## Synthetic and Characterization of Some new Schiff bases Complexes with Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Pd<sup>II</sup> ions

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### Abstract

A new ligand (H<sub>4</sub>L) and its complexes with (Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Pd<sup>II</sup>). This ligand was prepared in two steps, in the first step a solution of terephthaldehyde in methanol reacted under refluxe with 1,2-phenylenediamine to give precursore compound which reacted in the second step with 2,4dihydroxybenzaldehyde to give the ligand. The complexes were synthesized by direct reaction of the corresponding metal chloride with the ligand. The ligand and complexes were characterized by spectroscopic methods [FT-IR, UV-vis, <sup>1</sup>HNMR, HPLC and atomic absorption], chloride contant in addition to conductivity measurement. The stability constant K and Gibbs free energy  $\Delta G$  were calculated for [[Ni<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>], [Cu<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>] complexes using spectrophotometer method. The obtained values indicate that these complexes are stable in their solution. The data of these measurements suggest a distorted tetrahedral geometry for Co<sup>II</sup>, Ni<sup>II</sup>, and Cu<sup>II</sup> complexes and a square planar for Pd<sup>II</sup> complex. The biological activity for the ligand (H<sub>4</sub>L), and its complexes [Ni<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>], [Pd<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>] were studied.

Keywords: Schiff bases, complexes of Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Pd<sup>II</sup> ions.

### Introduction

Schiff's bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in many fields, e.g., biological, inorganic and analytical chemistry [1]. Schiff's bases of o-phenylenediamine and its complexes have a variety of applications including biological, analytical [2] and clinical [3]. Metal complexes of Schiff base are extensively studied due to synthetic flexibility and sensitivity toward a variety of metal atoms [4]. They are found useful in catalysis, in medicine as antibiotics and anti-inflammatory agent and in the industry as anti-corrosion [5]. It has been found that some the complexes are antimicrobially active and show higher activity than the free ligand. Metal chelation affects significantly the antimicrobial/bioactive behavior of the organic ligands [3]. In this paper the synthesis and characterization of Schiff base ligand (H<sub>4</sub>L) derived from the reaction of terephthaldehyde, o-phenylene diamine and 2,4 -Dihydroxy benzaldehyde and some of its complexes with (Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Pd<sup>II</sup>) are reported.

#### **Experimental**

Reagent grade terephthaldehyde and 2,4dihydroxy benzaldehvde obtained from Fluka and o-phenylene diamine obtained and from Aldrich used as received. while CoCl<sub>2</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuCl<sub>2</sub>.2H<sub>2</sub>O and PdCl<sub>2</sub> were available from (Riedial -Dehaen, Fluka, Merck and Hopkins & William LTD) respectively. The FT-IR spectra of compounds were recorded as (KBr) discs using Shimadzu 8300 FTIR Spectrophotometer in the range (4000-400) cm<sup>-1</sup>. Electronic spectra of the prepared compounds were recorded in the region (200-900) nm for 10<sup>-3</sup>M solutions in (DMF) as a solvent at 25 C° using a CECIL, CE7200 spectrophotometer. While metal contents of the complexes were determined by atomic absorption (A.A) technique using a Shimadz (AA .620) atomic absorption spectrophotometer. Electrical conductivity measurements of the complexes were recorded at 25 C° for  $10^{-3}$  M solutions of the samples (DMF) using а Wissenschaftlichin Techniche Werkstatten. D1820 Wilhelm LF 42 conductivity meter. Nuclear magnetic resonance spectra <sup>1</sup>HNMR for the ligand ( $H_4L$ ) were recorded using Burker (400MHz)

spectrophotometer with a tetramethylsilane (TMS) as an internal standard in DMSO-d<sup>6</sup>. Al-Baath University, Syria. The chloride contents for complexes were determined by potentiometric titration method on (686-Titro processor-665. Dosimat Metrohn Swiss) and melting point measurements by using Stuart melting point. The High Performance Liquid Chromatography (HPLC) analysis of the complexes were obtained by using HPLC type Shimadzu (LC-2010AHT) (UV-250Detector).

## Preparation

## Synthesis of the ligand (H<sub>4</sub>L):

The ligand was prepared by two steps

Step (1): preparation of the (precursore compound).

A solution of terephthaldehyde (0.15 g, 1.118 mmole) in methanol (5 ml), then (5) drops of glacial acetic acid were added slowly, then mixture was added to 1,2phenylenediamine (0.241 g, 2.23 mmole) in methanol (5ml). The mixture was refluxed for (15 hrs.) with stirring. The orange solution was left to dry for (24 hrs.) in room temperature, the precipitate was washed with an excess of methanol. dried. An orange solid was obtained. Yield (0.26)g, (74%), m.p (190 dec).

### Step (2): preparation of the ligand (H<sub>4</sub>L):

A solution of (precursore compound) (0.12 g, 0.38 mmole) in methanol (5 ml) and few drops of DMF were added to 2,4-dihydroxybenzaldehyde (0.1 g, 0.724 mmole) in methanol (5ml), then five drops of glacial acetic acid were added slowly to the reaction mixture. The reaction mixture was refluxed for (15 hrs.) with continuous stirring. The brown solution was left to dry for (72 hrs.) at room temperature, the precipitate was washed with an excess of methanol, dried. A brown solid was obtained. Yield (0.18)g, (85%), m.p (223 dec).

## Synthesis of $(H_4L)$ complexes with some metal ions

### Synthesis of [Co<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>] (1) complex:

A solution of  $(H_4L)$  (0.15 g, 0.27 mmole) in methanol (5 ml) with few drops of DMF were placed in a round bottomed flask. A solution of KOH (0.04g, 0.714 mmole) in (5ml) ethanol. A solution of CoCl<sub>2</sub>.6H<sub>2</sub>O (0.0429g, 0.18 mmole) in (10 ml) methanol was dropwise with stirring. The mixture was refluxed for (3 hrs.). A deep green precipitate was formed, which was filtered off, washed several times with methanol and dried at room temperature during (72 hrs.). Yield (0.051)g, (76 %), m.p. (270 dec).

## Synthesis of $[Ni_2(H_2L)Cl_2]$ (2), $[Cu_2(H_2L)Cl_2]$ (3) and $[Pd_2(H_2L)Cl_2]$ (4)complexes.

The method used to prepare these complexes was a similar method to that mentioned in preparation of  $[Co_2(H_2L)Cl_2]$  complex. Table (1) shows the stated weight of starting materials, % yield and some physical properties of the prepared complexes.

Table (1)Some physical properties of the complexesand their reactants quantities.

No.	Empirical Formula	colour	m.p °C	Yield %
	$H_4L$	brown	223 dec	85
1	[Co <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	deep green	270 dec	76
2	[Ni <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	deep green	240 dec	85
3	[Cu <sub>2</sub> ( H <sub>2</sub> L )Cl <sub>2</sub> ]	deep green	250 dec	74
4	[Pd <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	deep brown	280 dec	69

### **Results and Discussion**

Scheme (1) Preparation of the ligand  $(H_4L)$  and its complexes



Scheme (1) Synthesis route of the Schiff's base ligand  $(H_4L)$  and its complexes.

## <sup>1</sup>HNMR spectra analysis:

The <sup>1</sup>HNMR spectrum of ligand (H<sub>4</sub>L), Fig.(1) in DMSO–d<sup>6</sup> solvent shows a singlet signal at ( $\delta = 10.93$  ppm) equivalent to two protons assigned to (O–H) group[6]of carbon (C<sub>1,32</sub>). Two protons of (C<sub>5</sub>-OH,C<sub>30</sub>-OH) group appears as a singlet signal at ( $\delta = 13.1$  ppm) [6]. Two protons of (N=C<sub>7</sub>–H,N=C<sub>28</sub>–H) imine group appears as a singlet signal at  $(\delta=8.36 \text{ ppm})$ . Two protons of  $(N=C_{14}-H, N=C_{21}-H)$  imine group appears as a singlet signal at ( $\delta = 9.93 \text{ ppm}$ ). The multiplet signals at ( $\delta = 6.33 \text{ ppm}$ ), (7.23), (7.33), (7.46), (7.53), (7.63), (7.95) ppm are due to aromatic hydrogen of carbon (C<sub>2,33</sub>), (C<sub>3,34</sub>), (C<sub>6,31</sub>), (C<sub>9,12,23,26</sub>), (C<sub>10,11,24,25</sub>), (C<sub>16,17</sub>), (C<sub>19,20</sub>) respectively [7,8].



Fig. (1) <sup>1</sup>HNMR spectrum of the ligand (H<sub>4</sub>L).

## Infrarwd Spectra:

The FT-IR spectrum for the ligand  $(H_4L)$ , Fig.(2) displays two bands at (3429) cm<sup>-1</sup> and (3140) cm<sup>-1</sup> can be due to the v(O-H)stretching[7,8]. The bands at (1635)  $\text{cm}^{-1}$  and cm<sup>-1</sup> were assigned (1620)to the v(C=N)(Benz) and v(C=N) (Tere) stretching vibration. Appearance of imine (C=N) (Benz) band and the disappearance of carbonyl (C=O) band and (NH<sub>2</sub>) bands of 1.2phenylenediamine in the ligand spectrum confirm the formation of Schiff's base ligand [9]. The band at (1235) cm<sup>-1</sup> can be assigned to the (C–O) stretching vibration [10]. The FT-IR spectra for complexes (1-4). The assignments of the characteristic bands are summarized in Table (2). The strong band at (1635) and (1620) which referred to v(C=N) of imine groups in the free ligand (H<sub>4</sub>L) are shifted to lower frequency and appeared at (1627, 1604) cm<sup>-1</sup> Fig.(3), broad band at (1616) cm<sup>-1</sup>, (1620,1589) cm<sup>-1</sup> and (1600,1577) cm<sup>-1</sup> for the complexes [1-4] respectively [11]. The shift to lower frequency may be due to delocalisation of metal electron density into the ligand  $\pi$ -system [12] (HOMO $\rightarrow$ LUMO), (where HOMO: Highest Occupied Molecular Orbital, LUMO: Lowest

Unoccupied Molecular Orbital). The shift in v(C=N) confirms the coordination of the ligand through nitrogen atoms to the metal ions[13]. These bands were assigned to the v(C=N) stretching of reduced bond order. On the other hand, the band at (1235) cm<sup>-1</sup> in the free ligand (H<sub>4</sub>L) has been assigned to phenolic v(C-O) stretching. On complexation, this band has been shifted to lower frequency and appeared at (1215), (1226), (1203) and (1226) cm<sup>-1</sup> for complexes [1-4] respectively. The shift in v(C-O) confirms the coordination of the ligand through oxygen atoms to the metal ions [14]. The broad band at (3429) cm<sup>-1</sup> attributed to v(OH) in the free ligand are shifted to lower frequency appeared at (3329) cm<sup>-1</sup>, (3387) cm<sup>-1</sup>, (3356) cm<sup>-1</sup> and (3406) cm<sup>-1</sup> for complexes [1-4] respectively. The bands at (547), (513), (559), and (530) cm<sup>-1</sup> were assigned to v(M-N) for compounds [1-4] respectively, indicating that the imine nitrogen is involved in coordination with metal ions[11]. The bands at (421), (470), (497) and (497) cm<sup>-1</sup> were assigned to v(M-O)for compounds [1-4] respectively indicating that the phenolic oxygen of the ligand is involved in coordination with metal ions [11].

No.	Compounds	v(C=N)	v ( <b>O-H</b> )	v(C=C) arom.	v (C-H) arom.	v (C-H) aliph.	v (C-O)	v (M-N)	v (M-O)
	[H <sub>4</sub> L]	1635 1620	3429	1500	3062	2927	1235	-	-
1	[Co <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	1627 1604	3329	1527	3066	2924	1215	550 547	421
2	[Ni <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	1616	3387	1539	2950	2924	1226	551 513	470
3	[Cu <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	1670 1589	3356	1550	3089	2958	1203	578 559	497
4	[Pd <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	1600 1577	3406	1531	3059	2889	1226	565 530	497

Table	2 (2)
FT-IR spectral data (wave number v) cm <sup>-1</sup>	<sup>1</sup> for the ligand ( $H_4L$ ) and its complexes.



Fig. (2) FT-IR spectrum for the  $(H_4L)$ .



Fig. (3) FT-IR Spectrum of the complex  $[(Co_2(H_2L)Cl_2]]$ .

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 $(\varepsilon_{max}=1524)$ 

The

peaks

[15].

spectrum

at (291

Fig.(5).

showed

 $molar^{-1}.cm^{-1}$ )

 $(28985.5 \text{ cm}^{-1})(\epsilon_{\text{max}}=2374 \text{ molar}^{-1}.\text{cm}^{-1})$  these

peaks were assigned to ligand filed[15]. The

same spectrum exhibited two additional peaks,

the first high intense peak at (360 nm)

 $(27777.77 \text{ cm}^{-1})$  ( $\varepsilon_{\text{max}}=2000 \text{ molar}^{-1}$ . cm<sup>-1</sup>) is

Complex

(345

two

nm) (34364.689

(2):

intense

 $cm^{-1}$ )

 $cm^{-1}$ )

nm)

## **UV-Vis spectroscopic:**

The (UV-vis) spectrum for the (H<sub>4</sub>L). ligand Fig.(4)exhibits high absorption peak at (282 nm)  $(35460.99 \text{ cm}^{-1})$ molar<sup>-1</sup>  $.cm^{-1}$ ),  $(\varepsilon_{max}=2229)$ high absorption peak at (330 nm)  $(30303 \text{ cm}^{-1})$  $(\epsilon_{max}=2175 molar^{-1}.cm^{-1})$ and intense absorption peak at (348nm)  $(28571.4 cm^{-1})$  $(\varepsilon_{\text{max}} = 186 \text{ 1molar}^{-1}, \text{ cm}^{-1})$ , which were assigned to  $(\pi \rightarrow \pi^*)$ ,  $(\pi \rightarrow \pi^*)$  and  $(n \rightarrow \pi^*)$ transition respectively[11]. The (UV-vis) spectra for the complexes (1-4). The absorption data for complexes are given in Table (3). Complex (1): The spectrum showed two intense peaks at (292 nm)  $(34246.57 \text{ cm}^{-1})$  $(\varepsilon_{\rm max}=1861\,{\rm molar}^{-1}.{\rm cm}^{-1}),$  $(334 \text{ nm}) (29940 \text{ cm}^{-1})(\epsilon_{\text{max}}=2.230 \text{ molar}^{-1}.\text{cm}^{-1})$ these peaks were assigned to ligand filed[15]. The same spectrum exhibited three additional peaks, the first high intense peak at  $(351 \text{ nm})(28490 \text{ cm}^{-1})(\epsilon_{\text{max}}=2205 \text{ molar}^{-1}.\text{cm}^{-1})$ is due to charge transfer (C.T), while the second and third peaks at (608.6 nm)  $(16431.15 \text{ cm}^{-1})$   $(\epsilon_{\text{max}}=177 \text{ molar}^{-1}.\text{cm}^{-1})$ (14863.25  $cm^{-1}$ ) and (672.8 nm)  $(\varepsilon_{max}=205 \text{ molar}^{-1}.\text{cm}^{-1})$ , which assigned to (d-d) transitions in a distorted tetrahedral

due to (C.T), while the second weak broad peak at (628 nm)  $(15910.89 \text{ cm}^{-1})$  $(\varepsilon_{\text{max}} = 120 \text{ molar}^{-1}.\text{cm}^{-1})$  which assigned (d-d)transition in а distorted to tetrahedral geometry [15]. Complex (3): The spectrum showed two intense peaks nm) (34246.57 at (292  $(\varepsilon_{max} = 1367 \text{ molar}^{-1} \cdot \text{cm}^{-1})$  and (345 nm) $(28985.5 \text{ cm}^{-1})$  ( $\varepsilon_{\text{max}}=2374 \text{ molar}^{-1}.\text{cm}^{-1}$ ) these peaks were assigned to ligand filed [15]. The same spectrum exhibited three additional peaks, the first high intense peak at  $(351 \text{nm}) (28490 \text{cm}^{-1}) (\epsilon_{\text{max}} = 1790 \text{molar}^{-1} \text{.cm}^{-1})$ is due to charge transfer (C.T), while the second and third peaks at (582.2 nm)  $(17182.13 \text{ cm}^{-1})(\varepsilon_{\text{max}} = 443 \text{ molar}^{-1} \text{ .cm}^{-1})$  $(14660 \text{ cm}^{-1})$ and (682.1 nm)  $(\varepsilon_{max} = 433 \text{ molar}^{-1} . \text{cm}^{-1})$ , which were assigned to (d–d) transitions in a distorted tetrahedral geometry[15]. Complex (4): The spectrum showed two intense peaks at (284 nm) (35211.26 cm<sup>-1</sup>)( $\varepsilon_{max}$ =2267 molar<sup>-1</sup>.cm<sup>-1</sup>) and (320 nm)(31250cm<sup>-1</sup>) ( $\varepsilon_{max}$ =2400 molar<sup>-1</sup>.cm<sup>-1</sup>) these peaks were assigned to ligand filed [15]. The same spectrum exhibited two additional

peaks, the first intense peak at (400 nm) (25000 cm<sup>-1</sup>) ( $\epsilon_{max} = 1525 \text{ molar}^{-1} \text{ cm}^{-1}$ ) is due to (C.T), while the second peak at (423.8 nm) (23596 cm<sup>-1</sup>) ( $\epsilon_{max} = 1178 \text{ molar}^{-1} \text{ cm}^{-1}$ ) which were assigned to (d–d) transition in a square planar geometry [15].

Table (3)
Electronic data and molar conductivity for the ligand $(H_4L)$ and its Metal complexes.

No.	Compounds	λnm	ΰ cm <sup>-1</sup>	$\Lambda_m$ S.cm².mole-1	Assignments	Coordination	
		282	35460.99		$\pi  ightarrow \pi^{*}$		
	$H_4L$	330	30303		$\pi \rightarrow \pi^*$		
		348	28735.63		$n \rightarrow \pi^*$		
		292	34246.57		Ligand field		
		334	29940	25	Ligand field		
1	$[\mathrm{Co}_2(\mathrm{H}_2\mathrm{L})\mathrm{Cl}_2]$	351	28490		C.T	Distorted Tetrahedral	
		608	16431.15		d-d		
		672	14863.25		d-d		
		291	34364.689		Ligand field	Distorted	
	Ni <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	345	28985.5	24.9	Ligand field		
2		360	27777.7		C.T	Tetrahedral	
		628	15910.89		d-d		
		292	34246.57		Ligand field		
	[Cu <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	335	29850.74	27.9	Ligand field		
3		351	28490		C.T	Distorted Tetrahedral	
		582	17182.13		d-d	Tetraneurar	
		682	14660.6		d-d		
4	[Pd <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	284	35211.26		Ligand field		
		320	31250	10.50	Ligand field	Square planar	
		400	25000		C.T	Square planal	
		423.8	23596		d-d		



Fig. (4) Electronic spectrum of the ligand  $(H_4L)$ .



Fig. (5) Electronic spectrum of the  $[Co_2(H_2L)Cl_2]$ .

### Molar conductivity measurement:

The molar conductance for the ligand (H<sub>4</sub>L) complexes summarized in Table (3) was in the range (10.50-27.9) S.cm<sup>2</sup>.mole<sup>-1</sup>, indicating their non-electrolytic behavior [16].

# High Performance liquid Chromatography (HPLC):

The (H.P.L.C) for the compound (H<sub>4</sub>L),  $[Co_2(H_2L)Cl_2]$  and  $[Cu_2(H_2L)Cl_2]$  complexes, exhibits one sharp chromatogram at retention time (Rt=6.877), (Rt=7.036) and (Rt=7.048), respectively, indicating that, the complexes are pure, and appear as a single species in solution. The atomic absorption analysis and the chloride content results of the complexes are in a good agreement with suggested formula  $[M_2(H_2L)Cl_2]$ .

## Study of formation and stability of complexes:

The mole-ratio In this method the relative (L: M) was calculated depending on the measurement of the absorption to the series of solutions which contain increased molar concentrations of one component (ligand) with concentration to the other component (metal) at wave length which occurs in its higher absorption from the product complex and does not occur at the absorption to the chelate ligand alone or to the metal ion alone. The relationship between the absorption which was presented as (Y) axis and the concentration to the two reactants (metal: ligand) was drown, which was presented as (X) axis, also the rectum contiguity was drown until they intersect and from the intersection point equivalent metal was limitated. The absorbance as and Am for the solutions of first and second set were measured. On the basis of these measurement the stability constant K and Gibbs free energy  $\Delta G$  were calculated. The stability constant K and Gibbs free energy were calculated using the method shown above in the experimental. The equilibrium of the complex metal ion and the ligand for 2: 1 mole ratios and K for this ratio is expressed by:

$2M+L \longleftrightarrow M_2L$	(1)
$K = [M_2L] / [M]^2 [L]$	(2)
$K = (1 - \alpha)/4\alpha^{3}C^{2}$	(3)

Where C and  $\alpha$  the concentration and degree of decomposition of the complex respectively. The values a were determined from the equation

$$\alpha = (\mathbf{A}_{\mathrm{m}} - \mathbf{A}_{\mathrm{S}}) / \mathbf{A}_{\mathrm{m}}$$

- $A_s$  =The absorption at the eq. point of mole ratio.
- A<sub>m</sub>=The maximum absorption at the mole ratio.

As and Am, are the absorption of the M: L =1:1, M: L=2:1, M: L=3:1 and M: L=4:1 respectively. The calculation of  $\Delta G$  at 300°K was carried out according to following expression

 $\Delta G$ = -2.303RT log k

Where R=8.31J mole-1.K-1 and T=300°K.

The obtained date is listed in Table (4) which shows that the complexes are stable ( $\Delta G < 0$ ) and there stability increase in the order Cu (II) > Ni (II).

Table (4)
Stability constant and $\triangle G$ for the ligand (H <sub>4</sub> L) complexes
$[Ni_2(H_2L)Cl_2]$ and $[Cu_2(H_2L)Cl_2]$ .

Compounds	$A_s$	$A_m$	α	K	Log K	1/K	$\Delta \boldsymbol{G}$
$[Ni_2(H_4L)Cl_2]$	2.37	2.4	0.0125	1.26408090117×10 <sup>11</sup>	11	7.91×10 <sup>-12</sup>	-62.8
$[Cu_2(H_4L)Cl_2]$	1.82	1.83	0.00546	1.527711213517×10 <sup>12</sup>	12.18	6.55×10 <sup>-13</sup>	-69.5

#### **Biological activity:**

The biological activity of the ligands  $(H_4L)$ and  $[Ni_2(H_2L)Cl_2]$ ,  $[Pd_2(H_2L)Cl_2]$ , complexes was studied by using inhibition method for two types of pathogenic bacteria. One type of becteria was gram positive which is Bacillus Cereus. The second one was gram negative which is Psudomonus Sp. The biological effect of the chemical complexes as was shown, was studied for the (2) types of bacteria as shown in Table (5). The rate of inhibition diameter was varied according to the variation in the complex type and Bacterial type.

Table (5)

The inhibition circle diameter in millimeter for the bacteria after 24 hour incubation paid and 37 °C for  $H_4L$  and some complexes.

Compounds	<i>P.S.</i>	<i>B.C.</i>	
Control DMF	10.9	9.9	
$H_4L$	30	25	
[Ni <sub>2</sub> (H <sub>2</sub> L)Cl <sub>2</sub> ]	16	20	
$[Pd_2(H_2L)Cl_2]$	20	18	

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[16]Ketlle S.F.A., "Coordination Compounds", Thomas Nelson &Sons, London, 1975. بينما يتخذ معقد البلاديوم شكل المربع المستوي. تم اختبار الفعالية البايلوجية لليكاند المحضر ومععقداته و كانت ايجابية بالتثبيط.

#### الخلاصة

تضمن البحث تحضير الليكاند الجديد ٤,٤' – (٢,٢'- (٤,١ - فنيلين ثنائی (ميثان – ۱ – يل – ۱ – يلدين)) ثتائي (ازان-۱-يل-۱-يلدين) ثتائي(۲,۱-فنيلين)) ثنائي (ازان – ۱ – یل – ۱ – یلدین) نتائی (میثان – ۱ – یل – ۱ – یلدین) نتائی بنزین ۳,۱-ثنائے اول إذ حضر هذا اللبكاند بخطوتين: الخطوة الأولى مفاعلة (ترفثالديهايد) مع (٢,١-فنيلين داي امین) و تکوین ان'،ان'-(٤,١ فنيلين ثنائي(ميثان-١-يل يلدين)) ثنائي بنزین-۲٫۱ ثنائی امین الخطوة الثانية مفاعلة ان ،ان '-(٤,١ فنيلين ثنائي (ميثان-۱-يـل يلـدين)) ثنـائي بنـزين-۲٫۱ ثنـائي امـين مـع ٤,٢ثنائي هايدروكسي بنزالديهايد و تكوين الليكاند الجديد (H<sub>4</sub>L). ٤,٤' – (٢,٢'- (٤,١ - فنيلين ثنائي (ميثان – ١ – يل – ١ – يلدين)) ثنائی(ازان – ۱ – پل – ۱ – پلدین) ثنائی(۲٫۱ –فنیلین)) ثنائی (ازان – ۱ – یل – ۱ – یلدین) ثنائی (میثان – ۱ – یل – ۱ – یلدین) ثنائی بنزين ۳,۱-ثنائي اول تم مفاعلة هذا الليكاند مع بعض العناصر الفلزية باستخدام الميثانول وسطا للتفاعل وبنسبة (١:١) حيث تكونت معقدات جديدة لها الصيغة العامة [M<sub>2</sub>(H<sub>2</sub>L)Cl<sub>2</sub>] M=Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Pd<sup>II</sup> حيث:

شخصت جميع المركبات بالطرق الطيفية، الأشعة تحت الحمراء، والأشعة فوق البنفسجية – المرئية، (H.P.L.C) الحمراء، والأشعة فوق البنفسجية – المرئية، (H.NMR محتوى الكلور ودرجات الانصبار، مع قياس التوصيلية المولارية الكهربائية. كذلك تم استخدام طريقة (النسب المولية) لتعيين نسبة الليكاند الى الفلز وكانت النسبة ١: ٢ وايضا تم دراسة استقرارية المعقدات المحضرة وحساب الدوال الثرموديناميكية، ثابت الاستقرارية K و طاقة جبس الحرة م  $\Delta G$  بناء على نتائج البحث تم اقتراح الشكل الفراغي