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Synthesis, Characterization and Spectral Studies and Biological Screening Study of Transition Metal Complexes with New Heterocyclic Ligand Derived from Pyridoxal Hydrochloride

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ABSTRACT

The complexes of Co (II), Ni (II), Cu (II), Zn (II), Cd (II), Pt (II), and Au (III) were prepared from the complexation reaction between the new Schiff base ligand and metal ions. This ligand**(3,4-bis(((E)-1-(2-(((E)-(3-hydroxy-5-(hydroxymethyl)-2-methylpyridin-4-yl) methylene) amino) phenyl) ethylidene) amino) phenyl) phenyl) methanone** was derived from the condensation reaction of 3,4-diamino benzophenone, 2-amino acetophenone, and pyridoxal hydrochloride. The ligand and its complexes were characterized by C.H.N elemental analysis, Uv-Vis, FT-IR, ^{1}H-NMR and molar conductivity, Atomic absorption, magnetic moment measurements and mass spectra studies. The results of this studies show the coordination sites for the ligand with the metal ion were to be through oxygen of the hydroxyl group, and the nitrogen of azomethine groups. The electronic spectral and magnetic measurement data predict octahedral structure of the complexes, except for Pt (II) and Au (III) complexes were suggested a square planar geometry. All complexes show that nonelectrolytes properties, but Pt (II) and Au (III) complexes are electrolyte nature. New Schiff base ligand and its metal complexes were Screened against bacterial and fungal strains and their preliminary results compounds exhibited the high effect of activity. This may be attributed to the impact of both the chelate effect of new Schiff bases ligand and the role of the metal in these complexes.

Keywords: Pyridoxal, Azomethine, 3,4-diamino benzophenone, Transition metal complexes. Biological activity

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INTRODUCTION

Pyridoxal is organic molecule a close analog of pyridoxine (a form of vitamin B_6) and its transition metal complexes are investigated by many researchers. Schiff bases derivaties from pyridoxal constitute an important class of heterocyclic being the core fragment of different natural product and biological application $^{(1-4)}$. Many Schiff base ligands of pyridoxal have been synthesized semicarbazide–based derivaties$^{(5)}$,amino acid$^{(6)}$,aminoguanidine, cephalexin$^{(7,8)}$, 4-aminoantipyrine$^{(9)}$, Metal complexes of imine pyridoxal have various applications in different scientific fields $^{(10,11)}$ pyridoxal- semicarbazone (PLSC), it is show against microbial activity, anticancer and cytotoxicity$^{(12,13)}$. It is used in the preparation of azomethine groups, intramolacular hydrogen bonding between (OH) hydrogen and (C=N) nitrogen atom of Schiff bases determines the biological properties.

Here in, we report the Synthesis and Characterization spectral Studies and Biological Screening Studyof New Schiff base ligand and its metal complexes with Co (II), Ni (II), Cu (II), Zn (II), Cd (II), Pt (II), and Au (III) ions.

Experimental

MATERIALS AND METHODS

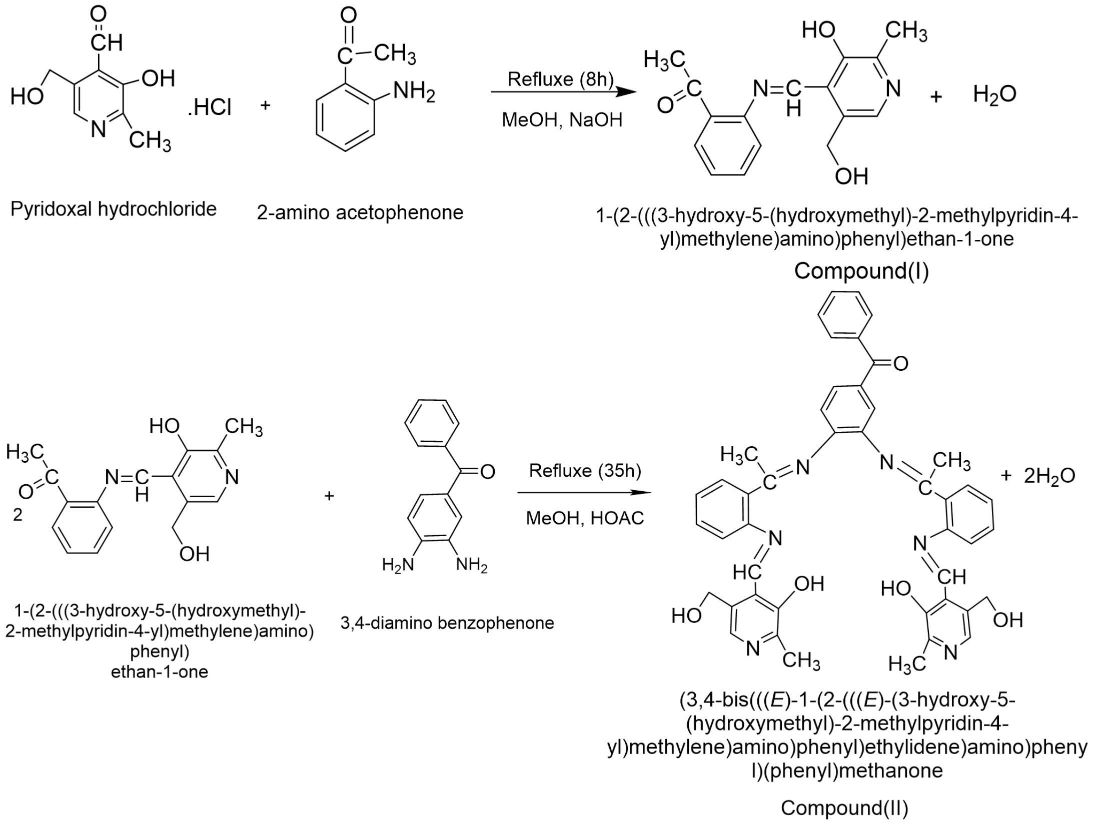
All chemicals were obtained from Sigma - Aldrich, and Merck and used without further purification. Melting point were determined using model 9300 of ligand and its complexes. The UV-Visible spectra were recorded on Shimadzu spectrophotometer double beam model 1700. IR spectra were recorded on Shimadzu FTIR 8400 spectrometer using KBr pellet in the wavelength range 4000-400 cm ^{1}. Magnetic susceptibility measurements were carried out on a balance magnetic MSB-MKI using faraday method. The diamagnetic corrections were made by Pascal's constants. Elemental analyses were performed by means of EURO 2012EA 300 C.H.N Elemental analysis. ^{1}HNMR spectra were recorded as solution in DMSO d^6 as solvent using (varian 500MHZ Spectrophotometer), and Mass Spectra were recorded on Shimadzu Agilent Technologies 5975C.

Synthesis of the new Schiff base ligand (BHMAPh)

Synthesis of (3,4-bis(((E)-1-(2-(((E)-(3-hydroxy-5-(hydroxymethyl)-2 methylpyridin -4-yl) methylene) amino) phenyl) ethylidene) amino) phenyl) phenyl) methanone

Equimolar quantity (1:1) mole ratio, (15ml) methanolic solution of pyridoxal hydrochloride (PL. HCl) (2.03g, 0.01mol) was added to solution of 2-amino acetophenone (1.35 ml,0.01mol) in same solvent were mixed thoroughly. To this mixture 0.1% methanolic NaOH was added to the reaction mixture as catalyst for adjust the (pH= 7-8) and refluxed with stirring for (8h). A clear yellow colored solution was collected for isolation of solution by crystallization. The product took place after volume reduction by evaporation, then dried over anhydrous $CaCl_2$, yield= 75%, melting point (m.p =165-167°C).

The solid product from standard method compound (I) has been taken and dissolved (5.6 gm, 0.02 mol) in (50ml) of methanolic with the same volume of methanolic solution of 3,4-diamino benzophenone (2.12g ,0.01 mol). To this mixture was added drops from glacial acetic acid. The reaction mixture was refluxed with stirring for (35h). Schiff base ligand was isolated after the volume of the mixture was reduced to half by evaporation and precipitated product was collected by filtered off, purified by crystallization from hot ethanol, and dried over anhydrous $CaCl_2$. Yield: 77% (mp: 175-177C°) As showing in Scheme. 1.



Scheme. 1. Preparation of the new Schiff base ligand (BHMAPh).

Preparation of metal complexes

Complexes of the ions Co (II), Ni (II), Cu (II), Zn (II), Cd (II), Pt (II), and Au (III) were prepared by the mixing of (50ml) ethanolic solution with (0.744g, 1mmol) of new Schiff base ligand dissolved in (50ml) from the same solvent in (1:1) (metal: ligand) ratio. The resulting mixture was refluxed for (2 hr) at (70-80°C) and cooled to room temperature. The final product was filtered and washed with ethanol, dried in air.

Determination of Antimicrobial Activity

The new Schiff base ligand and metal complexes were evaluated for their antimicrobial activity in vitro against bacteria and fungi, Bacteria activity Pathogenic biological, *Esherichia coli, Staphylococcus aureus and Klebsilla* were used to test the antimicrobial activity of the ligand and it is metal complexes. The nutrient agar (NA) medium was prepared and quantity of 10ml of the medium was poured into the sterilized petri plates and allowed to solidify. The plates were inoculated with spore suspension of pathogenic bactericides. By using the sterilized cork bore, well, s was dug in the center of the culture plates, the test complexes solution was added (0.5 ml) to these wells and the plates were incubated at 25^0C for (24 hr). Then the inhibition zone appeared around the wells in each plate was measured and recorded as the cyto toxic effect of the appropriate complexes.

Fungal activity the complexes and ligand were tested against fungi such as *Aspergillus niger, Aspergillus flavus*, cultured on potato dextrose agar as medium. In atypical procedure, as well was made on the agar medium inoculated with the fungi- the well was filled with the test. Solution using a micropipette and the plate was incubated at 30 ^{0}C for 72 hr. During this period, the test solution diffused, and growth of the inoculated fungi was affected.

RESULTS AND DISCUSSION

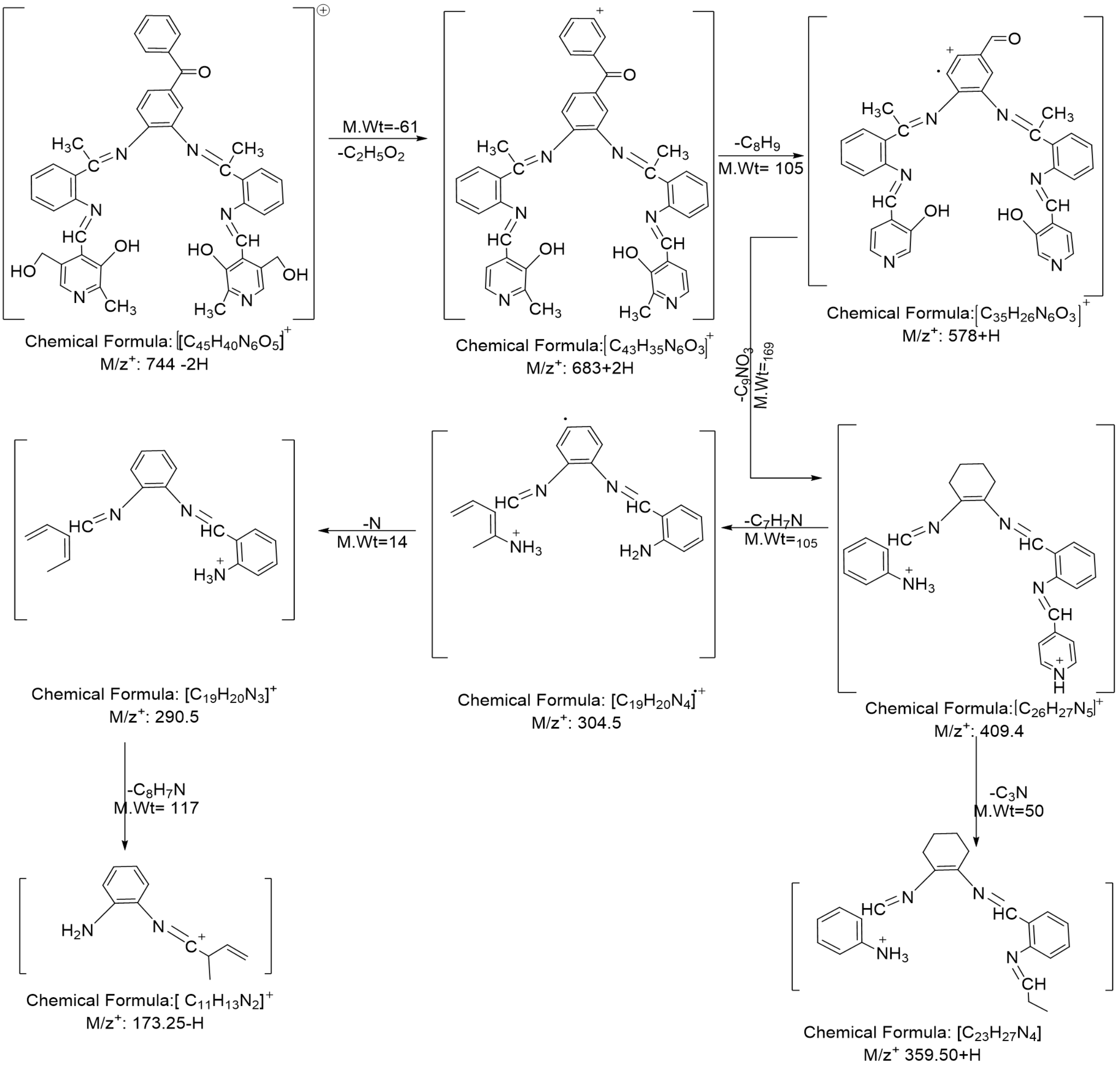
The new Schiff base ligand and the metal complexes are colored. All complexes are stable towards air. Generally, the complexes are insoluble in water, but they are soluble in common organic solvents (DMSO, DMF, Methanol and Ethanol). The metal complexes were characterization by (C.H.N) micro elemental analysis, FT-IR, UV-Vis, ^{1}HNMR, molar conductivities, mass spectrum and the magnetic moment. The analytical data of the complexes are agreed with the experimental data. The molar conductivity values show that of the complexes were (1:1) (metal: ligand), this result proves that complexes have non-electrolytic nature, except the Pt (II) and Au (III) complexes which showed higher conductivity value, this supports the electrolytic nature of the chelate complex. The magnetic moments value of the complexes is agreement with geometry structure. Some physical and chemical propertied for the new Schiff base ligand and it chelate complexes are recorded at Table 1.

Table 1. The analytical and the physic-Chemical data of the new Schiff base ligand and its metal complexes.

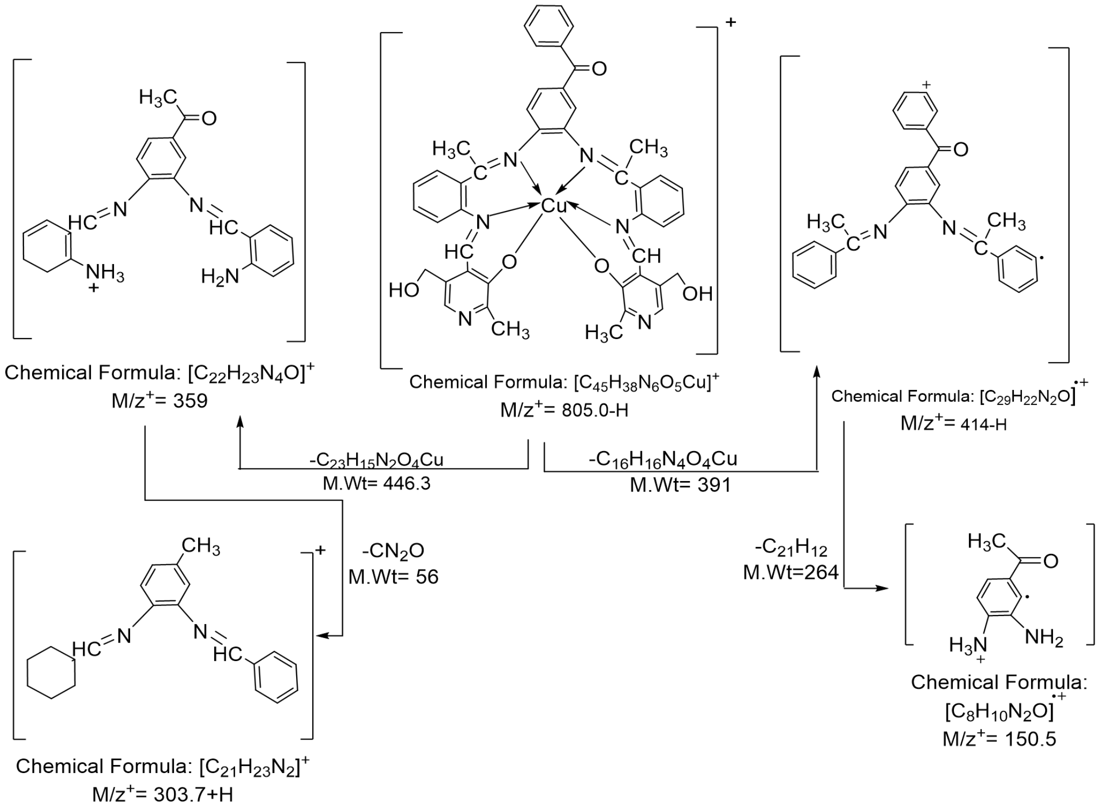
No.	Compound	Formula Weight	Colour	M.p.℃	Yield %	Found (Calc.) %			
						%C	%H	%N	%M
1	HL=$C_{45}H_{40}N_6O_5$	744	yellow	175-177	77	(72.58) 72.67	(5.37) 5.40	(11.29) 11.90	-----
2	[Co (HL)]	800.9	Green	221-223	78	(67.42) 67.81	(4.74) 4.92	(10.48) 10.78	(7.35) 7.50
3	[Ni (HL)]	800.7	Green	178-180	73	(67.44) 67.73	(4.74) 4.82	(10.49) 10.98	(7.33) 7.89
4	[Cu (HL)]	805.5	Green	200-201	80	(67.03) 67.61	(4.71) 4.82	(10.42) 10.92	(7.88) 7.95
5	[Zn (HL)]	807	Colorless	193-195	72	(66.91) 66.94	(4.70) 4.79	(10.40) 10.42	(8.05) 8.87
6	[Cd (HL)]	854	Colorless	202-204	74	(63.23) 63.89	(4.49) 4.54	(9.83) 9.91	(13.11) 13.50
7	[Pt (HL)] Cl_2	1010	Green	180-182	80	(53.46) 53.62	(3.96) 3.93	(8.31) 8.34	(19.30) 19.65
8	[Au (HL)] Cl_3	1047	Green	224-226	75	(51.57) 51.87	(3.82) 3.73	(8.02) 8.62	(18.76) 19.80

Mass spectra

Mass spectra of new Schiff base ligand and its Cu (II) complex are shown in **Figuer1,** and the suggested mass spectral fragmentations of the ligand and it chelate complex is shown in **schemes** 2 and 3. The mass spectrum ligand displays a base peak at m/+z=744 is attributed to the original molecular weight. The mass spectrum of Cu (II) complex displays base peak at m/z+=805 which corresponds to the exact molecular weight of the Cu (II) complex. which are in good agreement with their formula as expressed from micro analytical data



Scheme 2. Suggested fragmentation pathways of the new Schiff base ligand and structural assignments of fragments.



Scheme 3. Suggested fragmentation pathways of Cu- complex and structural assignments of fragments.

^{1}HNMR spectra

The data of ^{1}H-NMR spectra of Schiff base ligand and Zn (II) complex are show in **Figure 5,6** was measured in d^6-DMSO solvent with TMS as an internal reference. The ^{1}HNMR spectrum of the ligand shows the following signals: phenyl multiples at (7.2-8.0) ppm, CH-methane at (8.2) ppm**,** C-CH$_3$ acetyl acetone at (3.1) ppm, CH$_2$-pyridin ring at (4.8) ppm, CH$_3$-pyridin ring at (2.8) ppm **[14,15]**. Methanolic-OH at (5.8) ppm, Phenolic-OH at (9.3) ppm **[16]**. This signal disappear in spectra of Zn (II) complex indicates to absence (OH) proton and contribute the oxygen atom in complexity. There is no appreciable change in all other signals in complex.

FT-IR Spectra of new Schiff base ligand and its metal ion complexes

FTIR spectra the new Schiff base ligand and their metal complexes are scheduled in **(Figure 7 and 8).** In The infrared spectrum of the ligand exhibit broad band at (3412) cm^{-1} were assigned to **υ**(OH) group in the free Schiff base ligand confirmed that hydroxyl group was present in Schiff base ligand, which is absent in all metal complexes except the Pt (II) and Au (III) complexes, revealed that it was coordinated to metal ions via deprotonation of phenolic **υ**(OH)(17.18). Band at (1643) cm^{-1} due to carbonyl **υ** (C=O) of benzophenone group, unchanged this band in the complexes (19).

The bands at (1614, 1671) cm^{-1} that was assigned to **υ**(CH=N) in the free Schiff base ligand, the azomethine peak in ligand is shifted, indicating the coordination through **υ** (-C=N-) group (20,21). The appearance of new absorption bands at (503-588) cm^{-1} and (443-449) cm^{-1} refer to **υ**(M-N), **υ**(M-O) sequence in some complexes, confirmed that O and N atoms of the ligand banded with the metal ions (22). Results showed that The ligand behavior as a hexadentate chelating, bonded to the metal ion via the two nitrogen atoms azomethine υ(-C=N-), and phenolic υ(OH) groups and of the Schiff base ligand, except the Pt (II) and Au (III) complexes the ligand acts as a tetra dentate chelating agent, indicates that the coordination of azomethine nitrogen atoms. The spectra data of the new Schiff base ligand and the metal complexes were shown in table (2).

Table 2: FT-IR spectral data (cm^{-1}) of new Schiff base ligand and its complexes.

Compound	*υ(OH)*	*υ(OH) methanolic*	*υ(C=N)*	*υ(M-N)*	*υ(M-O)*
HL=$C_{45}H_{40}N_6O_5$	3412	3192	1641,1671	-----	-----
[Co (HL)]	-----	3066	1670, 1575	509	443
[Ni (HL)]	-----	3381	1614	588	443
[Cu (HL)]	-----	3412	1598	553	449
[Zn (HL)]	-----	3410	1698	567	447
[Cd (HL)]	-----	3415	1625	534	444

[Pt (HL)] Cl_2	3385	3105	1637, 1593	584	-----
[Au (HL)] Cl_3	3221	3192	1604	503	-----

Electronic spectra and magnetic susceptibility measurements

Ultraviolet – visible spectra are one of the important methods used in the field of coordination chemistry, is studied and compared metal complexes spectra with free ligand spectra.

The electronic absorption spectra of the new Schiff base ligand and its complexes were recorded in freshly ethanol solution (10^{-3}) at room temperature. The absorption region band assignment and the proposed geometry of the complexes Co (II), Ni (II), Cu (II), Zn (II), Cd (II) are octahedral except Pt (II), Au (III) complexes, which had square planar geometry. Their relevant data shown in **Table. 3**.

The new Schiff base ligand shows two charge transfer (CT) absorption bands in the region Uv-Visible at (352) nm (28409) cm^{-1}, and (436) nm (22935) cm^{-1}, which were allotted to (π -π^*) and (n-π^*) transition within molecule [(23)]. In the metal complexes, these absorption bands undergo of red-shifted indicating that the Schiff base ligand are coordinated to the metal ions [(24)].

The Co (II) complex exhibits new three absorption bands at (378) nm (26455) cm^{-1}, (655) nm (15267) cm^{-1}, (678) nm (14749cm^{-1}) assigned to MLCT, $^4T_1g \rightarrow ^4A_2g_{(F)}(\nu_2)$, $^4T_1g \rightarrow ^4T_2g_{(p)}(\nu_3)$ transitions respectively [(25)]. This indicates the present of an octahedral geometric structure of cobalt (II) complex. The magnetic moment value (3.3 B.M) may be taken as additional evidence for octahedral geometry [(26)].

The Ni (II) complex is brown in colour exhibits absorption bands at (765) nm (13071) cm^{-1}, (562) nm (17793) cm^{-1}, (480) nm (20833) cm^{-1} which assigned to $^3A_2g \rightarrow ^3T_2g_{(F)}(\nu_1)$, $^3A_2g \rightarrow ^3T_1g_{(F)}(\nu_2)$, $^3A_2g \rightarrow ^3T_1g_{(P)}(\nu_3)$ transition respectively [(27)]. The magnetic moment value of this complex is (2.4 B.M) which was very close to the octahedral geometry structure [(28)].

The spectrum of the Cu (II) [(29,30)] complex appears abroad absorption band around at (622) nm (16077cm^{-1}) described to electronic transition type $^2Eg \rightarrow ^2T_2g$, the magnetic moment of this complex was found at (1.78 B.M) which was very close to octahedral environment [(31)].

The spectrum of Zn (II), Cd (II) complexes showed band at (375) nm, (26666cm^{-1}), and (423) nm (23640cm^{-1}) respectively assigned to a charge transfer transition, no (d-d) field transition band but always shows prominent (C.T) transition [(32)]. The Zn (II), and Cd (II) complexes is diamagnetic moment because possess completely filled d^{10}configuration [(33)]. Based on these data, an octahedral geometry is proposed to the complexes.

The spectrum of the Pt (II) complex is green in colour display one absorption bands at (546) nm (18315 cm^{-1}) showed the spin-allowed transition which designed $^1A_1g \rightarrow ^1T_1g$ transition, the magnetic moment is diamagnetic nature because has ($5d^8$) system, according to electronic spectra data suggest a square planar geometry around Pt (II) ion [(34,35)].

The electronic spectrum of the Au (III) complex exhibits one band at (443) nm (22573cm $^{-1}$) which assigned to $^1A_1g \rightarrow$ ^1B_1g transition [(36)], the Au (III) complex is diamagnetic moment and a square planar [(37)]. The UV-Vis spectra of the new Schiff base ligand and Cu (II) complex are shown in **(Figure 9 and 10).**

Molar conductivity measurements

The conductivity values ($\Lambda\mu$) of prepared complexes in DMSO solvent at a concentration of (10^{-3}M) in room temperature are listed in **Table (3).** The result explained molar conductance in the range between (12 – 18) $S.cm^2.mol^{-1}$, indicating that the compounds are non-electrolytes [(38)], except the molar conductance of Pt (II) and Au (III) complexes are (74, 130) S. $cm^2.mol^{-1}$ respectively suggesting that the electrolytic nature, indicate are present chloride ions outside the coordination sphere [(39)].

Table 3: Electronic spectra (nm, cm ^{1}) magnetic moments, geometry, hybridization and conductivity.

Compounds	λ_{max} (nm)	Absorption Bands (cm ^{1})	Transitions	μ_{eff} (B.M)	Geometry	Hybridization	Conductivity $S.mol^{-1}. cm^2$
HL=$C_{45}H_{40}N_6O_5$	352	28409	$\pi \rightarrow \pi^*$	----	----	----	----
	436	22935	$n \rightarrow \pi^*$				
[Co (HL)]	678 655 378	14749 15267 26455	$^4T_1g \rightarrow ^4T_2g_{(F)}$ ν_1= ν_2= $^4T_1g \rightarrow ^4A_2g_{(F)}$ MLCT	3.3	Octahedral	Sp^3d^2	12
[Ni (HL)]	765 562 480	13071 17793 20833	ν_1=$^3A_2g \rightarrow ^3T_2g_{(F)}$ ν_2= $^3A_2g \rightarrow ^3T_1g_{(F)}$ $\nu_{3=}$ $^3A_2g \rightarrow ^3T_1g_{(P)}$	2.4	Octahedral	Sp^3d^2	15
[Cu (HL)]	622	16077	$^2Eg \rightarrow ^2T_2g$	1.78	Octahedral	Sp^3d^2	18
[Zn (HL)]	375	26666	$d\pi(Zn)^{+2} \rightarrow \pi^*(L)$	Dia	Octahedral	Sp^3d^2	13
[Cd (HL)]	423	23640	$d\pi(Cd)^{+2} \rightarrow \pi^*(L)$	Dia	Octahedral	Sp^3d^2	15
[Pt (HL)] Cl_2	546	18315	$^1A_1g \rightarrow ^1T_1g$	Dia	Square planer	dsp^2	74

[Au (HL)]Cl_3	443	22573	$^1A_1g \rightarrow ^1B_1g$	Dia	Square planer	dsp^2	130

Antimicrobial Activity
The data of antimicrobial activaties of the prepared new Schiff base ligand and its complexes are given in Table 4, the result showed that most of the complexes have more toxicity against the bacteria and fungi species, increased activity of the metal chelates can be explained on the basis of Chelation theory [40]. On Chelation, the polarity of the metal ion will be reduced to a greater extant due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. Further, it increases the delocalization of the complexes into lipid membranes and blocking of the metal Binding sites in the enzymes of microorganisms. These complexes also disturb the respiration process of the cell and thus block the synthesis of proteins, which restricts further growth of the organism [41]. Furthermore, the mode of action of compound may involve the (C=N) group and Heterocyclic Ligand with active centers of cell constituents resulting in the inter ference with normal cell process.

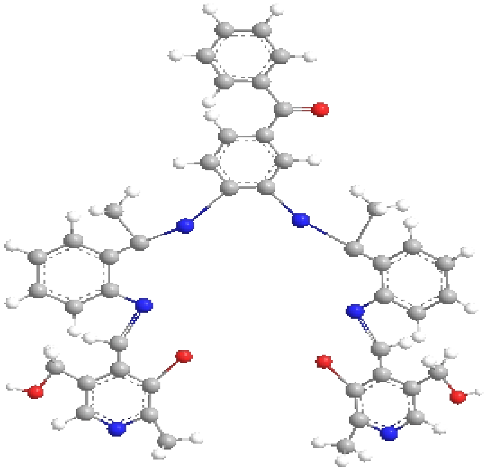


Fig. 1: Molecular structure of new Schiff base ligand

Table. 4: Antibacterial and Antifungal activities for ligand and its complexes

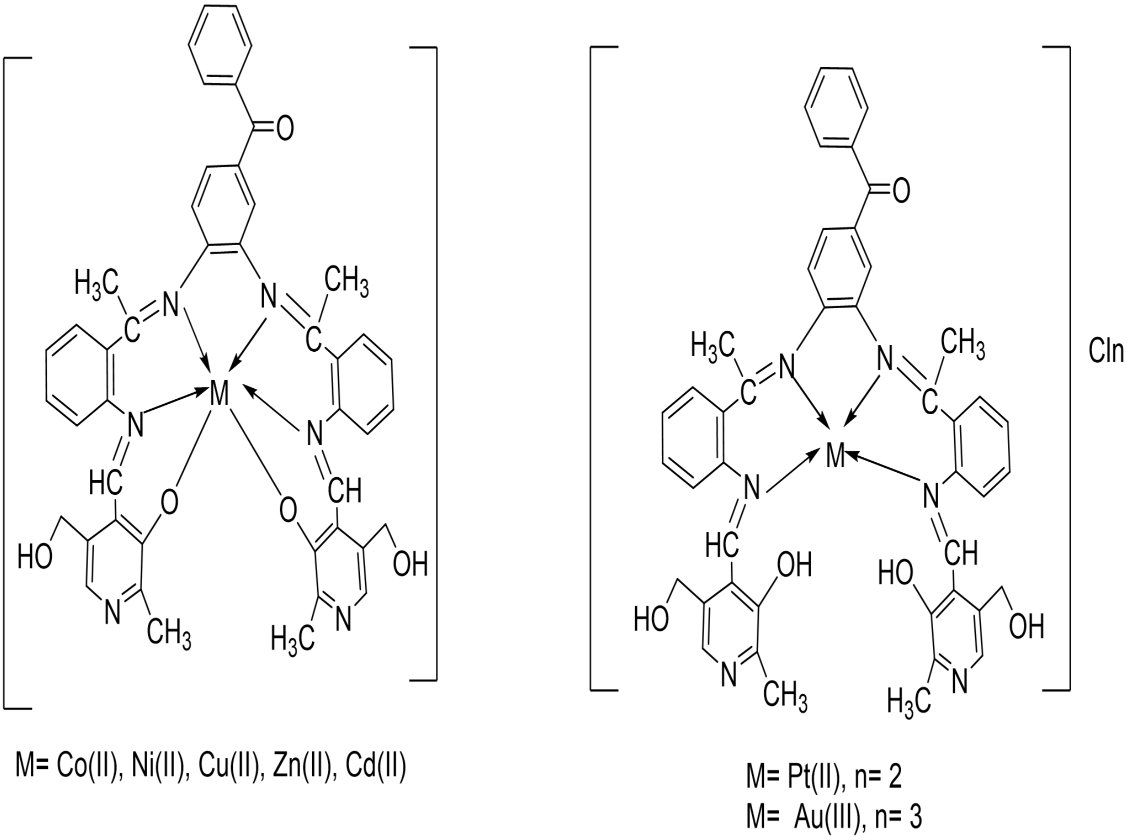
Compounds	Staphylococcusaureus(+) 1×10^{-4} M, 1×10^{-5}M	A. Flavus 1×10^{-4} M, 1×10^{-5}M	A. Niger 1×10^{-4} M, 1×10^{-5}M	Klebsilla 1×10^{-4} M, 1×10^{-5}M	Esherichia coli (-) 1×10^{-4} M, 1×10^{-5}M
HL=$C_{45}H_{40}N_6O_5$	_ _	_+	_+	_ _	_ _
[Co(HL)]	_ +	++	_+	_ +	+ +
[Ni (HL)]	+ +	_+	++	+ +	++ ++
[Cu (HL)]	+ +	_+	++	+ +	+ +
[Zn (HL)]	_+	_+	_+	_+	_+
[Cd (HL)]	_ +	_ +	_ +	_ +	_ +
[Pt (HL)] Cl_2	+ +	+ +	+ +	+ +	+ +
[Au (HL)] Cl_3	+ +	+ +	+ +	+ +	+ +

The suggested structures of the metal complexes
According to the analytical data and spectroscopic studies of the prepared metal complexes are show in **Fig. 2**. The new Schiff base ligand behaves as a hexadentate chelating agent coordination through the nitrogen atom of azomethine group, and oxygen atom of (-OH) phenolic group, except the Pt (II) and Au (III) complexes, the ligand acts as a tetradentate chelating agent.

CONCLUSION

In this study have reported the synthesis and characterization of Schiff base ligand containing(-C=N-) group and its Co (II), Ni (II), Cu (II), Zn (II), Cd (II), Pt (II), Au (III) complexes were synthesized and characterized by various spectroscopic techniques. The result show octahedral geometry around of the complexes with mole ratio (1:1) metal: ligand while the complexes of Pt (II) and Au (III) show a square planar geometry around the complexes with mole ratio (1:1) metal: ligand.

According to elemental analysis, Uv-Vis, FT-IR, spectroscopy, melting point, conductivity, magnetic susceptibility data Atomic absorption, magnetic moment measurements and mass spectrum studies. The following structures are proposed for the synthesized metal complexes as shown in Fig.2. Biological activity studies of the Schiff base ligand and its metal complexes against several organisms, staphylococcus aureus, Esherichia coli, Klebsilla, Aspegills niger, Aspegills flavus are reported. higher effect activity.



M= Co(II), Ni(II), Cu(II), Zn(II), Cd(II)

M= Pt(II), n= 2
M= Au(III), n= 3

Fig.2. The proposed structural formula of the complexes.

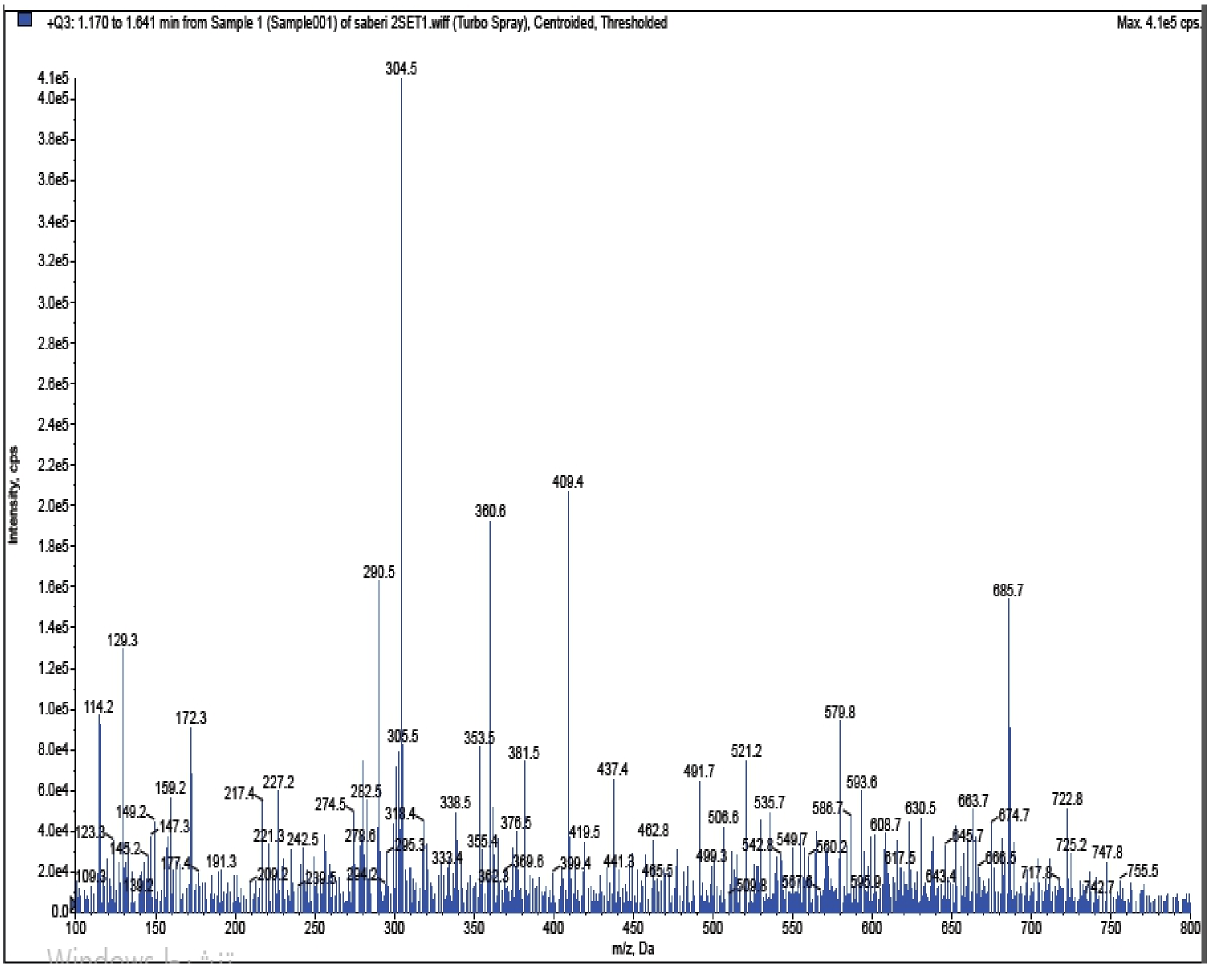


Fig. 3: the mass spectrum of the new Schiff base ligand

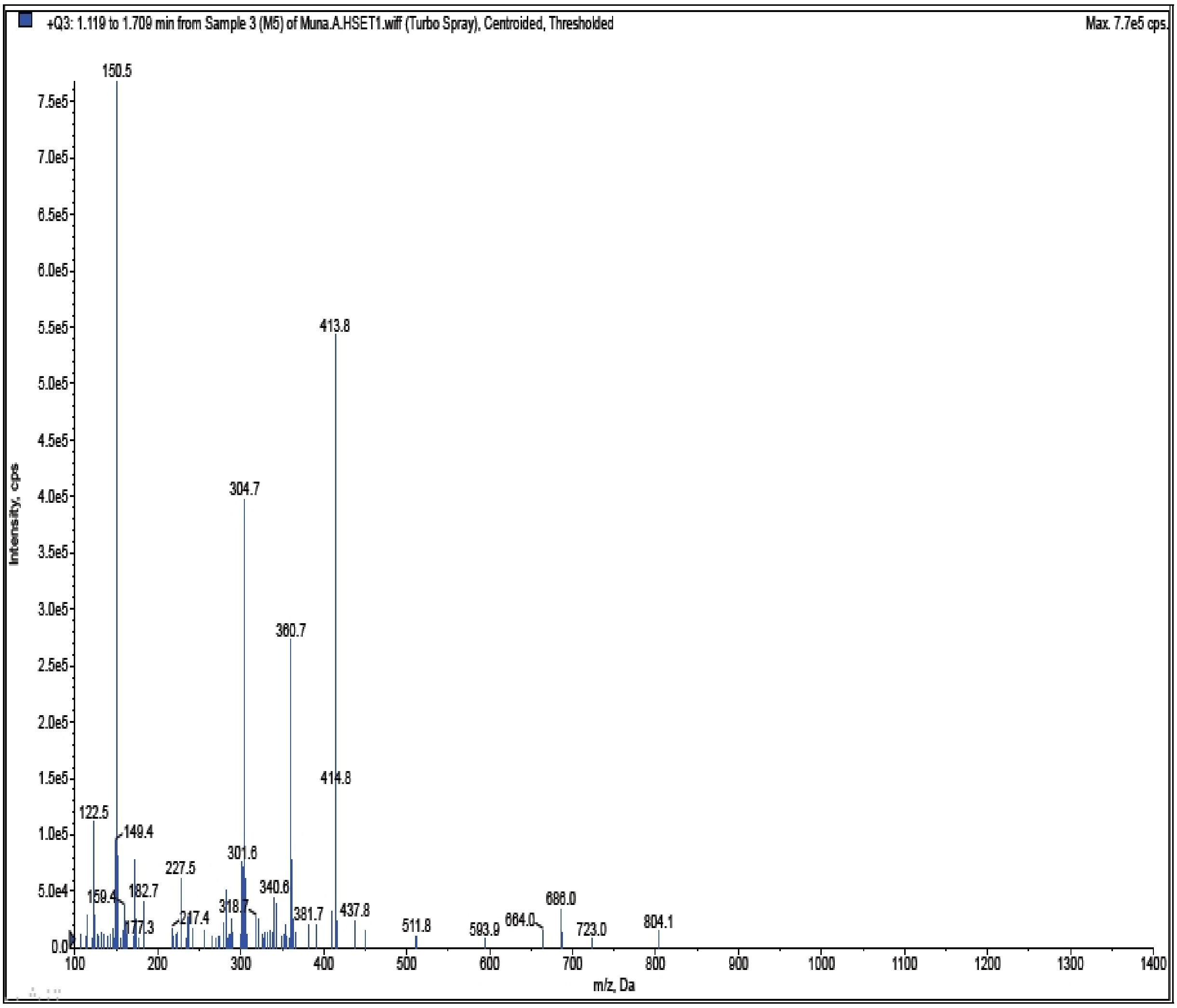


Fig. 4: the mass spectrum of the Cu (II) complex.

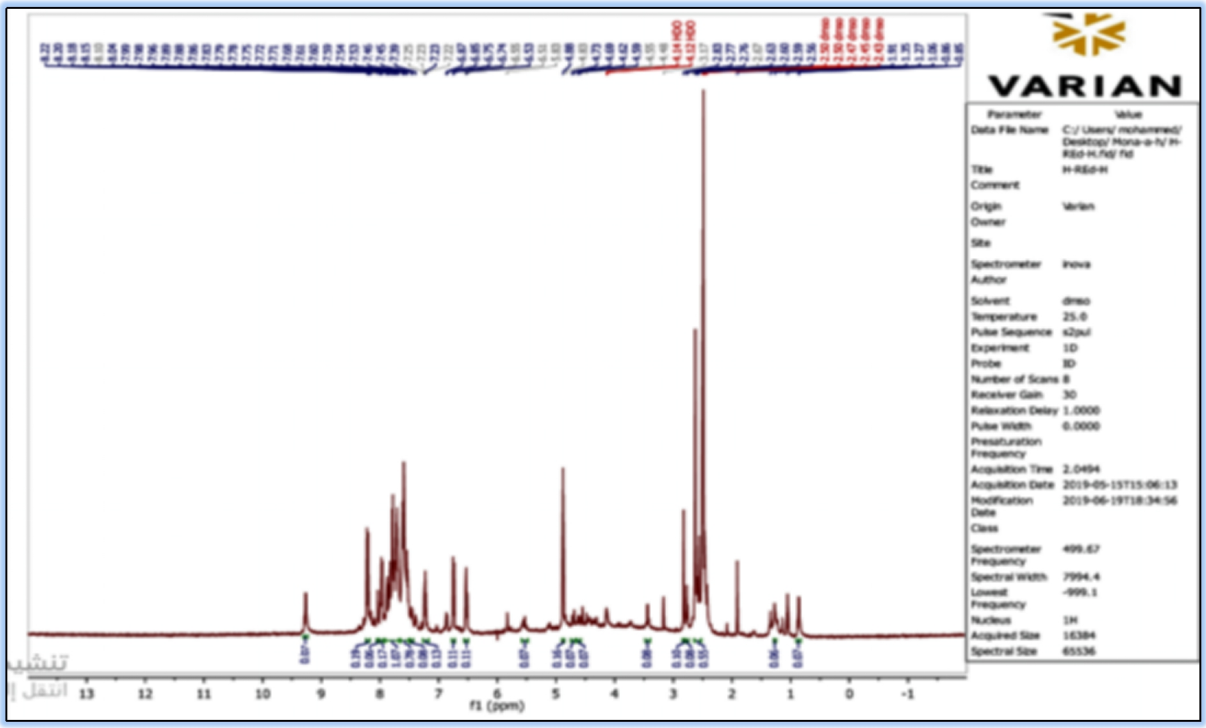


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Fig. 5: ^{1}H-NMR Spectrum of the new Schiff base.

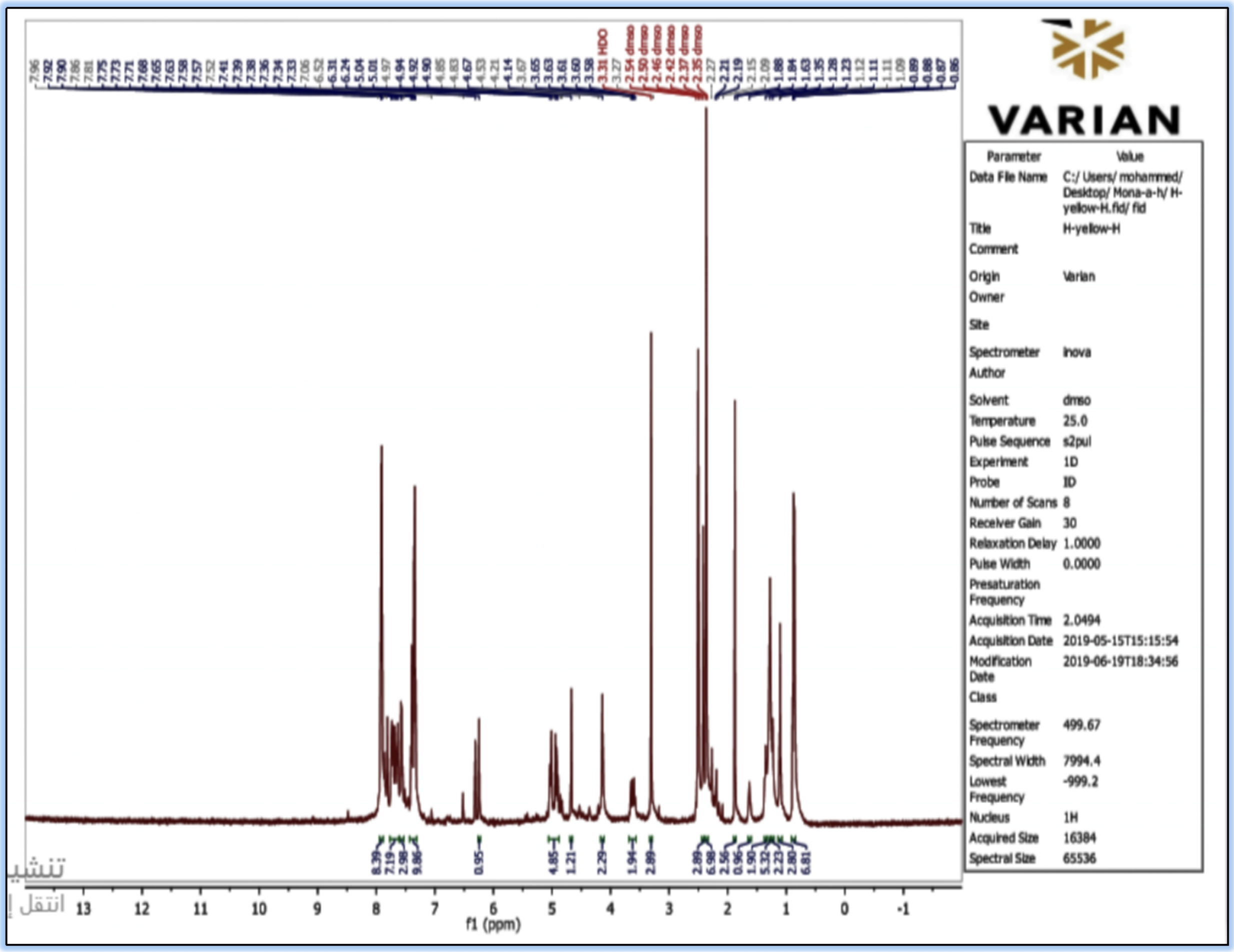

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Fig 6: ^{1}H-NMR Spectrum of Zn (II) complex.

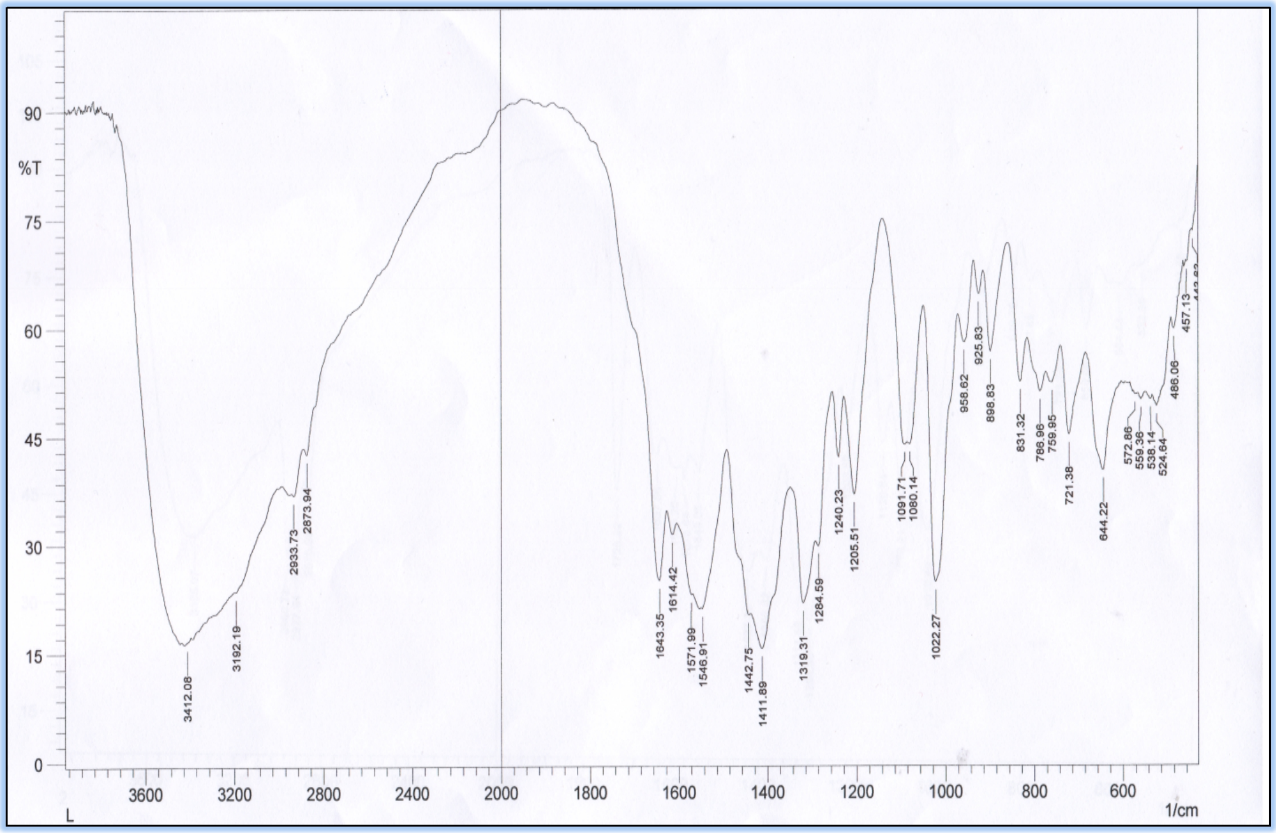


Fig 7: FT-IR bands of the new Schiff base ligand

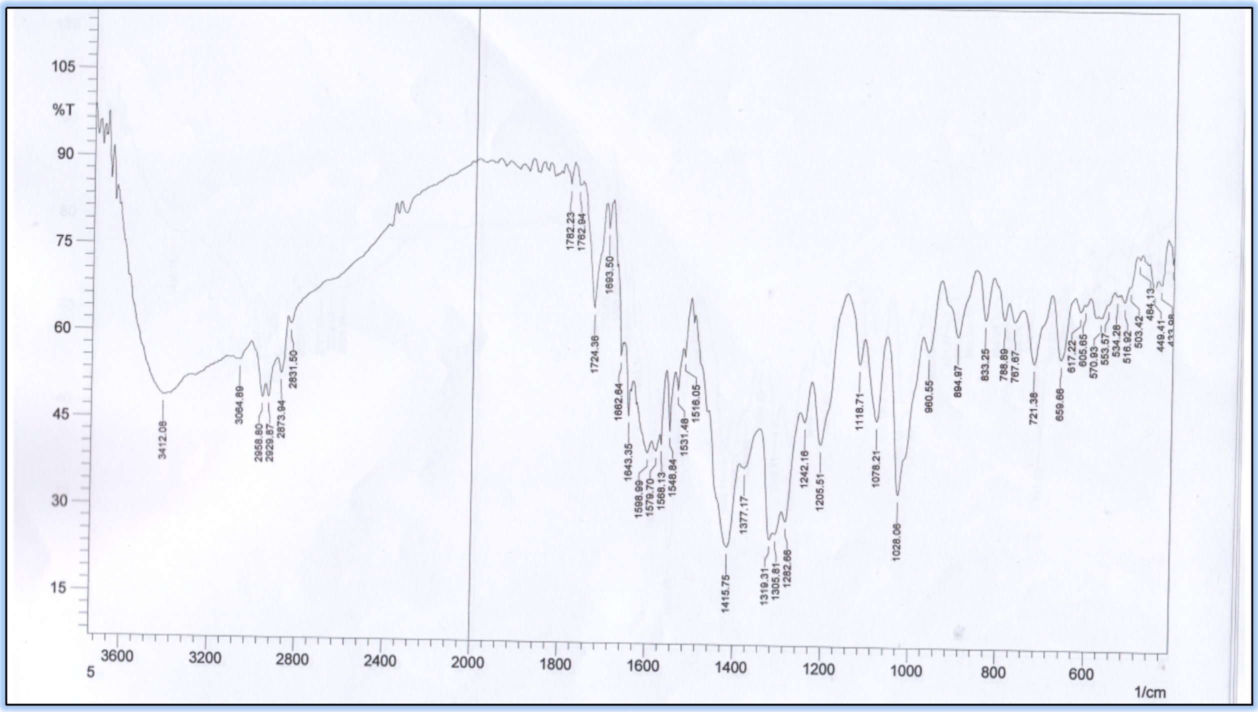
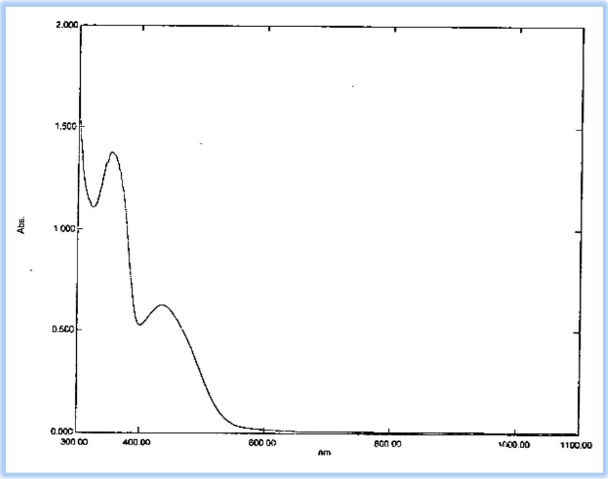


Fig 8: FT-IR bands of Cu (II) complex.



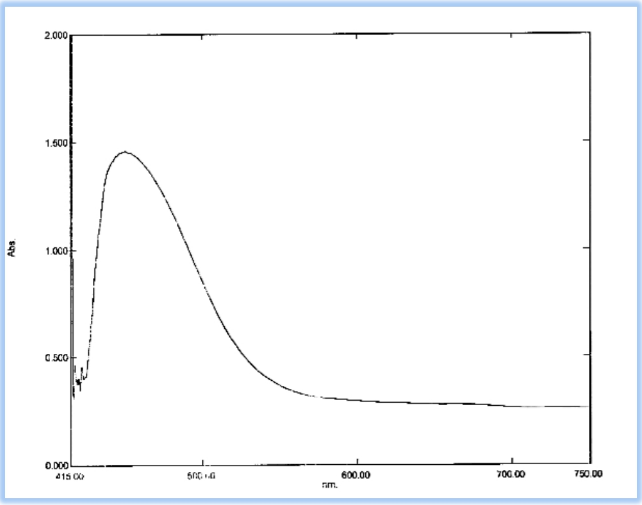


Fig 9: UV-Vis. Spectrum of the new Schiff base ligand

Fig 10: UV-Vis. Spectrum of Au (III) complex.

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