

## Mixed Ligand Schiff Base Complexes of Some Metal (II) Salts: Preparation, Spectral Characterization and Antibacterial Screening

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### Abstract

Schiff base ligand (E)-2-((3-hydroxyphenylimino) methyl)-6-methoxyphenol has been synthesized from m-aminophenol and o-vanillin. The geometry of Schiff base has been determined on the basis of Micro elemental analysis (C.H.N), molar conductance. Mass,  $^1\text{H}$ NMR, FT-IR and UV-Vis spectroscopic techniques. Metal mixed ligand complexes for manganese(II), cobalt(II), nickel(II), copper(II), zinc(II) and cadmium(II) for Schiff base ligand and paracetamol have been prepared in the molar ratio 1:1:1 (M:L: Para), (M= Metal, (L)= Schiff base ligand and (Para) = paracetamol) have been prepared and identified by using flame atomic absorption, FT-IR and UV-Vis spectral methods, on other than magnetic properties and conductivity measurements. Their electrical conductance values indicate that all complexes are electrolytes. From obtained data octahedral geometry were suggested with all prepared complexes. Schiff base ligand, paracetamol as well as metal (II) complexes were assayed by antibacterial activity against some pathogenic bacteria using disc diffusion method.

**Keywords:** Schiff base, Paracetamol, Mixed ligand complexes, Antibacterial screening.

### Introduction

Schiff bases are widely application in medicinal and pharmaceutical chemistry with several biological [1-3]. Schiff base ligand are large studies at the class from coordination chemistry at most due into their cursory synthesis, readily availability, electronic properties are solubility in good common solvents as well they easily shape stable compounds for most transition metal ion [4-5]. Metal complexes of Schiff base ligand were found to possess significant biological as well catalytic activity [6-7].

Schiff base compounds combining phenolic group like chelating moieties within the ligand are considered as models with performing important biological reactions and catalytic effectiveness from metalloenzymes [8]. Mixed ligand compounds were mimic found to act like an efficient catalyst to the reactions from industrial significance containing hydrogenation, hydro formation as well as oxidative hydrolysis from olefins and carboxylation for methanol. These compounds have so appear catalytic

efficacy by kind's oxidation reactions from peripheral and biological significance [9]. Paracetamol (Acetaminophen) (Para) is a mild analgesic with weak anti-inflammatory activity, commonly used for the relief of aches and pains, However, overdose of (Para) may cause liver damage [10]. (Para) has painkiller and antipyretic action. (Para) is beneficial into the treatment fromache such as headache, toothache rheumatism as well neuralgia [11]. (Para) is a well-known drug that finds inclusive applications into medicinal industries.

It is also utilized like an intermediate drug like a precursor at penicillin as well azo dyes, stabilizer with hydrogen peroxide, photographic chemicals. It is used as substitutional in to aspirin. It is one from the most vastly used from all drugs, for abundance from experience obviously fixing it has the standard hypothermia and analgesic with mild pain cases [12]. However, it is known that about medicines work via chelation or through reining metalloenzymes

however with most from the drugs that act like potential ligands, a lot from studies are being achieved to fulfill how metal binding impacts the efficiencies to the drugs [13]. The current implementation is to prepare Schiff base mixed ligand complexes derived of (L) with (m-aminophenol and o-vanilline) and (Para), the prepared complexes were performed using different physicochemical methods as well as antibacterial efficacy studies from novel transition metal complexes for Schiff bases (L) with (Para

## Experimental

### Materials and Methods

#### Chemical

M-Aminophenol, o-vanilline and paracetamol were purchased, metal (II) as well as solvents solution of (Merk, B.D.H. and Riedel). The reagents have been utilized without further refining.

#### Instruments

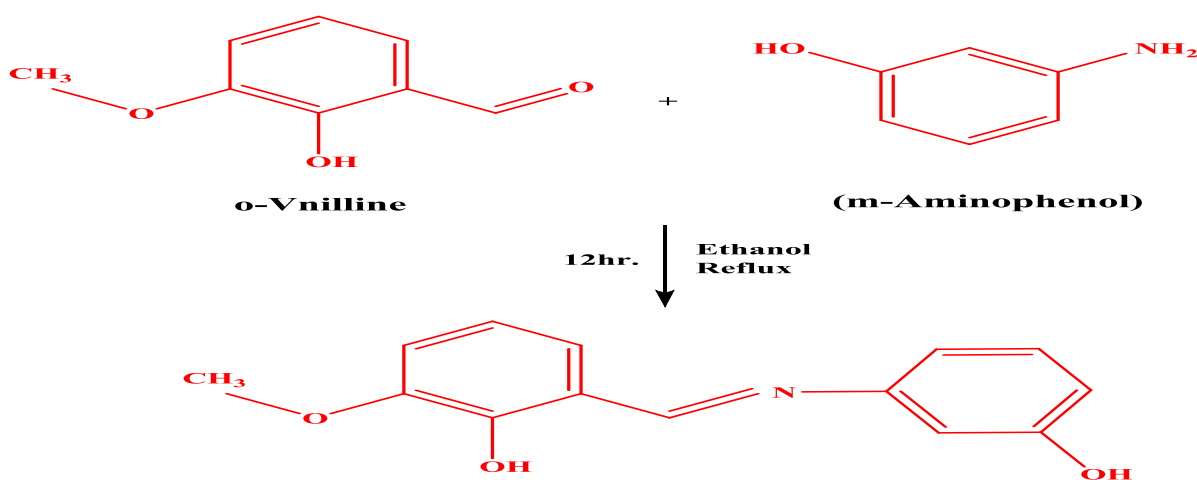
The measured molar conduct values ( $\Lambda_m$ ) at DMSO solution to the Schiff base ligand (L), (Para) and metal (II)- complexes were measured at 25°C with ( $10^{-3}$ M) solution of the samples which uses CON 510 Conductivity. The infrared (FT-IR) spectra have been registered at (4000-400)  $\text{cm}^{-1}$  range of Schiff base (L), (Para) and metal(II)- complexes on FT-IR-600 FT-IR Spectrophotometer using KBr pellets. The electric spectrum from the complexes in DMSO solution ( $10^{-3}$ ) were followed during 25°C in the (200-1000) nm range for a Shimadzu-U.V-160 Ultraviolet

spectrometer at  $1.000 \pm 0.001$  cm matched quartz cell. The C, H as well as N elements for Schiff base (L) as well as complexes have been nominated through the micro analytical unit at Euro EA Elemental Analyzer. When metal (II) contents of the complexes have been identified from before Atomic Absorption (A.A) Technique / Flame Emission Spectrophotometer using AA-680 Shimadzu. Magnetic susceptibility measurements were measured through Auto Magnetic Susceptibility Balance Sherwood Scientific.

Melting points have been determined by employing the Stuart Scientific melting point instrument. The  $^1\text{H}$ NMR for Schiff base (L) was recorded by Nmr bruker400mhz in DMSO- $d_6$  with TMS as the internal standard. Mass spectra was recorded for ligand Schiff base by the 5975C VLMSD with Triple -Axis Detector by the analyzer Quadrupole at 230 °C.

#### Synthesis of Schiff base (L)

o. Vanilline (0.152g, 1mM) in 40ml of ethanol was stirred with m-aminophenol (0.109g, 1mM) by heating under reflux, in the presence of 5 drops of acetic acid as a catalyst for 12hr. The deep brown solid (L) formed as shown in Scheme (1) was filtered and recrystallized from ethanol and with molecular weight is equal to 243.3.26 g/mol. The (L) was identified by micro elemental analysis (C.H.N),  $^1\text{H}$ NMR, FT-IR, Mass and UV-Vis spectroscopic mechanisms.

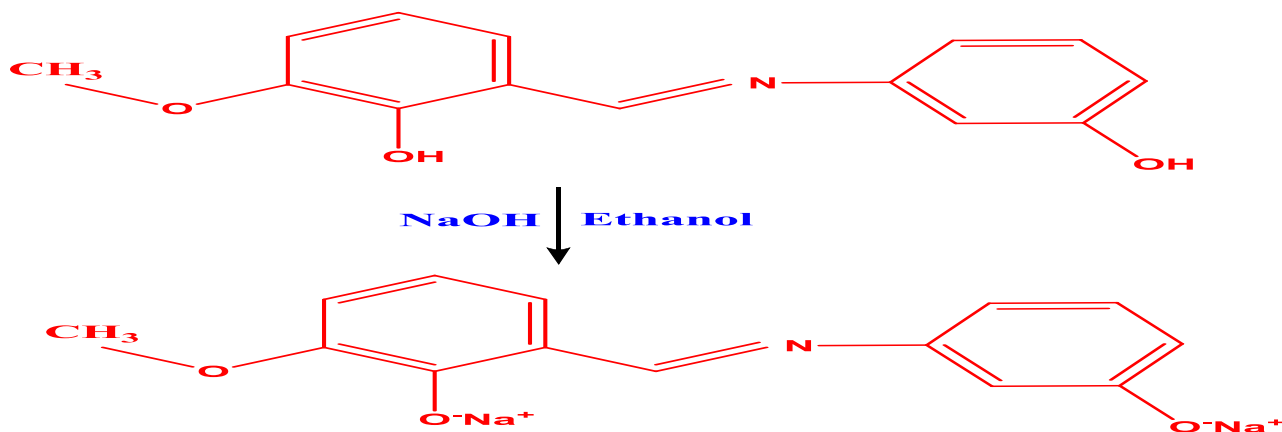


Scheme 1: Preparation of the Schiff base (L)

### Synthesis Metal (II) Complexes

#### Schiff Base (L) solution

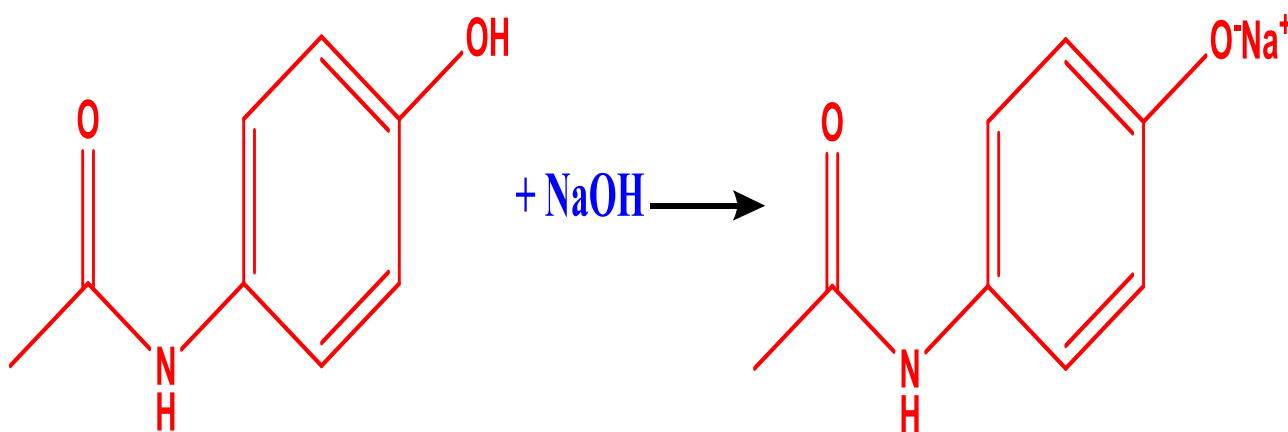
Dissolve (0.243g, 1mM) in 25ml of ethanol and added (0.04g, 1mM) of the sodium hydroxide according in Scheme (2).



Scheme 2: Schiff base (L) solution

### Paracetamol Solution

Dissolve (0.151g, 1mM) in 5ml of ethanol and added (0.04g, 1mM) of the sodium hydroxide according in Scheme (3).



Scheme 3: Paracetamol (Para) solution

### Synthesis of Metal (II)-complexes

The metal (II)-complexes were prepared by adding of the Schiff base (L) solution as well (Para) solution to the ethanol solution to the particular metal chloride of  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (0.197g, 1mM),  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  (0.238g, 1mM),  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (0.238g, 1mM),  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.171g, 1mM),  $\text{ZnCl}_2$  (0.136g, 1mM) and  $\text{CdCl}_2 \cdot \text{H}_2\text{O}$  (0.201g, 1mM).

The mixtures were heated on a water bath to reflux for 3hr. for each complex. The metal complexes were crystal residus observed. The precipitated complexes then were refined off and washed with ether followed by drying at room temperature and analyzing employing standard methods.

### Antibacterial Activity

Schiff base (L), (Para) and metal (II) - complexes were screened for antibacterial activity by agar well – spread method.

The antibacterial action for Schiff base (L), (Para) as well as metal (II) complexes have been checked with each other against Gram positive bacteria (G<sup>+</sup>ve), (*Staphylococcus aureus* and *Staphylococcus epidermidis*) and Gram negative bacteria (G<sup>-</sup>ve), (*Escherichia Coli* and *Kiebsiella sp.*) by means of nutrition's agar well - diffusion method. The solvent utilized for working exam samples and level was DMSO and sample of (1 - 200 µg/ml) were utilized.

Antibacterial activities of any compound were estimated by means of the well-diffusion method. 1cm<sup>3</sup> at a 24h broth the culture including 106CFU/cm<sup>3</sup> was placed in sterilized Petri-dishes. Molten nutritious agar (15cm<sup>3</sup>) was kept in ca.45 °C and teeming in the Petri-dishes and pliable into stiffen. Other holes of 6mm diameter were punctured accurately employing a sterilized cork borer and these were perfectly stuffed

for the exam solutions. The dishes were brood into 24h. Within 37 °C.

## Results and Discussion

### <sup>1</sup>H-NMR Spectrum of the Ligand

The <sup>1</sup>H-NMR spectra from Schiff base (L) in DMSO-d<sub>6</sub> solvent at Fig. (1), appears chemical shift from CH protons to the azomethine [14] group from (L) was displayed in 8.28 ppm.

The spectrum displays various signals in  $\delta=6.35\text{--}7.73$  ppm assigned into aromatic protons [15], the gesture in  $\delta=5.33$  and  $6.34$  ppm due into protons of phenol [16]. The resonance at  $\delta=1.91$  ppm assigned to  $\delta$  (CH<sub>3</sub>) group as well the signals in  $\delta=2.50$  ppm and  $\delta=2.50$  indicated into DMSO-d<sub>6</sub> and water (D<sub>2</sub>O) respectively [17].

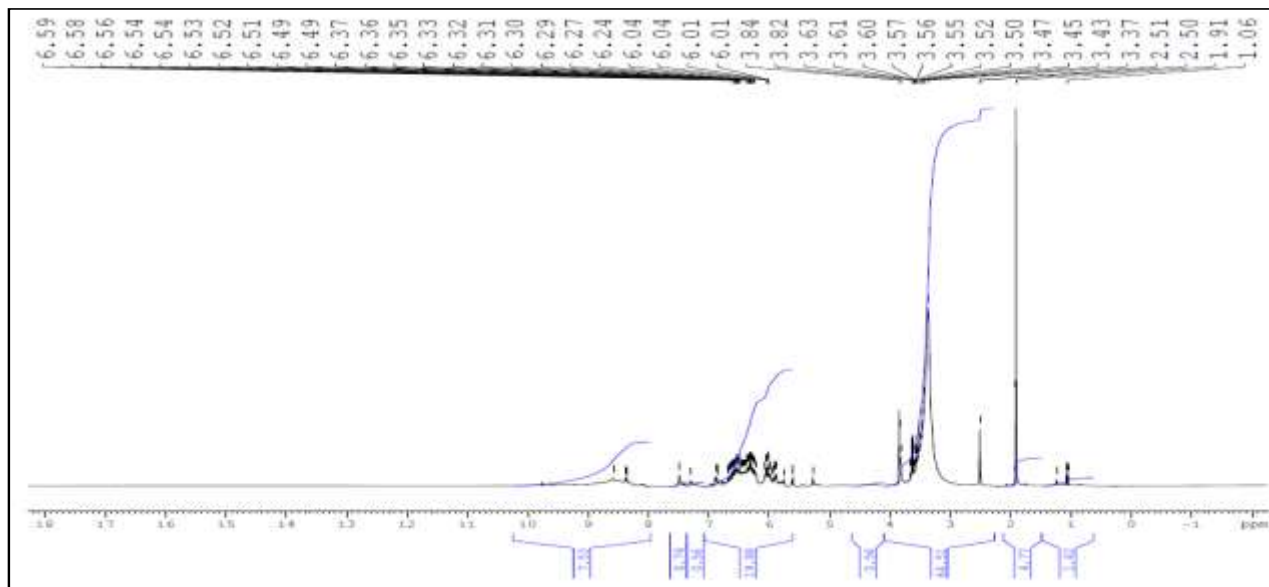


Fig 1: <sup>1</sup>H-NMR spectrum of Schiff base (L)

### Mass Spectrum of the Ligand

The prepared Schiff base (L) has been measured by mass spectroscopy to provide

the molecular weight of the Schiff base (L) shown in Fig. (2).

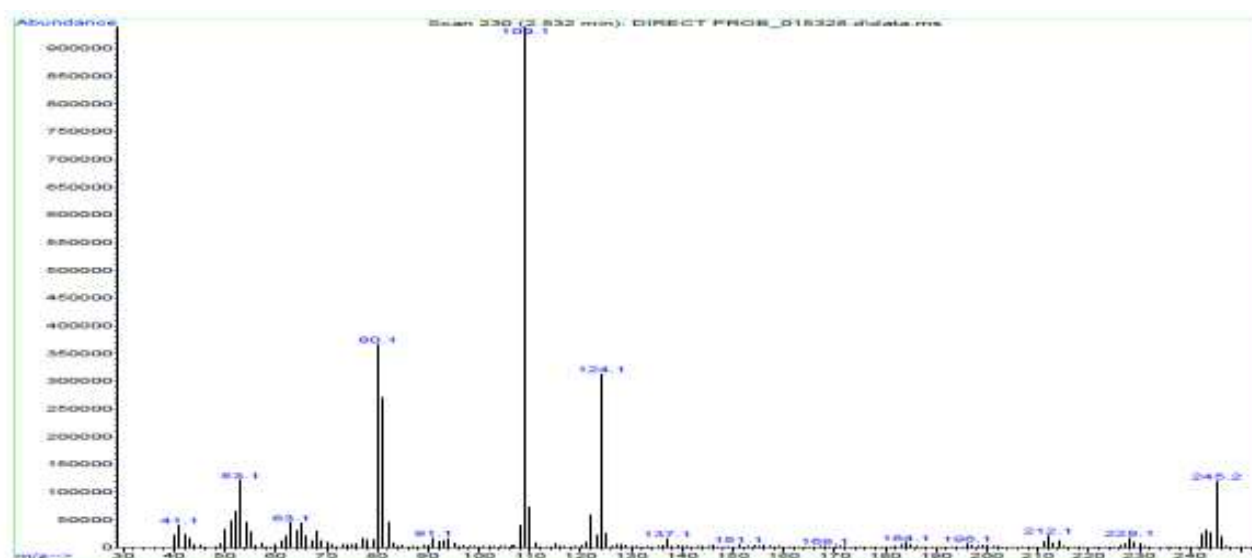
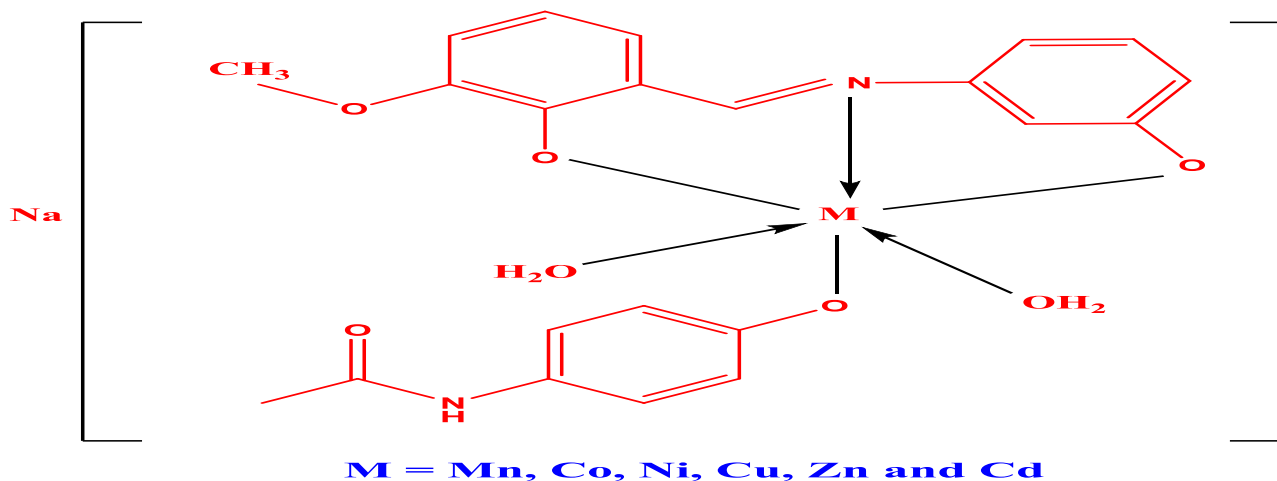


Fig. 2: Mass spectrum of Schiff base (L)

### Physical Properties

The analytical in formations and some of the physical characteristics of Schiff base (L), Para and metal (II) complexes are collected in Table (1) and are proportionate for their

generic form like (1:1:1) mixed ligand complexes of (M: L: Para) to the species of Na [M (L) (Para) (OH<sub>2</sub>)<sub>2</sub>], (M=Mn, Co, Ni, Cu, Zn and Cd) in the Scheme (4).



Scheme 4: The proposed structural formulae of metal (II) complexes

All complexes dissolvable in water, ethanol, acetone, DMF and DMSO, while non-soluble in ether, chloroform and benzene as well all complexes are colored. The molar conduct

values of the ligands and metal (II) complexes during DMSO in  $10^{-3}$  concentration and the type of electrolyte for each compound could be determined [18].

Table 1: Physical properties for compounds

Compounds	Color	M.P. <sup>°C</sup>	Analysis Calc (Found)			
			M%	C%	H%	N%
Schiff base (L)	Deep brown	200 dec.	-	69.13 (68.38)	5.39 (5.59)	5.76 (4.69)
(Para)	White	(174-176)	-	-	-	-
Na[Mn(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Brown	171 dec.	10.49 (9.76)	50.47 (40.10)	3.66 (3.49)	5.35 (5.59)
Na[Co(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Reddish brown	180 dec.	11.18 (10.87)	50.09 (49.59)	3.63 (3.07)	5.31 (3.78)
Na[Ni(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Green	162 dec.	11.13 (10.28)	50.11 (49.47)	3.63 (3.54)	5.31 (4.30)
Na[Cu(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Deep brown	168 dec.	11.94 (11.65)	49.66 (48.54)	3.40 (3.35)	5.26 (4.22)
Na[Zn(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Yellow	175 dec.	12.24 (11.41)	49.49 (48.15)	3.59 (3.18)	5.25 (4.73)
Na[Cd(L)(Para)(OH <sub>2</sub> ) <sub>2</sub> ]	Pale brown	135 dec.	19.34 (18.43)	45.48 (44.21)	3.30 (3.19)	4.82 (3.20)

Dec. = decompose

## FT-IR Spectra

The features frequencies of the ligands and complexes of the metals (II) are set in Table (2) for each of ligands and metal(II) complexes as well the [Schiff base (L) as well (Para)] and nickel(II) complex in Fig.(3). The FT-IR spectra from Schiff base (L) exhibited broad bands in 3375 and 3311  $\text{cm}^{-1}$ , that were assigned into the stretching vibration of u(OH) groups, these bands were absent in the spectra of the prepared complexes, which indicated deprotonation and involvement of the enol oxygen in chelation [19].

Strong band at 1591  $\text{cm}^{-1}$  in the ligand spectrum ascribed to azomethine group, suffered a great change to lower frequency was observed on complexation with metal ion [20]. The spectrum of (Para) show band at 3325  $\text{cm}^{-1}$  due to  $\nu(\text{OH})$ , this band also disappear in the all spectra of complexes lead

to coordinated from this group at coordination [21]. The bands at 3208 and 1655  $\text{cm}^{-1}$  in the (Para) spectrum due to secondary amine and  $\nu(\text{C}=\text{O})$  stretching vibration, since no significant change in these bands were noticed, the possibility that coordination occurs via the donating atoms in these groups were excluded in the spectra of the prepared complexes [22-23].

The presence of water in the spectra of all complexes were suggested by the very broad absorption bands around 3325 and 3327  $\text{cm}^{-1}$  and show peaks at (715-721)  $\text{cm}^{-1}$  because stretching, rocking as well wagging modes from coordination water molecules [24]. Several new bands weak density observed at the regions about (509-528)  $\text{cm}^{-1}$  as well (455-469)  $\text{cm}^{-1}$  may be attributed to  $\nu(\text{M}-\text{N})$  and  $\nu(\text{M}-\text{O})$  vibration respectively [25-26].



Table 2: FT-IR spectra data for ligands as well metal (II) complexes (cm<sup>-1</sup>)

Compounds	$\nu$ (OH)	$\nu$ (H <sub>2</sub> O) + $\nu$ (N-H)	$\nu$ (C=O) + $\nu$ (C=N)	$\delta$ (H <sub>2</sub> O)	$\nu$ (M-N)	$\nu$ (M-O)
Schiff base(L)	3375 br. 3311 br.	- -	- 1591 sh.	-	-	-
(Para)	3325 br.	- 3208 br.	- 1655 sh..	-	-	-
Na[Mn(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3325 br. 3207 br.	1655 sh. 1562 sh.	721 w.	519 w.	469 w.
Na[Co(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3327 br. 3203 br..	1658 sh. 1560 sh.	715 w.	526 w.	461 w.
Na[Ni(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3327 br. 3207 br.	1658 sh. 1560 sh.	715 w.	528 w.	455 w.
Na[Cu(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3327 br. 3207 br.	1653 sh. 1562 sh.	719 w.	513 w.	467 w.
Na[Zn(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3325 br. 3207 br.	1655 sh. 1562 sh..	717 w.	513 w.	465 w.
Na[Cd(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	-	3325 br. 3207 br.	1657 sh. 1560 sh.	717 w.	509 w.	461 w.

br = broad, sh=sharp, w=weak

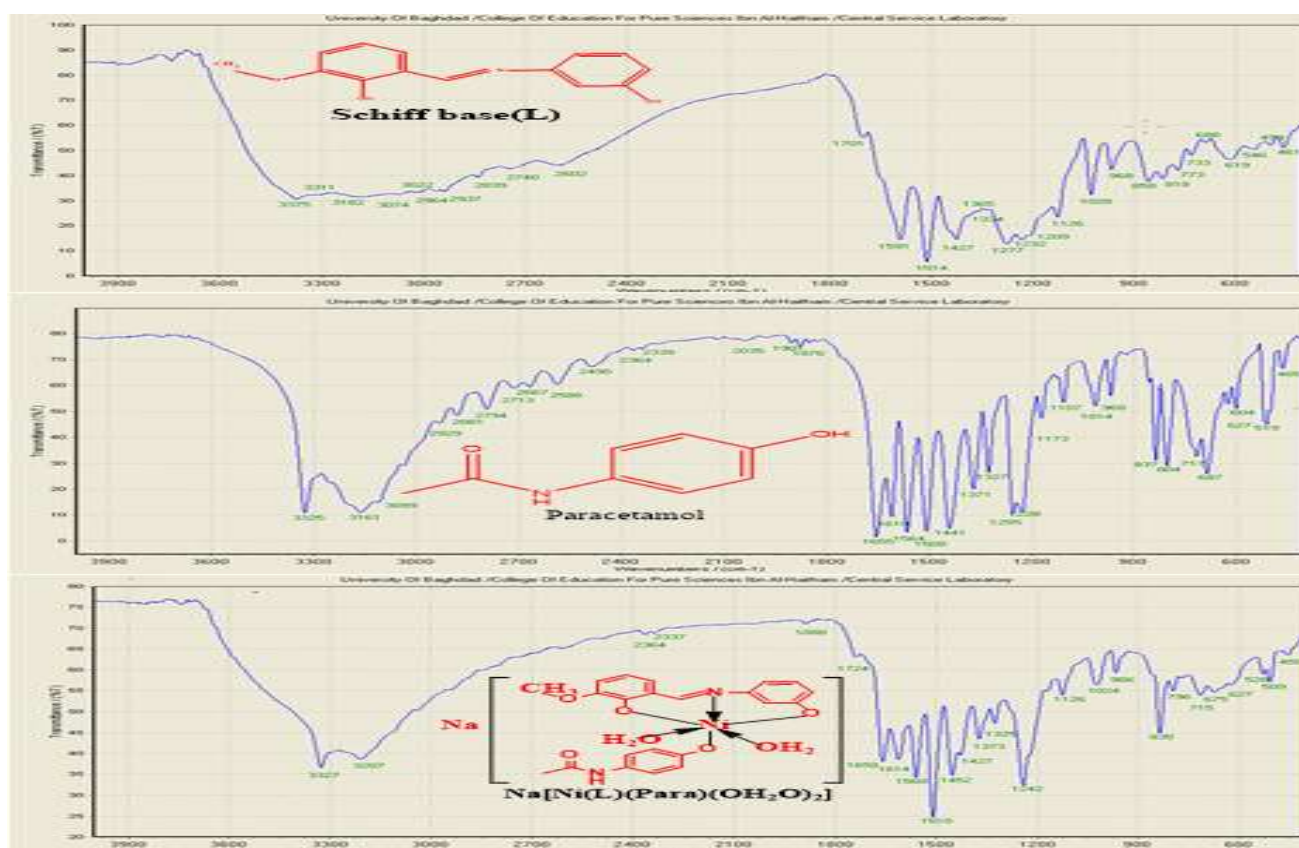


Fig 3: Infrared spectrum for Schiff base (L), (Para) as wellnickel (II) complex

## UV-Vis Spectra

The UV-Vis spectra of the ligands and metal(II) complexes dissolved in DMSO (10<sup>-3</sup> M) have been measured as well the datum obtained are included at Table(3).UV-Vis spectra for Schiff base(L) exhibit three high intense absorption peaks at 271, 345and 375nm which were assigned to ( $\pi$ - $\pi^*$ ) and ( $n$ - $\pi^*$ ) electronic transition respectively[27].

It can see that free (Para) has two distinct absorption bands. The first one at 270 nm may be attributed into ( $\pi$ - $\pi^*$ ) transition

from heterocyclic moiety and benzene ring. The second band observed at 348 nm was attributed to ( $n$ - $\pi^*$ ) electronic transition [21]. The electronic spectrum from manganese (II) complex exhibited five peaks, the first and second peaks at 270 and 348 nm were assigned to ligand field (L.F), other three peaks at 518,715 and 751 nm due to electronic transition type  ${}^6A_{1g} \rightarrow {}^4A_{1g}$ ,  ${}^4E_g(4G)$ ,  ${}^6A_{1g} \rightarrow {}^4T_{2g}(4G)$  and  ${}^6A_{1g} \rightarrow {}^4T_{1g}(4G)$  respectively [28].The manganese (II)- complex shows  $\mu_{eff}$  values 5.8 B.M. conforming to five unpaired electrons, which suggests a high spin to octahedral stereochemistry[29].The spectrum

of cobalt (II) complex appears three peaks at 270,322 and 348 nm due to ligand field, other three peaks at 521,714 and 753 nm which were assigned to electronic transition type  $^4T_{1g}(F) \rightarrow ^4T_{1g}(P)$ ,  $^4T_{1g}(F) \rightarrow ^4A_{2g}(F)$  and  $^4T_{1g}(F) \rightarrow ^4T_{2g}(F)$  respectively[30]. The magnetic moment value (4.87) B.M. of cobalt (II) complex are typical for proposes a high spin octahedral geometry [31].

The electronic spectrum of nickel (II) complex exhibited two absorption peaks at 271 and 346 nm due to ligand field. Other three peaks at 519, 748 and 864 nm were assigned to electronic transition type  $^3A_{2g} \rightarrow ^3T_{1g}(P)$ ,  $^3A_{2g} \rightarrow ^3T_{1g}(F)$  and  $^3A_{2g} \rightarrow ^3T_{2g}(F)$  respectively. These transitions are typical of octahedral geometry, a magnetic moment of 2.48 B.M. is usually observed for nickel (II) complex [32].

The electronic spectrum of copper (II) complex appears three peaks, the first and second at 271 and 364 nm due into ligand field, while third peak in 676 nm was described to electronic transition type  $^2E_g \rightarrow ^2T_{2g}$ . The magnetic moment of this complex was found at 1.76 B.M. which was very close to the octahedral environment [33].

Electronic spectra from zinc(II) and cadmium(II) complexes do show charge transfer, as well the magnetic susceptibility shows which three complexes have diamagnetic moments with  $d^{10}$  ions because d-d transition are not probable hence electronic spectra did not give any prolific information, at fact this result is a good agreement for former work from octahedral geometry[34].

**Table 3: Electronic spectral, magnetic susceptibility as well as conductance mensurations datum**

Compounds	( $\lambda$ nm)	ABS	Wave number)	$\epsilon_{\max}$ ( $L \cdot mol^{-1} \cdot cm^{-1}$ )	$\Lambda_m(S \cdot cm^2 \cdot mol^{-1})$ In DMSO	$\mu_{eff}$ (B.M)
Schiff base(L)	271	1.189	36900	1189	-	-
	345	1.917	28985	1917	-	-
	375	0.987	26666	987	-	-
(Para)	270	1.007	37037	1007	-	-
	348	0.148	28738	148	-	-
Na[Mn(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	270	0.951	37037	951	55.5	5.8
	348	0.810	28735	810		
	518	0.204	19305	204		
	715	0.076	13986	76		
	751	0.073	13315	73		
Na[Co(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	270	1.085	37037	1085	45.4	4.87
	322	0.724	31055	724		
	348	0.856	28735	856		
	521	0.275	19193	275		
	714	0.058	13986	58		
	753	0.054	13280	54		
Na[Ni(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	271	1.238	36900	1238	41	2.48
	346	1.941	28901	1941		
	519	0.261	19267	261		
	748	0.076	13368	76		
	864	0.068	11574	68		
Na[Cu(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	271	1.206	36900	1206	51.5	1.76
	364	1.217	27472	1217		
	676	0.164	14792	164		
Na[Zn(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	271	1.206	36900	1206	44.6	Dia
	360	1.217	27472	1217		
Na[Cd(L)(Para)(H <sub>2</sub> O) <sub>2</sub> ]	271	1.269	26900	1269	36.1	Dia
	344	2.342	29069	2342		
	351	1.223	27777	1223		

## Antibacterial Activity

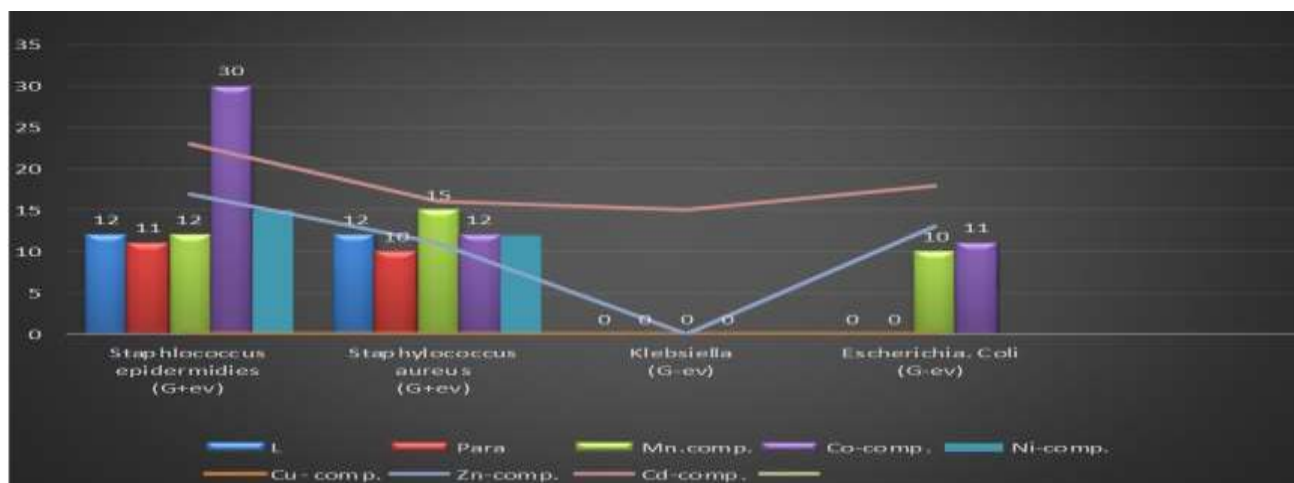
The conclusion gained into antibacterial experience models studies through agar good -diffusion bioassay detect biological efficacy to the Schiff base (L), (Para) and metal(II) complexes after 24h in Table (4) and Fig.(4). In situation of cobalt (II) complex the antibacterial activity at *Staphylococcus epidermidies* is higher activity of (30 mm) like contrast of the rest, but cadmium (II) complex has a higher activity of (17 mm) with *Staphylococcus aureus* as compared to (L),

(Para) and the remaining complexes. In case of the cadmium (II) complex shows higher inhibition area of (15 mm) with *Klebsiella*, Compared to the (L), (Para) and the remaining complexes that did not give any effect, as well as cadmium (II) complex shows higher inhibition area of (15 mm) with *Escherichia. Coli* of the rest. Cobalt (II) and cadmium (II) complexes that increased the value of inhibition of growth than were during the 24 hours. The consequences of biological checking reference that in several complexes are more effective than free

ligands, increased activity of the complexes can exist, illustrated at the fundamental in chelation theory [35].

**Table 4: Displayed the Inhibition Circle Dimeter at Millimeter into the Bacteria after 24 h**

Compounds	<i>Staphylococcus epidermidies</i> (G+ ev)	<i>Staphylococcus aureus</i> (G+ev)	<i>Klebsiella</i> (G-ev)	<i>Escherichia. Coli</i> (G-ev)
(L)	12	12	-	-
(Para)	11	10	-	-
Mn.comp.	12	15	-	10
Co-comp.	30	12	-	11
Ni-comp.	15	12	-	-
Cu- comp.	-	-	-	-
Zn.comp.	17	11	-	13
Cd.comp.	-	16	15	18

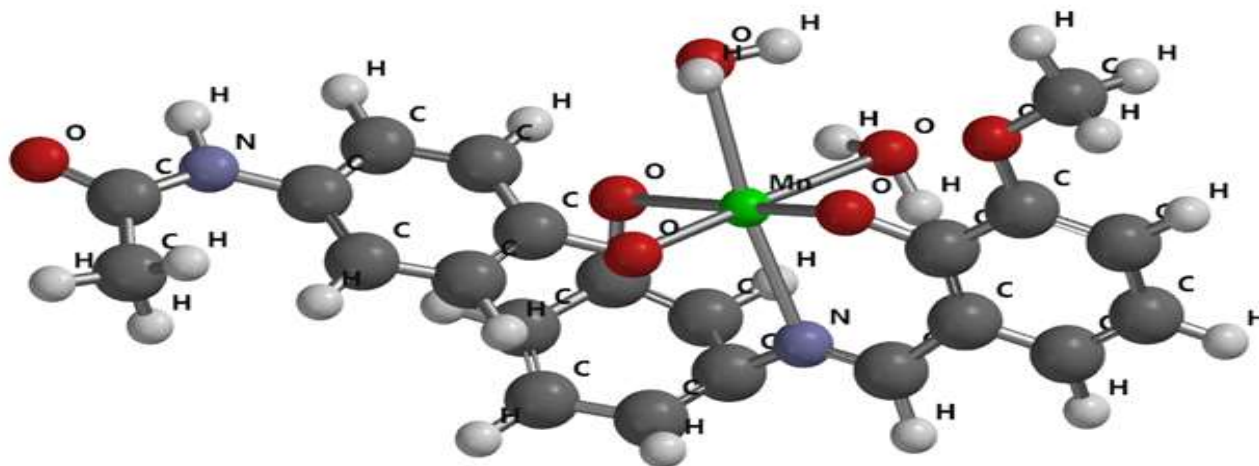


**Fig. 4:** Shows the antibacterial efficacy for Schiff base (L), (Para) as well metal (II) complexes appear the inhibition zones against pathogenic bacteria (*Staphylococcus epidermidies*, *Staphylococcus aureus*, *Klebsiella* and *Escherichia. Coli*)

## Conclusion

In this paper synthesis and spectroscopic characterization of Schiff base (L), (Para) and metal(II) complexes of (Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II)) were presented. The type of mixed ligand complexes were also synthesized, spectroscopic and structural characterized by using various physico-chemical of Schiff base (L) as well (Para) for M (II) ions [where M (II) = manganese (II), cobalt (II), nickel (II), copper (II), zinc (II) and cadmium (II)]. Schiff base (L) has been

derived from (o-vanilline and m-aminophenol) as a tridentate ligand and (Para) as a monodentate ligand. From spectral studies the geometry from mixed ligand complexes was explored as well found to have octahedral environment for mononuclear nature. 3D-structure is proposed for the manganese (II) complex according to Scheme (5). Biological activities are shown towards more efficient mixed ligand complexes than Schiff base (L) and (Para).



**Scheme 5: Proposed 3D-structure of the manganese (II) complex**



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