



Synthesis, Characterization Of New Schiff Base And Some Metal Complexes Derived From Glyoxylic Acid And *O*-Phenylenediamine

J. Sh. Sultan

Department of Chemistry, College of Education, Ibn-Al-Haitham, University of Baghdad

Email: ja.sultan@yahoo.com.

Received in : 27 May 2012 Accepted in : 7 August 2012

Abstract

The new Schiff base, namely (2-Amino-phenylimino)-acetic acid (L) was prepared from condensation of glyoxylic acid with *o*-phenylene diamine. The structure (L) was characterized by, IR, ^1H , ^{13}C -NMR and CHN analysis. Metal complexes of the ligand (L) were synthesized and their structures were characterized by Atomic absorption, IR and UV-Visible spectra, molar conductivity, magnetic moment and molar ratio determination (Co^{+2} , Cd^{+2}) complexes. All complexes showed octahedral geometries.

Key words: Synthesis, Characterization, Schiff base, glyoxylic acid, *O*-phenylenediamine and metal ions

Introduction

Glyoxylic acid and its derivatives play important roles in natural processes, participating in glyoxylate cycle which functions in plants and in some microorganism[1-4].

The presence of aldehyde in the glyoxylic acid allows numerous a cyclic derivatives containing C=N bond- azomethines and hydrazones[5-8].

The aim of this work is to synthesize and study the coordination behaviour of the new ligand (2-Amino-phenylimino)-acetic acid (L) and its complexes with Co^{+2} , Ni^{+2} , Cu^{+2} , Cd^{+2} , Hg^{+2} and Pb^{+2} .

Experimental

All chemicals were purchased from BDH, and used without further purifications.

Instrumentation

1. FTIR spectra were recorded in KBr on Shimadzu- 8300 Spectrophotometer in the range of ($4000\text{-}400\text{ cm}^{-1}$).
2. The electronic spectra in H_2O were recorded using the UV-Visible spectrophotometer type (spectra 190-900 nm) CECIL, England, with quartz cell of (1 cm) path length.
3. The melting point was recorded on "Gallen kamp Melting point Apparatus".
4. The Conductance Measurements were recorded on W. T. W. conductivity Meter.
5. Metal analysis. The metal contents of the complexes were determined by atomic absorption (A. A.) technique. Using a shimadzu PR-5. ORAPHIC PRINTER atomic absorption spectrophotometer.



6. Balance Magnetic Susceptibility model MSB-MLI Al-Nahrain University

7. The characterize of new ligand (L) is achieved by:

A: ^1H and ^{13}C -NMR spectra were recorded by using a Bruker 300 MHz (Switzerland).

Chemical Shift of all ^1H and ^{13}C -NMR spectra were recorded in $\delta(\text{ppm})$ unit downfield from internal reference tetramethylsilane (TMS), using D_2O as a solvent.

B: Elemental analysis for carbon, hydrogen was using a Euro Vector EA 3000 A Elemental Analysis (Italy).

C: These analysis (A and B) were done in at AL-al-Bayt University, Al- Mafrag, Jordan.

Synthesis

1. Synthesis of (2-Amino-phenylimino)-acetic acid (L)

To a hot solution of O-phenylenediamine (0.074g, 1m mole) in (5ml) of ethanol, a hot solution of glyoxylic acid (0.108 g, 1 m mole) in (5ml) of ethanol was added. The solution was refluxed for 3.5 hrs. Upon cooling a dark brown precipitate formed, was filtered off and recrystallized from a hot mixture of [(5ml) methanol, (5ml) acetone and (2ml) distilled water]. A dark brown precipitate, yield 85%, melting point 98- 100°C, CHN, C= 58.53 (58.51), H = 4.87 (4.69).

2. Synthesis of complexes

The complex $\text{LCoCl}_2 \cdot 2\text{H}_2\text{O}$ has been synthesized as follows:

To a hot solution of ligand (L) (0.164g, 1m mole) in (5ml) of ethanol, a hot solution of cobalt(II) Chloride. hexa hydrate (0.238g, 1m mole) in (5 ml) of ethanol was added. The precipitate immediately formed, the mixture was boiled and stirring for 10-15 min., filtered off. Recrystallized from a hot of (10ml) methanol, a dark green precipitate, yield 80%, decomposed at 110 D°.

The physical properties for synthesized ligand (L) and its complexes are shown in Table (1).

A similar method was used to prepare other complexes: $\text{LNiCl}_2 \cdot 2\text{H}_2\text{O}$, L(0.164g 1m mole), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.238g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 90% decomposed at 200 D°, $\text{LCuCl}_2 \cdot 2\text{H}_2\text{O}$, L(0.164g, 1m mole), $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.170g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 82% decomposed at 180 D°, $\text{LCdCl}_2 \cdot 2\text{H}_2\text{O}$, L(0.164g, 1m mole), $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ (0.202g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 95% decomposed at 160 D°, $\text{LHgCl}_2 \cdot 2\text{H}_2\text{O}$, L(0.164g, 1m mole), HgCl_2 (0.271g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 78% decomposed at 190 D°, $\text{LPb}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, L(0.164g, 1m mole), $\text{Pb}(\text{NO}_3)_2$ (0.331g, 1m mole), (10ml) ethanol, (10ml) methanol yield 82% decomposed at 210 D°.

IR spectrum of the ligand (L)

The IR spectrum of the (L) Fig. (1) shows new strong bands at (1737, 1668) cm^{-1} are due to $\nu(\text{C}=\text{O})$ of carboxylic group and $\text{HC}=\text{N}$ imine[8-9] compared with the precursors Figs. (2-3), Table (4), which indicate the ligand (L) has been obtained.

Bands corresponding to C-H aromatic stretching at (3061) cm^{-1} [1,5], νNH_2 at (3385, 3363) cm^{-1} are observed[1,3].

Absorption occurs as a sharp peak in the 3466 cm^{-1} is attributed to free (unassociated) hydroxyl- CH_2COOH group[5,7,9].



UV- spectrum of the ligand (L)

The UV- spectrum of (L) Fig. (4), Table (5) was recorded in distilled water with the range (210– 400) nm. The molar absorption at (261) nm may be assigned to an $\pi-\pi^*$ transition[10].

NMR spectrum for the ligand (L)

^1H - NMR spectrum of the ligand (L) in DMSO- d_6 Table (2), Fig. (5) is characterized by the appearance of chemical shift related to the NH_2 protons- aromatic $\delta-\text{NH}_2$ at 5.10 ppm., Chemical shift of aromatic protons showed at δ 6.77–7.539 ppm. The characteristic signals at 8.21 ppm. is assigned to $\text{HC}=\text{N}$. The COOH signal is found at 10.354 ppm.

^{13}C -NMR of the free ligand Table (3), Fig. (6) shows the $\text{HC}=\text{N}$ peak at 143.50 ppm., the COOH peak at 170 ppm. and carbon peaks for aromatic are detected at 110-125 ppm.[5,11,12].

The IR spectra for the complexes

The free ligand exhibits a strong absorption band at $(1737) \text{ cm}^{-1}$ due to the stretching vibration of $\nu(\text{C}=\text{O})$ of the carboxylic group. This band is disappeared in the spectra of its complexes accompanied by the appearance of two bands one in the $(1569-1514) \text{ cm}^{-1}$ range due to $\nu_{\text{asymm.}}(\text{COO}^-)$ and another bands in the $(1398-1375) \text{ cm}^{-1}$ range assigned to $\nu_{\text{symm.}}(\text{COO}^-)$, $\Delta\nu = (171-139) \text{ cm}^{-1}$. Fig. (7), Table (4). This indicates that the carboxylic group is monodentate coordinate[13-14].

The appearance of stretching modes assigned to NH_2 and $\text{HC}=\text{N}$ of $-\text{C}=\text{NH}$ groups was observed at $(3375-3456) \text{ cm}^{-1}$ and $(1660-1620) \text{ cm}^{-1}$ respectively in free ligand[1-3].

The stretching vibration of azomethine group of the ligand was shifted to lower frequencies in all spectra, whereas stretching vibrations of NH_2 getting broad indicating additional coordination of metal ions to NH_2 and $(\text{C}=\text{N})$ group[1-5].

Bands related to coordinate water were observed in all spectra; Cd^{+2} $(704) \text{ cm}^{-1}$, Ni^{+2} $(713) \text{ cm}^{-1}$, Hg^{+2} $(621) \text{ cm}^{-1}$, Cu^{+2} $(669) \text{ cm}^{-1}$, Co^{+2} $(775) \text{ cm}^{-1}$ and Pb^{+2} $(806) \text{ cm}^{-1}$. Additional bands were observed at lower frequencies $(600-400) \text{ cm}^{-1}$ and were attributed to $\text{M}-\text{N}$, $\text{M}-\text{O}$ stretching modes[1-2,5,8].

Lead complex shows band at 1660 cm^{-1} due to the NO_3^- group.

The electronic absorption spectral and magnetic studies

The Co(II) complex exhibited band around $(490) \text{ nm}$ $(20408) \text{ cm}^{-1}$ ($\epsilon_{\text{max}}=300 \text{ molar}^{-1} \text{ cm}^{-1}$) Table (5), which was assigned to $^4\text{T}_{1g(\text{F})} \rightarrow ^4\text{T}_{1g(\text{P})}$, for high- spin octahedral geometry.

The magnetic susceptibility measurements $(4.50) \text{ BM}$ Table (1), for the solid Co(II) complex is indicated of three unpaired electrons per Co(II) ion consistent with its octahedral environment[9,16-17].

The electronic absorption spectrum of the Ni(II) complex showed broad band center at $(460) \text{ nm}$ $(21739) \text{ cm}^{-1}$ ($\epsilon_{\text{max}}=400 \text{ molar}^{-1} \text{ cm}^{-1}$) assigned to the spin-allowed transition $^3\text{A}_{2g(\text{F})} \rightarrow ^3\text{T}_{1g(\text{P})}$ consistent with octahedral configuration[19,22]. The magnetic moment $(2.90) \text{ BM}$ suggested two unpaired electrons per Ni(II) also consistent with octahedral geometry. The electronic absorption spectrum of Cu(II) complex Fig. (8) showed broad band at $(800) \text{ nm}$ $(12500) \text{ cm}^{-1}$ ($\epsilon_{\text{max}}=224 \text{ molar}^{-1} \text{ cm}^{-1}$), which was assigned to $^2\text{E}_g \rightarrow ^2\text{T}_{2g}$ transition, typical for an octahedral



configuration. The magnetic moment (1.80) BM suggested one unpaired electron for Cu(II) consistent with its octahedral environment[18-19].

The spectra of Cd^{+2} , Pb^{+2} and Hg^{+2} complexes exhibited charge transfer bands, which were assigned to a ligand to metal charge transfer^[12,20].

Solutions chemistry

Molar ratio

The complexes of the ligand (L) with selected ions (Co^{+2} , Cd^{+2}) were studied in solution using water as solvents, in order to determine (M:L) ratio in the prepared complexes, following molar ratio method[21].

A series of solutions were prepared having a constant concentration (C) 10^{-3} M of the hydrated metal salts and the ligand (L). The (M:L) ratio was determined from the relationship between the absorption of the observed light and mole ratio (M:L) found to be (1:1). The result of complexes formation in solution are shown in Table (6–9), Fig. (9–10).

To determined ΔG [15]:

$$k = \frac{ML}{[M][L]} \quad (1)$$

$$\alpha = \frac{(A_m - A_s)}{A_m} \quad (2)$$

k = The equation (1) is written to mole ratio (1:1) as the following

$$k_f = \frac{(1 - \alpha)}{\alpha^2 C} \quad (3)$$

$$\Lambda = \epsilon_{\max} \cdot b \cdot c \quad (4)$$

k_f = stability constant

α = decomposition Degree

M = metal ion

L = The ligand

[] = concentration

A_s = The absorption of the equivalent point of mole ratio

A_m = The maximum absorption of the mole ratio

C = The complex concentration (mole. L^{-1}).

$$\Delta G = - 2.303 RT \text{ Log } K.$$

$$R = 8.303$$

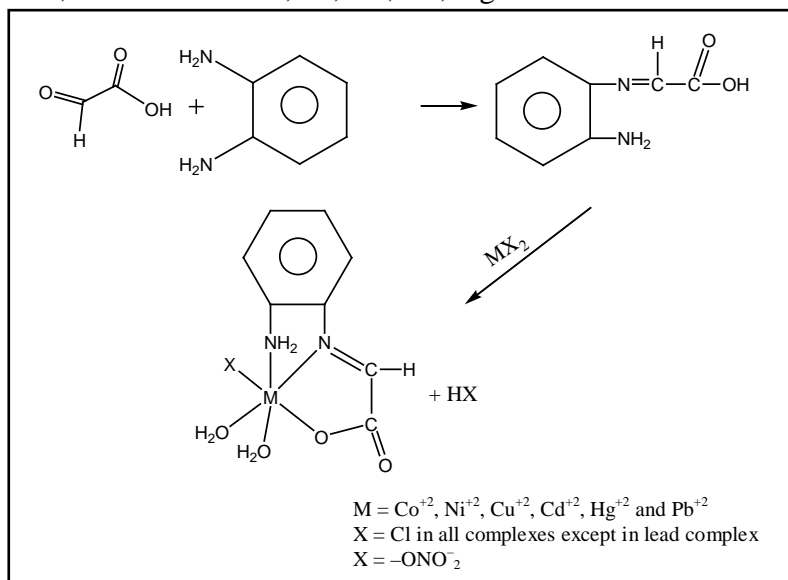
$$T = 273 + 25 = 298$$

Molar conductivity for the complexes of the ligand (L)

The molar conductance of the complexes in water Table (10) lies in the (3.40– 0.61) S. $\text{cm}^2 \text{ molar}^{-1}$ range, indicating their non– electrolyte nature, except for the Cu complex which its molar conductance lies in the (119) S. $\text{cm}^2 \text{ molar}^{-1}$ range, indicating its electrolytic nature with (1:1) ratio[22].

Conclusion

The Schiff base ligand (L) is prepared and characterised by C, H, N and ^1H , ^{13}C -NMR. The ligand (L) is behaved as tridentate mode: NH_2 , $\text{CH}=\text{N}$ and $\text{C}=\text{O}$ forming octahedral complexes with M^{+2} , where $\text{M}^{+2} = \text{Co}, \text{Ni}, \text{Cu}, \text{Cd}, \text{Hg}$ and Pb Scheme below:



References

- Mishchenko, A. V.; Lukov, V. V. and Popov, L. D. (2011) synthesis and physico-chemical study of complexation of glyoxylic acid arolydrazone, with $\text{Cu}(\text{II})$ in solution and solid phase, *Journal of coordination chemistry*, **64**(11):1963- 1976.
- Arif, M.; Qurashi, M. M. R. and Shad, M. A. (2001) metal- based antibacterial agents; synthesis, characterization, and in vitro biological evaluation of cefixime- derived Schiff bases and their complexes with $\text{Zn}(\text{II})$, $\text{Cu}(\text{II})$, $\text{Ni}(\text{II})$ and $\text{Co}(\text{II})$, *Journal of coordination chemistry*, **64**(11):1914-1930.
- Dominik, C. and Branko, K. (2011) schiff base derived from 2-hydroxyl-1-naphthaldehyde and liquid- assisted mechanochemical synthesis of its isostructural $\text{Cu}(\text{II})$ and $\text{Co}(\text{II})$ complexes, *crystengcomm*, **13**: 4351-4357.
- Abdulghani, A. J. and Abbas, N. M. (2011) synthesis characterization and biological Activity study of New Schiff and mannich bases and some metal complexes derived from isatin and dithiooxamide, Hindawi publishing corporation bioinorganic chemistry and applications, p. 1-15.
- Cemal, S.; Zeliha, H. and Hakan, D. (2011) synthesis, characterizations and structure of NO doner Schiff base ligands and nickel(II) and copper(II) complexes, *Journal of Molecular structure*, **977**: 53- 59.
- Anant, P. and Singh, K. K. (2011) synthesis, spectroscopy and biological studies of Nickel(II) complexes with tetradentate shicff basce having N_2O_2 donor group, *J. Dev. Biol. Tissue. Engineering*, **3** (2):13-19.
- Kamellia, N. and Razie, S. (2011) synthesis and mesomorphic of symmetric tetradentate shicff bases based on azo-containing salicylaldimines and their copper (II) complexes, *Journal of coordination chemistry*, **64**(11): 1859-1870.
- Sajjad, M.; Shokoh, B. and Asad, Sh. (2011) Hetero trinuclear manganese (II) and Vanadium (IV) Schiff base complexes, as epoxidation catalysts, *transition met chem.*, **36**: 425-431.

No.	3	Vol.	25	Year	2012	2012	السنة	25	المجلد	3	العدد
-----	---	------	----	------	------	------	-------	----	--------	---	-------

9. Raj, K. D. and Sharad, K. M. (2011) synthesis, spectroscopic and antimicrobial studies of new iron (III) complexes, containing Schiff bases and substituted benzoxazole ligands, *Journal of coordination chemistry*, 64(13):2292-2301.
10. Fleming, I. and William, D. H. (1966) "Spectroscopic methods in organic chemistry", Ed. McGraw Hill publishing company ltd, London.
11. Tajmir, R. (1990) coordination chemistry of vitamin c. part I. Interaction of L-Ascorbic Acid with Alkaline Earth Metal Ions in the Crystalline Solid and Aqueous Solution, *J. Inorg. Biochem*, 40:181-188.
12. Tajmir, R. (1991) Coordination chemistry of vitamin C. part (II). Interaction of L-Ascorbic Acid with Zn(II), cd(II), Hg(II), and Mn(II) Ions in the solid state and in Aqueous solution, *Int. J. Inorg. Biochem*, 42: 47-55.
13. Geeta, B. and Ravinder, V. (2011) synthesis, characterization and biological evaluation of mononuclear Co(II)m Ni(II) and Pd(II) complexes, with New N2O2 schiff base ligand, *chem., pharm. Bull.*, 95 (2):166-171.
14. Washed, M. G.; Refat, M. S. and Megharbel, S. M. (2009) "Synthesis spectroscopic and thermal characterization of some transition metal complexes of folic acid", *spectrochimia acta A*, 70(4): 916–922.
15. Sutton, D. (1968) *Electronic spectra of Transition Metal complexes* Mc GRAW-HILL., London.
16. Malcolm, J. A.; Gordonk, A. and Nigam, P. R. (1999) Synthesis and characterization of platinum (II) complexes of L- Ascorbic Acid, *Inorg. Chem.*, 38:5864-5869.
17. Orgel, L. (1966) "An Introduction to transition metal Chemistry", 2nd ed, Wiley, New York.
18. Rakesh, K. Sh.; Munirathnam, N. and Ashoka, G. S. (2008) Asymmetric allylic alkylation by palladium- bisphosphinites, *Tetrahedron; Asymmetry*, 19:555–663.
19. Lever, P. A. B. (1968) "In organic electronic spectroscopy", Elsevier publishing company, New York, 6:121.
20. Choi, K. Y.; Jeon, Y. M.; Lee, K. C.; Ryu, H.; Suh, M.; Park, H. S.; Kim, M. J. and Song, Y. H. (2004) Preparation and characterization of a bidentate carboxylate bridged dinuclear cadmium(II) complex with bis(2-pyridyl methyl) amino-3-propionic acid, *Journal of Chemical Crystallography*, 34:591-596.
21. Skoog, D. A. and Donald, M. (1974) *Fundamentals of Analytical chemistry* Altoit London Edition.
22. Kettle, S. F. (1975) "Coordination Compounds", Thomas Nelson and Sons, London, P. 165.



Table (1): The physical properties for synthesized lignad (L) and its complexes

Empirical formula	Yield %	M.P. C°	Colour	μ_{effect}	Found (Calc.) % metal	Solubility
L	85	98-100C°	Dark brown		-	Water, methanol, ethanol, ether, DMF, DMSO
LCoCl.2H ₂ O	80	110 D°	Dark green	4.50	(20.00) 19.66	Water, methanol, ethanol, DMF, DMSO
LNiCl.2H ₂ O	90	200 D°	Brown	2.90	(20.00) 19.31	=
LCuCl.2H ₂ O	82	180 D°	Dark brown	1.80	(21.07) 21.80	=
LCdCl.2H ₂ O	95	160 D°	brown	-	(32.18) 32.45	=
LHgCl.2H ₂ O	78	190 D°	pale brown	-	(45.93) 45.46	=
LPbNO ₃ .2H ₂ O	82	210 D°	brown	-	(44.13) 44.50	=

L=C₈H₈O₂N₂, DMF = dimethyl formamide, DMSO = dimethyl sulfoxide, °D = Decomposition

Table (2): ¹H-NMR Chemical shifts for L (ppm in D₂O)

NH ₂	Aromatic proton	HC=N	COOH
5.10 ppm.	6.77-7.539	8.221 ppm	10.354 ppm

Table (3): ¹³C-NMR Chemical shifts for L (ppm in D₂O)

HC=N	COOH	Aromatic carbons
143.50 ppm.	170 ppm	110-125 ppm.

Table (4): Infrared spectral data (wave number ν^-) cm⁻¹ for the ligand (L), precursors and its complexes

Compound	$\nu(\text{OH})$	$\nu(\text{C=O})$	$\nu(\text{NH}_2)$	$\nu(\text{C=N})$	$\nu(\text{C-H})$ Aromatic	$\nu_{\text{assm.}} \text{COO}^-$	$\nu_{\text{symm.}} \text{COO}^-$	Δcm^{-1}	Coordinate water	M-N M-O
Glyoxylic acid	3361	1745	-	-	-	-	-	-	-	-
O-phenylene diamine	-	-	3387 3363	-	3057	-	-	-	-	-
L	3466	1737	3385 3363	1668	3061	-	-	-	-	-
LCoCl.2H ₂ O	-	-	3408 3375	1640 1624	3080	1558 1516	1398 1380	170 136	775	594 468
LNiCl.2H ₂ O	-	-	3404 3375	1660 1614	3060	1560 1520	1396 1380	164 140	713	572 430
LCuCl.2H ₂ O	-	-	3448 3376	1653 1622	3134	1565 1825	1394 1385	171 140	669	549 457
LCdCl.2H ₂ O	-	-	3456 3379	1640 1620	3080	1550 1514	1386 1375	164 139	704	599 424
LHgCl.2H ₂ O	-	-	3450 3380	1650 1620	3064	1541 1516	1398 1375	143 141	621	580 420
LPbNO ₃ .2H ₂ O	-	-	3450 3376	1650 1630	3065	1569 1520	1384 1375	185 145	806	580 450

Table (5): Electronic spectral data of the ligand (L) and its metal complexes

Compound	λ nm	ν^- wave number cm^{-1}	(ϵ_{max} molar cm^{-1})	Assignments	Proposed structure
L	261	38314	325	$\pi \rightarrow \pi^*$	
LCoCl ₂ .2H ₂ O	490	20408	300	${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$	Octahedral
LNiCl ₂ .2H ₂ O	460	21739	400	${}^4A_{2g(F)} \rightarrow {}^4T_{1g(P)}$	=
LCuCl ₂ .2H ₂ O	800	12500	224	${}^2E_g \rightarrow {}^2T_{2g}$	=
LCdCl ₂ .2H ₂ O	267	37453	463	C. T.	=
LHgCl ₂ .2H ₂ O	268	37313	398	C. T.	=
LPbNO ₃ .2H ₂ O	268	37313	398	C. T.	=

Where L=C₈H₈O₂N₂,

C.T.= Charge Transfer

Table (6): VM, VL and Absorption of ligand (L),
VM = volume of metal in ml, VL= volume of ligand in ml

[LCoCl ₂ .2H ₂ O]			[LCdCl ₂ .2H ₂ O]		
VM	VL	Abs	VM	VL	Abs
1 ml	0.25	0.772	1 ml	0.25	0.783
1	0.50	1.251	1	0.50	1.092
1	0.75	1.624	1	0.75	1.455
1	1.00	1.950	1	1.00	1.755
1	1.25	2.050	1	1.25	1.964
1	1.50	2.174	1	1.50	2.084
1	1.75	2.289	1	1.75	2.250
1	2.00	2.404	1	2.00	2.403
1	2.25	2.500	1	2.25	2.558
1	2.50	2.601	1	2.50	2.695

Table (7): The absorbance values against mole- ratio values of complex [LCoCl₂.2H₂O] in solution (1×10^{-3} mole. L⁻¹) in water at λ (272.8) nm

No.	L: M	absorbance
1	0.5:1	1.251
2	1:1	1.950
3	2:1	2.404

Table (8): The absorbance values against mole- ratio values of complex [LCdCl₂.2H₂O] in solution (1×10^{-3} mole. L⁻¹) in water at λ 272.8 nm

No.	L: M	absorbance
1	0.5:1	1.092
2	1:1	1.775
3	2:1	2.403

Table (9): Stability constant and ΔG for the ligand (L) complexes

Compounds	As	Am	α	K	Log K	ΔG
[LCoCl ₂ .2H ₂ O]	1.950	2.404	0.19	22×10^9	10.43	-58.9
[LCdCl ₂ .2H ₂ O]	1.755	2.403	0.27	1×10^4	4	-22.7

[LCoCl₂.2H₂O] > [LCdCl₂.2H₂O]



Table (10): The molar conductance of the complexes*

Compound fragment ions	$\Lambda_m \text{ S.cm}^2 \text{ molar}^{-1}$	ratio
LCoCl.2H ₂ O	1.71	Neutral
LNiCl.2H ₂ O	0.61	Neutral
LCuCl.2H ₂ O	119	1:1
LCdCl.2H ₂ O	1.5	Neutral
LHgCl.2H ₂ O	3.40	Neutral
LPbNO ₃ .2H ₂ O	0.71	Neutral

* Recorded in (water) solvent, where L=C₈H₈O₂N₂

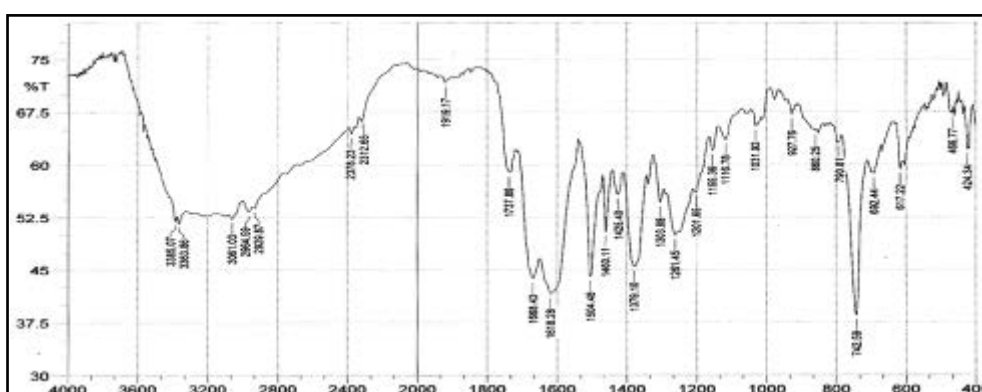


Fig. (1): The IR spectrum of the ligand (L)

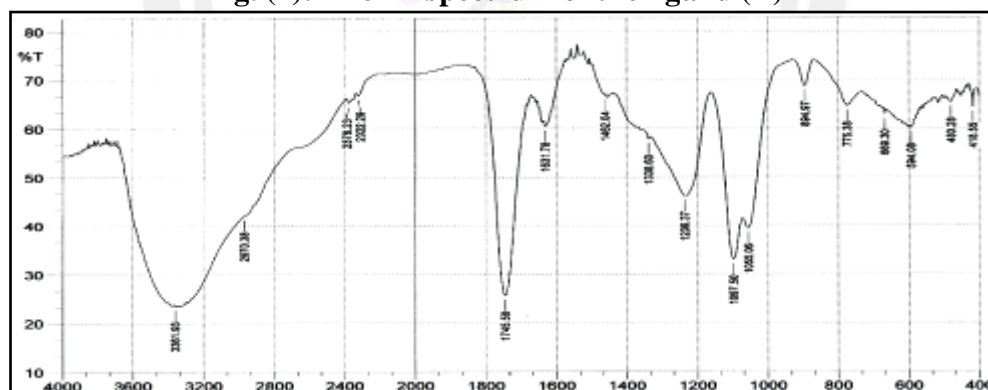


Fig. (2): The IR spectrum of glyoxylic acid

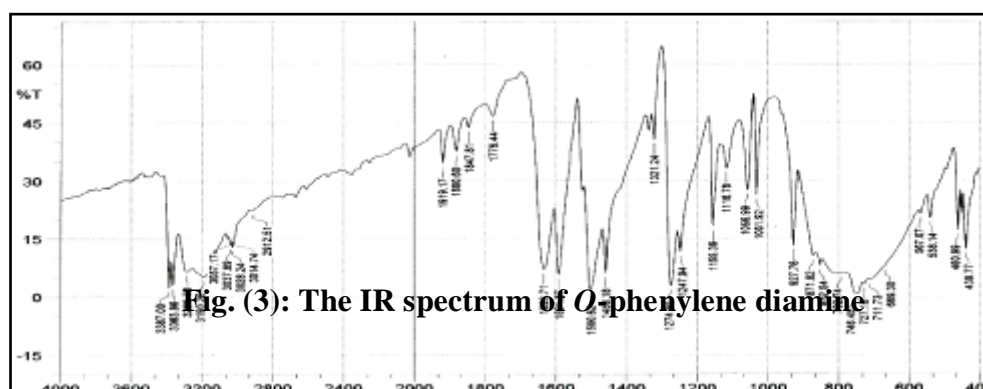


Fig. (3): The IR spectrum of O-phenylene diamine

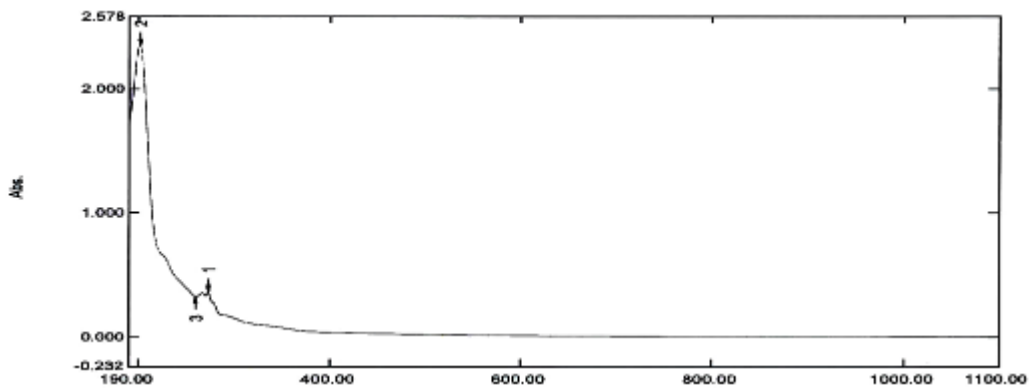


Fig. (4): Electronic spectrum of the ligand (L)

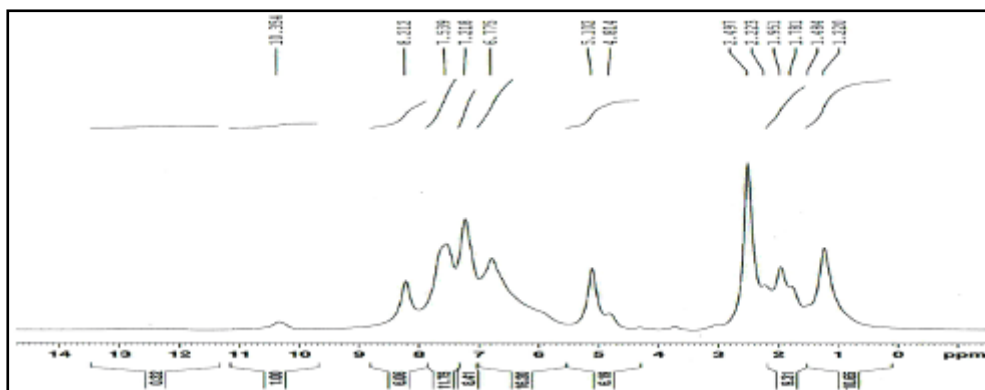


Fig. (5): The ¹H-NMR spectrum of the ligand (L)

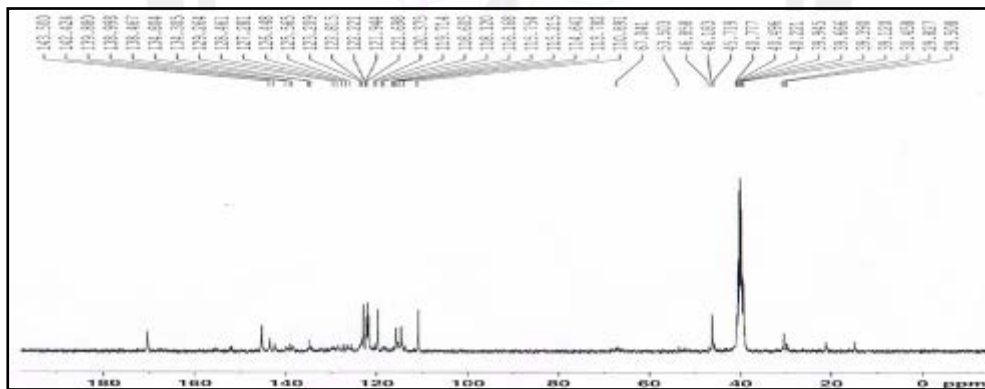


Fig. (6): The ¹³C-NMR spectrum of the ligand (L)

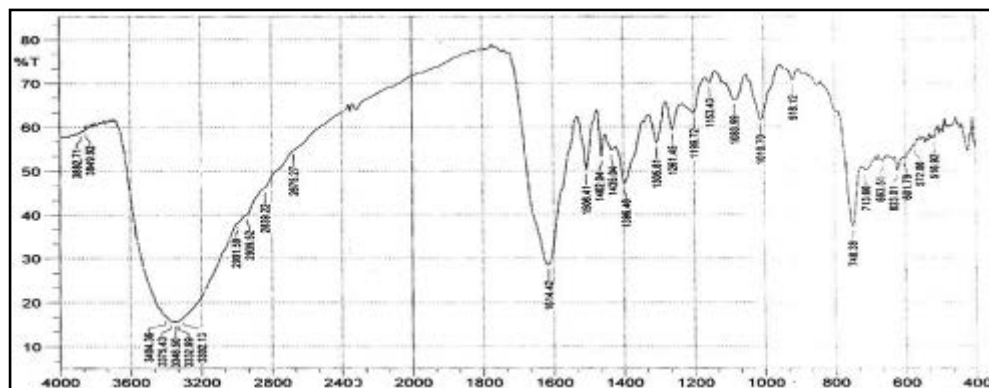
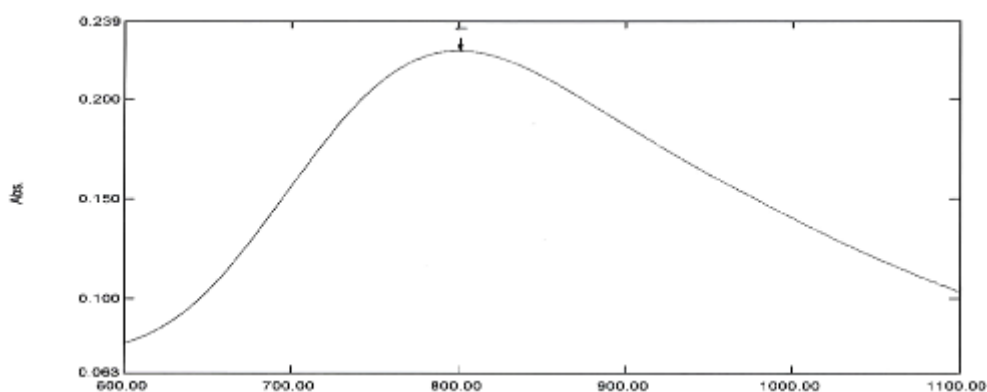
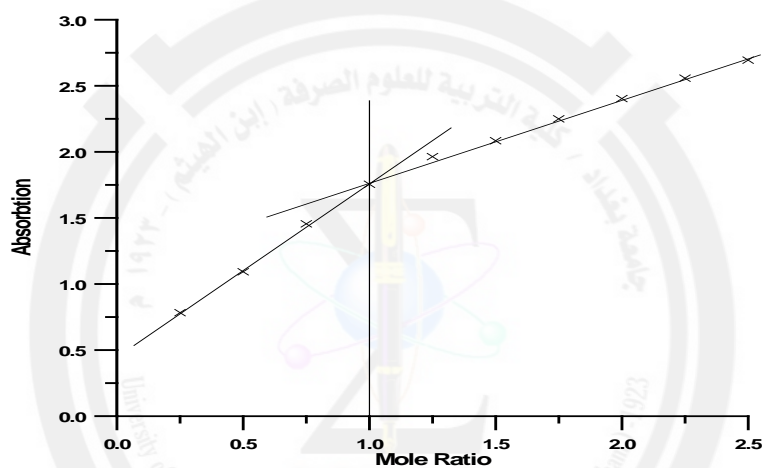
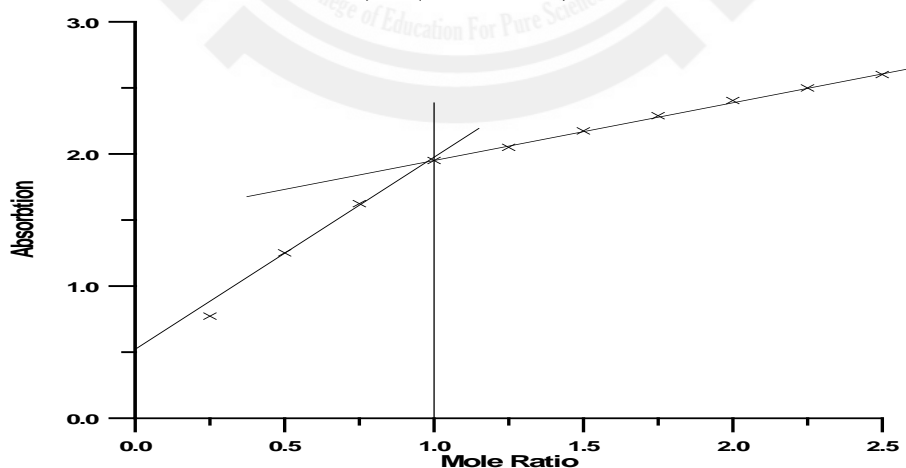


Fig. (7): The IR spectrum of the $(\text{NiCl}_2 \cdot 2\text{H}_2\text{O})$ complexFig. (8): Electronic spectrum of the $(\text{CuCl}_2 \cdot 2\text{H}_2\text{O})$ complexFig. (9): The mole ratio curve of complex $[\text{CoCl}_2 \cdot 2\text{H}_2\text{O}]$ in solution (1×10^{-3} mole. l^{-1}) at $(\lambda = 272.8 \text{ nm})$ Fig. (10): The mole ratio curve of complex $[\text{CdCl}_2 \cdot 2\text{H}_2\text{O}]$ in solution (1×10^{-3} mole. l^{-1}) at $(\lambda = 272.8 \text{ nm})$



تحضير، تشخيص قاعدة شف جديدة وبعض المعقدات الفلزية المشتقة من حامض الكلايوكسيلك واورثو- فنيلين داي امين

جاسم شهاب سلطان

قسم الكيمياء، كلية التربية، ابن الهيثم، جامعة بغداد

استلم البحث في: 27 آيار 2012 قبل البحث في: 7 آب 2012

الخلاصة

قاعدة شف جديدة، (2-أمينو-فنيل إمينو)- حامض الخليك (L) حضرت من تكاثف حامض الكلايوكسيلك مع اورثو- فنيلين داي امين، وتم تشخيصها بواسطة اطياف الأشعة تحت الحمراء، وطياف الرنين النووي المغناطيسي ^1H , ^{13}C -NMR وتحليل العناصر كاربون، هيدروجين و نيتروجين (C.H.N). حضرت وشخصت معقدات الليكاند (L) بواسطة الامتصاص الذري، طيف الاشعة تحت الحمراء، طيف الأشعة المرئية، التوصيلية المولارية، الحساسية المغناطيسية والنسبة المولية لمعقدي الكوبلت والكادميوم الثنائية التكافؤ. كل المعقدات أعطت شكل ثماني السطوح.

الكلمات المفتاحية: تحضير، تشخيص، قاعدة شف، حامض الكلايوكسيلك، ارثو- فنيلين داي أمين وايونات الفلزات.