

Synthesis, Characterization and Bioactivity Evaluation for Some Metal Ions Complexes with New Ligand Derived from 2-hydroxy-1-naphthaldehyd

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Abstract

The newly synthesized Schiff base ligand (E)-2-((2-phenylhydrazono)methyl)naphthalen-1-ol (phenyl hydrazine derivative), is allowed to react with each of the next mineral ion: Ni²⁺, Cu²⁺, Zn²⁺ and Cd²⁺ successfully resulting to obtain new metal complexes with different geometric shape. The formation of Schiff base complexes and also the origin Schiff base is indicated using LC-Mass that manifest the obtained molar mass, FT-IR proved the occurrence of coordination through N of azobenzene and O of OH by observing the shifting in azomethines band and appearing of M-N and N-O bands. Moreover, we can also detect by such apparatus, the presence of aquatic water molecule inside the coordination sphere. UV-Vis spectra of all resultants revealed the creation of coordination by noticing the shifting in electronic transitions that happened in ligand at ultra violet region. TGA and DSC measurements for ligand and Ni-complex also prove the complexation and presence of coordinated water molecule inside the coordination sphere. In addition, molar accessibility and FAA results were closer to the counting results. The diagnoses return gave mononuclear complexes, bidentate dental behavior and tetrahedral geometry for all complexes. Depending on the antibiotic property of Schiff base and its complexes, we have tested such property on various types of microorganisms as detailed in the paper.

Keywords: bioactivity, electronic transition, 2-hydroxy-1-naphthaldehyd, Schiff bases, spectroscopic studies.

Introduction

Schiff bases are organic compounds that contain an azomethine group (-CH=N-), are named after the German scientist (Hugo Schiff) who synthesized them for the first time in 1864 by the condensation reaction of aldehydes or ketones with primary amines^{1,2}. The general formula of which is [R₁R₂-C=N-R₃] and the naming of these bases depending upon R₁R₂R₃ groups either aliphatic or

aromatic, and related with the aldehyde, ketone and amine that are derived from which. The Schiff bases complexes involving O, N donating atoms of chelates own an uncommon configuration. In these cases, the presence of donating atoms at complexes could enhance antibiotic reactivity such as (anticancer, antifungal, antibacterial, etc.)³. Schiff bases are considered as (Imines), (Azomethines) and

(Anils)⁴⁻⁸. Generally, the Schiff bases derived from the condensation of aldehydes with primary amines, are referred to as (Aldimines), whereas the compounds that derived from the condensation of ketones with primary amines are referred to as (Ketimines). In the condensation of suitable acid hydrazines with ketones or aldehydes in the presence of suitable solvents, the result of this condensation is called (Hydrazones)^{9, 10}. These compounds are preferred to contain an aryl group attaches to either

Materials and Methods

The starting materials including (NH₂-ph, 2-hydroxy-1-naphthaldehyde, NiCl₂.6H₂O, CuCl₂.2H₂O, ZnCl₂.2H₂O, CdCl₂.2H₂O) were commercially abundant in addition to the employed catalysts including (dimethyl sulfoxide, abs. CH₃CH₂OH and diethyl-ether) that supplied from F-897ewluka, Sigma Aldrich. The employed FTIR apparatus operates in the range 4000-400 cm⁻¹ Shimadzu-3800 model. Electronic spectral inform were accomplished depending on Shimadzu160-meter. LC/MASS incomes are also established by Mass100P_Shimadzu contribution. Pyrolysis diagnosis were carried out depending on perkin_Elmer_pyris Diamond DSC/TG. Proton-NMR was published using Bruker 400-MHz-meter and elemental micro analysis were done on a perkin_Elmer_automatical instruments model 240B.minerals were determined obeying a Shimadzu (A-A) 680G AA_spectrometer. The Cl combination was estimated gravimetrically. Magnetic features were measured using balance magnetic susceptibility model MSR-MKi.

Preparation of the Ligand (E)-2-((2-phenylhydrazono)methyl)naphthalen-1-ol

An ethanolic solution of 2-hydroxy-1-naphthaldehyde (which formed by dissolving (0.5 g, 0.003 mol) of 2-hydroxy-1-naphthaldehyde in 10ml of ethanol with continuous stirring, then adding the hydrobromic acid HBr (about 3 drops as a catalyst) onto the extremely dissolved solution and adding 5 ml of ethanol (for dilution purposes because of its exothermic reaction), finally ,adding 5ml of phenyl hydrazine with continuous stirring (for about 5 min.), a yellow precipitate will be formed immediately, as shown in scheme 1.

the nitrogen of amino group or the carbon of aldehyde or ketone molecule in order to increase their stability and keep them from decomposition or polymerization. This work investigates the synthesis and characterization of a new -Schiff base ligand (E)-2-((2-phenylhydrazono)methyl)naphthalen-1-ol and all complexes were formed and diagnosed by ¹H-NMR, Mass-spectroscopy, IR and UV Vis spectra and TGA. Also, we evaluated its vital reactivity.

Synthesis of Ni²⁺ Complex

Nickle complex was synthesized as shown in scheme 1 by dissolving (0.1g, 0.0004 mol) NiCl₂.6H₂O as metal salt in 5ml of ethanol with continuous stirring at room conditions 25 °C. Then adding metal salt solution onto 5ml of ethanolic solution of (0.1 g, 0.0003 mol) from ligand. Light brown precipitate will be formed immediately for Ni²⁺. The precipitate was filtered and washed by di ethyl ether and weighted, the weight was found 0.16 gm for 64% Ni²⁺ and the organic complex was found to be 1:1.

Synthesis of Cu²⁺Complex

Copper complex was synthesized as shown in scheme 1 by dissolving (0.1g, 0.0004 mol) CuCl₂.2H₂O as metal salt in (5ml) of ethanol with continuous stirring at room conditions 25 °C. Then adding metal salt solution onto (5ml) of ethanolic solution of (0.1 g, 0.0003 mol) from ligand. dark green precipitate will be formed 6 hours of stirring. The precipitate was filtered and washed by di ethyl ether and weighted, the weight was found 0.06 gm for 44% Cu²⁺ and the organic complex was found to be 1:1.

Synthesis of Cd²⁺Complex

Cadmium complex was synthesized as shown in scheme 1 by dissolving (0.1g, 0.0004 mol) CdCl₂.2H₂O as metal salt in 5ml of ethanol with continuous stirring at room conditions 25 °C. Then adding metal salt solution onto 5ml of ethanolic solution of (0.1 g, 0.0003 mol) from ligand. Green precipitate will be formed immediately. The precipitate was filtered and washed by di ethyl ether

and weighted, the weight was found 0.05 gm for 55% Cd^{2+} and the organic complex was found to be 1:1.

Synthesis of Zn^{2+} Complex

Zinc complex was synthesized as shown in scheme 1 by dissolving (0.1g, 0.0004 mol) $\text{ZnCl}_2 \cdot 2\text{H}_2\text{O}$ as metal salt in 5ml of ethanol with continuous stirring at room conditions (25 °C). Then adding metal salt solution onto 5ml of ethanolic solution of (0.1 g, 0.0003 mol) from ligand. reddish-brown precipitate will be formed 6 hours of stirring. The precipitate was filtered and washed by di ethyl ether and weighted, the weight was found 0.03 gm for 46% Zn^{2+} and the organic complex was found to be 1:1.

Bioactivity Processor

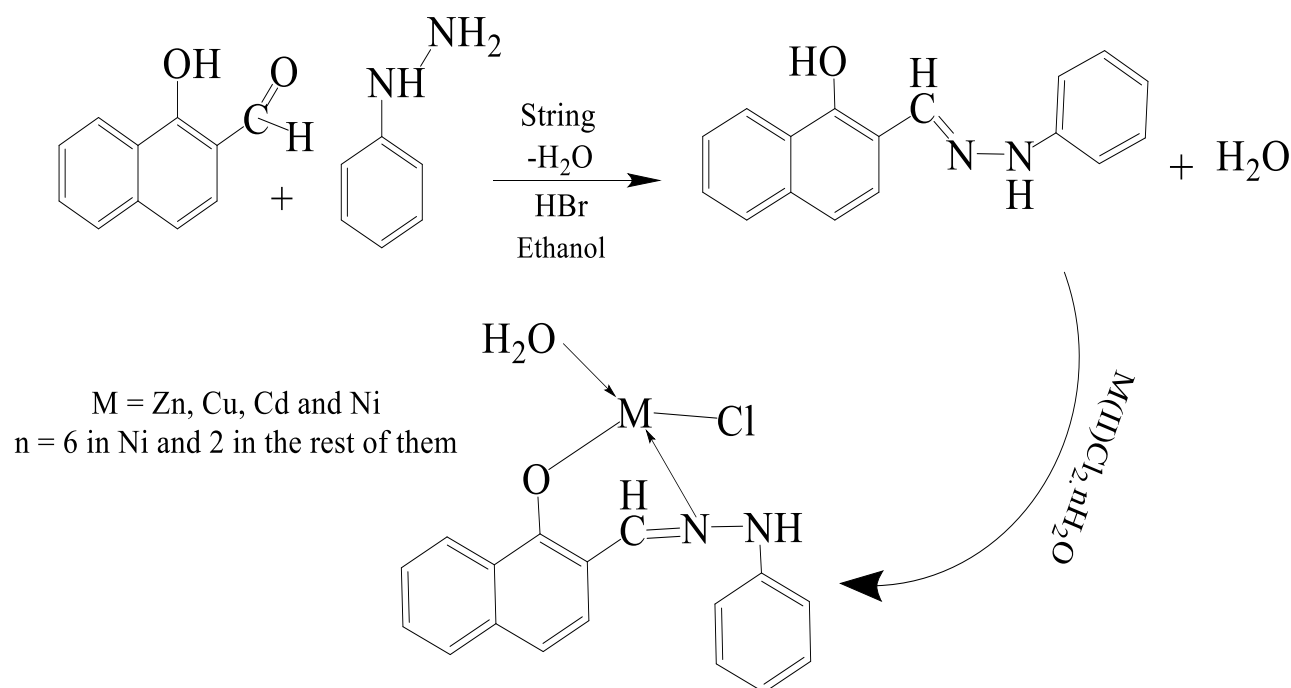
1- The Measurements of the Inhibitory Action on Bacteria

Bauer and his colleagues' method was followed in testing the activity of the synthesized ligand and its complexes on the bacteria by Well Diffusion Assay Method. The plates containing the agar middle were cultured in the bacterial suspension in 1×10^{-3} M with 1.5×10^8 cell/ml by using glass diffuser, then bores with 6mm diameter for each bore were carried out on the surface of the cultured middle by using sterile

cork piercing, the synthesized extracted concentrations were carried to the bores of 50 mm in each bore, one bore contains the solvent as control. The activity of the extracted was detected by measuring the inhibition zone which formed around the bore after 24 hours incubation at 37 °C.

2- The Measurement of the Inhibitory Action of Fungi

Synthesis of the sabouraud dextrose agar or potato dextrose agar was with 1×10^{-3} M in glass bottles with circular stirring so that the culture medium can be homogeneous and it is poured into Petri dishes with a diameter of 9 cm and then left to harden and using the spore suspension method, where a volume of 0.01 ml was taken from the spore suspension and the culture medium was received by stabbing method, it is the easiest and best method for most fungi, with a control sample free of extract for comparison, then the dishes were incubated in an inverted position at a temperature (28 ± 2), then the fungal growth area and the spore density of the fungi were measured after three days. The results were taken by calculating the average of two perpendicular diameters for each colony. Each treatment was three replicated, and each replicate had one dish.



Scheme 1. Pathway creation of Schiff-base and minerals

C₁₇H₁₅ClN₂ZnO₂	199-201	46	orange	54.01	2.98	8.11	7.71	18.81	10.00
379.88				(53.71)	(3.98)	(7.37)	(8.42)	(17.20)	(9.33)

FTIR Studies

FT-IR spectral data of obtained ligand demonstrates that, the absence of C=O vibrational, absence of asymmetric vibrational mode of NH₂ group for starting materials and presence of unique vibrational mode at 1602 cm⁻¹ ¹³. Attributed to C=N stretching vibrational mode which proves the formation of ligand through the mentioned groups. In addition to other vibrations that denoted in Table 3, C-H aldehydic 2555cm⁻¹, C-H aromatic 3020-3097 cm⁻¹, O-H phenolic 3461 cm⁻¹, C=N 1602 cm⁻¹, N-H 3228 cm⁻¹, C=C 1496 cm⁻¹. Moreover, in FT-IR data of our complexes, we can apparently notice the shifting in C=N vibrational mode, which manifest the creation

of coordination through this group to be observed at (1620, 1610, 1625 and 1604) cm⁻¹ for metal complexes Cu²⁺, Cd²⁺, Ni²⁺ and Zn²⁺ respectively. Moreover, some new bands appeared such as M-N observed at 550, 578, 582 and 570 cm⁻¹ for the mentioned complexes respectively. Such vibrations attributed to the coordination with N of Schiff base group, M-O (the presence of this band results in absence of stretching vibrational mode of hydroxyl group) observed at 478, 495, 495 and 495 cm⁻¹ for the mentioned complexes respectively. Such vibrations may be attributed to the coordination with O of OH group. Finally, aqua water molecule is presented inside the coordination sphere ¹⁴. All spectral data are displayed in Table 2.

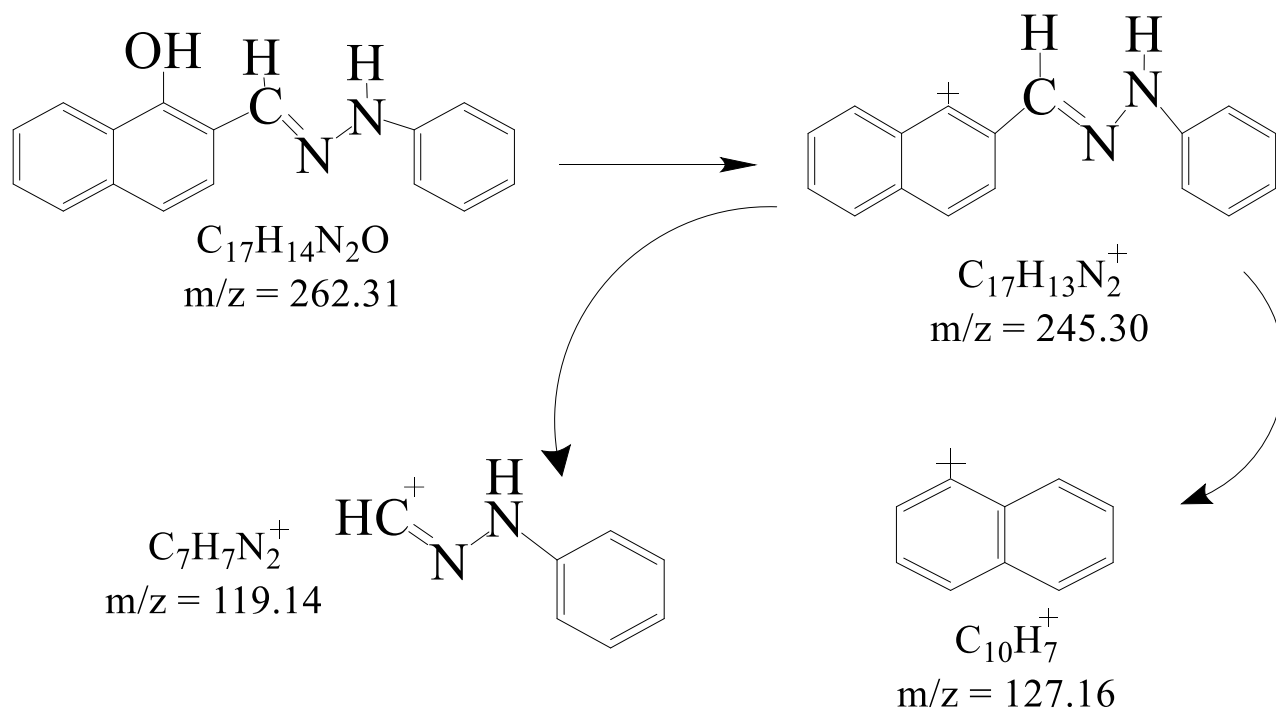
Table 2. FTIR inform of Schiff and minerals

Compound	C_H aldehydic	C_H Aromatic	O-H Phenolic	C=N	N-H	C=C	M-N	M-O	H ₂ O aquatic
C₁₇H₁₄N₂O	2555	3020-3097	3461	1602	3228	1496			
C₁₇H₁₅ClCuN₂O₂	2700	3050	-	1620	3429	1550	550	478	3464
C₁₇H₁₅CdClN₂O₂	2638	3001	-	1610	3020	1494	578	495	3485 1543
C₁₇H₁₅ClN₂NiO₂	2700	3057	-	1625	3265	1492	582	495	3381 1587
C₁₇H₁₅ClN₂ZnO₂	2675	3101	-	1604	3277	1498	570	495	3460

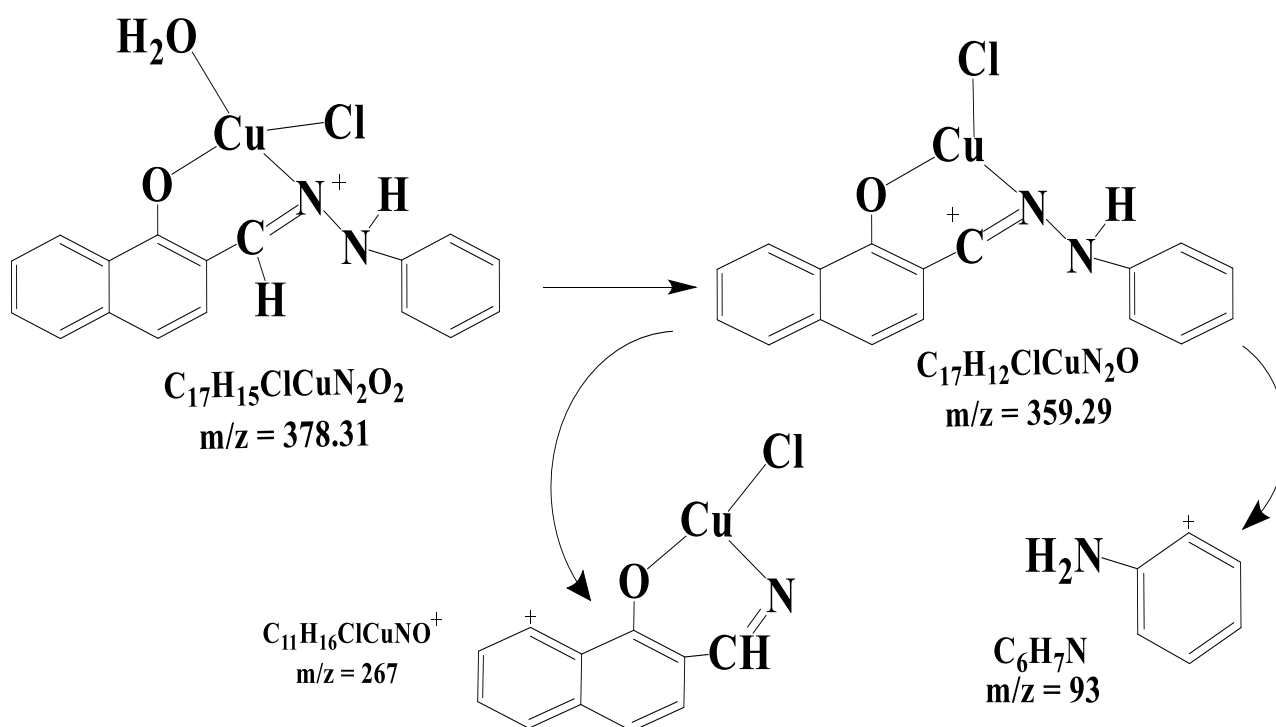
Mass Spectral Investigations

The mass spectral records of Schiff base ligand and its Copper complex are demonstrated in Fig. 3 and Fig. 4, denoted a pack at m/z= 262 contributes to [M⁺] and matching the molar mass of the ligand. In

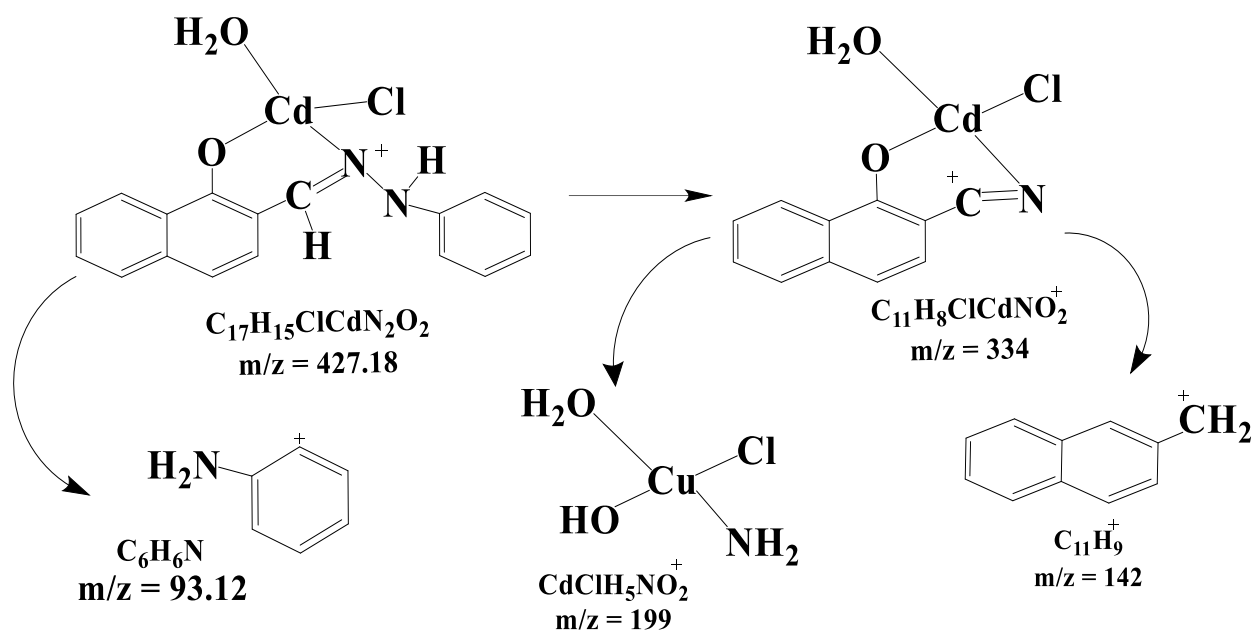
addition to other peaks that observed at 378, 373 and 427 m/z. which corresponds with its formula weight. The suggested fractionation forms of the ligand, Ni, Cu and Cd complexes are illustrated in Schemes 2, 3, 4 and 5. Table 3, illustrates mass spectral records for ligand and related complexes ¹⁵.



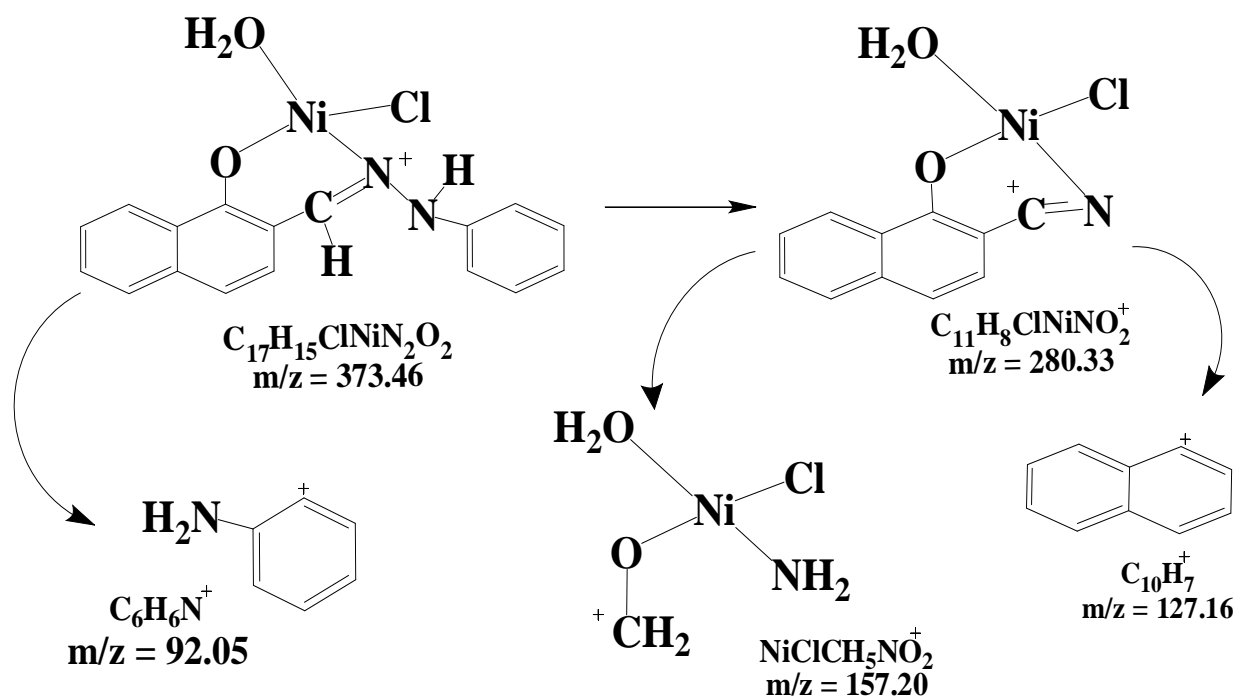
Scheme 2. Partitioning modes of Schiff-base



Scheme 3. Partitioning modes of Cu-Schiff



Scheme 4. Partitioning modes of Cd-complex



Scheme 5. Partitioning modes of Ni-complex

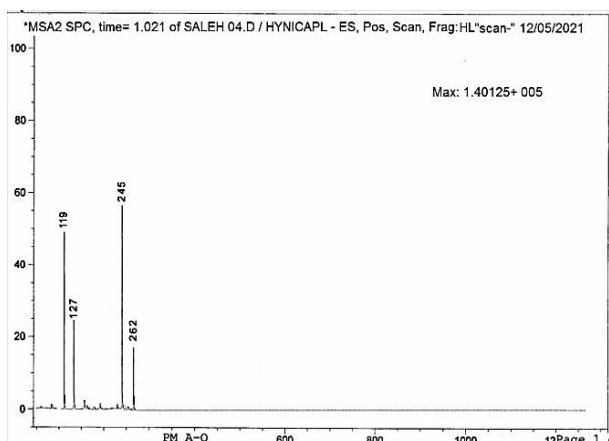


Figure 3. Mass spectrum of Schiff-base

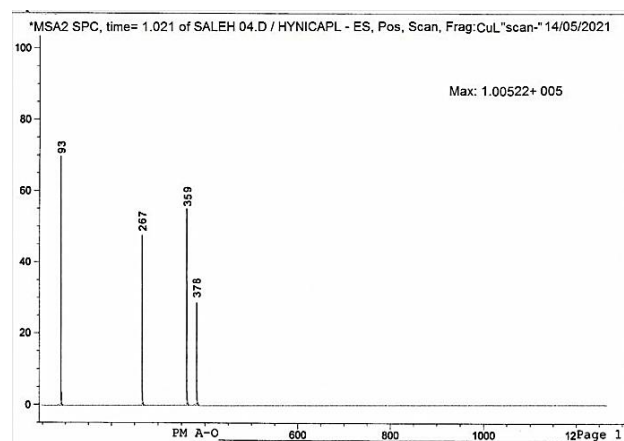


Figure 4. Mass spectrum of Cucomplex

Table 3. LC/Mass spectral inform of some resultants

Assignment	Peak	Abundance	Assignment	Peak	Abundance %
Ligand (HL)	m/z	%	Cu-L	m/z	
$C_{17}H_{14}N_2O$	262.31	17%	$C_{17}H_{15}ClCuN_2O_2$	378.31	28%
$C_{17}H_{13}N^+_2$	245.30	60%	$C_{17}H_{12}ClCuN_2O$	359.29	55%
$C_{10}H_7$	127.16	25%	$C_{11}H_6ClCuNO$	267	50%
$C_7H_7N_2$	119.14	50%	C_6H_7N	93	70%
Assignment	Peak	Abundance	Assignment	Peak	Abundance %
Cd-L	m/z	%	Ni-L	m/z	
$C_{17}H_{15}CdClN_2O_2$	427.18	27%	$C_{17}H_{15}ClNiN_2NiO_2$	373.46	27%
$C_{11}H_8CdClNO_2$	334	85%	$C_{11}H_8ClNiNO_2$	280.33	85%
$CdClH_5NO_2$	199	60%	$CH_5ClNiNO_2$	157.20	60%
$C_{11}H_9$	141	50%	$C_{10}H_7$	127.16	55%
C_6H_6N	92.12	80%	C_6H_6N	92.05	80%

Ultra Violet-Visible for the Ligand and Its Complexes

The electronic spectrum of the ligand showed intense absorption at 260, 283 nm and 339, 354 nm which belong to ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$) respectively. The electronic spectrum of Ni(II) complex showed four peaks at 280, 443, 616 and 678 nm assigned to ($\pi \rightarrow \pi^*$), $\nu_3(^3T_1 \rightarrow ^3T_{1F})$, $\nu_2(^3T_1 \rightarrow ^3T_{1P})$ and $\nu_1(^3T_1 \rightarrow ^3A_1)$ respectively¹⁶. The magnetic moment value 3.9 B.M supports the tetrahedral geometry for the complex. The theoretical value of magnetic moment is 2.98 describes only the spindle

moment because of the presence of two single electrons. Whereas the experimental value 3.9 B.M, describes the spindle moment combined with orbital moment. Knowing that the molar conductivity was found to be 12 S.cm².mol⁻¹. And according to the value of magnetic moment and the presence of two single electrons, the ligand can be considered as weak ligand with high spin. The ultra violet –visible spectrum of copper(II) complex illustrated three peaks at 224, 292 and 887 nm attributed to ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$), ($^2T_2 \rightarrow ^2E$) respectively and the magnetic moment 1.63 B.M support tetrahedral geometry of the complex in addition to molar conductivity which

equal to $10 \text{ S.cm}^2\text{mol}^{-1}$ ¹⁷. And according to the value of magnetic moment and the presence of one single electron, the ligand can be considered as weak ligand with high spin. The electronic spectrum of Zn (II) (diamagnetic support tetrahedral geometry of the complex in addition to molar conductivity which equal to $17 \text{ S.cm}^2\text{mol}^{-1}$. Cd (II) complex (diamagnetic support tetrahedral geometry of the

complex in addition to molar conductivity which equals $19 \text{ S.cm}^2\text{mol}^{-1}$. It showed three peaks at 261, 264 nm, 338,338 nm and 375, 379 nm assigned to ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$) and (C.T) respectively¹⁸, suggesting a tetrahedral geometry of all complexes. All the detected spectral incomes of the products are recorded in (Table 4).

Table 4. Transitions in UV-vis spectra, conductivity and μ_{eff} of ligand and its metal complexes

Compound	Abs	λ_{max}	$\nu_{\text{cm}^{-1}}$	ϵ_{max}	Transition	Λ S.cm ² mol ⁻¹	μ_{eff} B.M found (calc.)	geometry
C₁₇H₁₄N₂O	0.934	354	28248.587	934	* $\rightarrow \pi\pi$	-	-	
	0.974	339	29498.525	974	* $\rightarrow \pi\pi$			
	1.467	283	35335.689	1467	* $\pi \rightarrow \pi$			
	1.691	260	38461.538	1691	* $\pi \rightarrow \pi$			
C₁₇H₁₅ClN₂NiO₂	0.19	678	14727.541	190	³ T ₁ \rightarrow ³ A ₁			Td
	0.18	616	16155.088	180	³ T ₁ \rightarrow ³ T _{1p}	12	3.9	
	0.2	443	22624.434	200	³ T ₁ \rightarrow ³ T _{2F}		(2.98)	
	2.25	280	35714.285	2250	* $\pi \rightarrow \pi$			
C₁₇H₁₅ClCuN₂O₂	0.074	887	11273.957	74	² T ₂ \rightarrow ² E			Td
	3.141	292	34246.575	3141	* $\rightarrow \pi\pi$	10	1.63	
	0.349	224	44662.857	349	* $\pi \rightarrow \pi$		(1.7)	
C₁₇H₁₅ClN₂ZnO₂	0.464	375	26666.666	464	C.T		Dia	Td
	0.443	338	29585.798	443	* $\rightarrow \pi\pi$	17	(0)	
	1.388	261	38314.176	1388	* $\pi \rightarrow \pi$			
C₁₇H₁₅CdClN₂O₂	0.524	379	26385.224	524	C.T		Dia	Td
	0.453	338	29585.798	453	* $\rightarrow \pi\pi$	19	(0)	
	1.463	264	37878.788	1463	* $\pi \rightarrow \pi$			

Dia= diamagnetic

Thermal Analysis

The thermogravimetric (TG) curve of ligand L shows relatively one step decomposition in the range 50–595 °C with four DSC peaks and estimated and

calculated mass loss equal 97.47 and 98.36, respectively¹⁹. The TG curve of Ni complex shows four steps decomposition with four DSC peaks as shown in (Table 5) and Fig. 5 and 6.

Table 5. Thermogram incomes of ligand and Nickle

Molecule.	Thermograv imetri T_°C	D_S_C max °C	% Est. (calculated)		Assignment
			Mas lost	Tot. mas lost	
C₁₇H₁₄N₂O 262.31g/mole		89.3(Endo)			
		188.9(Exo)	97.47(98.36)	97.47	C ₁₆ H ₁₄ N ₂ O
	50-595	303.5(Exo)	2.53 (1.52)	(98.36)	C
		567.3(Exo)			
[Ni complex] 373.46g/mole	50-160	85.3(Endo)	7.39 (4.82)	91.79	H ₂ O
	160-240	265.2(Exo)	27.33(26.24)	(86.18)	C ₈ H ₂
	240-390	299.5(Exo)	43.6(41.64)		C ₇ H ₈ N ₂ Cl
	390-595	400(Endo)	13.47(13.48)		0.5NiO+CH
			8.21(10.0)		0.5NiO

Endo= Endothermic

Exo= Exothermic

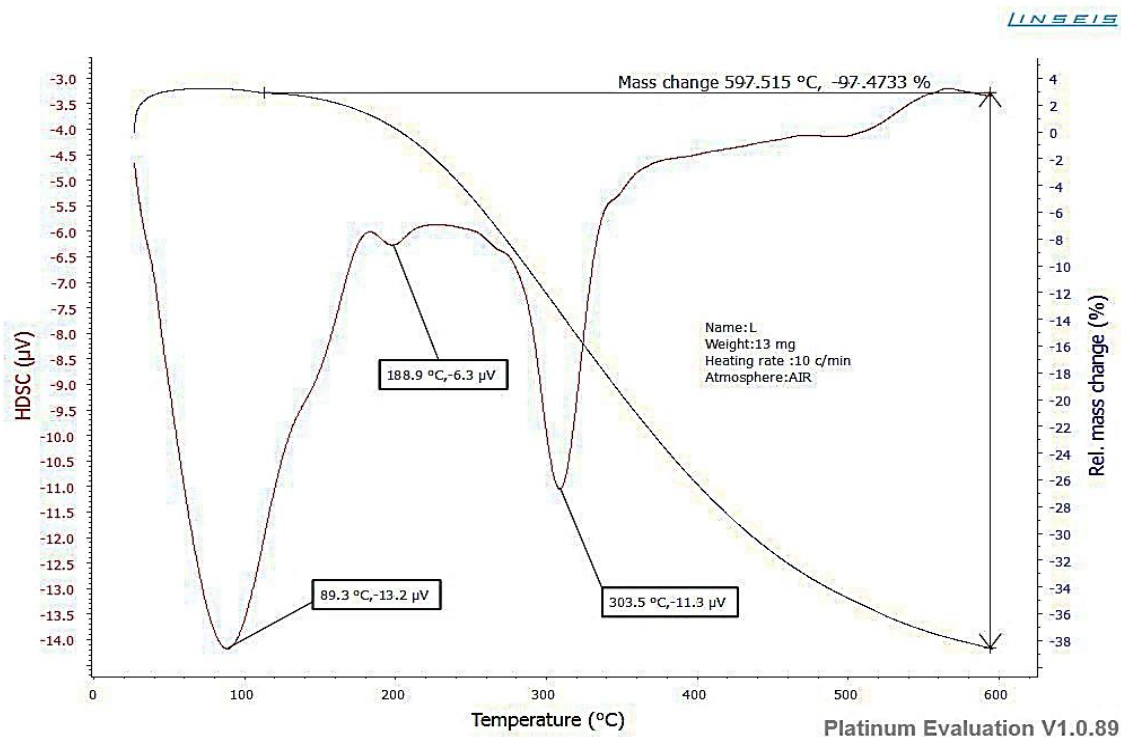


Figure 5. Ligand-thermogram

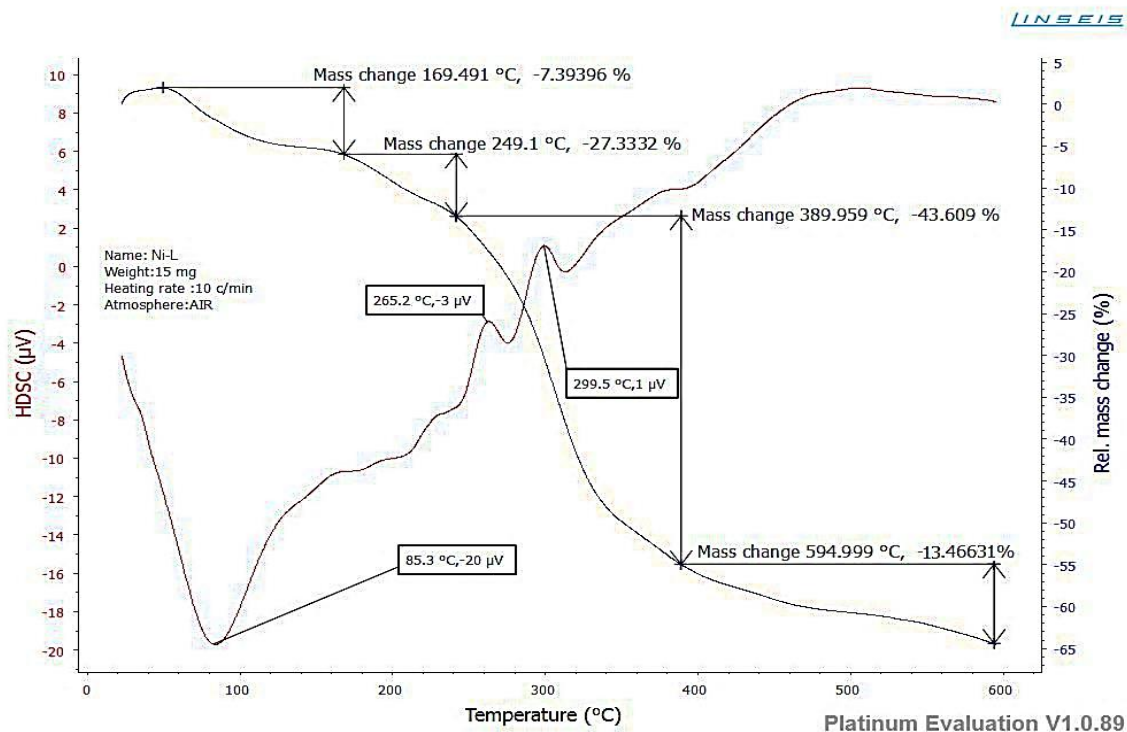


Figure 6. Nickel-thermogram

Biological Diagnosis

Two types of bacteria were tested, the gram-positive bacteria (*Staphellococcus aureus*) and gram negative bacteria (*E. coli*), and two types of fungi (*Candida*

albicans and *Rhizopus*), the effect of the synthesized ligand and its complexes on the previous bacteria and fungi were tested and compared in 1×10^{-3} M DMSO (solvent) as control. We could manifest their activity

on such organisms as demonstrated in Fig. 7. Additionally, we can also notice the variations in inhibitory radius between bacteria and fungi and according to these variations; we have detailed their action in Fig. 7 and Table 6. The ligand, copper and

nickel complexes are more effective than other complexes in their inhibition toward both types of fungi because of the mutual effect between their corresponded metal salts and ligand²⁰.

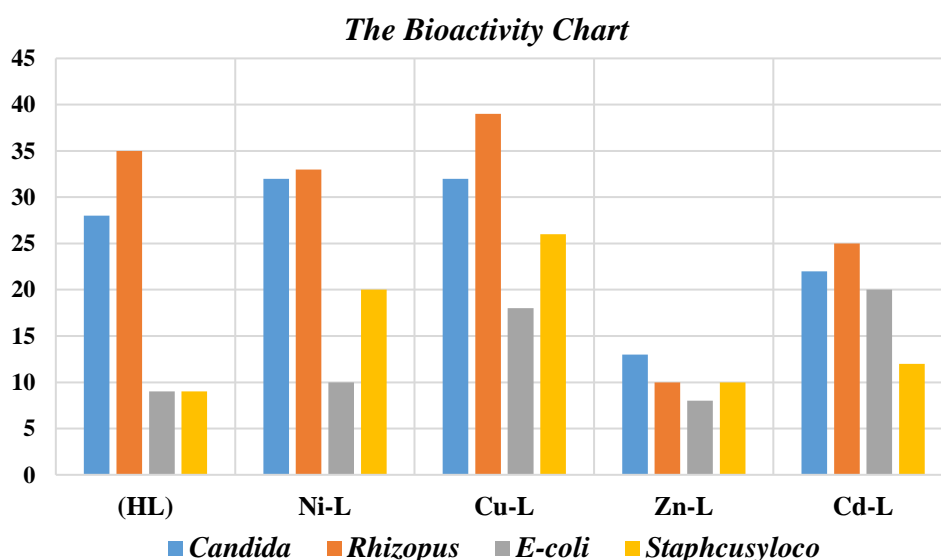


Figure 7. Biological diagnosis incomes

Table 6. Inhibition zone (mm) of ligand and its complexes

Compound	<i>fungi</i> <i>E-coli</i>	<i>Staphylococcus aureus</i> (gram positive)	<i>Candida albicans</i> (gram negative)	<i>Rhizopus</i> <i>fungi</i>
$C_{17}H_{14}N_2O$	9 mm	9 mm	28 mm	35mm
$C_{17}H_{15}ClN_2NiO_2$	10 mm	20 mm	32 mm	33mm
$C_{17}H_{15}ClCuN_2O_2$	18 mm	26 mm	30 mm	37mm
$C_{17}H_{15}ClN_2ZnO_2$	8 mm	10 mm	11 mm	8 mm
$C_{17}H_{15}CdClN_2O_2$	20 mm	27 mm	22 mm	25mm

Conclusion

In this paper, the formation of Schiff-base (E)-2-((2-phenylhydrazono)methyl)naphthalen-1-ol (phenyl hydrazine derivative) and their metal complexes including Ni^{2+} , Cu^{2+} , Zn^{2+} and Cd^{2+} ions were prepared and proved successfully. Spectral combination using various apparatus could manifest the formation of the resultants including (FTIR, UV-VIS, LC/MSS, (TGA and DSC) and 1H -NMR). Each

apparatus can prove the results individually depending on its own features as detailed in the article. On the other hand, the bidentate behavior of ligand was detected spectrally. Finally, we could manifest successfully the biological activity of compounds toward several shapes of microorganisms.

Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- The author has signed an animal welfare statement.
- Authors sign on ethical consideration's approval.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Authors' Contribution Statement

This work carried out in collaboration between all authors. A. A.S., W. A. M. and Z. H. A. participated in doing the tests and analyzing data with revision.

A. M. A. prepared the samples, wrote and edited the manuscript with revision.

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تحضير وتشخيص وتقييم الفعالية الحيوية لبعض معقدات ايونات المعادن مع ليكاند جديد مشتق من 2- هيدروكسي-1- نفتالديهايد

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الخلاصة

تم مفاعلة قاعدة شيفف المُحضرة حديثاً (E)-2-((2-phenylhydrazono)methyl)naphthalen-1-ol (مشتق فينيل هيدرازين) مع كل من الأيونات المعدنية التالية: Ni^{2+} ، Cd^{2+} ، Cu^{2+} ، Zn^{2+} للحصول على معقدات فلزية بأشكال هندسية مختلفة. تم الاستدلال عن تكوين معقدات قاعدة شيفف وأيضاً قاعدة شيفف الأصلية باستخدام كروماتوغرافيا السائل-مطياف الكتلة التي تتفق مع الكتلة المولية التي تم الحصول عليها نظرياً، أثبتت دراسات مطيافية الأشعة تحت الحمراء حدوث التناسق من خلال N من azobenzene و او كسجين مجموعة ال OH من خلال ملاحظة الانزياح في قمة azomethines والظهور كل من قمتي M-N و N-O. علاوة على ذلك، يمكننا أيضاً أن نكتشف بواسطة هذا الجهاز وجود جزيء ماء داخل مجال التناسق. كشفت أطيف UV-Vis لجميع النتائج عن حدوث تناسق من خلال ملاحظة الانزياح في الانتقالات الإلكترونية التي حدثت في الليكاند في المنطقة فوق البنفسجية. تثبتت قياسات TGA و DSC لكل من الليكاند و معقد النيكل أيضاً تكون المعقد ووجود جزيء ماء داخل مجال التناسق. بالإضافة إلى ذلك، كانت نتائج التوصيلية المولارية و الامتصاص الذري اللهبى FAA أقرب إلى نتائج المحسوبة نظرياً. أعطت نتائج التشخيص معقدات أحادية النواة وسلوك ثنائي السن وأشكال هندسية رباعي السطوح لجميع المعقدات. اعتماداً على خاصية المضادات الحيوية لقاعدة شيفف ومعقداتها، قمنا بدراسة الفعالية الحيوية لقاعدة شيفف ومعقداتها كما مفصل في المخطوطة.

الكلمات المفتاحية: الفعالية الحيوية، الانتقال الإلكتروني، هيدروكسي-1- نفتالديهايد، قاعدة شيفف، الدراسات الطيفية.