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Co(II),Cu(II),Ni(II),Fe(II)and Cr(III) Complexes of N,O-Type Schiff base ligand derived from 4,4'-methylenedianiline Synthesis, Characterization and antibacterial studies

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Summary:-

Complexes of Co(II),Cu(II),Ni(II),Fe(II)and Cr(III) were prepared from reaction between (E)-2-(((4-(4-aminobenzyl)phenyl)imino)methyl)phenol and hydrated metal ions such as $CoCl_2.6H_2o$,$NiCl_2.6H_2O$.$FeCl_2.6H_2O$ and $CrCl_3.H_2O$ characterization by analytical and spectra chemical methods .the metal:ligand stoichiometric ratio is 1:1 n the all complexes.it was determined that bidentate behavior of the ligand is accomplished via the phenolic oxygen and the azomethine nitrogen atom .the presence of water revealed by thermogram and supported by the presence of relevant bonds in their IR Spectra. These new complexes showed areasonable amount antibacterial activity.a ligand was synthesized by reaction (4,4'-Methylenedianiline and Salicylaldehyde. The newly synthesized compounds were characterized by Fourier Transform Infrared (FTIR) Spectroscopy, Nuclear Magnetic Resonance Spectroscopy (^1H NMR), elemental analyses (C, H, N), mass spectral, conductivity measurement, magnetic sensitivity measurement and atomic Absorption. The mass spectrum of the ligandexhibits a molecular ion peak $[M]^{+\cdot}$at 302 m/z.

Keywords: Schiff base, Antibacterial, Transition metal complexes.

Introduction:-

Schiff base are formed when any primary amine reacts with an aldehyde or a ketone under specific condition. Structurally, a Schiff base (also known as imine or azomethine) is a nitrogen analogue group (C.O) has been replaced by and imine or azomethine group (Da Silva *et al*., 2011). Schiff base are some of the most widely used organic compound. They are used as pigments and dyes, intermediates in organg synthesis, and as polymer stabilizers (Dhar and Taploo., 1982) .Imine or azomethine group are present in various natural .natural-derived, and non-natural compounds.the imine group present in such compounds has been shown to be critical to theirbiological activities (Guo *et al*., 2007; de Souza *et al*., 2007; Przylbylski *et al*., 2009). However their metal complexes have also been shown to exhibit abroad rang of biological activities, including antifungal, antibacterial, antimalarial, antipro-liferative, antiflammatory, antiviral and antipyretic properties (Dhar and Taploo., 1982; Bringman *et al*., 2004;). In addition, because of the ability to be coordinated of oxygen and nitrogen atoms an the complexes are effective and stereospecific catalysts for epoxidation reactions, catalytic hydrogenation of olefins and hydrolysis (Ali *et al*., 2003; Maurya *et al*., 2003; Ramesh and sivagamasundari., 2003). It is known that some drugs have greater activity when implemented as metal complexes than as free ligands (Ramesh and sivagamasundari, 2003).

Experimental:-

Preparation of the Ligand (LH):-

Salicylaldehyde (1.2g, 0.01mol) and 4, 4-Methylenedianiline (1.98g, 0.01mol) were mixed gently and heated under reflux for (30min) with (30ml) of methanol and few drops from glacial acetic acid (Najeeb., 2011). The crude product was filtered and washed with ethanol to give the desired product. The product color was light yellow (m.p: 188-190) °C, yield 91%, molecular formula: ($C_{20}H_{18}N_2O$), M.Wt: (302). Preparation of the ligand is shown in the scheme below.

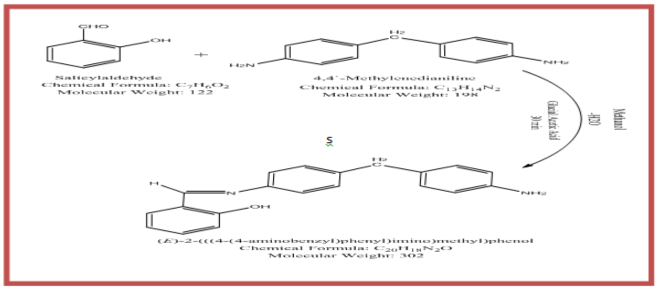


Figure (1) Preparation of the ligand (LH)

Preparation of Complexes:-

The Cr(III), Co(II), Ni(II), Cu(II) and Fe(II) complexes wereprepared by refluxing the respective hydrated metal chloride (0.001mol) in (10ml) Of ethanol with (25ml) of an ethanolic solution of the ligand (0.001mol) for 2 hrs. The separated solids were filtered and washed with ethanol to with water then removed unreacted salts or ligand, then precipitated complexes dried in air (Menati *et al*., 2015).

Biological activity:-

Staphylococcus aureus (positive to gram stain) and Escherichia coli (negative to gram stain) were used as pathogenic bacteria and they were identified in central public health laboratory Misan health director. these type of bacteria were chosen because of their importance in the medical field, were it caused many different diseases. Bacteria suspension of each tested bacteria was spread the surface of Muller-Hinton agar plates. 5mm cork borer was used to punch holes into the plates and 0.1ml of each sample (0.05g) compound dissolve in 1ml of (DMSO) as well as were applied to each hole. The plates were incubated for 24h at 37^0C. The inhibition zones diameter for each extract was measured (Jeremih *et al*., 2007).

Analytical procedure:-

Infrared absorption spectra were recorded on FTIR spectrophotometer on a model (Shimadzu FT-IR Spectrometer) in the range (200-4000) cm^{-1}. ^{1}HNMR spectra were recorded at 500 MHZ utilize a model Bruker DRX, TMS used as standard, DMSO-d^6 used as solvent. Mass spectra (MS) were registered in the range (0-800) m/e on 5973 network mass selective detector. Elemental C, H, Nanalysis were carried out on a Eager 300 for EA1112 elemental analyzer/CHNS. Melting points were specified in open capillary tubes using an electro thermal melting point/SMP31 device. Magnetic sensitivity measurement was done using a device M.S.B.Auto, atomic absorption measured was using a device (pg instruments-model AA500AFG).

Result and Discussion:-

The purity of 2-(((4-(4-aminobenzyl) phenyl) imino) methyl) phenoland its complexes were checked by TLC. The theoretical values for prepared ligand were in a good agreement with the experimental values as shown in Table(1). Some physical properties of prepared ligand and its complexes are listed in Table(2).

Infra-Red Spectroscopy study:-

The FTIR spectra assignments of 2-(((4-(4aminobenzyl) phenyl) imino)-methyl) phenolas well as its bonding sites (Table3**)** and figures 1,2,3,4,5 and 6 have been determined by careful comparison of the spectraof the ligand with those of its metal complexes and by considering our previous publications (Mostafa., 1983; Shokry *et al*., 2000). The ligand spectrum in KBr shows band at 3418cm-^{1} (O-H), 3028cm-^{1} (C-H,aromatic). (Williams and Fleming, 2004; Issa *et al*., 2008) 2920cm^{-1} (C-H, aliphatic) (Parameswari *et al*., 2013), 1640cm-^{1} (C=N) (Aman et al., 2013; Gomathi, 2013) 1566cm-^{1} (C=C, aromatic), 1153cm-1 (C-O, phenolic). There are some bands appeared in the complex spectra which areattributed to M-Cl and M-N, this bands are confirm presence the complex formation. The IR data are shown in table (3) and figures (1-6)**.**

Nuclear Magnetic Resonance (^{1}HNMR):-

The ^{1}HNMRspectrumof the ligand showed signals at (13.2ppm, S, 1H, OH-Phenolic) (Aderoju *et al*., 2003) (9ppm, 1H, S, C=N-H, azomethene) (6.8-7.7ppm,14H,m**,**) 12H return aromatic ring (Shelka *et al*., 2013) and 2H return to (NH_2) associated with the European ring, (4ppm, 2H, S, Ar-CH_2-Ar), (2.5ppm, S, DMSO) (Cao and Wang, 2006;George *et al*., 2008; Kamble *et al*., 2008) (3.5ppm,1H,S,D2O). The proton NMR of the ligand shown in figure (7).

Mass spectra:-

The mass spectrum of the ligand exhibits a molecular ion peak $[M]^+$. (Denizlil *et al.*, 2013; Balasubramanian and Suijathaat, 2013) 302 m/z. the ligand spectra shows fragments at (106,196,286, 120,182,209,93) m/z dueto $[C_7H_8N]^+$, $[C_{13}H_{10}NO]^+$, $[C_{20}H_{16}NO]^+$, $[C_7H_6N]^+$, $[C_{13}H_{12}N]^+$, $[C_{14}H_{13}N_2]^+$, $[C_6H_5]^+$.

respectively as shown in figure (8). The mass spectrum of the complex $[Ni(L_1)\ (Cl_2)\ (H_2O)_2]^+$. shows a molecular ion peak $[M]^{+\cdot}$at(468) m/zwhich is equivalent to molecular mass of the complex and shows another fragments at (450,415,379,361) m/z due to $[Ni(L_1)(H_2O)]^+$, $[Ni(L_1)(H_2O)(Cl)]^+$, $[Ni(L_1)\ H_2O]^+$, $[Ni(L_1)]^+$**.** respectively shown in figure(13). The mass spectrum of the complex $[Fe(L_1)\ (Cl_2)\ (H_2O)_2]^+$**.** shows a molecular ion peak $[M]^+$. At (465)m/zwhich is equivalent to molecular mass of the complexand shows another fragments at (394,412,376,358)m/z due to $[Fe(L_1)(H_2O)_2]^+$. $[Fe(L_1)(H_2O)(Cl)]^+$. $[Fe(L_1)H_2O]^+$, $[Fe(L_1)]^+$ respectivelyshown in figure(12). The mass spectrum of the complex $[Cu(L_1)(Cl_2)(H_2O)_2]^+$. shows a molecular ion peak $[M]^+$. at(472) m/zwhich is equivalent to molecular mass of the complex and shows another fragments at (437, 419,383,365)m/z due to $[Cu(L_1)(H_2O)_2(Cl)]^+$, $[Cu(L_1)(H_2O)(Cl)]^+$, $[Cu(L_1)H_2O]^+$, $[Cu(L_1)]^+$ respectivelyshown in figure(11). The mass spectrum of the complex $[Co(L_1)(Cl_2)(H_2O)_2]^+$. shows a molecular ion peak $[M]^+$ at(468) m/zwhich is equivalent to molecular mass of the complex and shows another fragments at (433,415,379,361) m/z due to $[Co(L_1)(H_2O)_2Cl]^+$, $[Co(L_1)(H_2O)(Cl)]^+$, $[Co(L_1)H_2O]^+$,$Co(L_1)]^+$respectively shown in figure(9). The mass spectrum of the complex $[Cr(L_1)(Cl_2)(H_2O)_2]^+Cl$ shows a molecular ion peak $[M]^+$ at (497) m/zwhich is equivalent to molecular mass of the complex and shows another fragments at (389,444,408,390)m/z due to$[Cr(L_1)(H_2O)_2]^+$, $[Cr(L_1)(H_2O)(Cl)]^+$, $[Cr(L_1)H_2O]^+$, $[Cr(L_1)]^+$ respectively shown in figure(10). The mass spectra of the complexes shown in figures (9-13).

Magnetic susceptibility:-

The results of prepared shown in table (4) show that the magnetic torque values of the prepared complexes eared low because there are no single electrons,ie,the complexes prepared are of allow type .This confirms that the ligand is a strong ligand.

Atomic absorptions:-

The transition elements of the prepared complexes were estimated using the flame atomic absorption technique and the practical results were close to the theoretical values as shown in the table(5).

Molar Electrical Conductivity:-

Molar conductivity of the solid solutions of complexes was measured for (Co(II), Cu(II), Ni(II), Fe(II) and Cr(III)) with ligand (LH). Results are shown in table(6), indicating that the complex solutions of salts (Co(II), Cu(II), Ni(II), Fe(II)) with ligand behave as neutral compounds (non-Electrolyte) while the (Cr(III) solution with ligand behave as ionic compounds (Electrolyte).

Antibacterial activity:-

This research Studies the effectiveness of ligand (LH) and its complexities prepared against the two type of pathogenic bacteria negative and positive for the gram dye a dye treated by bacteria that respond to this dye takes dye and not posed outside the cell wall is positive and that do not respond to this dye so take dye and posed outside the cell wall is negative towards this character,and this difference between bacteria due to the nature of the external walls,For this reason used two types of bacteria the first is sensitive topics and positive for the gram stain Staphylococcus aureus and the second sensitive and negative to gram stain a Escherichia coli. it was prepared by a certain concentration of these compounds from the dissolve 0.05g of ligand or complex in 1ml of DMSO (as solvent) and injected bacterial dishes after making holes where by corkborer 0.1ml of these solution in each hole and incubated for 18 hours at a temperature 37^0C then extracted compared the inhibition of these compounds with standard inhibition is Cipro and the same concentration (Kurdir, 2013). From results shown in the table(7) and figures(14)-(17) can be concluded the following notices:

1-the ligand(LH) showed lower efficiency than the standard inhibition trendstaph. Bacteria.

2-the ligand(LH) showed higher efficiency than the standard inhibition trend bacteria E.coli.

3-The prepared complexes from ligand appeared low effective than the effectiveness of the ligand itself.

Table(1) The practical and theoretical ratio of ligand (LH)

Name	Molecular Formula	Molecular weight	C%		H%		N%	
			Theo.	Prac.	Theo.	Prac.	Theo.	Prac.
L_H	$C_{20}H_{18}N_2O$	302	79.47	79.17	5.96	5.91	9.27	9.17

Table(2) some physical properties of ligand and its complexities

No	Chmical Formula	M.wt	Color	m.p. °c	Yiehd%
L_1	$C_{20}H_{18}N_2O$	302	Light yellow	188-190	91
1	$[Fe(L_1)(Cl_2)(H_2O)_2]$	465	Bright yellow	220-222	89
2	$[Ni(L_1)(Cl_2)(H_2O)_2]$	468	Bright yellow	216-218	86
3	$[Cr(L_1)(Cl_2)(H_2O)_2]CL$	496	Yellow	223-225	71
4	$[Cu(L_1)(Cl_2)(H_2O)_2]$	472	Dark brown	214-215	85
5	$[Co(L_1)(Cl_2)(H_2O)_2]$	468	Yellow	218-219	84

Table(3) FT-IR peaks value of the ligand and its complexes

رمز الليكاند	O-H	C-H Ar.	C-H Aliph.	C-O	C=N	C=C	M-O	M-N
L_1	3418	3028	2920	1153	1640	1566	-----	-----
L_1-Co	3405	3017	2924	1188	1620	1566	571	440
L_1-Fe	3412	3001	2924	1181	1620	1573	578	509
L_1-Cu	3451	3011	2899	1223	1612	1600	697	404
L_1-Cr	3450	3032	2916	1280	1620	1566	517	436
L_1-Ni	3350	3020	2950	1195	1622	1569	580	439

Table(4) Effective magnetic torque of the complexes

Number	Complexes	μ_{eff} (B.M)	Coordination
1	$[Cr(L_1)(Cl_2)(H_2O)_2]Cl$	3.8	Octahedral
2	$[Fe(L_1)(Cl_2)(H_2O)_2]$	0.32	Octahedral
3	$[Ni(L_1)(Cl_2)(H_2O)_2]$	2	Octahedral
4	$[Cu(L_1)(Cl_2)(H_2O)_2]$	1.9	Octahedral
5	$[Co(L_1)(Cl_2)(H_2O)_2]$	2.2	Octahedral

Table(5) Determination of the percentage of elements in the complex using the atomic atomic absorption technique

Compound	Theoretical Data%	Practical Data%
$[Cr(L_1)(Cl_2)(H_2O)_2]Cl$	10.4	9.7
$[Fe(L_1)(Cl_2)(H_2O)_2]$	12	11.5
$[Ni(L_1)(Cl_2)(H_2O)_2]$	12	11.8
$[Cu(L_1)(Cl_2)(H_2O)_2]$	13.3	12.77
$[Co(L_1)(Cl_2)(H_2O)_2]$	12.6	11.8

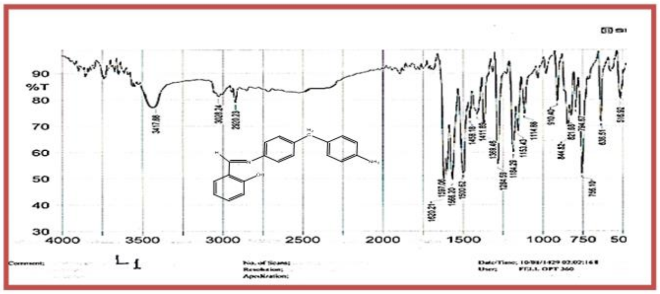
Table(6):The values Molar Conductivity Λm of ligand complexes in solvent DMSO concentration of 10^{-3}M at a temperature of 298K

Number	Complexes	Λm (S .cm2 .mole-1)	Electrolyte Type
1	$[Cr(L_1)(Cl_2)(H_2O)_2]Cl$	36	1:1
2	$[Fe(L_1)(Cl_2)(H_2O)_2]$	10	non Electrolyte
3	$[Ni(L_1)(Cl_2)(H_2O)_2]$	13	non Electrolyte
4	$[Cu(L_1)(Cl_2)(H_2O)_2]$	19	non Electrolyte
5	$[Co(L_1)(Cl_2)(H_2O)_2]$	12	non Electrolyte

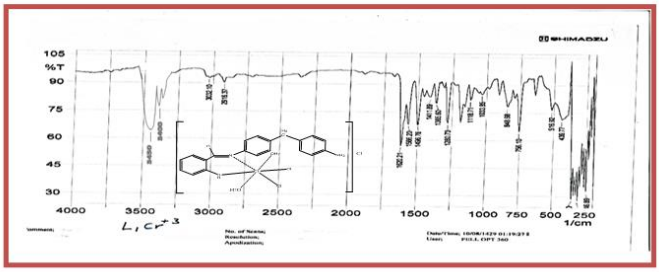
Table(7)Effectiveness biological of studied compounds

NO.	Compound	Escheria Coli Inhibtion Zone(mm)	Staphylococcs aureus Inhibtion Zone(mm)
1	L_H	25+++	22+++
2	L_HNi	14++	12++
3	L_HCo	15++	13++
4	L_HFe	12+	10+
5	L_HCu	13++	12+
6	L_HCr	10+	8+
18	Cpro.	25++++	30+++
19	DMSO	----------	----------

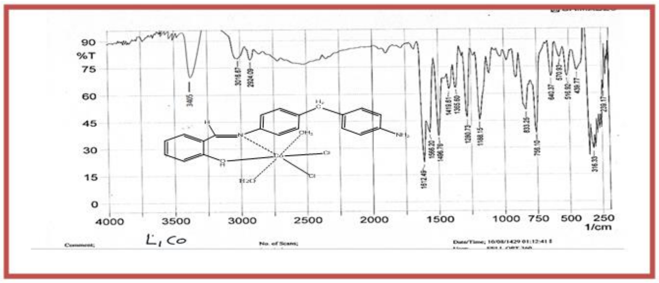
Note:++++ Very good inhibition, +++Good inhibition, ++Middle inhibition, +Weak inhibition, - No inhibition



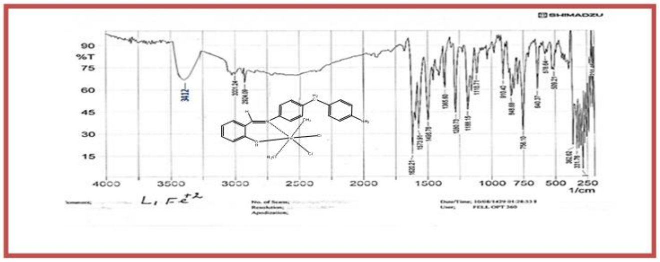
Figure(1) IR spectra of the ligand (LH)



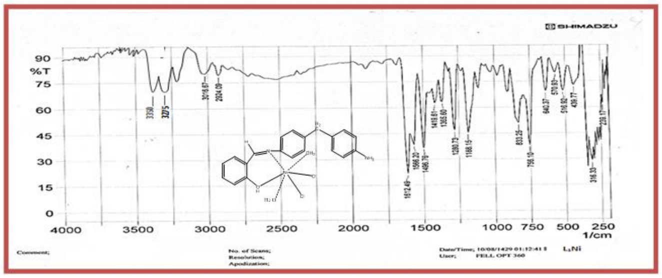
Figure(2) IR spectra of complex(LH-Cr)



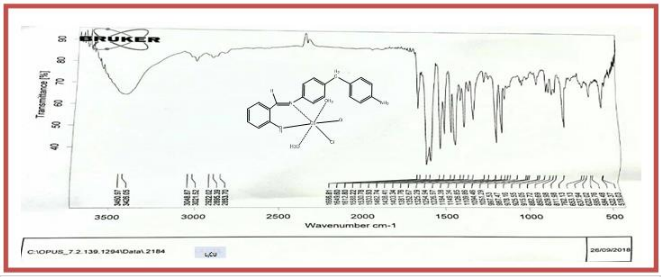
Figure(3) IR spectra of complex(LH-Co)



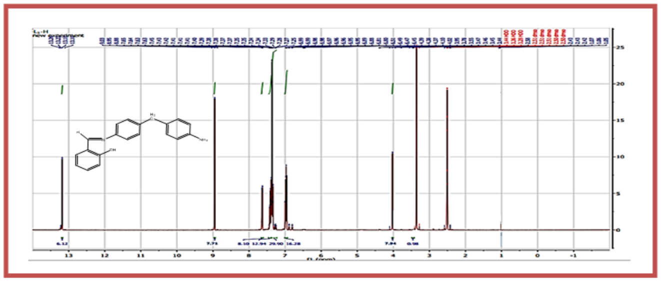
Figure(4) IR spectra of complex(LH-Fe)



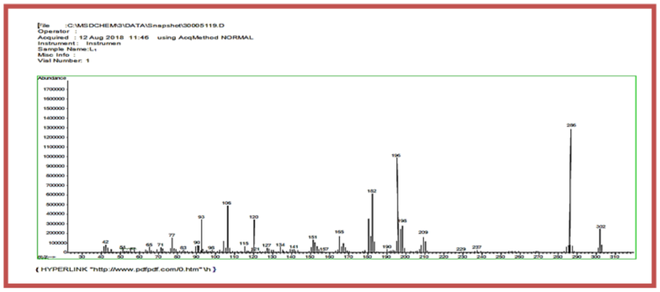
Figure(5) IR spectra of complex(LH-Ni)



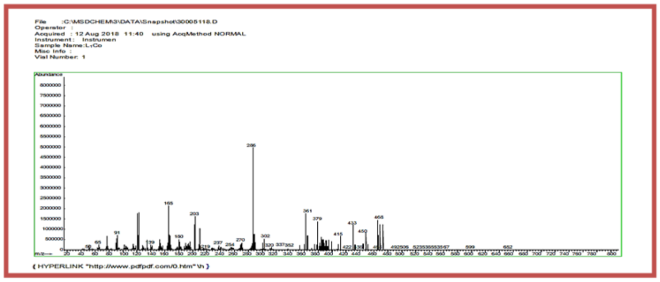
Figure(6) IR spectra of complex(LH-Cu)



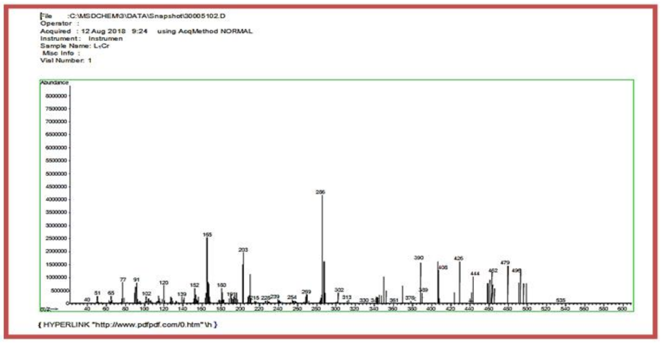
Figure(7) 1HNMR spectra of ligand(LH)



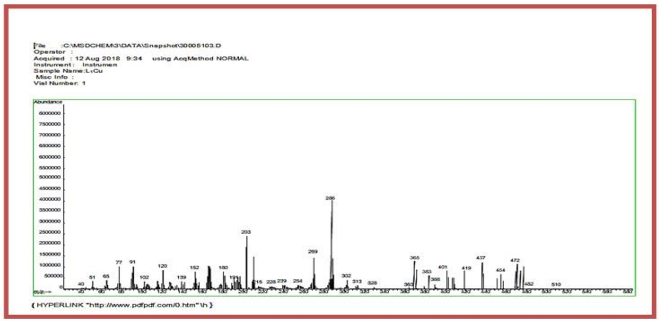
Figure(8) Mass spectra of the ligand (LH)



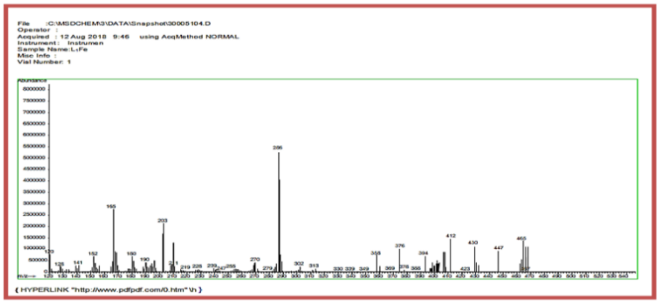
Figure(9) Mass spectra of the complex $[Co(L_1)(Cl_2)(H_2O)_2]$



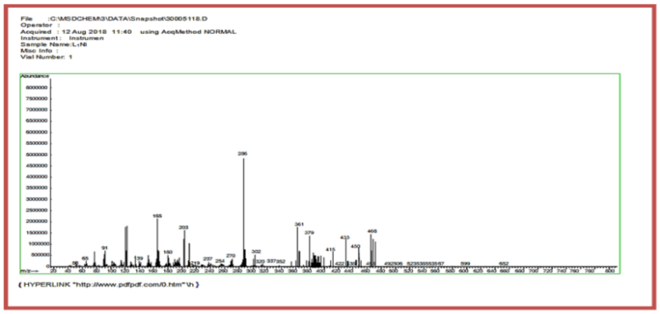
Figure(10) Mass spectra of the complex $[Cr(L_1)(Cl_2)(H_2O)_2]Cl$



Figure(11) Mass spectra of the complex $[Cu(L_1)(Cl_2)(H_2O)_2]$



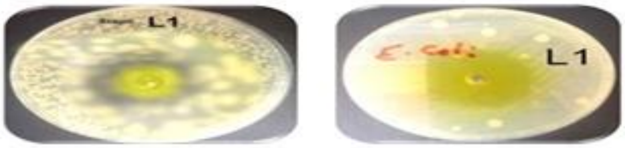
Figure(12) Mass spectra of the complex $[Fe(L_1)(Cl_2)(H_2O)_2]$



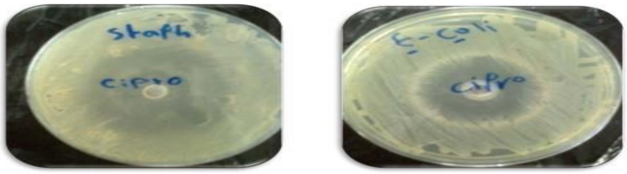
Figure(13) Mass spectra of the complex $[Ni(L_1)(Cl_2)(H_2O)_2]$

*1=L_1Ni, 2= L_1Co, 3= L_1Fe, 4= L_1Cu, 5= L_1Cr

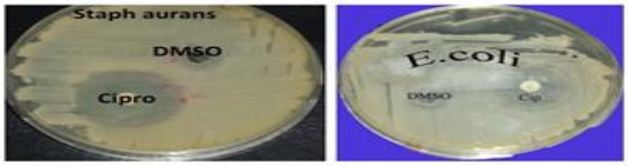
Figer (14) Biological activity of ligandcoplexes (LH)



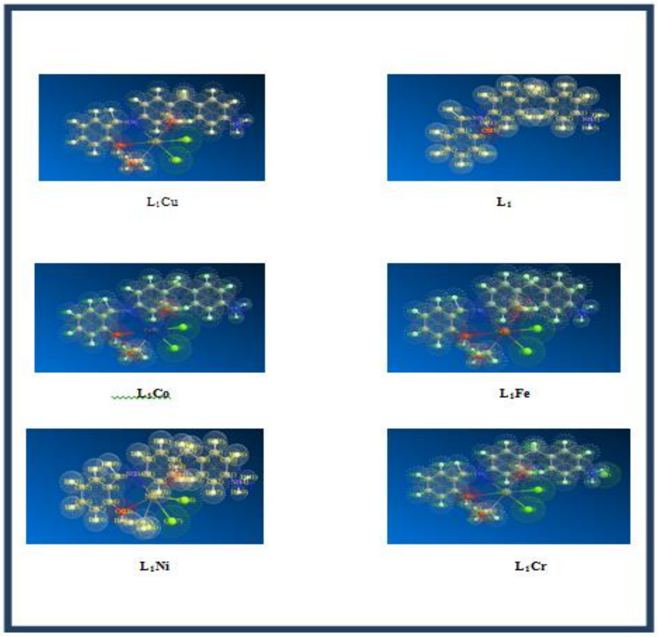
Figer (15) Biological activity of a ligand (LH)



Figer (16) Biological activity of cipro(LH)



Figer (17) Biological activity of DMSO



The three- dimensional shape of the ligand and its complexes

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