Microwave Preparation, Spectral Studies and Antimicrobial Activities Evaluation of Mn$^{(II)}$,Ni$^{(II)}$, Hg$^{(II)}$,Co$^{(II)}$ and Cu$^{(II)}$ Complexes with Schiff Base Ligand

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Abstract

New Schiff base and their Mn$^{(II)}$,Co$^{(II)}$,Ni$^{(II)}$, Cu$^{(II)}$ and Hg$^{(II)}$ complexes formed by the condensation of O-phathaldehyde and ethylene diamine (2:1) to give ligand (L^{1}) in the first step ,then the ligand (L^{1}) with 2- aminophenol (1:2) to give ligand (L^{2}) were prepared by classic addition through microwave method . These compounds (Ligands and complexes) have been diagnosed electronic spectra, FT-IR, ^{1}H-&^{13}C-NMR (only ligand), magnetic susceptibility, elemental microanalysis and molar conductance measurements. Analytical values displayed that all the complexes appeared (metal: ligand) (1:1) ratio with the six chelation. All the compounds appear a high activity versus four types of bacteria such as; (*Escherichia coli*), (*Staphylococcus aureus*),(*Bacillus btilis*), (*Staphylococcus aureus*) and *(Pseudomonas aeruginosin)***.**

Keywords: Microwave Preparation, Complexes for Schiff base, O- Paraldehyde, and Antimicrobial activities.

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Introduction

In every day of life, the improved pharmaceuticals in coordination chemistry have a large interdisciplinary relation for transition metal ion that are used to control the harmful effects of fungi and bacteria [1-8]. The studies about behavior ligands in the chelation with transition metal ions by using the microwave as assisted were considered as one of the important properties in green chemistry [9]. The application of microwave- supported preparation continues to develop in organometallic [10], coordination [11] and organic chemistry pace [12]. The reactions in Microwave-irradiated show using shorter reaction times, less amount of solvent or solvent-free conditions present low cost, good yield, minimum pollution, simplicity in handling and processing[13].By microwave approaches, there is little literature on the preparation of metal complexes [14,15]

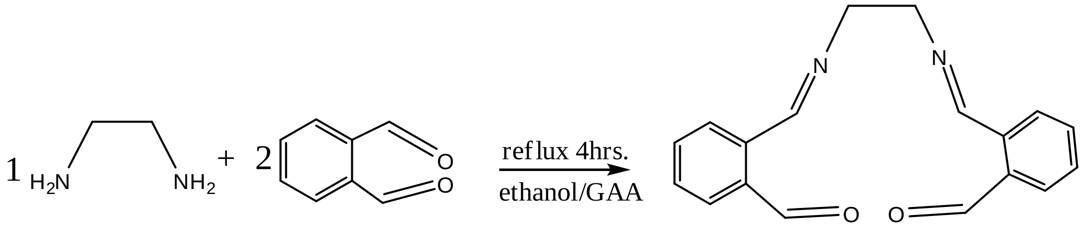
Materials: All the used chemicals O-phathaldehyde, ethylene diamine, 2-amino phenol, glacial acetic acid, HBr ,$CaCl_2$ and reagents and solvents such as ethanol, methanol in addition to various metal chlorides were obtained from Sigma-Aldrich.

Instrumentation

Electronic spectra were recorded using UV-Vis. spectrophotometer type CECIL, England, by using quartz cell has path length (1cm) in range (200-1000)nm in DMSO at room temperature. Melting point was measured by "Gallenkamp Melting point Apparatus". Elemental microanalysis C.H.N. was carried out using Euro Vector EA 3000 A Elemental Analysis (Italy). FT-IR measurements were calculated on Shimadzu- 8300, Spectrophotometer in the range of (4000-400cm^{-1}) as KBr disc. In DMSO by using a Bruker 300 MHZ (Switzerland) were obtained on (^1H and^{13}C-) NMR spectra, Chemical shift was obtained in δ(ppm) unit downfield internal reference (TMS), Conductivity measurements were obtained from WTW conductivity meter by using ethanol as a solvent of $10^{-3}M$ concentration at room temperature. Magnetic susceptibility measurements were obtained at room temperature on the solid state applying Faraday's Method using Bruker BM6 instrument. Metal analyses of complexes were determined by using a Shimadzu PR-5.GRAPHIC PRINTER atomic absorption spectrophotometer.

Conventional technique for the preparation of the ligand [5]

(L^1) was synthesized by the condensation of (1:2) molar ratio of ethylenediamine (0.060g, 0.001mmole) and O-paraldehyde (0.268g, 0.002 moles) with 3droup glacial acetic acid dissolved in(15mL) ethanol. The product mixture was refluxed for 4 h on a water bath and then left to cool overnight. The colored solid result of the ligand was filtered, washed with hot ethanol many times and dried at room temperature in air and finally desiccated under low pressure in desiccators. m.p. 171°C. Yield: 67 %.



Synthesis of [L^2]: (L^2) was synthesized by the condensation of (1:2) molar ratio of (L^1) (0.0292 g,0.001 mmole) with 2-aminophenol (0.218 g,0.001 mmole) dissolved in (15mL) ethanol. The product reaction mixture was refluxed for 3 h on a water bath and then left to cool overnight. The colored result of the ligand was filtered, washed with hot ethanol several

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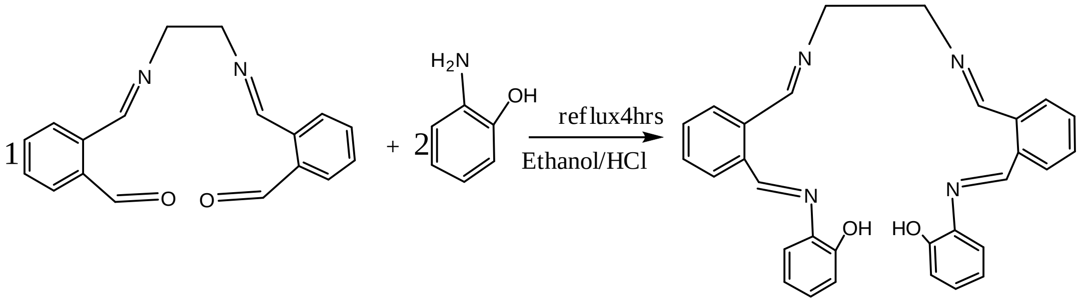
times and dried in air at room temperature and finally stored under low pressure in desiccators. m.p. 183 °C; Yield: 70 % scheme(**2**)



Microwave technique for the synthesis of the Schiff bases

Synthesis of [L^1] :(2:1) molar ratio of O-paraldehyde (0.268g) with ethylenediamine (0.06 g) was mixed in a grinder completely. The mixture in the microwave oven was then irradiated by taking 3-4 mL of absolute ethanol as a solvent. The reaction was finished in a distilled time (4–5 min) with more yields than were got in the conventional procedure. Then the results were recrystallized from ethanol and finally dried in desiccators over anhydrous $CaCl_2$under low pressure. (yield: 80 %).

Synthesis of [L^2]: The [L^2] has prepared according to the method synthesis of [L^1] scheme(**2**) by(2:1)(2-aminophenol :L^1:) molar ratio (0.380g) was mixed in a grinder completely. The mixture in the microwave oven was then irradiated by taking (3–4 mL) of absolute ethanol as a solvent. The reaction was finished in a shortened time (4–5 min) with more yields than were got in the conventional procedure. Then the results were recrystallized from ethanol and lastly dried in desiccators over anhydrous $CaCl_2$under low pressure.

Conventional technique for the synthesis of metal complexes

All the complexes were synthesized by blending 10 mL of a methanolic solution of (10 mM) salt {(1.98 g $MnCl_2.4H_2O$, 2.38 g $CoCl_2.6H_2O$, 2.37 g $NiCl_2.6H_2O$, 1.72g $CuCl_2.2H_2O$, and 2.71 g $HgCl_2$)} with 10 mL a methanolic solution of (10 mM) the Schiff base (L^2) in{ (metal: ligand) (1:1) }ratio, then refluxed mixture for 1 h on a water bath. The precipitated complexes were filtered, washed several times with ethanol and dried under low pressure in a desiccator over $CaCl_2$. Also by using an electric oven they were further dried the results were at 55–70 °C.

Microwave technique for the synthesis of metal complexes

The metal salts and the ligand and were mixed in a grinder in a {(metal: ligand) (1:1)} ratio. In the irradiated oven, the mixture was irradiated by taking 3–4 mL ethanol solvent. The mixing was finished in a shortened time (2-3 min) with more yields than were get in the classic method. Then the results were recrystallized from 2ml of {ethanol, methanol, and distilled water} and finally dried in desiccators over anhydrous $CaCl_2$under low pressure.

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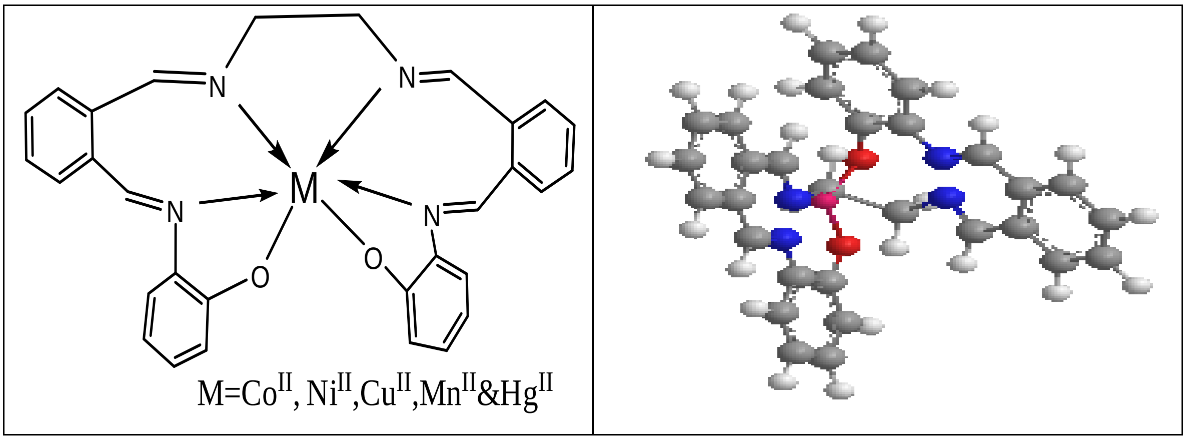

Antimicrobial activity

The antimicrobial activity(*in vitro*)of the realized ligand and their metal chelates were examined against the four types of bacteria as (*Escherichia coli* and *Staphylococcus aureus, Pseudomonas aeruginosin)* by the disc diffusion method using nutrient agar as the medium using a micropipette in ethanol and the plate was incubated at 37 °C 24 h. After incubation, we measured the samples that considered as the inhibitory power of the specified test bacterial [4].

Results and Discussion

As an outcome of the irradiated -supported preparation, the time of reaction showed that it was diminished from more hours to little minutes also got good yields of the results were received looked at those fulfilled by the customary preparation method. In the microwave method, rotation of mixture platform tray makes reaction mixture more homogeneity. The repetition of the preparation process to make sure the results [3] . All the metal complexes are characterized by the following data. Based on the elemental analysis the composition of metal complexes and percentage of various elements help to formulate the complexes. They were solid, colored and disintegrated on warming at rising of the temperatures and were less or more solvent in basic organic solvents. The data of a comparison of the two preparation techniques showed that reactions which were completed 5.2–6.8 min by the microwave methods but were demanded 4.6–7.20 h by the conventional method and the yields were got better 61.8–75.8 % to 76.2–85.4 %. .

Molar conductivity

All the complexes had stoichiometry for (ligand: metal) was (1:1). The watched molar conductance of metal chelates at room temperature in ethanol, were shown in Table 5. They were consistent with the non-electrolytic nature of all the complexes [2,3].

NMR spectra

^{1}HNMR spectrum: All signals in the integral intensities of the ^{1}HNMR spectrum of ligand Figure (3) were found to agree with the number of the following signals: DMSO at δ_H 2.479, $C\underline{H}_2$ at δ_H 5.38; C_6H_5 as multiple at δ_H7.31~8.28; $\underline{H}$CO carbonyl group at δ_H10.38 .The peaks observed at δ_H 8.78 and δ_H 8.85 are attributable to the $\underline{H}$C=N amine group present in the ligand [5].

^{13}C NMR spectrum: in DMSO-d_6 solution shows the following signals Figure (4): DMSO at 40.8;$\underline{C}H_2$ at 56.70, 61.84; =$\underline{C}$-N at 101.41, $\underline{C}_6H_5$ as multiple at 128.50~141.13. The peaks observed at 160.05 and 192.87are attributable to the $\underline{C}$=N imine group, and $\underline{C}$=O carbonyl group $\underline{C}$=N imine group, respectively [6].The characteristics frequencies of the free and mixed ligand complexes of the metals are given in Table (4).

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The IR data support these assignments

IR spectrum of the ligand displayed the generality characteristic frequencies due to(C=N) azomethine and (C–O) groups and showed in the formed ligand, NH_2, and C=O bonds were disappeared. The free ligand spectrum, Figure (1) displayed band at (3441) cm^{-1} is attributed to the deformation and stretching of the phenolic OH. These bands were disappeared in the spectra of the complexes [7]. The two bands at (1678, 1620) cm^{-1} attributed to the two azomethine groups of the ligand, after complexation, these bands were shifted to lower frequencies in the region (1668-1661) cm^{-1} and (1617-1614) cm^{-1}, elucidating the bonding of nitrogen atoms of (C=N) imine groups to the metal ions. This can be illustrated by the granting of electrons from nitrogen to the vacuous d-orbital of the metal atom[8]. The phenolic C–O stretching vibration that showed at (1211) cm^{-1} in the ligand, this band was shifted towards higher frequencies about (22–16) cm^{-1}in the complexes, Figure (2). This suggests deprotonation of the phenolic OH group after its coordination with the metal ion[9]. This shift confirms the participation of oxygen in the C–O–M bond. In the low-frequency region, the band of weak intensity observed for the complexes in the region (567-555) cm^{-1} is attributed to M–O and in the region (474-438) cm^{-1} is attributed to M–N. The IR data of both the Schiff base and their metal complexes showed that the Schiff base was the type of hexadentate ligand through chelated to the metal ion [10].

Electronic spectra and magnetic moment

The U.V- Vis spectra values of the metal complexes in ethanol solution are observed in Table 6. The (U.V- Vis) spectrum for the ligand, exhibits a high intense absorption band at {(275) nm}{ (36363) cm^{-1}} and a small peak at (385) nm (25974) cm^{-1}, which assigned to ($\pi \rightarrow \pi^*$), and ($n \rightarrow \pi^*$) transitions, respectively [11].

The electronic spectrum [Co(L^2)], displays five peaks, the peaks at υ_1={(910) nm}{(10989)cm^{-1}},υ_2={(657)nm}{(15220)cm^{-1}}and{υ_3=(486)nm} (20576)cm^{-1}}due to{$^4T_1g_{(F)} \rightarrow {^4T_2g_{(F))}}$},{$^4T_1g_{(F)} \rightarrow {^4A_2g_{(F)}}$}and{$^4T_1g_{(F)} \rightarrow {^4T_1g_{(P)}}$} transitions, and the high absorptions at {(292) nm}{(34246) cm^{-1}}and {(397)nm}{ (25188)cm^{-1}}are assigned to the (L.F) and (C.T) transitions, which suggest the octahedral geometry for Co(II) complex [12]. The room temperature magnetic moment (μ_{eff} = 4.58B.M) corresponded to a high spin octahedral symmetry.

The (U.V- Vis) spectrum [Ni(L^2)], exhibits four bands, the peaks at {υ_1= (668) nm} {(14970)cm^{-1}}due to{$^3A_2g_{(F)} \rightarrow {^3T_1g_{(F)}}$},{$\upsilon_2$=(543)nm}{(18416)$cm^{-1}$},{$^3A_2g_{(F)} \rightarrow {^3T_1g_{(P)}}$ } transitions and the high peaks at {(280) nm}{(35714)cm^{-1}}and {(392)nm}{ (25510) cm^{-1}} for the (L.F) and (C.T) transitions[13] . Ni $^{(II)}$ complex showed an amount of μ_{eff} = 2.98 B.M, which suggests an octahedral geometry around the central metal ion.

The (U.V- Vis) spectrum [Cu(L^2)] displays three peaks, the band at{ υ_1= (530)nm}{(18867)cm^{-1}} due to{$^4Eg \rightarrow {^4T_2g}$} transitions and the high peaks at {(280)nm}{ (35714) cm^{-1}} and {(392)nm}{(25510) cm^{-1} and {(269) nm}{(37174)cm^{-1}} due to (L.F) and (C.T) transitions, on an octahedral geometry [14]. Cu (II) complex exhibited a value of μ_{eff} =1.83 μB.

The (U.V- Vis) spectrum of [Mn(L^2)] showed three peaks, the peak at{ υ_1= (553)nm}due to {(18083)cm^{-1}}{$^6A_1g(s) \rightarrow {^4T_1g_{(G)}}$},and the high peaks at {(279)nm} {(35842)cm^{-1}}and {(394)nm}{(25380)cm^{-1}}are assigned to the (L.F) and (C.T) transitions in an octahedral geometry[15] . Mn(II) complex exhibited a value of μ_{eff} =5.51μB.

The (U.V- Vis) spectrum [Hg(L^2)], exhibits two high peaks at{(310) nm}{(32258) cm^{-1}} and {(412)nm}{ (24271)cm^{-1}} are due (L.F) and (C.T) transitions .The Hg (II), complex did not

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show any peak in the vis. The region, no ligand field absorptions band were appeared, therefore the absorptions observed in the spectrum of the complex could be assigned to transition from type charge transfer. In fact this produced a good convention with past study of octahedral geometry[16].Hg(II)complex measured magnetic susceptibility and showed diamagnetic as a perspective from their electronic arrangement (d^{10}) .

Antimicrobial activities

The antimicrobial activities in vitro of the prepared ligand and their corresponding metal complexes against bacteria such as *Escherichia coli* and *Staphylococcus aureus* , *Pseudomonas aeruginosin* were established in Table(5). All compounds are checked and displayed better antimicrobial activity versus the microorganism [17]. On comparing it was shown that the activity of the complexes had moderate as compared to the standard but all the complexes were more active than their single ligand. The greater inhibition zone of the complexes than the free ligand can be expounded based on the chelation theory and the Overtone concept. The interfere of the partial sharing of the positive charge of the metal ion and the ligand orbital with donor groups are due to reduce the polarity of the metal ion in upon chelation. Furthermore, this enhances the blocking of the metal binding sites and the penetration of the complexes into lipid membranes in the enzymes of microorganisms that the delocalization of the π-electrons increases up to the full coordination ring[18].

Conclusion

One of a green chemical path is the microwave technique. The prepared compounds were diagnosed by spectral analyses and different chemical-physic. In route of the microwave helped syntheses, it was spotted that the time of reaction was decreased from many hours to few minutes and the results were obtained in good yield as a comparison to those of the conventional method. The metal ions bound with the diagnosed hexadentate ligand type N_4O_2 in six donor sites are present four N- azomethine and two O- phenylic.

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Table (1): The physical and analytical data of ligand(L^2) and metal complexes[$M(L^2)$]

compounds	Formula	Molecular Weight	Colour	Yeild% *Conventional*	M.P.	%Elemental Analysis Found % (Calculated)			
				Microwave		C	H	N	M
L^2	$C_{30}H_{26}N_4O_2$	474.55	Light brown	70.5 82	201-203	75.93 (75.23)	5.52 (5.31)	11.81 (10.85)	-
[$Co(L^2)$]	$C_{30}H_{24}CoN_4O_2$	531.47	Brown	74 80.3	229-231	67.80 (59.50)	4.55 (4.84)	10.54 (8.67)	11.09 (9.12)
[$Ni(L^2)$]	$C_{30}H_{24}N_4NiO_2$	531.23	Brown	65.8 76.2	220-222	67.83 (59.52)	4.55 (4.84)	10.55 (8.68)	11.05 (9.09)
[$Cu(L^2)$]	$C_{30}H_{24}CuN_4O_2$	536.08	Deep brown	61.8 78	235-237	67.21 (59.07)	4.58 (4.80)	8.34 (8.61)	9.45 (9.77)
[$Mn(L^2)$]	$C_{30}H_{24}MnN_4O_2$	642.0	Brown	72 85.4	226-228	59.08 (59.87)	4.26 (4.87)	8.33 (8.73)	7.89 (8.56)
[$Hg(L^2)$]	$C_{30}H_{24}HgN_4O_2$	788.17	Off-White	75.8 79.7	222- 224	48.00 (48.80)	3.64 (3.97)	8.89 (8.32)	25.45 (25.47)

Table (2)^{1}H-NMR chemical shifts for ligand (L^2) (ppm in DMSO)

DMSO	CH_2	C_6H_5	HC=N	HCO
2.47	5.38	7.31-8.28	8.78 8.85	10.38

Table (3):^{13}C-NMR chemical shifts for ligand (L) (ppm in DMSO)

DMSO	CH_2	C_6H_5	C=N	C=O
40.82	56.70 61.84	128.50-141.13	160.05	192.87

Table (4): The important IR bands of the Ligand(L^2) and it's metal complexes[$M(L^2)$]

Compound	υ(OH)	υ(CH)$_{aroma.}$	υ(CH)$_{alpha}$	υ(C=N)	υ(C=C)	υ (C-O)	υ (M–N)	υ(M–O)
L^2	3441	3032	2916	1678 1620	1598	1211	-	-
[$Co(L^2)$]	-	3012	2920	1670 1617	1590	1227	567	468
[$Ni(L^2)$]	-	3006	2931	1666 1616	1595	1233	558	474
[$Cu(L^2)$]	-	3024	2947	1662 1614	1592	1229	561	438
[$Mn(L^2)$]	-	3003	2918	1664 1615	1597	1228	565	459
[$Hg(L^2)$]	-	3005	2927	1663 1613	1589	1230	555	461

Table (5): Zone of inhibition of growth in 100 millimeters after 24 hours of incubation

Comp.	*Escherichia. Coli*	*Staphylococcus aureus*	*Bacillus*	*pseudomonas*
L^2	2	-	-	1
[$Co(L^2)$]	6	10	12	11
[$Ni(L^2)$]	8	11	10	9
[$Mn(L^2)$]	10	9	11	7
[$Cu(L^2)$]	7	7	8	6
[$Hg(L^2)$]	9	6	7	8

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Table (6):Electronic spin resonance parameters of the complexes

Compound	μ_{eff}	Λ_{m} S.Cm^{2} molar^{-1}	λnm	υ–wave number cm^{-1}	Assignments
L^2	-	-	275	36363	$\pi \rightarrow \pi^*$
			385	25974	$n \rightarrow \pi^*$
$[Co(L^2)]$	3.98	17.8	292	34246	L.F
			397	25188	C.T
			486	20576	$^4T_1g_{(F)} \rightarrow {}^4T_1g_{(P)}$
			657	15220	$^4T_1g_{(F)} \rightarrow {}^4A_2g_{(F)}$
			910	10989	$^4T_1g_{(F)} \rightarrow {}^4T_2g_{(F)}$
$[Ni(L^2)]$	3.57	14.5	280	35714	L.F
			392	25510	C.T
			543	18416	$^3A_2g_{(F)} \rightarrow {}^3T_1g_{(P)}$
			668	14970	$^3A_2g_{(F)} \rightarrow {}^3T_1g_{(F)}$
$[Cu(L^2)]$	1.83	12.6	286	34965	L.F
			390	25641	C.T
			530	18867	$^4Eg \rightarrow {}^4T_2g$
$[Mn(L^2)]$	5.51	12.7	279	35842	L.F
			394	25380	C.T
			553	18083	$^6A_1g_{(s)} \rightarrow {}^4T_2g_{(G)}$
$[Hg(L^2)]$	Dia	16.9	310	32258	L.F
			412	24271	C.T

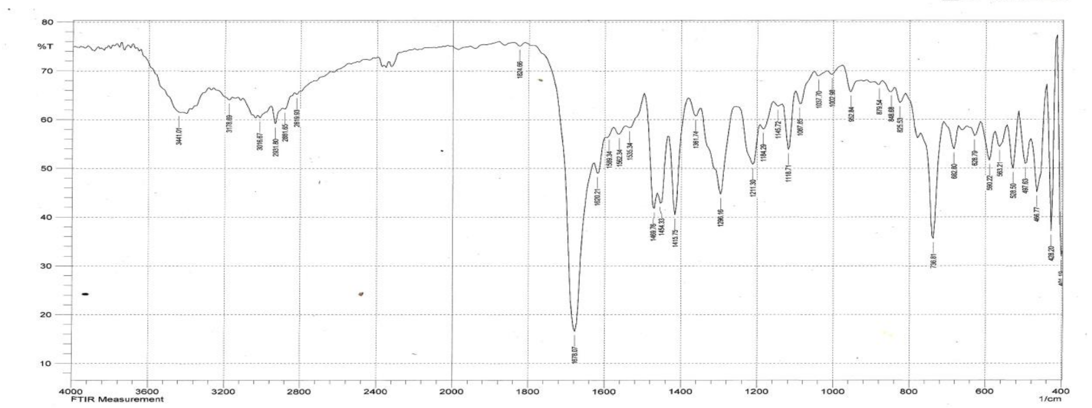

Figure (1): The IR spectrum of (L^2)

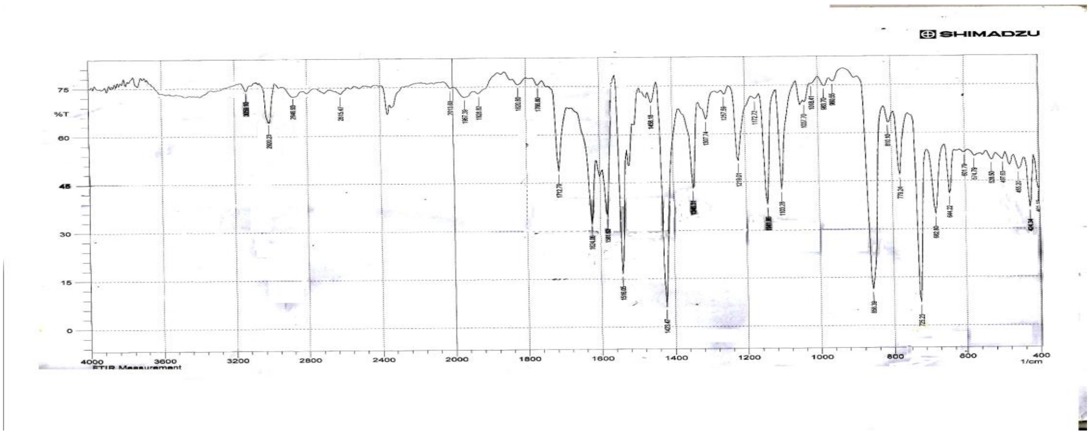

Figure (2): The IR spectrum of the $[Co(L^2)]$ complex

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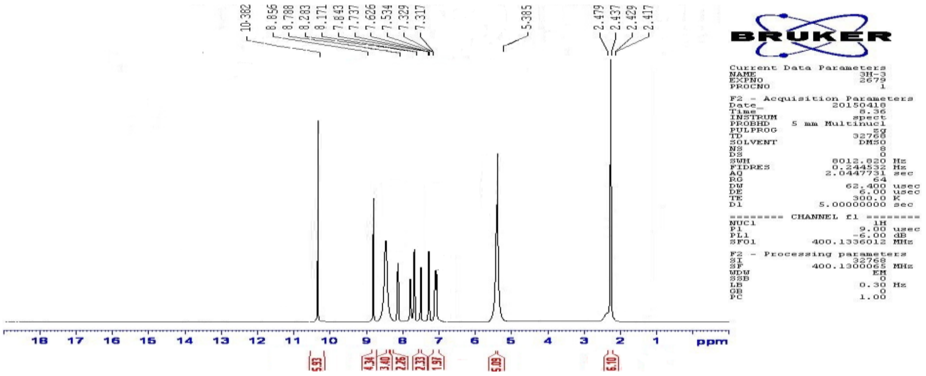

Figure (3): The^{1}H-NMR spectrum of (L^2)

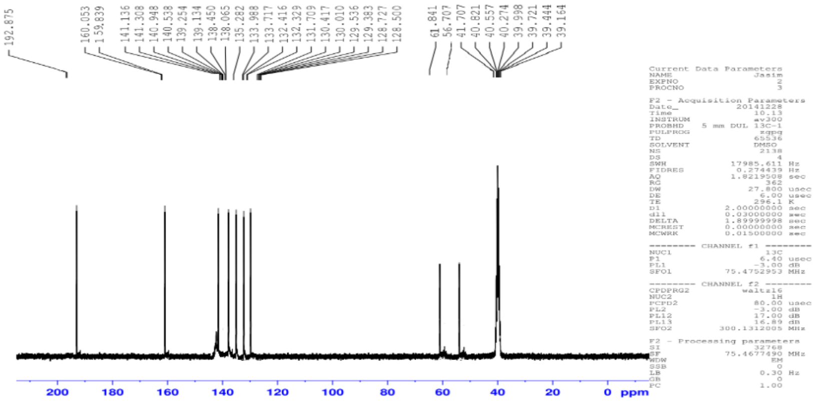

Figure (4): The^{13}C-NMR spectrum of (L^2)

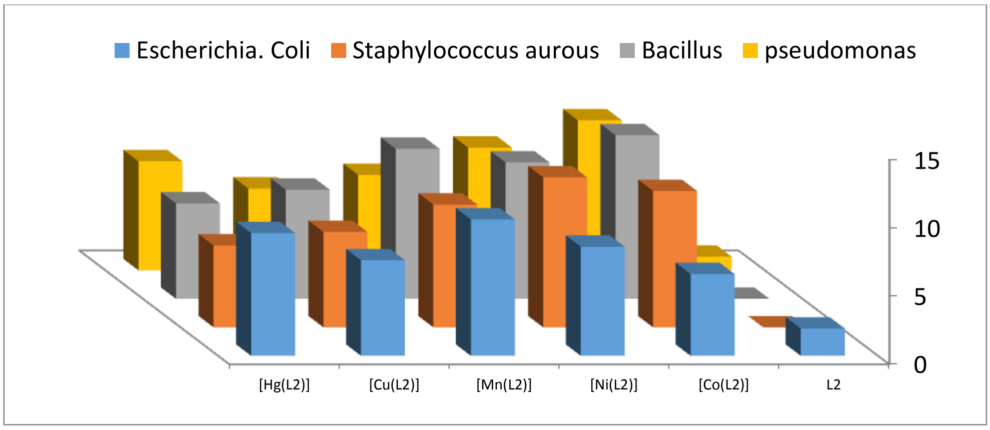

Figure (5): Variance between the antimicrobial activity of (L^2) and their complexes