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Synthesis, characterization and evaluation biological activity of new Schiff base compounds and their complexes with some transition elements

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Abstract:

The novel Schiff base ligand **L_1** (Z)-N'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide and **L_2** (Z)-N'-(2-hydroxy-3-methoxybenzylidene)nicotinohydrazide obtained by the condensation of isonicotinic hydrazide and benzahydrazide with o-Vanilin (2-hydroxy-3-methoxybenzaldehyde) and its Co(II), Ni(II) and Cd(II) complexes were synthesized and characterized by elemental analysis and various physico-chemical techniques like, FT-IR, ^{1}H-NMR, Electrospray Ionisation mass(ESI-MS), UV-visible and molar conductance. The ligand involving oxygen atom of amide carbonyl, azomethine nitrogen and oxygen of hydroxyl via deprotonation. Spectral analysis indicates octahedral geometry for all the complexes. has 2:1 stoichiometry ratio of the type[$M(L_2)_2Cl$]. Schiff bases ligands and their complexes has been studied the biological activity where the schiff bases L_1, L_2 and their complexes showed lower efficiency than the standard inhibitor (Cipro.) trend three types of bacteria *E-coli, salmonella spp.* and *staph. aureus*, the prepared complexes appeared high effective than the effectiveness of the Schiff bases itself.

Keywords: Schiff base, o-Viniline, Elements analyses, FT-IR, ESI mass, ^{1}H NMR.

تحضير وتشخيص وتقيم الفعالية البيولوجية لبعض مركبات قواعد شف الجديدة ومعقداتها مع بعض العناصر الانتقالية

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قسم الكيمياء – كلية العلوم – جامعة ذي قار

الخلاصة

تم تحضير ليكاندات قواعد شف الجديدة (**L_1**) (Z)-N'-(2-hydroxy-3-methoxybenzylidene)isonicotinohydrazide و(**L_2**) (Z)-N'-(2-hydroxy-3-methoxybenzylidene)nicotinohydrazide من تكاثف isonicotinic hydrazide و benzahydrazide مع *o*-Vanilin (2-hydroxy-3-methoxybenzaldehyde) ومن ثم تم تعقيدهم مع Co(II) و Ni(II)و Cd(II) شخصت المعقدات المحضرة باستخدام التحليل الدقيق للعناصر CHNS و FT-IR و ^{1}H-NMR و ESI mass و UV-visible و Molar conductance. تشير التحاليل الطيفية الى ان جميع المعقدات المحضرة ثمانية السطوح. درست الفعالية البيولوجية لقواعد شف المحضرة ومعقداتها حيث أظهرت قاعدتا شف L_1 و L_2 والمعقدات المحضرة منهما فعالية بيولوجية اقل من المثبط القياسي (Cipro.) اتجاه الأنواع الثلاثة من البكتريا المدروسة *E-coli* و *salmonella spp* و *staph. aureus*، أظهرت المعقدات المحضرة فعالية بيولوجية اعلى من قواعد شف المحضرة منها تلك المعقدات.

الكلمات المفتاحية: قاعدة شف، *o*-Viniline، CHNS، FT-IR، FT-IR، ESI mass، Molar conductance.

1. Introduction

Schiff bases are condensation products of primary amines and carbonyl compounds and they were discovered by a German chemist Nobel Prize Winner, Hugo Schiff in 1864 (Salimon et al. 2011).

Schiff bases are of the general formula $R^1R^2C=NR^3$, where R^3 is aryl or alkyl that makes the Schiff base a stable imine. A Schiff base is derived from aniline, where R^3 is a phenyl or substituted phenyl, can be called on anil (Sahu et al. 2012). Schiff bases and their compounds are important intermediates for the synthesis of various bioactive compounds (Yang et al. 2006). A large number of different Schiff base ligands have been used as cation carriers in potentiometric sensors (Upadhyay et al. 2008). Studies in terms of catalytic properties of catalytic activity in hydrogenation of olefins and transfer of an amino (Jirjees et al. 2016). One of the more interesting applications of these compounds is the possibility to use them as effective corrosion inhibitors. This phenomenon is the spontaneous formation of a monolayer on the surface to be protected (Jirjees et al. 2015). Schiff base complexes have a broad range of biological properties antitumor, antiviral, antifungal, antibacterial (Radecka-Paryzek et al. 2007). They are also used in the treatment for diabetes and AIDS. As biological models, they help in understanding the structure of biomolecules and biological processes occurring in living organisms (Brodowska et al. 2014), and there are many studies on the effectiveness of the Schiff base complexes of the types of bacteria and fungi (Gwaram, N.S et al. 2012)، (Jana, G. K. et al. 2012). A large number of Schiff base complexes of various transition metal ions with a variety of donor atoms have been reported (Uddin 2014).

Synthesis, spectroscopic and electrochemical properties of some complexes of a new symmetric bidentate Schiff base ligands of ((Z)-N'-(2-hydroxy-3-methoxybenzylidene) isonicotinohydrazide) benzohydrazide (L_1) and (Z)-N'-(2-hydroxy-3-methoxybenzylidene) nicotinohydrazide (L_2) with a general formula of ML_2Cl_2 (M= Ni(II), Cd(II) and Co(II)) are reported.

2. Methods and Materials

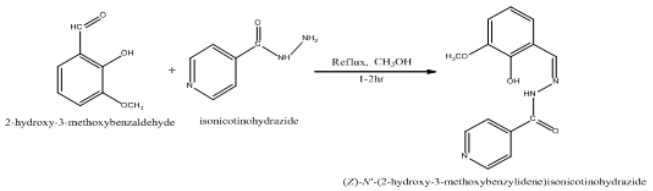
2.1. Chemicals

Chemicals are purchased from BDH, and used without further purifications. The purity of the synthesized formazan derivative was checked by thin layer chromatography (TLC). FT-IR spectra are recorded using BRUKER FT-IR affinity spectrophotometer in the range of 3800-200 cm-1. Melting points are measured with an electrothermalsturat apparatus, model SMP30. 1H-NMR spectra are recorded on a BRUKER Ultra shield spectrophotometer 400 MHz using DMSO-d6, chemical shift in ppm relative to internal Me4Si. The elemental analysis (CHN) data was recorded using Leco (Model: 932) element analyzer. Mass spectra are recorded with HPLC-LCQ Fleet/Thermo Scientific Mass spectrophotometer (ESI).

2.2. Preparation of Schiff bases

2.2.1. Synthesis of Schiff (Z)-N'-(2-hydroxy-3-methoxybenzylidene)isonicotino hydrazide (L_1)

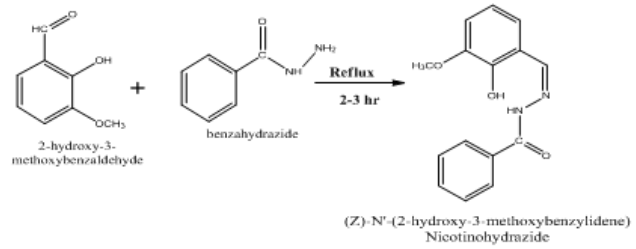
The compound **(L_1)** was prepared by condensation of isonicotinohydrazide (1.37g, 0.01mole) dissolved in 20ml methanol was added to 2-hydroxy-3-methoxybenzaldehyde (1.52g, 0.01) added (3-4) drops of glacial acetic acid the reaction mixture was refluxed for (1-2) hrs. The progress of the reaction was followed by TLC using hexane: ethyl acetate 1:3 as eluent. After completion. It was filtered and recrystallized by using absolute ethanol (Mohammed et al. 2013; Abdullahi O. 2014). Elemental analysis for **(L_1)** $C_{14}H_{13}N_3O_3$ Calc. (%): C, 61.99; H, 4.83; N, 15.49. Found (%): C, 61.83; 4.96; N, 51.53.



Scheme (1) Formation of Schiff Base Ligand L_1

2.2.2 Synthesis of Schiff base (Z)-N'-(2-hydroxy-3-methoxybenzylidene) nicotinohydrazide (L_2)

The compound **(L_2)** was prepared by condensation of benzahydrazide (1.36g, 0.01) dissolved in 20ml methanol was added to 2-hydroxy-3-methoxybenzaldehyde (1.52g, 0.01) added (3-4) drops of glacial acetic acid the reaction mixture was refluxed for (2-3) hrs. The progress of the reaction was followed by TLC using hexane: ethyl acetate 1:3 as eluent (Mohammed et al. 2013; Abdullahi O. 2014). After completion. It was filtered and recrystallized from ethanol. Elemental analysis for **(L_2) $C_{15}H_{14}N_2O$** Calc. (%): C, 66.66; H, 5.22; N, 10.36 Found (%): C, 65.99; H, 5.32; N, 9.89.

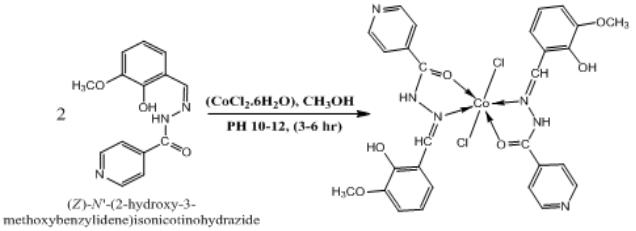


Scheme (2) Formation of Schiff Base Ligand L_2.

2.3. Synthesis of the Schiff base complexes

2.3.1. Cobalt complex synthesis (1b)

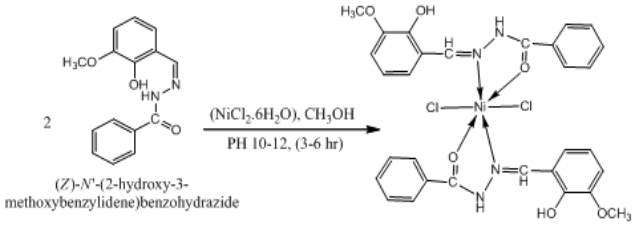
Schiff base (0.27g, 0.001mole) of the ligand **(L_1)** dissolved in (20 ml) of hot methanol with (0.12g, 0.0005mole) each of the salt {$CoCl_2.6H_2O$} dissolved in (25 ml) of hot methanol or chloroform, then mixture reflux for (3-6hr). It has been monitoring the reaction using thin layer chromatography (TLC). The solid product separated was filtered and washed with hot ethanol (NITIKA 2014; Jirjees et al. 2015).



Scheme (3) Metal complexes fromthe prepared ligand **L_1**.

2.3.2. Nickel **complex synthesis (2b)**

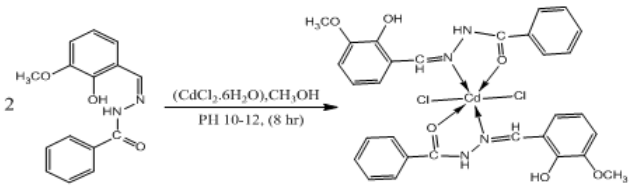
Schiff base (0.27g, 0.001mole) of the ligand **(L_2)** dissolved in (20 ml) of hot methanol with (0.12g, 0.0005mole) of each of the salts {$NiCl_2.6H_2O$} dissolved in (25 ml) of hot methanol or chloroform, then mixture reflux for (2-4hr). It has been monitoring the reaction using thin layer chromatography (TLC). The solid product separated was filtered and washed with hot ethanol (NITIKA 2014).



Scheme (4) Metal complexes from the prepared ligand L_2.

2.3.3. Cadmium complex synthesis (3b)

Schiff base (0.27g, 0.001mole) of the ligand (L_2) dissolved in (20 ml) of hot methanol with (0.12g, 0.0005mole) of each of the salts {$CdCl_2.6H_2O$} dissolved in (25 ml) of hot methanol or chloroform, then mixture reflux for (8hr).It has been monitoring the reaction using thin layer chromatography (TLC).The solid product separated was filtered and washed with hot ethanol (NITIKA 2014).



Scheme (5) Metal complexes from the prepared ligand L_2.

Table (1) Some chemical and physical properties of the Schiff base ligands and their complexes.

Compound	Chemical formula	M.wt	Melting point (°C)	Color	Yield (%)
L_1	$C_{14}H_{13}N_3O_3$	271.27	189-191	White yellowish	80.2
1b	$Co(L_1)_2Cl_2$	672	206-208	Green	88
L_2	$C_{13}H_{14}N_2O$	270.28	190-192	White	79.5
2b	$Ni(L_1)_2Cl_2$	670	210-212	Green yellow	86.5
3b	$Cd(L_1)_2Cl_2$	724	148-150	Yellow	84.2

3. Results and discussion

The analytical data of the Ni, Co, Cd complexes of Schiff bases indicate 1:2 metal to ligand stoichiometry. The Prepared complexes are stable in room temperature, don't soluble in EtOH and MeOH, but soluble in DMSO. Attempts to propose the structure of the isolated complexes come from full investigation using the following studies.

3.1. UV-Visible spectra

Diagnosed ligands Schiff bases prepared **L1, L2** and their complexes by ultraviolet-visible radiation and the existence of ethanol as a solvent and a blank as shown in table (3-3) It was clear that the UV-Visible spectrum of ligand **(L1)** showed absorption bands in 372nm back to the first transmission $n\rightarrow\pi^*$ and 337nm back to transmission $\pi\rightarrow\pi^*$ while showed Ligand **(L2)** absorption bands in 383 nm back for the first transmission $n\rightarrow\pi^*$ and 352, 340nm back for transmission $\pi\rightarrow\pi^*$ (Subhi A Al-Jibori et al. 2015).
Spectrum UV-visible showed peaks absorption of prepared complexes ranging 384-416 nm and these peaks are attributable to the charge transfer of type

M→L between the metal and ligand which confirms formation the complexes.

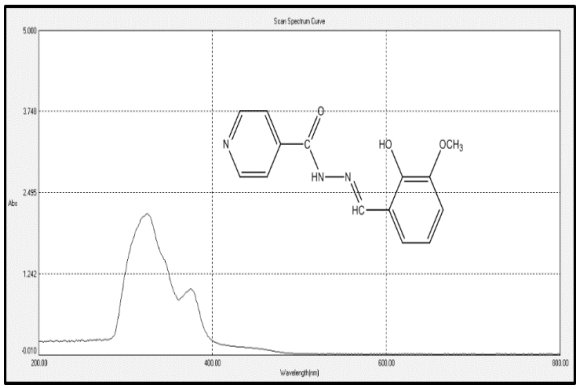


Figure (1) UV-Visible spectra of (L_1).

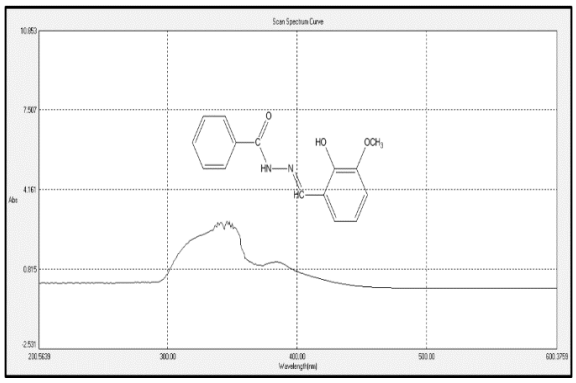


Figure (2) UV-Visible spectra of (L_2).

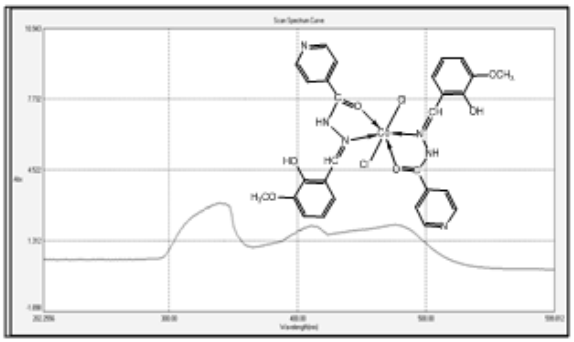


Figure (3) UV-Visible spectra of (1b).

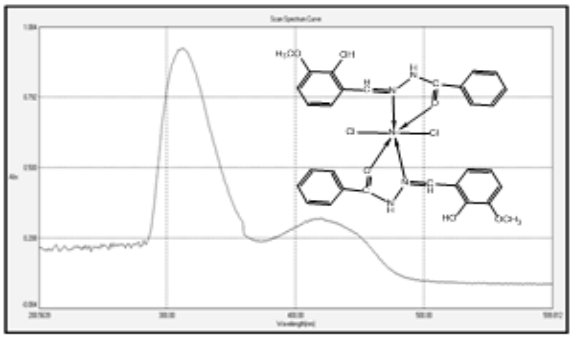


Figure (4) UV-Visible spectra of (2b).

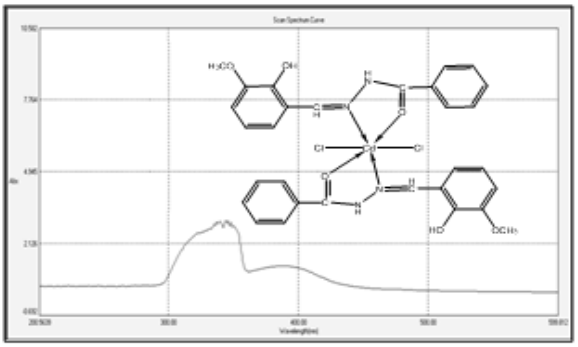


Figure (5) UV-Visible spectra of (3b).

3.2. IR Spectra

The Figures (6), (7) which representative the IR spectra of Schiff bases compounds L_1 and L_2 represent the absorption sites for prepared Schiff bases ligands because these Schiff bases have the characteristic absorbance bands in the infrared spectrum in the spectral range (1602-1605cm^{-1}) attributable swing stretch of the bond (C=N) (M.J. Mahmoud 2013) also owns bands at the range (3202-3548cm^{-1}) back to the swing stretch of the bond (O-H), the other hand, these Schiff bases have a strong bands within the range (1531-1577cm^{-1}) back to swing stretch of the bond (C=C) of the aromatic rings (Rajavel 2013). As well as all prepared Schiff bases containing the bands at the range (3006-3079 cm^{-1}) (Muter et al. 2011) back to the swing stretch for a group (C-H) aromatic, and contains a band at (2993-2829cm^{-1}) back to the swing stretch group (C-H) aliphatic (H. Sie-Tiong et al. 2008).

Table (2): FT-IR spectra of the Schiff bases

Symbol	Structural Formula	O-H	N-H	C-H (Ar.)	C-H (Alp.)	C=O	C=N	C=C (Ar.)
L_1	$C_{14}H_{13}N_3O_3$	3373s	3207s	3074-3006w	2940-2863m	1656s	1605s	1572m
L_2	$C_{15}H_{14}N_2O_3$	3473b	3186m	3079m	2993-2858m	1660s	1602w	1574-1531s

b: broad, s: strong, m: medium, w: weak

The Figures (8) to (10) describes the most important absorption prepared Schiff base complexes bands sites as this spectrum characterized by obtaining displacement in the spectral range for all the bands that were prominent in the spectrums of the Schiff bases that synthesis from them these complexes , it has shifted to a lesser range for swing stretch for a group (C=N) from (1602cm^{-1}) to (1560, 1607cm^{-1}) in the prepared complexes from the first ligand **L2**, and got shifted from (1605cm^{-1}) to (1604cm^{-1}) in the prepared complex from the second

ligand **L1**, and a group (C=O) (M. J. Mahmoud 2013) shifted from(1660cm^{-1}) to (1602, 1652 cm^{-1}) in the prepared complexes from the first ligand **L2**, and got shifted from (1656 cm^{-1}) to (1604 cm^{-1}) in the prepared complex from the second ligand **L1**.

Complexes spectrum identify appearance of new bands were not present in the spectrum of ligand back to stretching vibration **(M-N)** appears at (674, 691 and 684 cm^{-1}) in the prepared complexes 1b, 2b and 3b respectively, **(M-O)** appears at (444, 444 and 489 cm^{-1}) in the prepared complexes 1b, 2b and 3b respectively and **(M-Cl)** appears at (420, 410 and 0545 cm^{-1}) in the prepared complexes 1b, 2b and 3b respectively, which confirms obtain complexity process.

Table (3): FT-IR spectra of the Schiff base

Symbol	O-H	N-H	C-H (Ar.)	C-H (Alp.)	C=O	C=N	C=C (Ar.)	M-N	M-O	M-Cl
1b	3446b	3208m	3068m	2941-2840w	1668w	1604s	1573-1535m	674w	444w	420w
2b	3406b	3194m	3061-3027w	2953-2837m	1602s	1560s	1469s	691w	444m	410w
3b	3573s	3384-3217b	3089-3068m	2971-2841s	1652s	1607m	1471-1539m	684w	489m	405w

b: broad, s: strong, m: medium, w: weak

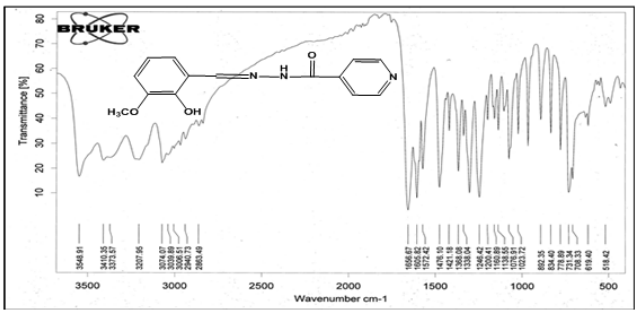


Figure (6) IR Spectra of (L_1)

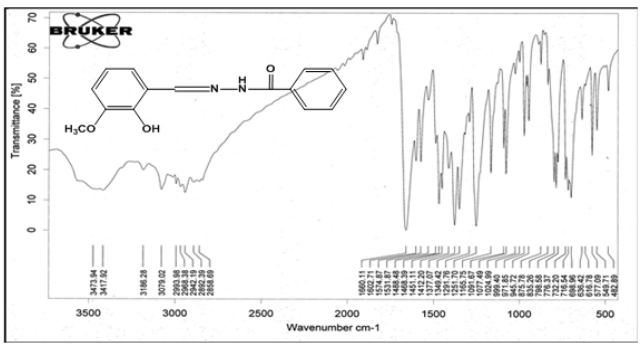


Figure (7) IR Spectra of (L2).

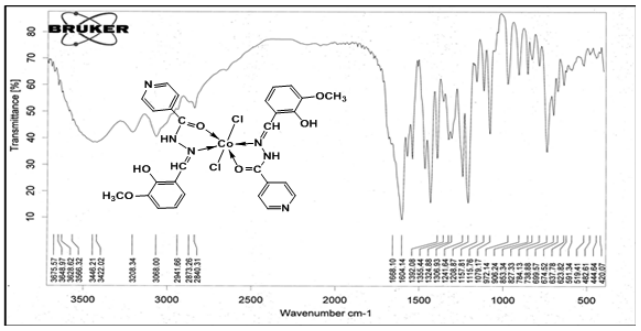


Figure (8) IR Spectra of (1b).

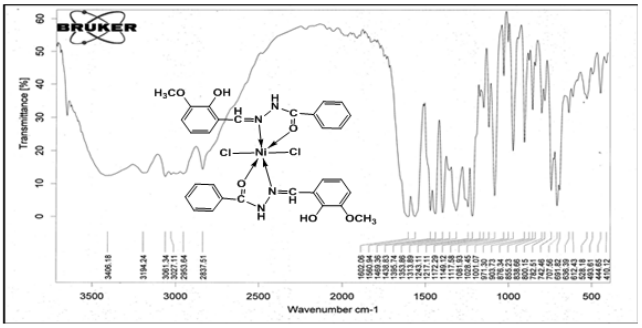


Figure (9) IR Spectra of (2b**).**

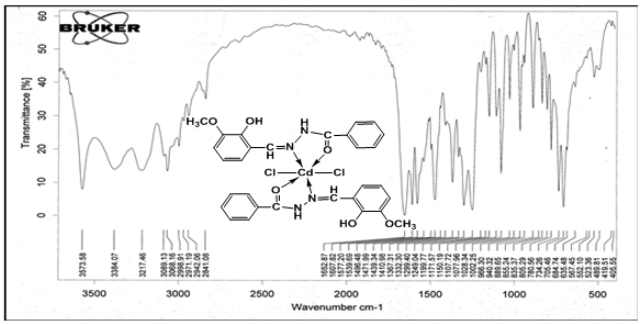


Figure (10) IR Spectra of (3b**).**

3.3. ^{1}H-NMR Spectra

The spectra of proton Nuclear Magnetic Resonance ^{1}H-NMR of the prepared Schiff bases ligands **(L_1, L_2)** shown in Figures (11), (12) characterize ^{1}H-NMR spectrum appearance of multiplet signal at chemical shift (δ) (6.9 - 8.7 ppm) due to the aromatic ring protons (Mohamed et al. 2009) (Ar-H), and the appearance of a singlet signal at (9.1, 9.8 ppm) due to the proton -NH group of **L_1** and **L_2** respectively, the appearance of a singlet signal at (10.8, 11.1 ppm) due to the proton (-OH) group phenolic of **L_1** and **L_2** respectively.

Characterized protons of aromatic rings substitutes groups -OH and -O-CH3 chemical shift least expected and this goes back to mesomeric effect

the motivation for these two groups making protons ring with high electronic intensity therefore appear less chemical shift of the rings is not substituting these groups (Ebraheem Abdu Musad 2010).

The ligands L1 and L2 characterized by the appearance of a singlet signal at (3.4 ppm) due to -O-CH3 group, and also showed the ligand **L_1** multiplet signal at the range (6.8 – 6.9 ppm) due to the pyridine ring protons. In the compounds **L_1** and **L_2** singlet signal at appeared (8.8, 8.7 ppm) due to the azomethine proton group (-CH=N) of **L_2** and **L_2** respectively. All spectra showed a signal at δ 2.5 ppm for the DMSO-d6 solvent (A. Rana et al. 2011).

Table (4) ^{1}H – NMR spectra data of prepared

Symbol	δ (ppm)					
	O-CH$_3$	Ar-OH	-NH	-CH (Aromatic)	-CH (pyridine ring)	-CH=N
L_1	3.4 s	10.8 s	9.1 s	7.0-8.3 m	6.8-6.9 m	8.8 s
L_2	3.4 s	11.1 s	9.8 s	6.9-8.7 m	——	8.7 s

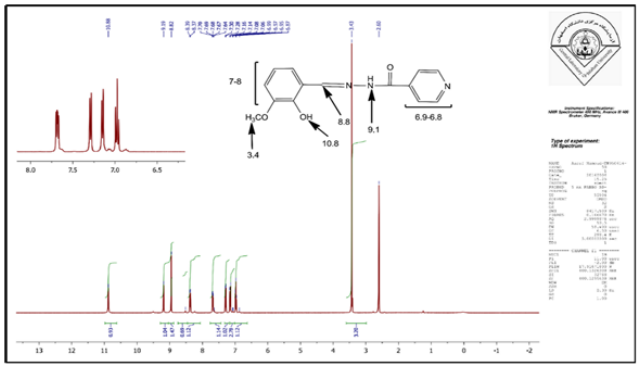


Figure (11) ^{1}H-NMR Spectra of (L_1).

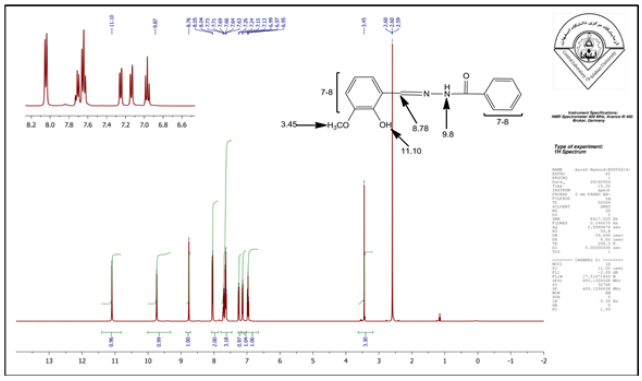


Figure (12) ^{1}H-NMR Spectra of (L_2).

3.4. Mass spectra

Characterize mass spectra of the formazans L_1, $L_2$and their complexes which appearance of molecular ion ($M^{+\cdot}$) at (271, 270 m/z) respectively, and the fragmentation of (L_1) showed the peaks in 256, 228, 200, 174, 122 and 96 m/z due to **$C_{13}H_{10}N_3O_3^+$, $C_{12}H_{10}N_3O_2^+$, $C_{11}H_{10}N_3O^+$, $C_9H_8N_3O^+$, $C_5H_4N_3O^+$** and **$C_3H_2N_3O^+$** respectively, and the fragmentation of (L_2) showed the peaks in 269, 241, 147, 120, 105 and 95 m/z due to **$C_{15}H_{13}N_2O_3^+$, $C_{14}H_{13}N_2O_2^+$, $C_8H_7N_2O^+$, $C_7H_7NO^+$, $C_7H_5O^+$, $C_6H_7O^+$ a**nd **$C_5H_5^+$** respectively. The following figures show the mass spectra of the formazans and their complexes.

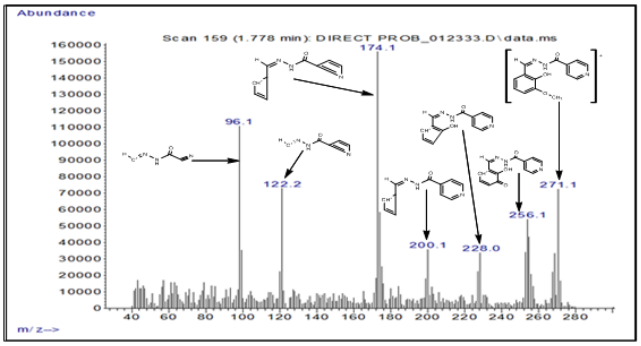


Figure (13) Mass spectra of (L_1).

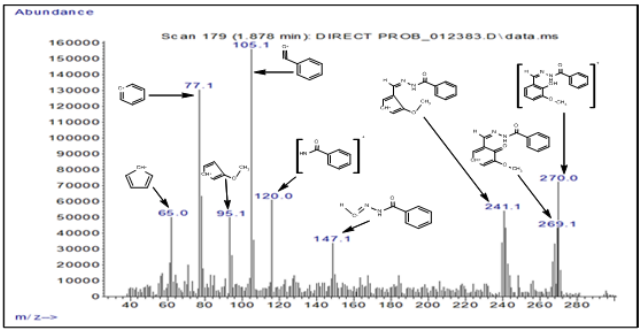


Figure (14) Mass spectra of (L_2).

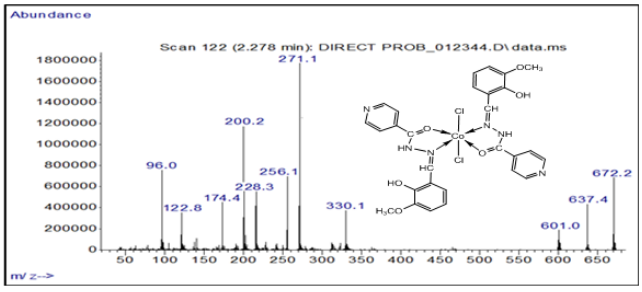


Figure (15) Mass spectra of the complex (1b).

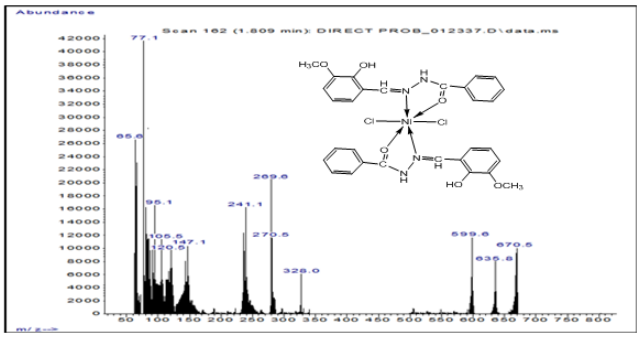


Figure (16) Mass spectra of the complex (2b).

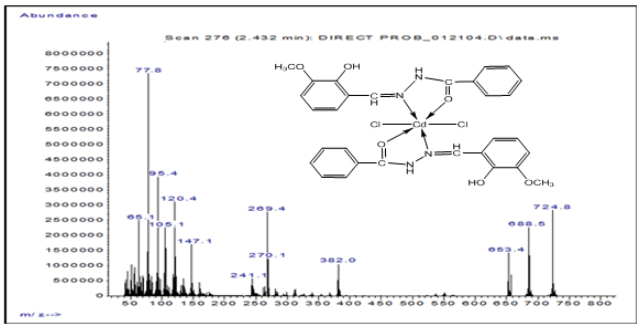


Figure (17) Mass spectra of the complex (3b).

3.5. Molar Electrical Conductivity

The electrical conductivity is regarded as one of the important and simple means for knowing the ionic formulas of the compounds (FELTHAM et al. 1964).Molar conductivity measured for solid complexes solutions of ions Ni (II) and Co (II) and Cd (II) with $L_1$and L_2 formazans concentration of 10^{-3} M dissolved in Dimethylsulfoxide (DMSO) each separately at a laboratory temperature, has been used distilled water as a reference. The results are included in Tables (2) was found from the conductivity values of the complexes behave neutral compounds (non-electrolytic) the lack of any adjective-ionic, because of the lack of chloride ions out of the coordination sphere as counter ions of the central ion. The obtained results were appropriate with the molecular formulas and stereochemistry proposed of the prepared complexes.

Table (5) Molar electrical conductivity of the complex.

Complexes	Λ m (S. cm^2 .mole^{-1}) In (DMSO)	Electrolyte Type
$[Co(L_1)_2Cl]$	15	Non Electrolyte
$[Ni(L_2)_2Cl_2]$	11	Non Electrolyte
$[Co(L_2)_2Cl_2]$	13	Non Electrolyte

3.6. Effectiveness Biological

Study in this research the effectiveness of formazans (L_1, L_2) and their complexes against the three types of bacteria two sensitive topics and positive for the Gram stain *Salmonella spp* and *Staphylococcus aureus* and third sensitive and negative to Gram stain a *Escherichia coli* (J.P. Tierney 2015), 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 cork borer 0.1ml of these solutions in each hole and incubated for 18 hours at a temperature 37˚C then extracted from the incubator and measured diameters of inhibition zones and compared the inhibition of these compounds with the standard inhibitor is Cipro and the same concentrate (Khudir 2013).

The Schiff base ligands L_1, L_2 and their complexes showed lower efficiency than the standard inhibitor trend three types of bacteria *E-coli* and *staph. aureus*. The prepared complexes appeared high effective than the effectiveness of the formazans itself (RAY 1994). The results showed in the table (6) and Figures (18) to (20).

Table (6) Effectiveness biological of studied formazans and their complexes.

Symbol	*Salmonella spp.* Inhibition zone(mm)	*Escherichia coli* Inhibition zone(mm)	*Staphylococcus aureus* Inhibition zone(mm)
L_1 (30)	0+	0+	0-
1b (1)	5+	5+	26++
L_2 (31)	2+	0-	0-
2b (6)	7+	0-	15++
3b (7)	10++	0-	16++
Cipro.	30+++	15++	36++++

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

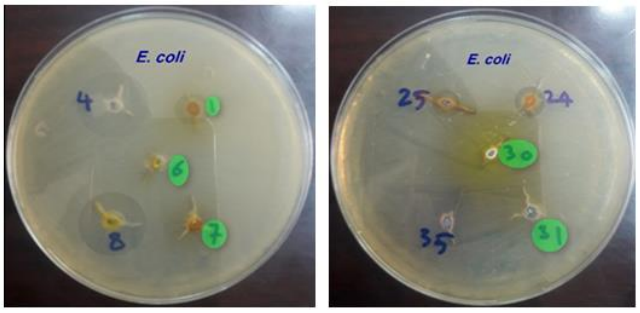


Figure (18) Biological effectiveness of complexes 1,6 and 7 against E. coli on the left and the schiff bases 30 and 31 against E. coli on the right.

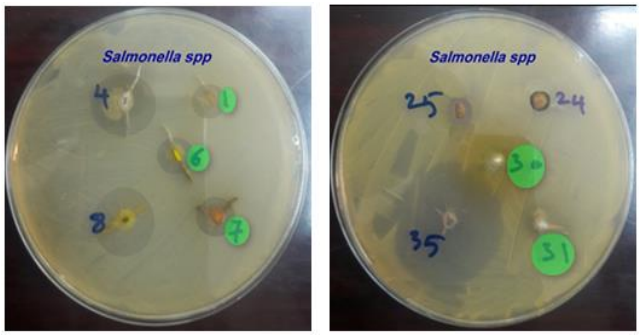


Figure (19) Biological effectiveness of complexes 1,6 and 7 against *salmonella spp.* on the left and the schiff bases 30 and 31 against *salmonella spp.* on the right.

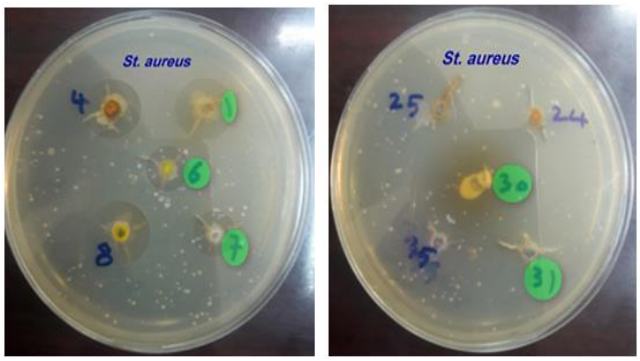


Figure (20) Biological effectiveness of complexes 1,6 and 7 against *St. aureus* on the left and the schiff bases 30 and 31against *St. aureus* on the right.

4. Conclusions

In this work Schiff base ligands forms stable complexes with transition metals such as Nickel (II), Cobalt (II) and Cadmium (II) are given. The ligands and their complexes are characterized using spectral. These analytical and spectral data suggests octahedral geometry.

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