

Synthesis and Characterization of [2-(carboxy methylene-amino)-phenyl imino] acetic acid (L) and its some metal complexes

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Abstract

New Schiff base, namely [2-(carboxy methylene-amino)-phenyl imino] acetic acid (L) and its some metal complexes [LCo.2H₂O], [LNi.2H₂O], [LCu].3H₂O, [LCd.2H₂O], [LHg.2H₂O] and [LPb.2H₂O], were reported and characterized by elemental analysis, metal content, spectroscopic methods, magnetic moments and conductivity measurements, it is found that the geometrical structures of these complexes are octahedral [Co(II), Ni(II), Cd(II), Hg(II), Pb(II)] and square planar Cu(II). The complexes have been found to possess 1:1 (M:L) stoichiometry

Key words: Synthesis, characterization, New Schiff base and its complexes.

Introduction:

N-substituted imines, also known as Schiff bases, have been used extensively as ligands in the field of coordination chemistry, furthermore the Schiff bases are very important tools for the inorganic chemists as these are widely used to design molecular ferromagnets, in catalysis, in biological modeling applications, as liquid crystals and as heterogeneous catalysts[1,2]. Glyoxilic acid and its derivatives play important roles in natural processes, participating in glyoxylate cycle which functions in plants and in some microorganisms[3]. Physical-chemical study of complexation of glyoxilic acid aroyl hydrazones with Cu(I) in solution and solid phase is reported[4]. In continuation of earlier work[5], we report Schiff base derived by the condensation of o-phenylenediamine with glyoxylic acid (1:2) and its metal complexes.

Materials and Methods:

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 (4000-400 cm⁻¹).
2. The electronic spectra in H₂O 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.

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6. Balance Magnetic Susceptibility model MSB-MLI Al-Nahrain University
7. The characterization 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 analyses for carbon, hydrogen for ligand and its

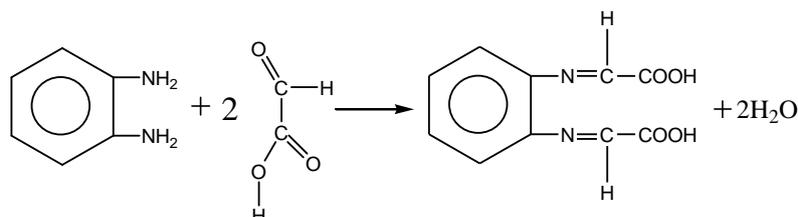
complexes were using a Euro Vector EA 3000 A Elemental Analysis (Italy).

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

Synthesis 1.

Ligand Synthesis

The ligand (L) has been synthesized by the condensation of glyoxylic acid with *o*-phenylenediamine as a Scheme:



To a hot solution of *o*-phenylenediamine (0.108g, 1m mole) in (5ml) of ethanol, a hot solution of glyoxylic acid (0.15 g, 2 m mole) in (15ml) of ethanol was added. The solution reflux for 3.5 h., upon cooling a precipitate formed, was filtered off and recrystallized from a mixture of methanol: acetone: distilled water (5:5:2) giving light brown crystals, yield (86%), m.p. (170) $^{\circ}\text{C}$

2. Synthesis of complexes

The complex $[\text{LCo}.2\text{H}_2\text{O}]$ has been synthesized as follows:

To a hot solution of ligand (L) (0.220g, 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 precipitation immediately formed, the mixture was boiled and stirring for 10-15min., filtered off. Recrystallized from a hot of (10ml) methanol, a dark green precipitate, yield 90%, melting point at 170 $^{\circ}\text{D}$.

A similar method was used to prepare other complexes: $[\text{LNi}.2\text{H}_2\text{O}]$, L(0.220g, 1m mole), $\text{NiCl}_2.6\text{H}_2\text{O}$ (0.238g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 92% decomposed at 120 $^{\circ}\text{D}$, $[\text{LCu}].3\text{H}_2\text{O}$, L(0.220g, 1m mole), $\text{CuCl}_2.2\text{H}_2\text{O}$ (0.170g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 88% melting point 150 $^{\circ}\text{C}$, $[\text{LCd}.2\text{H}_2\text{O}]$, L(0.220g, 1m mole), $\text{CdCl}_2.\text{H}_2\text{O}$ (0.202g, 1m mole), (10ml) ethanol, (10ml) methanol, $[\text{LHg}.2\text{H}_2\text{O}]$, L(0.220g, 1m mole), HgCl_2 (0.271g, 1m mole), (10ml) ethanol, (10ml) methanol, yield 80%, melting point 140 $^{\circ}\text{C}$, $(\text{LPb}.2\text{H}_2\text{O})$, L(0.220g, 1m mole), $\text{Pb}(\text{NO}_3)_2$ (0.331g, 1m mole), (10ml) ethanol, (10ml) methanol yield 85% melting point 230 $^{\circ}\text{C}$.

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

Results and Discussion:

The Schiff base (L) was synthesized in one step. The structure

of (L) was checked and confirmed by elemental analyses data which are in good agreement with proposed formula $C_{10}H_8N_2O_4$. The 1H -NMR illustrated in Fig. (1) and Table (2a) is complicated multiplets corresponding to aromatic four protons at 7.37- 7.79 ppm.

The proton of carboxylic group is slightly broad at 12.5 ppm. the two imine proton $HC=N$ is singlet and appeared at 8.20 ppm., the peaks at (5.2, 3.5 and 2.5) ppm. are due to water[6], water in DMSO and undeuterated DMSO.

^{13}C -NMR is shown in Fig. (2) and Table (2b) the peak at 159 ppm. belong to $HC=N$, the aromatic carbon peaks at (110-130) ppm, the COOH peak at 170 ppm[7-8]. IR spectrum at the (L) Fig. (3) shows disappearance of νNH_2 (3381, 3363) cm^{-1} and appeared new strong bands at (1699 and 1683) cm^{-1} are due to $\nu(C=O)$ of carboxylic group and $HC=N$ imine[9-10]. The stretching band of middle intensity at (3600-3330) cm^{-1} attributed to $\nu(OH)$ of carboxylic group compared with precursors Fig. (4-5). Table (3), which indicates the ligand (L) has been obtained. Bands corresponding to C-H aromatic stretching at (3118- 3000) cm^{-1} .

Band at 750 cm^{-1} is due to ortho disubstituted phenyl[1,11].

The UV- spectrum of (L) Fig. (6) Table (4) was recorded in distilled water gave molar absorption at (340) nm (29411) cm^{-1} ($\epsilon_{max}=409$ molar $^{-1}$ cm^{-1}) and (229) nm (43668) cm^{-1} ($\epsilon_{max}=2347$ molar $^{-1}$ cm^{-1}) may be assigned to the $n-\pi^*$ and $\pi-\pi^*$ [12].

The IR, UV spectral and magnetic moments of complexes

Elemental analysis with metal analysis (A.A) compatible with the formula ($C_{10}H_8N_2O_4$) M. nH_2O where (n=2 or 3).

The bonding of the synthesized ligand (L) to metal ions was investigated by comparing FT-IR spectra of complexes with the free ligand (L). Some important absorption bands, and their assignments are given in Table (3) and Fig. (7).

The spectra of these complexes exhibited a broad band around (3363-3120) cm^{-1} assigned to water[8] $\nu(OH)$ associated with the complexes.

In addition to these modes coordinated water exhibited $\nu(H_2O)$ rocking at (979-894) cm^{-1} all complexes except Cu (II) complex[13].

The band attributed to $\nu HC=N$ shifted to low frequencies by (71-46) cm^{-1} Table (3), supporting the idea that the ligand coordinate through the imine nitrogen[10-11].

The $\nu(C=O)$, ν_{asym} . (COO) and ν_{sym} (COO) stretching vibrations of the carboxylate O are observed at (1699,1541 and 1373) cm^{-1} for the free ligand (L), these stretching vibrations are shifted to lower or higher frequencies at (1674-1612) cm^{-1} , (1577-1546) cm^{-1} and (1400-1384) cm^{-1} for all the complexes, ($\Delta_{asym}-\Delta_{sym}$)=(187-146) cm^{-1} , supporting the idea that the ligand coordinate through deprotonated oxygen of carboxylate [14-15].

New bands are found in the spectra of the complexes in the regions (540-489) cm^{-1} which are assigned to $\nu(M-O)$ mode[7].

The bands at (459 – 424) cm^{-1} have been assigned to $\nu(M-N)$ mode[10].

Therefore from IR spectra, it is concluded that the ligand behave as anion tetradentate and bind to the metal ions via the two imine N and two carboxylate O.

The electronic spectrum of the Co(II) complex is observed multiple bands in the visible region near (462)nm (21645 cm^{-1}) ($\epsilon_{max}=3895$ molar $^{-1}$ cm^{-1}) and (485) nm

(20618) cm^{-1} ($\epsilon_{\text{max}}=3505 \text{ molar}^{-1} \text{ cm}^{-1}$) Fig. (8) Table (4). This may be assigned to the ${}^3\text{T}_{1\text{g}(\text{P})} \leftarrow {}^4\text{T}_{1\text{g}(\text{v}_3)}$ transition in admixture with spin forbidden transition to doublet states derived principally from the free ion ${}^2\text{G}$ and ${}^2\text{H}$ terms for a high spin octahedral geometry[16].

The magnetic susceptibility measurement for the solid Co(II) complex is (4.90) B.M. Table (1) also is indicative of four unpaired electron per Co(II) ion suggesting consistency with its octahedral environment.

The electronic spectrum of the Ni(II) complex showed d-d bands in the regions (651-718)nm center at (684)nm (14619 cm^{-1} ($\epsilon_{\text{max}}=64 \text{ molar}^{-1} \text{ cm}^{-1}$) and (404-486)nm center at (445)nm (22471 cm^{-1} ($\epsilon_{\text{max}}=3000 \text{ molar}^{-1} \text{ cm}^{-1}$) and are assigned to ${}^3\text{T}_{1\text{g}(\text{F})} \leftarrow {}^3\text{A}_{1\text{g}(\text{F})}$ and ${}^3\text{T}_{1\text{g}(\text{P})} \leftarrow {}^3\text{A}_{2\text{g}(\text{F})}$ transition, consistent with its octahedral configuration[17].

Ni(II) complex showed the magnetic moment value (3.20) B.M.[18-19].

The electronic spectrum of the Cu(II) complex showed a broad band center at (804) nm (12437 cm^{-1} ($\epsilon_{\text{max}}=339 \text{ molar}^{-1} \text{ cm}^{-1}$) attributed to ${}^2\text{A}_{1\text{g}} \leftarrow {}^3\text{B}_{1\text{g}}$ transition and a broad band center at (458) nm (21834 cm^{-1} ($\epsilon_{\text{max}}=3999 \text{ molar}^{-1} \text{ cm}^{-1}$) attributed to

${}^2\text{E}_{\text{g}} \leftarrow {}^2\text{B}_{1\text{g}}$ transition of square planar environment[20].

The magnetic susceptibility measurement of Cu(II) complex is (1.90) B.M., which suggests the presence of one unpaired electron with square planar configuration.

The spectra of the Cd, Hg and Pb exhibited charge transfer bands only which is a common phenomenon for metal complexes, where d-d transitions are excluded[21].

According to the elemental analysis Table (1) and FT-IR spectra, the structures of these complexes can be suggested octahedral[22] except Cu(II) complex is square planar.

Solutions chemistry

Molar ratio

The complexes of the ligand (L) with selected ions (Cu^{+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[23].

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 show in Table (5-8), Fig. (9-10).

To determined ΔG [24]:

$$k = ML/[M][L] \quad \dots \quad (1)$$

$$\alpha = (A_m - A_s)/A_m \quad \dots \quad (2)$$

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

$$k_f = (1 - \alpha)/\alpha^2 C \quad \dots \quad (3)$$

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

$k_f =$ stability constant

$\alpha =$ 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)

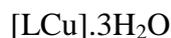
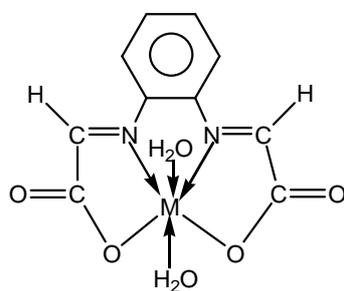
The molar conductance of the complexes in water Table (9) lie in the (130-180) $S.cm^2 \text{ molar}^{-1}$ range, indicating its electrolytic nature with (1:1) ratio[25].

Conclusion:

The new Schiff (L) and metal complexes where prepared [LCo.2H₂O], [LNi.2H₂O], [LCu].3H₂O, [LCd.2H₂O],

[LHg.2H₂O] and [LPb.2H₂O]. The metal (II) ions are coordinated by two carboxylate -O atoms and two imine (H C= N) atoms.

Spectroscopic, structural and magnetic data show that all complexes are six-coordinate metal complexes owing to the ligation of tetradentate Schiff base moieties with two coordinated water except [LCu].3H₂O showed square planar geometry as follow:



Where $M^{II} = Co, Ni, Cd, Hg, Pb$

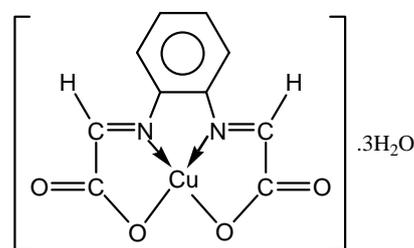


Table (1): The physical properties for synthesized lignad (L) and its complexes

Empirical formula	Yield %	M.P. C°	Colour	μ_{effect}	Found (Calc.) %				Solubility
					C	H	N	metal	
$L=C_{10}H_8N_2O_4$	86	170	Light brown	-	(54.60) 54.54	(3.64) 3.63	(13.06) 12.72	-	Water, methanol, ethanol, ether, acetone, DMF, DMSO
[LCo.2H ₂ O]	90	170	Dark green	4.90	(37.61) 38.09	(3.61) 3.19	(9.21) 8.94	(18.20) 18.84	Water, methanol, ethanol, cetone DMF, DMSO
[LNi.2H ₂ O]	92	120 D°	Pale brown	3.20	(37.63) 38.09	(3.61) 3.19	(8.91) 8.94	(18.31) 18.84	=
[LCu].3H ₂ O	88	150	Redish brown	1.90	(36.84) 37.85	(2.09) 2.73	(7.62) 7.65	(30.11) 30.60	=
[LCd.2H ₂ O]	80	240 D°	Pale brown	-	(32.00) 32.78	(2.09) 2.73	(7.62) 7.65	(30.11) 30.60	=
[LHg.2H ₂ O]	82	140	Pale brown	-	(26.68) 26.43	(2.25) 2.20	(6.76) 6.16	(44.80) 44.11	=
[LPb.2H ₂ O]	85	230	Pale brown	-	(25.71) 26.03	(1.86) 1.30	(6.61) 6.07	(44.20) 44.90	=

Table (2a): ¹H-NMR Chemical Shifts for Ligand (L) (ppm in D 20)

Aromatic protons	Water	Water in DMSO	Undeaurated DMSO	HC=N	COOH
7.37-7.79 ppm	5.2 ppm	3.5 ppm	2.5 ppm	8.20 ppm	12.5 ppm

Table (2b): ¹³C-NMR Chemical Shifts for Ligand (L) (ppm in D 20)

Aromatic carbons	COOH	HC =N
110-130 ppm	170 ppm	159 ppm

Table (3): Infrared spectral data (wave number ν^-) cm^{-1} for the ligand, precursors and its complexes

Compound	$\nu(\text{OH})$	$\nu(\text{NH}_2)$	$\nu(\text{C}=\text{N})$	$\nu(\text{C-H})$ Aromatic	$\nu(\text{C}=\text{O})$	$\nu_{\text{asym.}} \text{COO}^-$	$\nu_{\text{symm.}} \text{COO}^-$	Δcm^{-1}	Coordinate water	M-N	M-O
Glyoxylicacid	3361				1745						
o-phenylenc diamine		3387 3363		3057							
$\text{L}=\text{C}_{10}\text{H}_8\text{N}_2\text{O}_4$			1683	3118 3000	1699	1541	1373				
$[\text{LCo}.2\text{H}_2\text{O}]$	3163		1614	3188 3014	1660	1554	1396	158	894	511	424
$[\text{LNi}.2\text{H}_2\text{O}]$	3363		1635	3100 3010	1635	1546	1400	146	894	511	459
$[\text{LCu}].3\text{H}_2\text{O}$	3182		1637	3057 3020	1674	1577	1390	187	896	520	440
$[\text{LCd}.2\text{H}_2\text{O}]$	3120		1614	3109 3100	1614	1590	1388	146	914	489	426
$[\text{LHg}.2\text{H}_2\text{O}]$	3122		1612	3122 3047	1612	1546	1386	160	979	516	424
$[\text{LPb}.2\text{H}_2\text{O}]$	3124		1620	3124 3059	1620	1546	1384	162	902	540	424

Table (4): Electronic spectral data of the Ligand (L) and its complexes

Compound	λnm	ν^- wave number cm^{-1}	$(\epsilon_{\text{max}} \text{molar}^{-1} \text{cm}^{-1})$	Assignments	Proposed structure
$\text{L}=\text{C}_{10}\text{H}_8\text{N}_2\text{O}_4$	340 229	29411 43668	409 2347	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	
$[\text{LCo}.2\text{H}_2\text{O}]$	462 485	21645 20.618	3825 3505	${}^4\text{T}_{1g(\text{P})} \leftarrow {}^4\text{T}_{1g}$	Octahedral
$[\text{LNi}.2\text{H}_2\text{O}]$	684 445	14619 22471	64 3000	${}^3\text{T}_{1g(\text{F})} \leftarrow {}^3\text{A}_{2g(\text{F})}$ ${}^3\text{T}_{1g(\text{P})} \leftarrow {}^3\text{A}_{2g(\text{F})}$	Octahedral
$[\text{LCu}].3\text{H}_2\text{O}$	804 458	12437 21834	339 3999	${}^2\text{A}_{1g} \leftarrow {}^2\text{B}_{1g}$ ${}^2\text{E}_g \leftarrow {}^2\text{B}_{1g}$	Square planar
$[\text{LCd}.2\text{H}_2\text{O}]$	342	29239	664	C. T.	Octahedral
$[\text{LHg}.2\text{H}_2\text{O}]$	342	29239	664	C. T.	Octahedral
$[\text{LPb}.2\text{H}_2\text{O}]$	342	29239	664	C. T.	Octahedral

Were C.T. = Charge Transfer

Table (5): VM, VL and absorption of ligand (L).
 VM= volume of metal in ml, vl= Volume of ligand in ml

[LCu].3H ₂ O			[LCd.2H ₂ O]		
VM	VL	Abs	VM	VL	Abs
1ml	0.25	0.829	1ml	0.25	0.494
1	0.50	1.100	1	0.50	0.621
1	0.75	1.140	1	0.75	0.763
1	1.00	1.280	1	1.00	0.862
1	1.25	1.283	1	1.25	0.865
1	1.50	1.288	1	1.50	0.868
1	1.75	1.285	1	1.75	0.869
1	2.00	1.285	1	2.00	0.867
1	2.25	1.286	1	2.25	0.868
1	2.50	1.285	1	2.50	0.869

Table (6): The absorbance values against mole-ratio values of complex [LCu].3H₂O in solution (1×10^{-3} mole. L⁻¹) in water at λ (270.5) nm

NO.	L: M	Absorbance
1	0.5:1	0.100
2	1:1	1.280
3	2:1	1.285

Table (7): The absorbance values against mole- values of complex [LCd.2H₂O] in solution (1×10^{-3} mole. L⁻¹) in water at λ (272.5) nm

NO.	L: M	Absorbance
1	0.5:1	0.621
2	1:1	0.862
3	2:1	0.867

Table (8): stability constant and ΔG for the Ligand (L) complexes

Compounds	As	Am	α	K	Log K	1/K	ΔG
[LCu].3H ₂ O	1.280	1.285	0.004	6.2×10^7	7.7	0.13	-43
[LCd.2H ₂ O]	0.862	0.867	0.006	2.7×10^7	7.4	0.13	-42

[LCu].3H₂O > [LCd.2H₂O]

Table (9): The molar conductance of the complexes

Compound fragmentations	Λ_m S.cm ² molar ⁻¹	ratio
[LCo.2H ₂ O]	160	1:1
[LNi.2H ₂ O]	180	1:1
[LCu].3H ₂ O	130	1:1
[LCd.2H ₂ O]	170	1:1
[LHg.2H ₂ O]	135	1:1
[LPb.2H ₂ O]	180	1:1

Recorded in (water) solvent, where L= C₁₀H₈N₂O₄

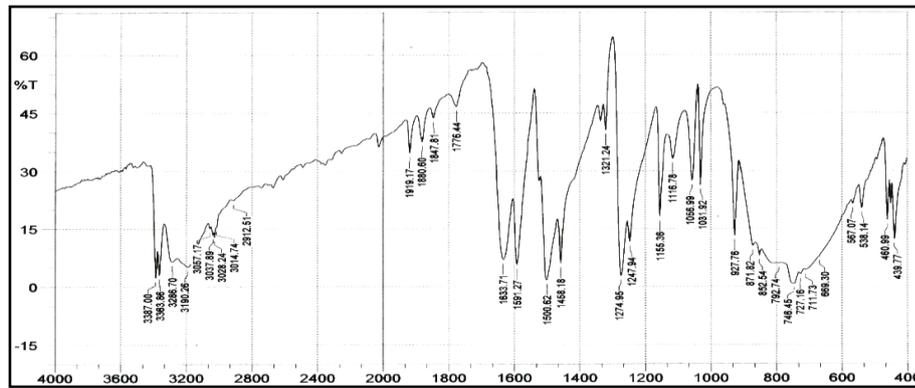


Fig. (5): The IR spectrum of *O*-phenylene diamine

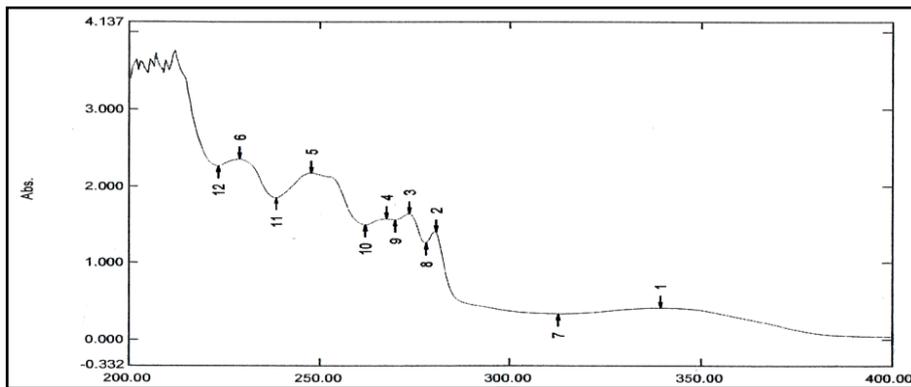


Fig. (6): Electronic spectrum of the ligand (L)

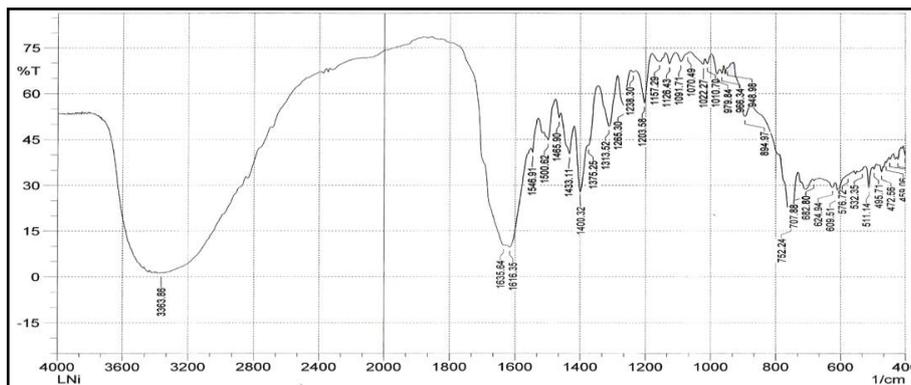


Fig. (7): The IR spectrum of the [LNi.2H₂O] complex

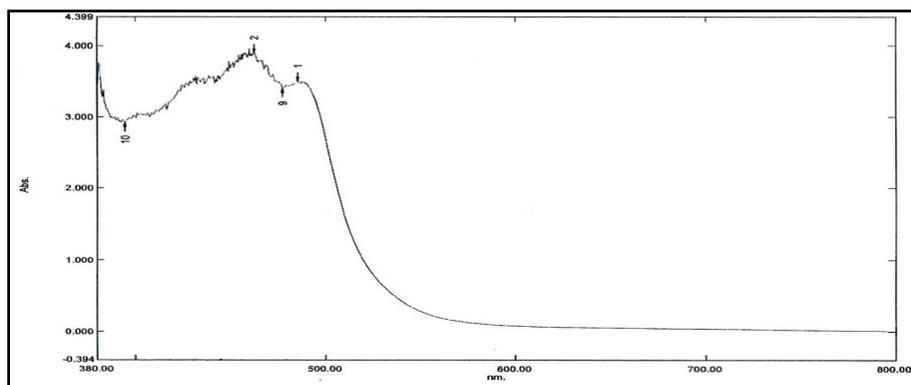


Fig. (8): Electronic spectrum of the [LCo.2H₂O] complex

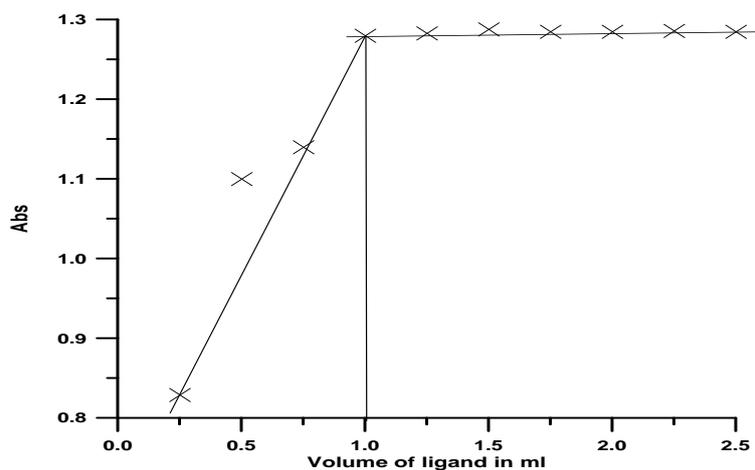


Fig. (9): The mole ratio curve of complex [LCu].3H₂O in solution (1×10^{-3} mole. L⁻¹) at ($\lambda=272.8$ nm)

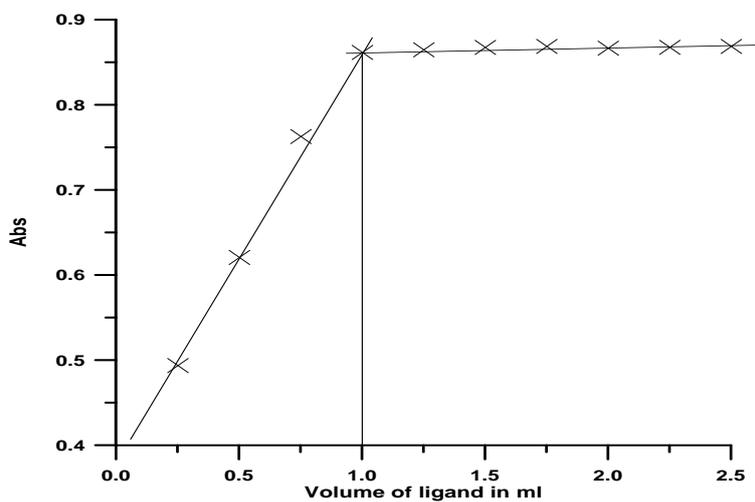


Fig. (10): The mole ratio curve of complex [LCd].2H₂O in solution (1×10^{-3} mole.L⁻¹) at ($\lambda=270.5$ nm)

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تحضير، تشخيص بعض المعقدات الفلزية
لـ[2-(كابوكسي مثيلين- أمينو)- فنيل إمينو] حامض الخليك (L)

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الخلاصة:

حضرت وشخصت قاعدة شف جديدة، [2-(كابوكسي مثيلين- أمينو)- فنيل إمينو] حامض الخليك (L) وبعض معقداته الفلزية [LHg.2H₂O]، [LCd.2H₂O]، [LCu].3H₂O، [LNi.2H₂O]، [LCo.2H₂O]، [LPb.2H₂O] بتحليل العناصر، محتوى الفلز، الطرق الطيفية، العزوم المغناطيسية وقياسات التوصيلية. التراكيب الهندسية للمعقدات الكوبلت (II)، النيكل (II)، الكاديوم (II)، الزئبق (II)، والرصاص (II) ثمانية السطوح والمربع المستوي للنحاس (II)، نسبة الفلز إلى اللكاند (I:I).