في رسم اشكال المعقدات. كما تم دراسة الفعالية البايولوجية للمعقدات ., قيم العزوم المغناطيسية والأطياف الالكترونية لجميع المعقدات دلت على أن جميع المعقدات لها بنية ثماني السطوح.

INTRODUCTION

The transformations of organic compounds belong to one of the following two broad categories: carbon–carbon bond-forming reactions and redox processes. Over the years, remarkable progress has been achieved in design and applications of novel metal-based complexes in oxidation chemistry(1). The oxidation of aromatic amines in human erythrocytes is very useful for producing phenoxazine compounds via the intervention of human oxyhaemoglobin .One of the main objectives has been the elucidation of the oxidation product of the aromatic amines, which has been shown to be a phenoxazine (2,3). Synthetic methodologies for the preparation of aziridines include: (1) nitrene addition to olefins (4), (2) carbene (5) and ylid (6) addition to imines; and (3) cyclization of 1,2-aminoalcohols, 1,2-aminohalides, and 1,2-azidoalcohols (7). Olefin aziridination reactions are typically accomplished via metal-mediated transfer of a nitrene fragment to the olefin (8). These metal-catalyzed reactions originate from Mansuy's study on the Fe-porphyrin and Mn-porphyrin complexes (9).
Among a wide variety of nitrogen heterocyles that have been explored for developing pharmaceutically important molecules, the quinazoline have played an important role in medicinal chemistry and subsequently have emerged as a pharmacophore (10).
In the present work, we have synthesized the6,6'-(1,4-phenylenebis(azanediyl) bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol) ligand (L), Then, its new M= Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) and Hg(II) complexes were synthesized by reaction of (L) and MCl_2 salts. The complexes were formulated on the basis of analytical, spectral and magnetic data.

MATERIALS AND METHODS

1. Chemical and Instrumentals:

All chemicals used were of reagent grade and were used without further purificon $MnCl_2.4H_2O$, $CoCl_2.6H_2O$ $FeCl_2.9H_2O$,$NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $HgCl_2$ were supplied by (Fluka) chemical DMF , THF and Ethanol ,from Merck (pure) and used without further purification.

b -UV-Vis spectra were recorded on a (Shimadzu UV- 160A) Ultra Violet-Visible Spectrophotometer. IR- spectra were taken on a (Shimadzu, FTI R- 8400S) Fourier Transform Infrared Spectrophotometer (4000-400) cm^{-1} with samples prepared as KBr discs. 1H NMR spectra of intermediate material(1)and ligand(L) using DMSO-d6 solvent were scanned on (EOL ltd) Model. Dalta2-NMR-400MHz , while metal contents of the complexes were determined by atomic absorption(A.A)technique using a Shimadzu AA 680G atomic absorption spectrophotometer.. Conductivities were measured for 10^{-3}M of complexes in DMF at 25^{o}C using (Philips PW- Digital Conduct meter). Magnetic measurements were recorded on a Bruker BM6 instrument at 298°K following the Farady's method . In addition melting points were obtained using (Stuart Melting Point Apparatus). The proposed molecular structure of the complexes were drawing by using chem. office program,3DX (2006).

2. Preparation of the ligand(L) and its complexes:

2.1 Preparation of intermediate material(1) (11):
Intermediate material (Dimethyl3,3'-(1,4phenylenebis(azanediyl))dibut-2-enoate) (1) was Prepared according to the general method shown in figure (1).
(90%) The Product was collected by filtration, and recrystallized from benzene.
The melting point of the product found to be (182 ^{o} C)

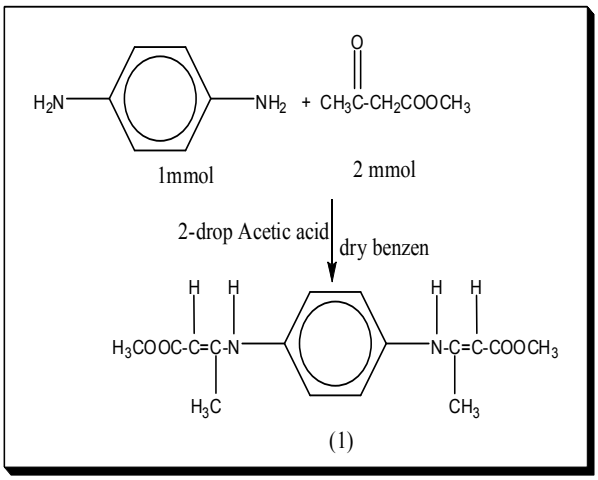

Figure (1):Schematic representation Preparation of the intermediate material(1) Dimethyl3,3'-(1,4phenylenebis(azanediyl))dibut-2-enoate

NMR Spectra. 1H NMR spectra of intermediate material(1) exhibits a singlet at δ $\sigma_{=}$10.30ppm due to amino group protons and $\sigma_{=}$7.69ppm due to the aromatic ring protons., $\sigma_{=}$4.69ppm
due to CH_3 attached (carbon atom number (4) and at $\sigma_{=}$ 3.67ppm due to (6H)ester(CH_3).Figure. (2).
Selected IR data (KBr): v 3255cm^{-1} (NH) , v2978c m^{-1} aliphatic (CH) , v1600cm^{-1}-1512cm^{-1} C=C(Ar) , v1253cm^{-1}(C=O)ester group ,strong band v1157cm^{-1} (C-N))v1658 cm^{-1} olefin(C=C) (13,14). Figure (3).

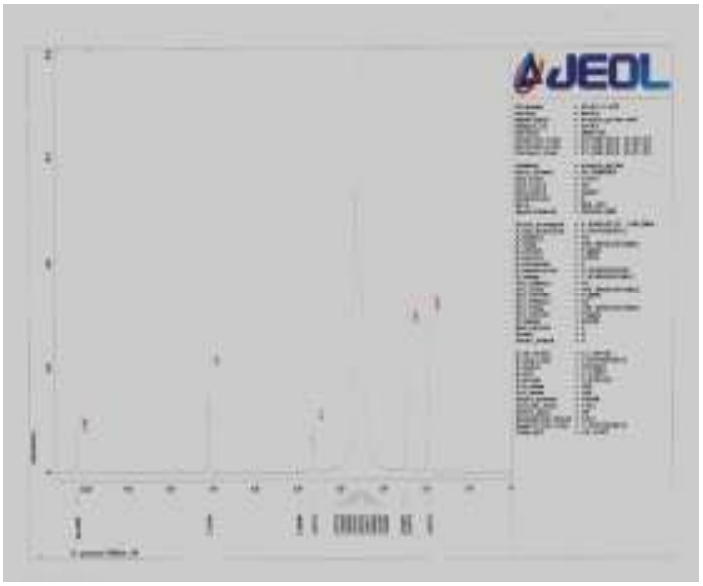

Figure (2). ^{1}H NMR spectrum of Dimethyl3,3'-(1,4phenylenebis(azanediyl)dibut-2-enoate (1)

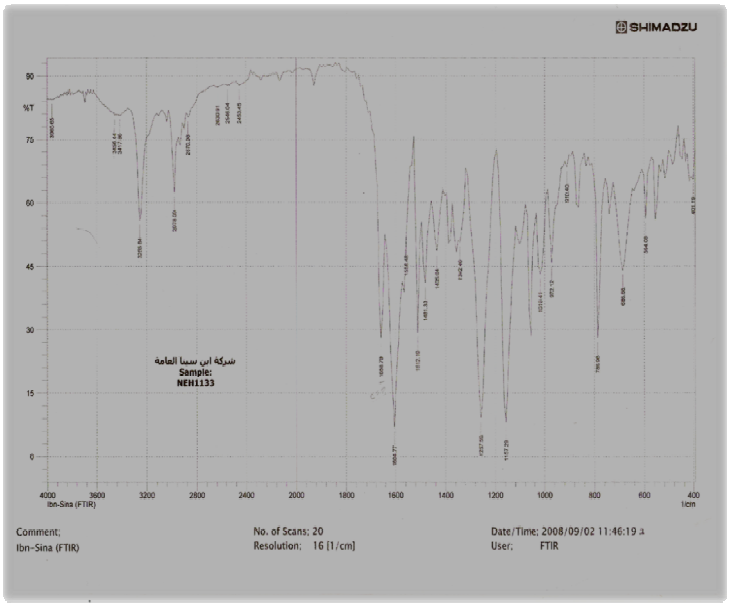

Figure (3) FTIR spectrum of Dimethyl3,3'-(1,4phenylenebis(azanediyl)dibut-2-enoate(1)

2. Preparation of ligand (L) (11):
the ligand(L) 6,6'-(1,4-phenylenebis(azanediyl) bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol) was Prepared according to figure (4).
NMR Spectra: 1H NMR spectra of (L) exhibits a singlet at δ σ=10.30ppm due to (NH_2) group protons , and σ=7.69ppm due to the aromatic ring protons., σ=10.22pmm for proton above nitrogen atom ,and σ=4.65ppm . Figure. (5)
(H) for proton(OH)group. Figure.(6).
selected IR data (K Br)v3271.2cm^{-1} (OH), v3271.2cm^{-1}(N-H), v1512-1600cm^{-1}(C=C)(Ar)
v 1161cm^{-1} (C-O-C), v 1481cm^{-1}(NH_2) , v 1235cm^{-1} (C-N) , v 1661cm^{-1} (C=N [11-13]. Figure. (7)

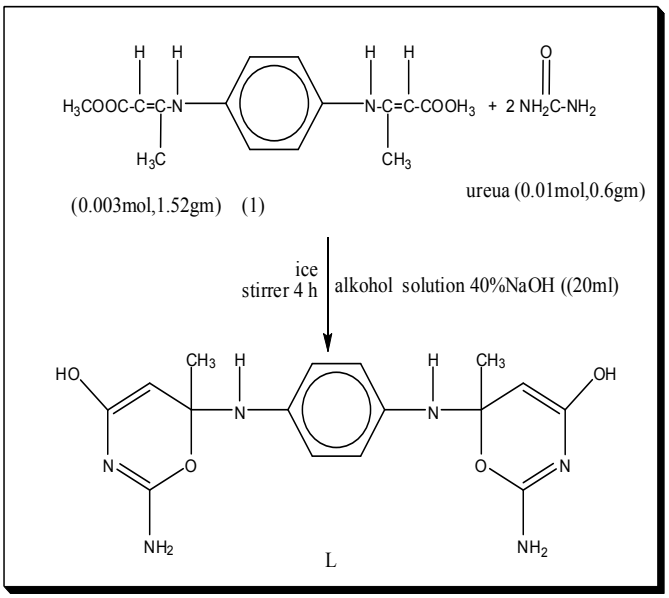

Figure (4): Schematic representation Preparation of the ligand (L) 6,6'-(1,4-phenylenebis(azanediyl))bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol)

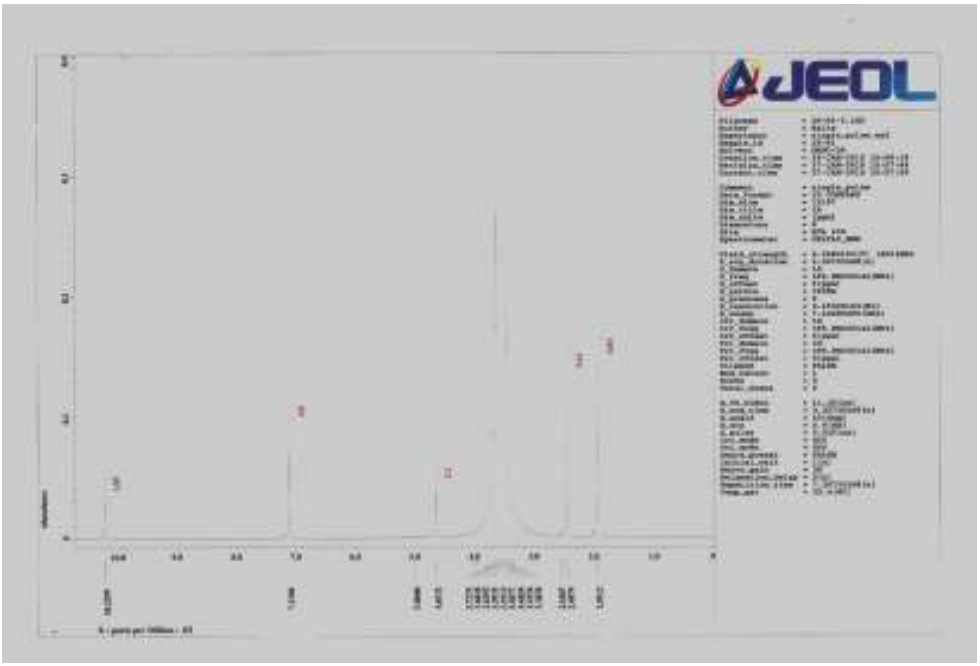

Figure (5) ^{1}H NMR spectrum of 6,6'-(1,4-phenylenebis(azanediyl))bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol)(L)

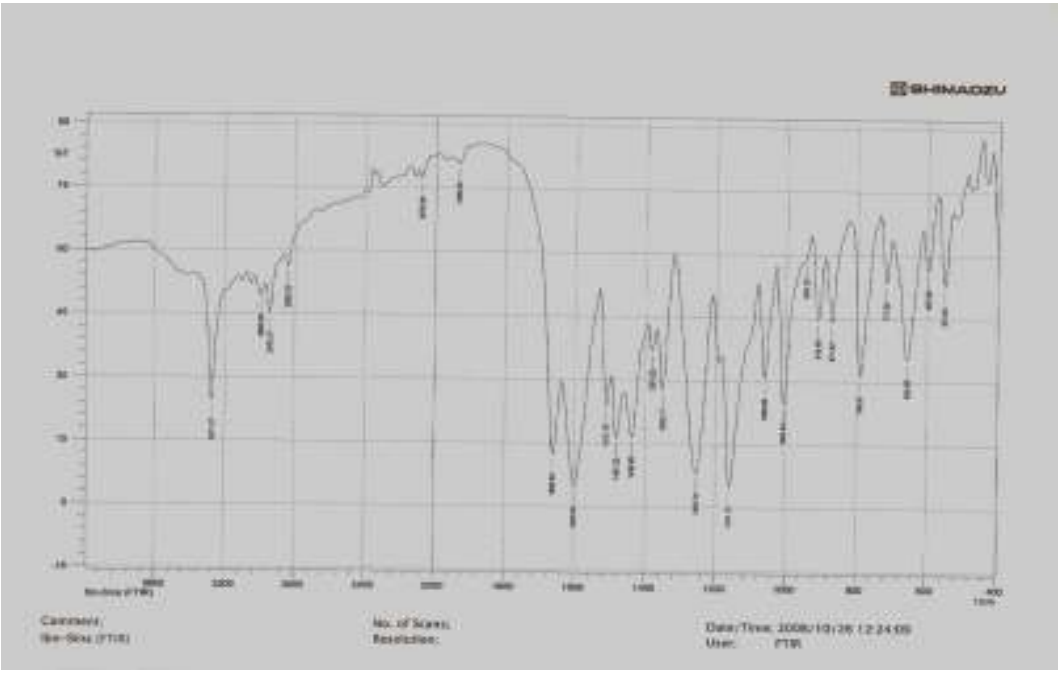

Figure (6) FTIR spectrum of 6,6'-(1,4-phenylenebis(azanediyl))bis(2-amino-6-methyl-6H-1,3-oxazin-4-ol) (L)

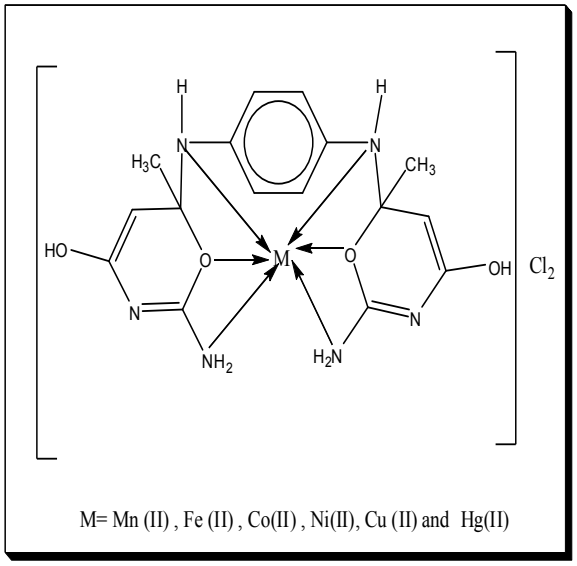

Figure (7): Suggested structure of the octahedral M(II)complex of the ligand, (L).

3 Synthesis of the Complexes*:*

All complexes were prepared by dissolving 0.39g 0.098 g , 0.118 g , 0.118 g , and 0.085 g ,0.271g 1mmole) of $FeCl_2.9H_2O$,$MnCl_2.4H_2O$, $CoCl_2.6H_2O$,$NiCl_2.6H_2O$ $CuCl_2.2H_2O$ and $HgCl_2$ respectively in (20 ml) THF in a 100 ml round-bottom flask. .A solution of(1mmol of L) in(20 ml) ethanol were added simultaneously to a solution of $MCl_2.nH_2O$ (1 m mol) in (20 ml) THF in the stoichiometric ratio (1:1)(M:L). The above solution was heated for 6o minute. The mixture was refluxed for 2 hrs. The reaction mixture was then further stirred for 2 hrs at room temperature. The product formed was filtered off ,washed with aqueous ethanol (1:1) and dried in air ,and analyzed employing standard method . Decomposition. temp: >300 ºC,

4. Preparation of Microorganism suspension (15):

A) The micro- organism suspension was prepared by taking 2–4 colonies from all the studied micro- organism. Then it was inserted in the physiological solution in 0.85% concentration and was compared with Macferr land tube number 0.5 which is equal to 1.5×108 cell/mm. It is used for Petri dish preparation for the examination of biological activity against the under studied chemical compound.

B) Inhibition Activity Selection for the complexes in studied Micro-organism.

The agar well diffusion method was used to see the effect of under studied chemical complexes on the micro-organism growth. This is done by using 20–25 ml from Nutrient agar medium for each Petri dish. The dish was incubated in incubator for 24 hours at (37°C) to make sure that no contamination would occur in the dish.

The dish was wetted in 10 milliliters of micro-organism which was prepared as mentioned in the previous paragraph which include 1.5×108 cell/mm. Distributed evenly on the Nutrient Agar medium surface by using spreader. Bore was made on the cultured medium surface by using cork borer. The chemical complexes were made as 100 m ml per bore and left the central bore containing only DMF. The dishes were left for 1/2 hour in refrigerator at 4°C (12). The dishes were incubated at (37°C) for 24 hours. The biological activity for the complexes was defined by measuring the diameter of the inhibition area surrounding each bore in millimeters.

RESULTS AND DISCUSSION

The Physical properties listed in(Table -1). Some the complexes are colored, non-hygroscopic, and appears as powders with high melting points .They are not soluble in water. All complexes dissolved in dimethyl formamide (DMF) solvent.

The complexes were analyzed for their metal by atomic absorption measurements and chloride contents were determined by standard

methods (16).(Table-1) for all complexes gave approximated values for theoretical values. The observed molar conductance (Table 1) values measured in DMF in 10-3M solution lie in the (133-164 Ω^{-1} cm^2 mol^{-1} range, indicating their electrolytic nature with(1: 2). (17)

Table (1) The physical properties of the compounds

Compound	Yield %	M. wt	Color	Mp °c (de)	*M.C µS.cm^{-1} in DMF	Metal%	
						theory	Exp
M			Pale –yellow	182	-	-	-
L= $C_{16}H_{20}N_6O_4$	75	360.4	Yellow	>300(de)	-	-	-
$[MnL]Cl_2$	79	486.21	Pink	(de)200	138	11.30	
$[FeL]Cl_2$	85	487.12	Dark red	(de)260	149	11.46	
$[CoL]Cl_2$	75	490.21	Dark green	(de)300 >	164	12.02	
$[NiL]Cl_2$	85	489.97	Red	(de)300 >	164	11.98	
$[CuL]Cl_2$	80	494.82	Green	(de)250	133	12.84	
$[HgL]Cl_2$	70	631.86	yellow	(de)260	143	31.75	

M.C = Molar Conductivity, L= $C_{16}H_{20}N_6O_4$, de = decomposition

Magnetic Susceptibility:

The magnetic moments obtained at room temperature for the complexes of Cu(II), Ni(II) , and Co(II) are listed (Table 1). Cu(II) complex exhibits magnetic moment 1.98 B.M. which is less than the normal value17 (1.84-2.20 B.M.). The lowered magnetic moment value observed for Cu(II) complex under present study is due to distorted octahedral geometry (15) .The Co(II) complex shows magnetic moment of 4.86 B.M. the spin free octahedral complex of Co(II) are reported to exhibit magnetic moment in the range of 4.46-5.53 B.M.19. Hence observed magnetic moment for the Co(II) complex under study indicates it has an octahedral configuration. The Ni(II) complex shows magnetic moment of 2.90 B.M. The magnetic moment of octahedral Ni(II) complex are reported to exhibit magnetic moment in the range of 2.80 – 3.40 B.M.20 including spin orbital coupling contribution from 3A2g and higher 3T2g states. Hence the observed magnetic moment for the Ni(II) complex suggest that it may have octahedral geometry (14,18,19).

Fourier-transform infrared spectra and mode of coordination :

As shown in Table 2, the IR absorption frequencies were different for free (L) and M(II) complexes with different functional groups. In the IR spectrum of ligand, the characteristic peaks are at 3271-2993 cm^{-1}, which are assigned to ν(N-H) and ν(-NH_2) and 1161 cm^{-1} that is assigned to the ν(C-O-C) group and shows strong band in the 1661 cm^{-1} due to (-C=N-) (17,18).Some new bands of weak intensity observed in the regions aroundν (684-570) cm^{-1} and ν (489-526) cm^{-1} may be ascribed to the ν(M-N) and ν(M-O) vibrations respectively. It may be noted that these vibration bands are absent in the infrared spectra of ligand (19,20-24).

Electronic spectra :

The UV-Visible Spectroscopy and Magnetic measurements shown in Table (3) . The electronic spectral data of the free ligand (L) absorption bands appears at 31250 cm^{-1} due to $n \rightarrow \pi^*$ transition.

The Co(II) complex (dark green) of the electronic absorption bands appears at 34965 cm^{-1} Ligand field**,** 24271 cm^{-1}, 13054 cm^{-1}, and10775 cm^{-1}, due to 4T1g (F) → 4A2g, 4T1g (F) → 4A2g(P) and 4T1g (F) → 4A2g (F) transition, respectively, in an octahedral environment .

The electronic spectra of complexes 1, show multiple bands, which are assigned to $2Eg \rightarrow 2T2g$ and CT transition characteristics of the *d*9 system. Hence, a distorted octahedral geometry was proposed for the copper complexes (23-25).

The electronic spectra of the nickel(II) complexes exhibited three typical absorption bands at-10270, 19055 and 27085 cm^{-1}, corresponding to the transitions 3AEg → 3T2g (VI), 3AEg→ 3Tlg (F)(V2), and 3A2g → 3TEg (F)(V3), respectively, characteristic for their octahedral environment. Also, the values of the magnetic moment (2.95)may be taken as additional evidence for their octahedral structure (21-24).

On the basis of the above observations, it is tentatively suggested that all of the complexes show an octahedral geometry in which the ligand act as sixdentates . Figure (7) These possibly accommodate themselves around the metal atom in such a way that a stable chelate ring is formed giving in turn, stability to the formed metal complexes. (23–25)

Finally, the diamagnetic Hg (II) show absorption band at 350 nm (28571 cm^{-1}) for the ligand metal charge transfer MLCT as the electronic configuration of these complexes confirmed the absence of any d-d transition and this confirms the presence of an octahedral geometry in the Hg (II) complex.

Biological evaluation:

The newly synthesized metal complexes were screened *in vitro* for their antibacterial activity against bacteria: *Salmonella Typhi*,*Escherichia coli and* Staphylococcus aureus.

The antibacterial activity results revealed that the complexes shown weak to good activity. Table (4).

Table (2) :- Data from the Infrared Spectra for the Free Lingand and its Metal Complexes (cm^{-1}).

Compound	OH , N-H	NH_2	C=N	(C=C) arom	C-N	C-O-C	(Ar-CH)	M-N	M-O
M	3255	1481	1658 s	1512-1600	1235	1161m	3022	-	
L	3271	2993	1661 m	1512-1600	1235	1161	-	-	-
$[MnL]Cl_2$	3423	2959	1654 s	1560s	1240	1116m		623s	489 m
$[FeL]Cl_2$	3383vs	2958	1635 m	1558m	1205w	1112m	-	650m	503 m
$[CoL]Cl_2$	3376vs	2964s	1628 s	1560s	1240	1159m	3025	659m	526 m
$[NiL]Cl_2$	3442	2958	1629 s	1558m	1205w	1159m	3024	684w	507 s
$[CuL]Cl_2$	3356s	2958	1624 s	1579-1624	1276vs	1159m	3032s	570w	495m
$[HgL]Cl_2$	3373 s	2956	1647 s	1525-1498	1274	1180m	3035	623 s	515

Table (3): The Electronic Spectra for the Free Ligand and its Complexes in (10^{-3}M) in DMF

Complexes		λ_{nm}	$\upsilon'(cm^{-1})$	Assignments	μ_{eff} (BM) (temp. K)	
	(Ligand)	320	31250	$n\rightarrow\pi^*$	-	
1	$[MnL]Cl_2$	301 417 827	33222 23980 12091	Ligand field $^6A1g \rightarrow {}^4T2g$ (G) $^6A1g \rightarrow {}^4T1g$ (G)	4.90	octahedral
2	$[FeL]Cl_2$	309 388 799	32362 25773 12515	Ligand field C.T $^5T_{2g} \rightarrow {}^5E_{2g}$	4.90	octahedral
3	$[CoL]Cl_2$	286 412 766 928	34965 24271 13054 10775	Ligand field ^4T1g (F) $\rightarrow {}^4A2g$(P) ^4T1g (F)$\rightarrow {}^4A2g$ (F) ^4T1g (F) $\rightarrow {}^4T1g$ (P)	4.86	octahedral
4	$[NiL]Cl_2$	369 524 928	27085 19055 10775	$^3AEg \rightarrow {}^3T2g$ $^3AEg\rightarrow {}^3Tlg$ (F) $^3A2g \rightarrow {}^3TEg$ (F)	2.90	octahedral
5	$[CuL]Cl_2$	263 600	38008 16666	CT $2Eg \rightarrow 2T2g$	2□01	distorted octahedral
6	$[HgL]Cl_2$	350	28571	C.T	Diamag	octahedral

C.T= Charge transfer

Table (4): Antimicrobial activity of the ligands and metal complexes Against *Staphylococcus aureus (+ve) and (Escherichia coli, Salmonella typhi) (-ve*)

Complexes	**Inhibition Zone (mm)**		
	Salmonella typhi (-ve)	***Escherichia coli (-ve)***	***Staphylococcus aureus (+ve)***
DMF	-	-	+
$[MnL]Cl_2$	+	+	++
$[FeL]Cl_2$	+	+	+
$[CoL]Cl_2$	++	+	++
$[NiL]Cl_2$	+	++	+
$[CuL]Cl_2$	+	++	+++
$[HgL]Cl_2$	+	+++	++

(0-6)mm =- (Non active)
(6-9)mm =+ (Slightly active)
(9-12)mm=++ (Moderately active)
(12-17)mm=+++ (Highly active)

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