

## Synthesis and study of new Macrocyclic ligand type $N_2O_2$ and its complexes with $(Co^{(II)}, Ni^{(II)}, Cu^{(II)}, Zn^{(II)}$ and $Cd^{(II)})$ ions.

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### Abstract

A new Schiff base ligand [L] [3-methyl-9,10 phenyl -6,7 dihydro-5,8 -dioxo-1,2 diazo -cyclo dodecu 2,11-diene ,4-one ] and its complexes with  $(Co^{(II)}, Ni^{(II)}, Cu^{(II)}, Zn^{(II)}$  and  $Cd^{(II)})$  were synthesis. This ligand was prepared in three steps, in the first step a solution of salicylaldehyd in methanol reacted under refluxed with hydrazine monohydrate to give an (intermediate compound 1) which reacted in the second step with sodium pyruvate to give an (intermediate compound 2) which gave the ligand [L] in the three step when it reacted with 1,2- dichloro ethane. The complexes were synthesized by direct reaction of the corresponding metal chloride with the ligand. The ligand and complexes were characterized by spectroscopic methods [IR, UV-Vis, HPLC and atomic absorption], chloride content in addition to conductivity measurement. From the obtained data the propose chemical formula for complexes  $[M(L)]Cl_2$  ( $M= Co^{(II)}, Ni^{(II)}, Cu^{(II)}, Zn^{(II)}$  and  $Cd^{(II)}$ ) are distorted tetrahedral structure about metal ions for the studied complexes.

### Introduction

Schiff base play a central role as chelating ligands in main group an transition metal coordination chemistry(1,2). Transition metal complexes of tetradented Schiff base ligands find applications as models of certain metal enzymes and in catalysis and materials chemistry(3). during the past two decades, considerable attention has been paid to the chemistry of metal complexes of Schiff bases containing nitrogen and other donor atoms (4,5), this may be attributed to their stability, biological activity (6) and potential application in many fields such as oxidation catalysis (7) and electrochemistry (8). In 2005 Halabi and Co-worker (9) prepared a Schiff base ligand derived from  $(N_2O_2)$  from amino - 1,2,3,6 - oxatrizain and salicylaldehyd with some transition metal complexes ( $Ni^{(II)}, Cu^{(II)},$  and  $Pd^{(II)}$ ). In this paper we report the synthesised and characterise of new ligand [3-methyl-9, 10 phenyl -6,7 dihydro-5,8 -dioxo-1,2 diazo -cyclo dodecu 2,11-diene ,4-one] [L] and its complexes with  $Co^{(II)}, Ni^{(II)}, Cu^{(II)}, Zn^{(II)},$  and  $Cd^{(II)}$ . The ligand was prepared in a three steps. To prepare the ligand, the solution of salicylaldehyd in methanol was mixed with hydrazinemonohydrate (1:1) then the resultant of reaction [(1-ortho hydroxy benzyldiene) hydrazine] was added to Sodium pyruvate to give the [2-Sodium pyruvalidene hydrazine, 1-(ortho hydroxy benzyldiene)] was added to 1,2- dichloro ethane to give the mentioned ligand.

## Experimental

Reagents were purchased from Fluka and Rediel – Dehenge Chemical Co.I.R spectra were recorded as (KBr) discs using a Shimadzu 8400 FTIR Spectrophotometer in the range (4000-450)  $\text{cm}^{-1}$ . Electronic spectra of the prepared compounds were measured in the region (200-900) nm for  $10^{-3}\text{M}$  solution in (DMSO) at  $25^{\circ}\text{C}$  using a Shimadzu 160 spectrophotometer with  $1.000\pm 0.001$  cm matched quartz cell . Metal contents of the complexes were determined by atomic absorption (A.A) technique using a Shimadzu A.A 680G atomic absorption spectrophotometer. The chloride contents for complexes was determined by potentiometric titration method on (686-titro processor-665) ,Dosinat-metrom Swiss . The (HPLC) chromatograms of the complexes were obtained by using a Shimadzu 2020. Electrical conductivity measurements of the complexes were recorded at  $25^{\circ}\text{C}$  for  $10^{-3}\text{M}$  solutions in(DMSO) as a solvent using aPW9526digital conductivity meter.

## Preparation

### Synthesis of the ligand [L]

**Step (1):** Preparation of the [(1-ortho hydroxy benzylidene) hydrazine] (intermediate compound 1).

A solution of salicylaldehyde 0.6 g,( 4.913 mmole) in methanol (5ml) was added to hydrazine monohydrate 0.245 g, (4.89 mmole) which was dissolved in methanol (5ml), and then 2-4 drops of glacial acetic acid was added slowly to the reaction mixture. The mixture was refluxed for 5 hrs, then allowed to dry at room temperature for 24 hrs. Deep yellow solid was obtained by evaporation of methanol. Yield (80%), 0.53 g, m.p ( $178^{\circ}\text{C}$ ).

**Step (2):** Preparation of the [2-Sodium pyruvalidene hydrazine, 1-(ortho hydroxy benzylidene)] (intermediate compound 2).

A solution of [(1-ortho hydroxy benzylidene) hydrazine] 0.4g, (2.941mmole) in methanol (5ml) was added to sodium pyruvate 0.32 g, (2.941 mmole) which was dissolved in methanol (5ml), then 2-4 drops of glacial acetic acid was added slowly to the reaction mixture .The reaction mixture was refluxed for 5 hrs with stirring, filtered and then dried at room temperature for 48 hours. A pale yellow solid was obtained. Yield (88%), (0.59) g, m.p  $225^{\circ}\text{C}$ .

**Step(3) :**Preparation of the ligand [L] [3-methyl-9,10 phenyl -6,7 dihydro-5,8 dioxo-1,2diazocyclo dodeca 2,11-diene ,4-one].

A Solution of [2-Sodium pyruvaliden hydrazine, 1-(ortho hydroxy benzylidene) ] 0.6g, (2.631mmole) in methanol (5ml) with 0.10g,( 2.631mmole ) NaOH in methanol was added to 1,2- dichloro ethane 0.26g, (2.631mmole) in methanol .The reaction mixture was refluxed for 5hrs with stirring ,filtered and dried at room temperature for 48 hrs .A orange solid was obtained .Yield (85%), 0.52 g, m.p  $265^{\circ}\text{C}$  .

## Synthesis of (L) complexes .

### Synthesis of [Co (L)] Cl<sub>2</sub>

A solution of (L) 0.2g, (0.862mmole) in methanol (5ml) was added to (5ml) solution of CoCl<sub>2</sub>.6H<sub>2</sub>O 0.2g, (0.862mmole). The resulted mixture was heated under reflux for 3 hrs. The mixture was filtered and the precipitate was washed with an excess of methanol and dried at room temperature for 24 hrs. A mustard solid which decomposed at (175) °C was obtained. Yield (77%), 0.24g, .

### Synthesis of [Ni (L)]Cl<sub>2</sub> (2),[Cu (L)]Cl<sub>2</sub> (3),[Zn (L)]Cl<sub>2</sub> (4) and [Cd (L)]Cl<sub>2</sub> (5) Complexes:-

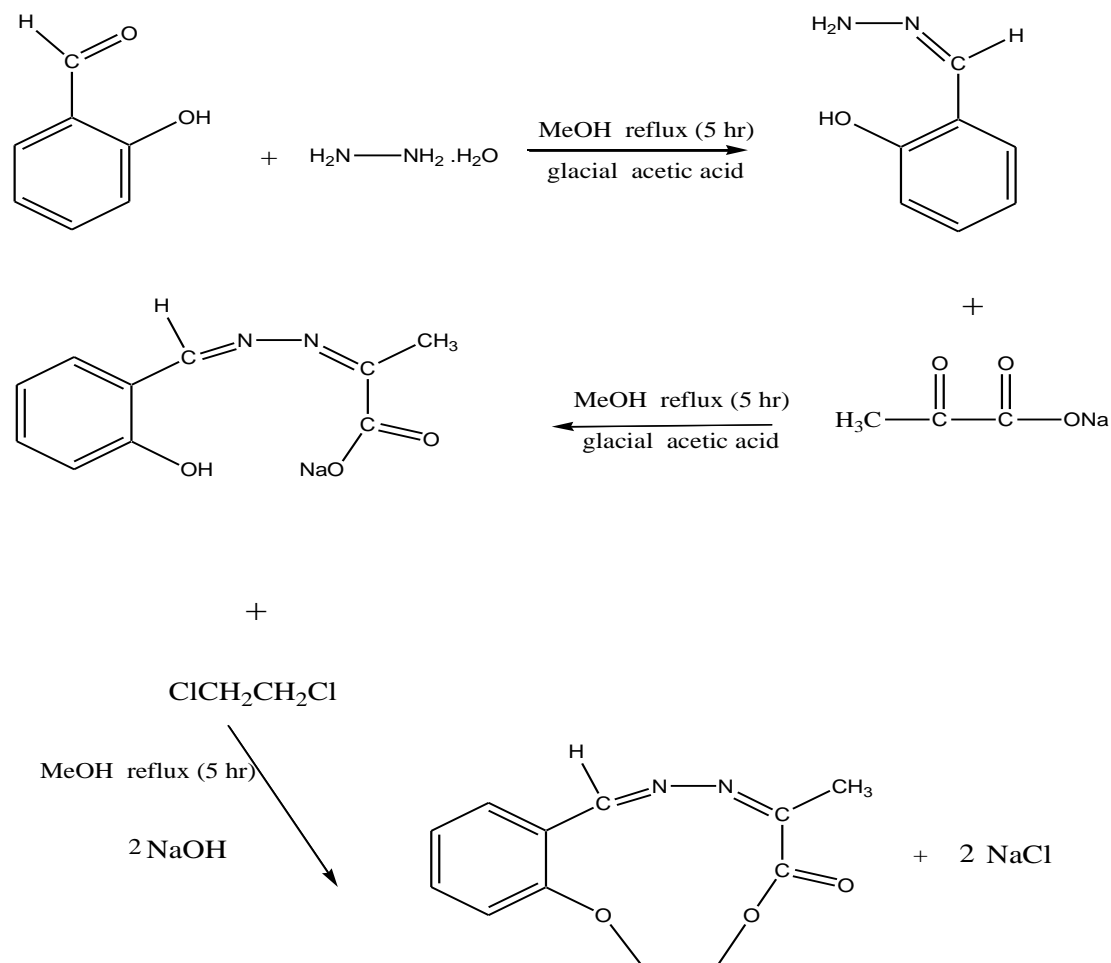
The method used to prepare these complexes was similar to that mentioned in the case of preparation [Co (L)]Cl<sub>2</sub> complex. Table (1) shows the stated weight of starting materials, % yield and some physical properties of the prepared complexes.

**Table (1) some physical properties of the complexes and their reactants quantities.**

Compound	decomposition temperature °C	Color	Metal chloride salt	Weight of chloride salt		Weight of product (g)	Yield %	chloride content	Metal ion % (Prac.) (Theo.)
				g	mmole				
[Co (L)] Cl <sub>2</sub>	170	mustard	CoCl <sub>2</sub> .6H <sub>2</sub> O	0.2	0.862	0.24	77	nil	15.53 (16.28)
[Ni(L)] Cl <sub>2</sub>	260	Yellow green	NiCl <sub>2</sub> .6H <sub>2</sub> O	0.2	0.862	0.22	70	nil	14.44 (16.23)
[Cu(L)] Cl <sub>2</sub>	200	Brown	CuCl <sub>2</sub> .2H <sub>2</sub> O	0.14	0.862	0,21	66	nil	16.57 (17.34)
[Zn(L)] Cl <sub>2</sub>	240	Yellow	ZnCl <sub>2</sub> .2H <sub>2</sub> O	0.14	0.862	0.23	72	nil	16.76 (17.75)
[Cd (L)]Cl <sub>2</sub>	220	Yellow	CdCl <sub>2</sub> . 2H <sub>2</sub> O	0.2	0.862	0.27	77	nil	26.16 (27.06)

## Results and discussion

The new ligand [L] pro-ligand was prepared in three steps according to the general method of preparation of Schiff base ligands as shown in Scheme (1). The (I.R) spectrum for [L] Fig (2) , displays the band at  $1731\text{ cm}^{-1}$  which was attributed to the  $\nu(\text{C}=\text{O})$  stretching vibration (10). The broad band at  $3423\text{ cm}^{-1}$  is due to the  $\nu(\text{HO-H})$  stretching of the hydrogen bonding between oxygen atom in (C=O) group and hydrogen in (CH<sub>3</sub>) group (10,11). The two bands at  $1647$  and  $1620\text{ cm}^{-1}$  are attributed to  $\nu(\text{CH}_3\text{-C}=\text{N})$  and  $\nu(\text{H-C}=\text{N})$  stretching frequency for the imine group vibration(12-14) .The sharp band at  $931\text{ cm}^{-1}$  is attributed to  $\nu(\text{N-N})$  stretching vibration(15). (U.V-Vis) spectrum of the ligand Fig (3) showed three high intense absorption peaks at (264) nm, (  $34013\text{ cm}^{-1}$ ) (  $\epsilon_{\text{max}} = 1846\text{ molar}^{-1}\cdot\text{cm}^{-1}$  ), (344) nm (  $29069\text{ cm}^{-1}$ ) (  $\epsilon_{\text{max}} = 1187\text{ molar}^{-1}\cdot\text{cm}^{-1}$ ) and (372) nm (  $26881\text{ cm}^{-1}$ ) (  $\epsilon_{\text{max}} = 1180\text{ molar}^{-1}\cdot\text{cm}^{-1}$ ) which assigned to overlap of ( $\pi \rightarrow \pi^*$ ), ( $n \rightarrow \pi^*$ ) and ( $n \rightarrow \pi^*$ ) transitions (16).



Scheme (1) the synthesis route of the ligand

The synthesis of the complexes was carried out by the reaction of [L] with  $[MCl_2 \cdot XH_2O]$  [where  $M^{II} = (Co, Ni, Cu, Zn \text{ and } Cd)$  in methanol under reflux. These complexes are stable in solution and electrolyte (1:2) systems in DMSO (Table-3). The analytical and physical data (Table-1) and spectral data (Table-2) are compatible with the suggested structures. The I.R spectra of complexes are presented in (Table-2). The I.R spectra of the complexes(1), (2), (3) (4) and (5) show the bands at 3396, 3415, 3419, 3410 and  $3417\text{cm}^{-1}$  respectively which assigned to  $\nu$  (HO...H ) stretching vibration of the hydrogen bonding between oxygen atom in (C=O) group and hydrogen in (CH<sub>3</sub>) group . This band was shifted to lower frequency in comparison with that of the free ligand at  $3423\text{cm}^{-1}$  (10,11) . The bands at 1720, 1716, 1725, 1718 and  $1716\text{cm}^{-1}$  which are due to  $\nu$ (C=O) Stretching vibration for all complexes respectively.Were shifted to lower

frequency in comparison with that of the free ligand at  $1731\text{ cm}^{-1}$  (17,18). These shifting can be attributed to delocalization of metal electronic density in the  $\pi$  system (19). The strong band in free ligand (L) at  $1647\text{ cm}^{-1}$  for the imine group ( $\text{CH}_3\text{-C=N}$ ) was shifted to lower frequency by  $1616, 1620, 1626, 1606$  and  $1625\text{ cm}^{-1}$  for the complexes (1), (2), (3) (4) and (5) respectively (12-14). The same shifting of ( $\text{H-C=N}$ ) group was appeared in the lower frequency at  $1591, 1606, 1606, 1597$  and  $1606\text{ cm}^{-1}$  showing a reducing in the bond order. This can be attributed to delocalization of metal electronic density in the  $\pi$  system of the ligand ( $\text{HOMO} \rightarrow \text{LOMO}$ ) (19).

Where HOMO = highest occupied molecular orbital.

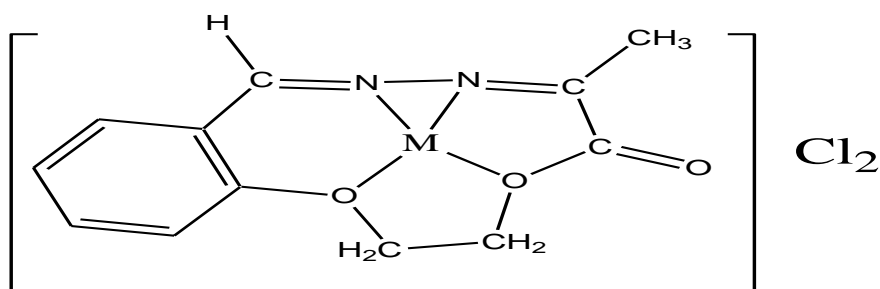
LUMO = lowest unoccupied molecular orbital

The bands at  $962, 945, 946, 970$  and  $985\text{ cm}^{-1}$  were assigned to  $\nu$  (N-N) stretching vibration (12) in the complexes (1), (2), (3), (4) and (5) respectively. The bands at  $(487-588), (586-516), (532-590), (547-570)$  and  $(520-565)\text{ cm}^{-1}$  were assigned to  $\nu$ (M-N) for complexes (1),(2),(3),(4) and (5) respectively indicating that the imine nitrogen is in addition to the oxygen involved in coordination with metal ions (20,21). The bands at  $(435-459), (453-472), (446-497), (440-451)$  and  $(405-459)\text{ cm}^{-1}$  were assigned to  $\nu$ (M-O) for complexes (1),(2),(3),(4) and (5), indicating that the phenolic oxygen of the ligand was involved in coordination with metal ions (21-23). Figs.(2b), (2d) and (2e) represent the (I.R) spectra of  $[\text{Ni(L)}]\text{Cl}_2$ ,  $[\text{Zn(L)}]\text{Cl}_2$ , and  $[\text{Cd(L)}]\text{Cl}_2$ . The (U.V-Vis) absorption spectra data for all complexes are given in (Table-3). In general, the electronic spectra show two intense peaks in the U.V region at  $(300, 322), (299, 344), (300, 343), (293, 344)$  and  $(294, 344)\text{ nm}$  for complexes (1), (2), (3), (4) and (5). These peaks were assigned to ligand field and charge transfer transition respectively (24). The electronic spectrum of complex (1) (Fig-3a) exhibited peak at  $525\text{ nm}$ , which can be attributed to (d-d) transition type ( ${}^4\text{A}_{2(\text{F})} \rightarrow {}^4\text{T}_{1(\text{P})}$ ). The observed weak peak in spectrum of complex (2) is at  $758\text{ nm}$  is assigned to (d-d) transition type ( ${}^3\text{T}_1 \rightarrow {}^3\text{T}_{1(\text{P})}$ ). The spectrum of complex (3) exhibited weak peak at  $404\text{ nm}$ . They can be attributed to (d-d) transition type ( ${}^2\text{B}_2 \rightarrow 2\text{E}$ ). The absence of absorption peaks in the range  $(360-1000)\text{ nm}$  for (U.V-Vis) spectra of complexes (4) and (5) indicate no (d-d) electronic transition happened ( $\text{d}^{10}$ - system) in the visible region (Fig.3b). These U.V-Vis data suggest a distorted tetrahedral configuration around the metal ion for the five studied complexes Fig (1) (25). The molar conductance values were determined in (DMSO) solvent in  $10^{-3}\text{ M}$  at  $298\text{ K}$  which are found at range  $(75-89)\text{ }\Omega\text{m}$  ( $\Omega^1.\text{cm}^2.\text{Mole}^{-1}$ ) (Table -3) indicated electrolytic nature with (1:2) ratio (26). The (HPLC) results of the complexes are presented in table(3). Figs.(4a and 4b) exhibit the chromatograms of  $[\text{Co(L)}]\text{Cl}_2$  and  $[\text{Ni(L)}]\text{Cl}_2$  complexes which show one signal at ( $\text{tr} = 7.679$  and  $6.008\text{ min}$ ) for  $[\text{Co(L)}]\text{Cl}_2$  and  $[\text{Ni(L)}]\text{Cl}_2$  respectively, indicating the purity of the complexes and appear as a single species in solution. The atomic absorption measurements (A.A) and chloride content results for all complexes (table -1) are in a good agreement with the calculated values.

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$M^{(II)} = \text{Co, Ni, Cu, Zn and Cd}$

Figure (1) the suggested structure for the complexes

Table (2) I.R spectral data of the ligand and it's complexes

Compound	$\nu$ (HO..H) Hydrogen bonding	$\nu$ (C=O)	$\nu$ (CH <sub>3</sub> C=N)	$\nu$ (HC=N)	$\nu$ (N-N)	$\nu$ (C-O-C)	M-O M-N	Other bands
[L]	3423	1731	1647	1620	931	1278	- -	$\nu$ (C=C) 1452 $\nu$ (c-H)alph2986 $\nu$ (CH)arom 3050
[Co (L)] Cl <sub>2</sub>	3396	1725	1616	1591	962	1251	435 459 487 588	$\nu$ (C=C) 1446 $\nu$ (c-H)alph2980 $\nu$ (C-H)arom 3010
Ni (L)] Cl <sub>2</sub>	3415	1716	1620	1606	945	1253	453 472 486 516	$\nu$ (C=C) 1442 $\nu$ (c-H)alph 2989 $\nu$ (C-H)arom 3015
Cu (L)] Cl <sub>2</sub>	3419	1725	1626	1606	946	1255	464 497 532 590	$\nu$ (C=C) 1438 $\nu$ (c-H)alph 2927 $\nu$ (C-H)arom 3025
[Zn (L)] Cl <sub>2</sub>	3410	1718	1606	1597	970	1258	440 451 547 570	$\nu$ (C=C) 1390 $\nu$ (c-H)alph 2920 $\nu$ (C-H)arom 3035



[Cd (L)] Cl <sub>2</sub>	3417	1716	1625	1606	985	1248	405 459 520 565	$\nu$ (C=C) 1390 $\nu$ (c-H)alph 2923 $\nu$ (C-H)arom 3045
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**Table (3): Electronic spectral data, and conductance measurement for the ligand [L] and it's complexes**

Compound	$\lambda$ nm	Wave number $\text{Cm}^{-1}$	$\epsilon_{\text{max}}$ Molar $\text{Cm}^{-1}$	Assignment	HPLC Min	$\Delta\epsilon$ ( $\Omega^1 \cdot \text{cm}^2 \cdot \text{Mole}^{-1}$ )	Propose structure
[L]	264	34013	1846	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ $n \rightarrow \pi^*$	-	-	-
	344	29069	1187				
	372	26881	1180				
[Co(L)] Cl <sub>2</sub>	300	33333	1902	charge transfer ${}^4T_{1(P)} \leftarrow {}^4A_{2(F)}$	7.679	75	distorted tetrahedral
	319	31347	1235				
	389	25706	1125				
	525	19047	35				
[Ni (L)] Cl <sub>2</sub>	301	33222	1335	charge t transfer ${}^3T_{1(P)} \leftarrow {}^3T_1$	6.008	82	distorted tetrahedral
	345	28985	1281				
	366	27322	868				
	758	13192	43				
[Cu(L)] Cl <sub>2</sub>	300	33333	2437	charge transfer ${}^2B_2 \rightarrow {}^2E$	-	89	distorted tetrahedral
	343	29154	2302				
	404	24752	549				
[Zn (L)] Cl <sub>2</sub>	293	34129	1625	charge transfer	-	87	distorted tetrahedral
	344	29069	1651				
[Cd (L)] Cl <sub>2</sub>	294	34013	2867	Charge transfer	-	79	distorted tetrahedral
	344	29069	2353				

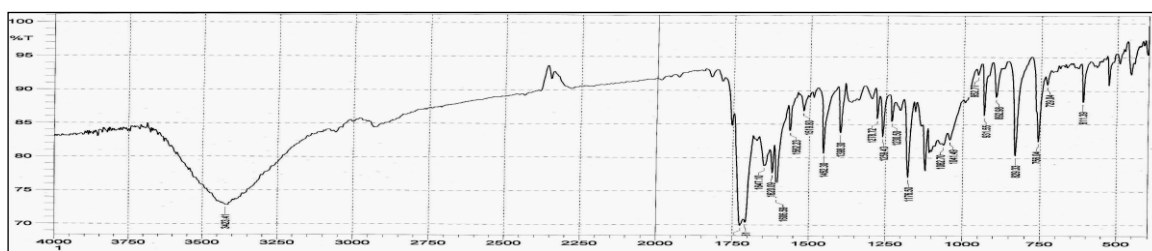


Fig (2) .The I.R. Spectrum of the ligand [L]

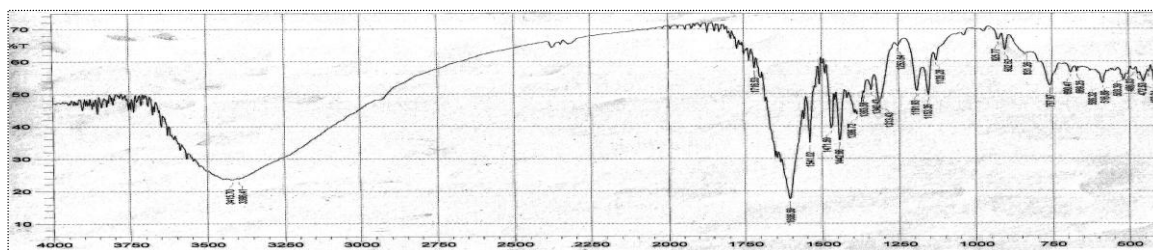


Fig (2b) .The I.R. Spectrum of [Ni (L)] Cl<sub>2</sub>

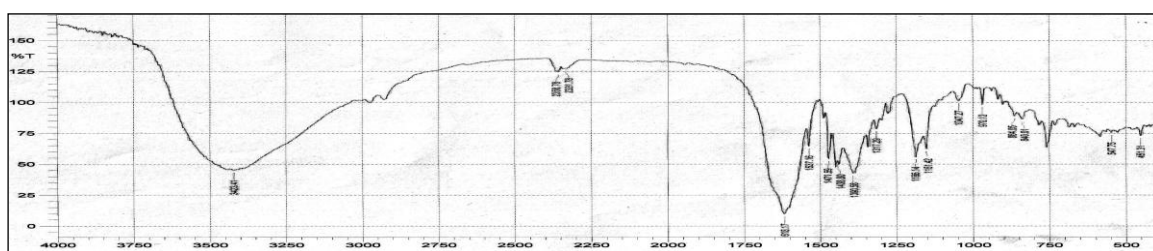


Fig (2d) .The I.R. Spectrum of [Zn (L)] Cl<sub>2</sub>

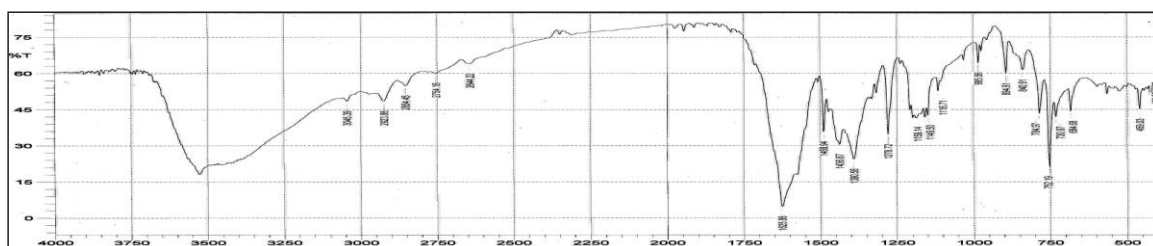


Fig (2e) .The I.R. Spectrum of [Cd (L)] Cl<sub>2</sub>

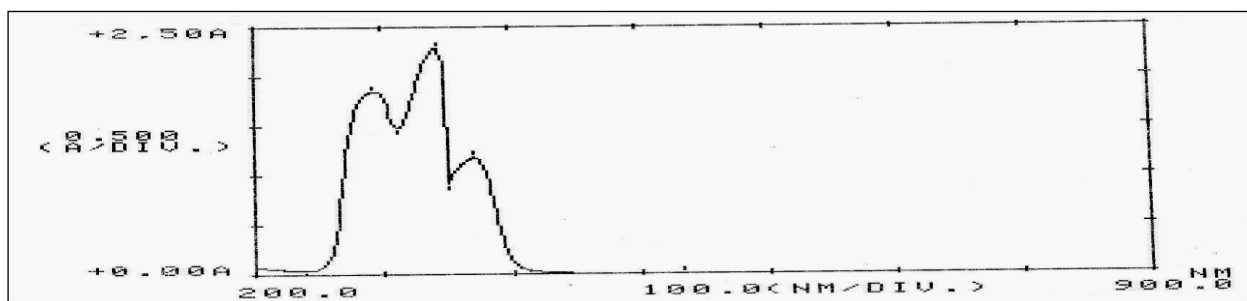


Fig (3) .The U.V Spectrum of the ligand [L]

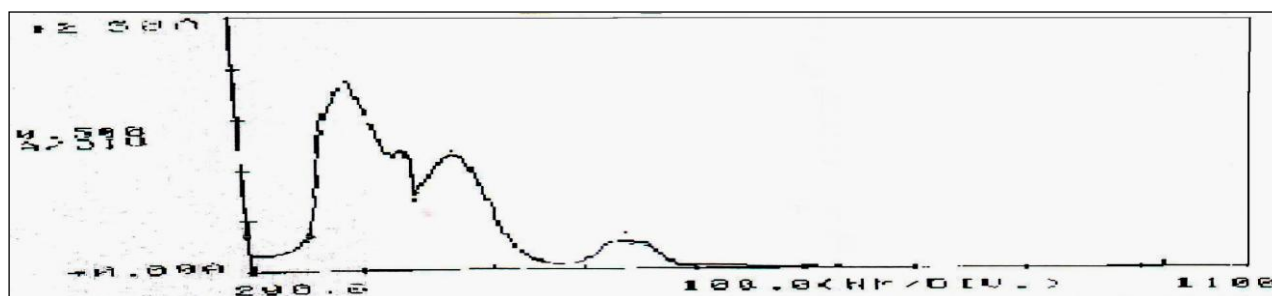


Fig (3a) .The U.V Spectrum of [Co (L)]Cl<sub>2</sub>

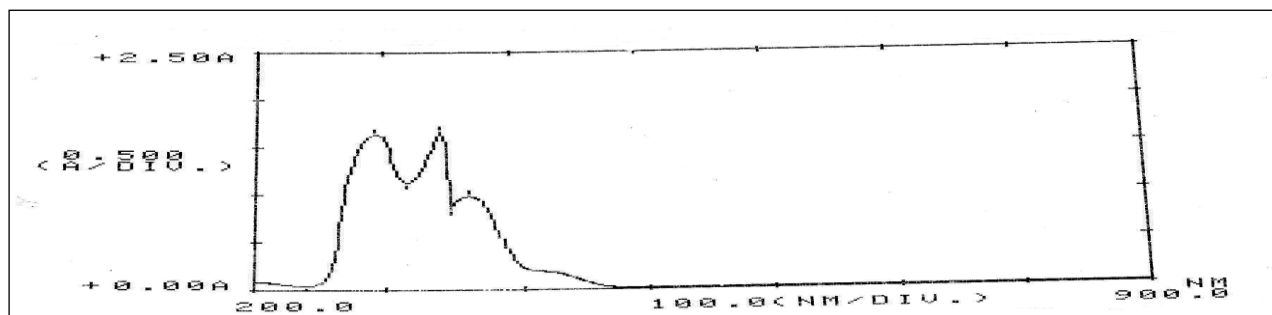


Fig (3b) .The U.V Spectrum of [Zn (L)] Cl<sub>2</sub>

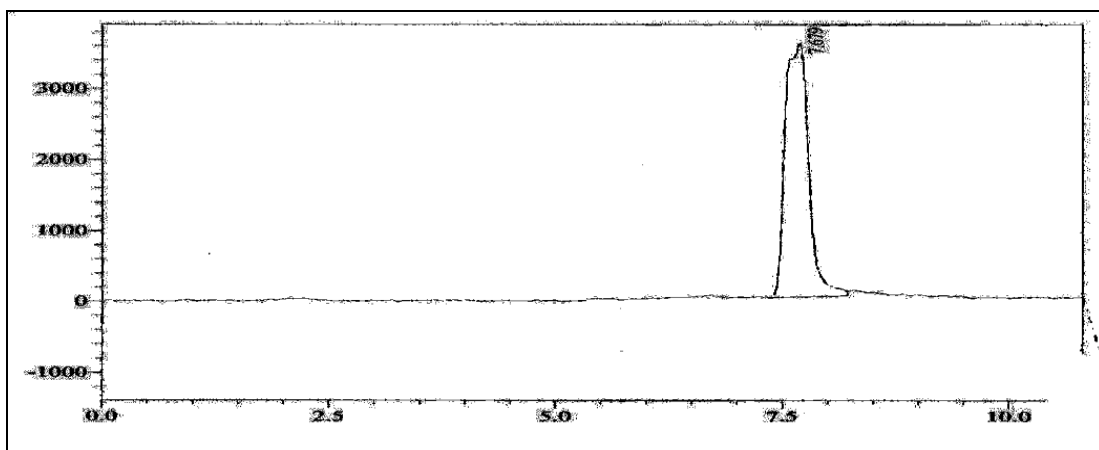


Fig (4a) .The HPLC chromatogram of [Co (L)] Cl<sub>2</sub>

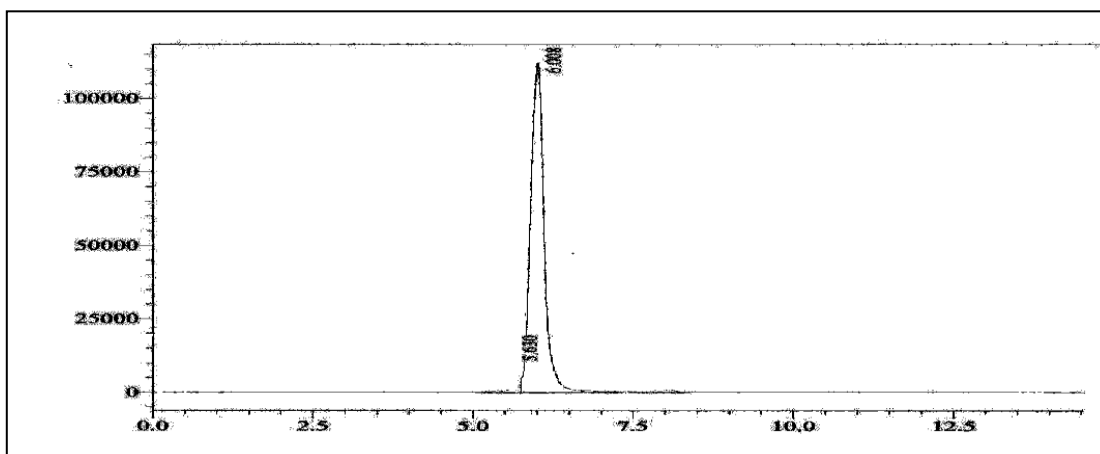


Fig (4b) .The HPLC chromatogram of [Ni (L)] Cl<sub>2</sub>

دراسة و تحضير ليكاند جديد حلقي نوع N<sub>2</sub>O<sub>2</sub> ومعقداته  
مع ايونات  
(Co<sup>(II)</sup>, Ni<sup>(II)</sup>, Cu<sup>(II)</sup>, Zn<sup>(II)</sup>, and Cd<sup>(II)</sup>)

\* أحمد ثابت نعمان ، إنعام إسماعيل يوسف وسامر سعد كاظم

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كلية التربية ابن الهيثم قسم الكيمياء  
العراق - بغداد - الاعظمية P.O.4150

الخلاصة

تضمن البحث تحضير الليكاند الجديد  
[3-methyl-9,10 phenyl -6,7 dihydro-5,8 -dioxo-1,2 diazo -cyclo dodecu 2,11-diene ,4-one ]  
بثلاث خطوات:

الخطوة الاولى مفاعلة ( salicyladehyed ) مع (hydrazine monohydrate)

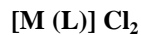
وتكوين [ (1-ortho hydroxy benzylidene )hydrazin ]

والثانية مفاعلة [ (1-ortho hydroxy benzylidene )hydrazine ] مع [ Sodium pyruvate ]

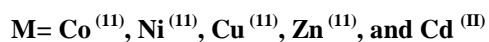
وتكوين [(1-ortho hydroxy benzylidene),1-(2-Sodium pyruvalidene hydrazine)] والثالثة مفاعلته (1,2 dichloro ethane)

مع [2-Sodium pyruvalidene hydrazine ,1-(ortho hydroxy benzylidene)] وتكوين اليكثاند الجديد ثم مفاعلة مع بعض

العناصر الفلزية باستخدام الميثانول وسطا للتفاعل وبنسبة (1:1) حيث تكونت معقدات جديدة لها الصيغة العامة:



حيث:



شخصت جميع المركبات بالطرق الطيفية الأشعة تحت الحمراء ، والأشعة فوق البنفسجية – المرئية ، مطيافية الامتصاص الذري للعناصر وتم تعيين محتوى الكلور ودرجات الانصهار ، مع قياس التوصيلية المولارية الكهربائية . من النتائج أعلاه فإن الشكل الفراغي المقترح للمعقدات هو رباعي السطوح المشوه.