

**Synthesis and characterization of Co(II), Ni(II), Cu(II), Cd (II) and Hg(II) complexes with new derivative of L-ascorbic acid**  
**تحضير وتشخيص معقدات Co(II), Ni(II), Cu(II), Cd (II), Hg(II) مشتق جديد لـ L- حامض الاسكوربيك**

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**Abstract**

Stable new derivative (L) Bis[O,O-2,3;O,O-5,6(carboxylic methyliden)]L-ascorbic acid was synthesized in good yield by the reaction of L-ascorbic acid with dichloroacetic acid with ratio (1:2) in presence of potassium hydroxide. The new (L) was characterized by <sup>1</sup>H, <sup>13</sup>C-NMR, elemental analysis (C,H) and Fourier Transform Infrared (FTIR). The complexes of the ligand (L) with metal ion, M<sup>+2</sup>= (Cu, Co, Ni, Cd and Hg) were synthesized and characterized by FTIR, UV-Visible, Molar conductance, Atomic absorption and the Molar ratio. The analysis evidence showed the binding of the metal ions with (L) through bicarboxylato group manner resulting in six-coordinated metal ion.

**KEYWORDS** Synthesis; L-ascorbic acid; New derivative; Complexes; Analysis

**الخلاصة**

حضر بس [O,O-2,3;O,O-5,6 كاربو كسيلك مثيلدين ] L-حامض الاسكوربيك بناتج جيد بتفاعل L-حامض الاسكوربيك مع ثنائي كلورو حامض الخليك بنسبة (2:1) في وسط هيدروكسيد البوتاسيوم الكحولي. شخض اللكند بمطيافية <sup>1</sup>H, <sup>13</sup>C الرنين النووي المغناطيسي. تحليل العناصر (كاربون, هيدروجين) والاشعة تحت الحمراء. حضرت معقدات للكند مع ايونات الفلزات ثنائية التكافؤ (Hg(II), Ni(II), Cu(II), Cd (II), Co(II)). وتم تشخيصها بمطيافية الاشعة تحت الحمراء. الاشعة فوق البنفسجية, التوصيلية المولارية, الامتصاص الذري والنسبة المولارية. اظهرت التحاليل أن ارتباط الايون الفلزي مع اللكند كان من خلال مجموعة الكربوكسيل ثنائية السن معطية شكل سداسي التناسق.

**Introduction**

L-ascorbic acid, commonly known as Vitamin C, is widely distributed in aerobic organisms and is involved in several biological processes. The biological and pharmacological activity, as well as the therapeutic potential of L-ascorbic acid and its derivatives has been studied extensively[1]. 2-O and 3-O-alkylated derivatives are known to protect against peroxidation of lipids of the bio-membrane[2,3]. L-Ascorbic acid has four hydroxyl groups at carbon 2, 3, 5, 6. These hydroxyl groups have different chemical activities. Their steric environment are open to derivative toward producing a number of compounds with interesting chemical and physical properties.

Musa-etal, synthesized derivatives of L-ascorbic acid such as 5,6-O-isopropylidene-2,3-(2-X,1carboxyl) deoxy-L-ascorbic acid, where X = H, Cl., 1,2-dihydroxyl ethyl-1-(2-mercaptophenyl)-5-(2-mercaptophenyl)-2,5-dihydro-1H-pyrrol-3,4-diol. Finally 5,6-O, 2,3-O L-ascorbic acid were substituted by acetic acid.

Above derivatives of L-ascorbic acid derivatives transition and alkali metal ions complexes were also synthesized and characterized[4,5]. As past of our continuing efforts to synthesize and characterize metal chelates using polydentate ligands[6,7,8]. We describe here the synthesis and spectral investigation of new ligand Bis[O,O-2,3;O,O-5,6(carboxylic methyliden)]L-ascorbic acid.

## 2. Experimental

### 2.1. Instruments, materials and methods

All chemicals were purchased from BDH, and without further purifications. FTIR spectra were recorded in KBr on Shimadzu- spectrophotometer in the range of (4000-400) $\text{cm}^{-1}$ . Electronic spectra in distilled water were recorded using the UV-visible spectrophotometer type Shimadzu in the rang of (200-1100)nm with quartz cell of (1cm) path length. Melting points where measured with an electrothermal Stuart apparatus, model SMP30. Electrical Conductivity Measurements of the complexes were recorded at (25°C) for  $10^{-3}$  mol.L<sup>-1</sup> solution of the samples in distilled water using Ltd 4071 digital conductivity meter. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker 300-MHz spectrometer in DMSO-d<sub>6</sub>. Chemical shifts are in ppm relative to internal Me<sub>4</sub>Si was performed at AL-al-Bayt University, Jordon. Elemental microanalyses of the ligand were carried out by using Euro Vectro- 3000A, AL-al-Bayt University, Jordon. Metal content of the complexes were measured using Atomic absorption technique by Perkin-Elmer 5000, University of Baghdad collage of science. While Hg metal determined using Biotech Eng. Management Co. Ltd. (UK), the University of Mustansiriyah college of science. Magnetic susceptibility values were obtained at room temperature using the Gouy method, Johnson Matthey, England, were performed at Al-Nahrain University. Thin Layer Chromatography (TLC): the (TLC) was performed on aluminum plates coated with silica gel (Fluka), and were detected by iodine.

### 2.2. Synthesis of ligand (L)

L-ascorbic acid (0.18gm, 1mmole) was dissolved in mixture of (15mL ethanol + 5mL water). Potassium hydroxide (0.23gm, 4mmole) in ethanol (10mL) solution was added. The solution was stirring for 30 minutes. The dichloroacetic acid (0.26gm, 2mmole) was added drop wise, stirring was continued for one hour. The precipitate was filtered and washed from mixture of (20mL ethanol + 5mL water) giving a brown precipitate melting point (162-164°C), yield 71%.

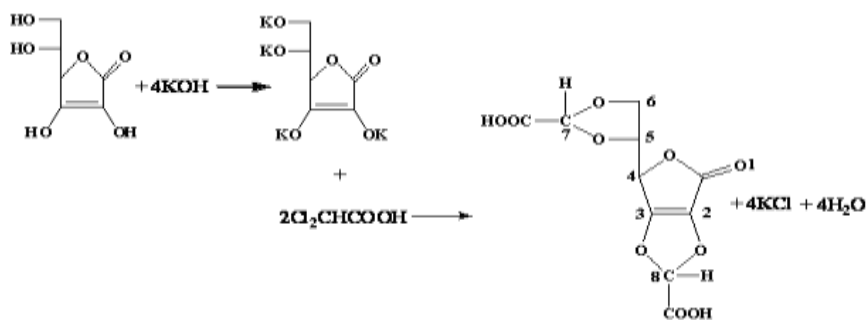
### 2.3. Synthesis of complexes ( $M^{II} = \text{Cu, Co, Ni, Cd, Hg}$ )

To a solution of the (L) (0.29gm, 1mmole) in mixture of (15mL ethanol + 5mL water) was added, a solution of 2mmole of metal chloride in 20mL ethanol 0,34gm CuCl<sub>2</sub>.2H<sub>2</sub>O, 0.24gm NiCl<sub>2</sub>.6H<sub>2</sub>O, 0.48gm CoCl<sub>2</sub>.6H<sub>2</sub>O, 0.46gm CdCl<sub>2</sub>.H<sub>2</sub>O, 0.54gm HgCl<sub>2</sub>. The solutions were stirring for one hour and left it to evaporate slowly to bring down the complexes. The complexes were washed from mixture of ethanol + water (4:1).

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

## 3. Results and Discussion

Bis[O,O-2,3;O,O-5,6(carboxylic methyliden)]L-ascorbic acid ligand (L) was synthesized in good yield by the reaction of L-ascorbic acid with dichloroacetic acid in the ratio (1:2) in presence of four mole of potassium hydroxide (Scheme 1). The isolated complexes are colored solids, stable in air and insoluble in common organic solvents but completely soluble in water and coordinating solvents (DMSO and DMF). The molar conductivity values were measured for 0.001 M of complexes in water. The values are found in 12.5–23.3  $\Omega^{-1} \text{cm}^2 \text{mol}^{-1}$  range which indicates their non-electrolytic nature consequently to the strong covalent character of the carboxylate group.



**Scheme 1** The reaction of L-ascorbic acid with dichloroacetic acid in presence of potassium hydroxide

### 3.1. The FT- IR spectral analysis

The IR spectrum of L-ascorbic acid as starting material is compared with the new ligand (L) (Figure 1), the results are summarized in Table (2). L-ascorbic acid exhibits bands at 3525, 3410, 3313 and 3213  $\text{cm}^{-1}$  caused by  $\nu(\text{OH})$  positions (C-5,6,2,3) respectively, these are disappeared in the spectrum of the new ligand accompanied by appearance of three bands in the 3371 (broad), 1551 (asym.), 1371 (sym.) are due to carboxylic group. (C-1=O) stretching vibration appeared at (1720  $\text{cm}^{-1}$ ). The bands at (1678,1647)  $\text{cm}^{-1}$  in starting L-ascorbic acid are due to the  $\nu(\text{C}=\text{C})$ ,  $\nu(\text{C}=\text{O})$  appeared as broad band centre at 1630  $\text{cm}^{-1}$  in the new ligand.

The bands located at (1551,1371)  $\text{cm}^{-1}$  which was assigned to  $\nu(\text{C}=\text{O})$  stretching vibration for (COOH) in free ligand, was shifted to lower frequency and appeared at [1410,1320]  $\text{cm}^{-1}$  (Cu) (Figure 2), [1392,1319]  $\text{cm}^{-1}$  (Co), [1410,1311]  $\text{cm}^{-1}$  (Cd), [1440,1384]  $\text{cm}^{-1}$  (Ni), [1400,1320]  $\text{cm}^{-1}$  (Hg).

Each two bands assignable to asymmetric and symmetric stretching frequencies of the carboxylate ion with average separation  $\Delta\nu_{\text{COO}^-}$ ; 90, 73, 99, 56, 80)  $\text{cm}^{-1}$  respectively indicating the deprotonation of the carboxylic proton and suggests that coordination occurs through the carboxylate ion as a bidentates bonding nature[9,10,11]. New bands appeared in the range 420-511  $\text{cm}^{-1}$  in the all complexes assignable to  $\nu(\text{M}-\text{O})$  vibrations[12]. A band due to  $\nu(\text{C}=\text{O})$  of the lacton ring appeared as a shoulder with range (1725-1735)  $\text{cm}^{-1}$  in each complexes. A strong broad absorption band appeared around (3500-3377)  $\text{cm}^{-1}$  associated of water molecules in these metal complexes. Coordinated  $\text{H}_2\text{O}$  appeared at range (810-825)  $\text{cm}^{-1}$  in all complexes[13].

### 3.2. NMR spectrum for the ligand

$^1\text{H}$ -NMR spectrum of the ligand (L) (Figure 3) in  $\text{DMSO-d}_6$  exhibited at  $\delta$ (5.898,5.598) ppm are due to methane (CH-8,7) and (CH-4) lactone, the CH-6,CH-5 appeared as a broad 4.133 and 3.783 ppm respectively. The OH- carboxylic showed broad center at 8.4 ppm.

The  $^{13}\text{C}$ -NMR of the ligand (L) (Figure 4) showed the following data.

- The signal at  $\delta=178$  ppm is due to carboxylic acid
- The signal at  $\delta=165$  ppm is due to C=O lactone
- The signal at  $\delta=(135, 75, 72, 68)$  ppm are due to (C-3, C-4, C-5, C-6)

The results were compared with those obtained by chem. Office program and with  $^1\text{H}$ ,  $^{13}\text{C}$ -NMR previous obtained on derivatives of L-ascorbic acid[14,15].

### 3.3. The UV-vis. spectral studies

The electronic absorption bands are summarized in Table 3. The UV-visible spectrum of the ligand (L) (Figure 5) showed one absorption at (40160 cm<sup>-1</sup>, 249 nm) is due to  $\pi-\pi^*$  transition[16].

The electronic spectrum of Co-complex in water solution exhibited two bands appeared at (16129 cm<sup>-1</sup>, 620 nm) and (19607 cm<sup>-1</sup>, 510 nm) were assigned to the  ${}^4T_{1g} \rightarrow {}^4A_{2g}$  ( $\nu_2$ ) and  ${}^4T_{1g} \rightarrow {}^4T_{1g}(p)$  ( $\nu_3$ ) transitions respectively of octahedral geometry[17]. From the ratio of ( $\nu_3$ )/( $\nu_2$ ) (1.22) the value of Dq/B (0.95) was obtained. The value of B<sub>complex</sub> (889.17) as well as position of  $\nu_1$  (7371 cm<sup>-1</sup>) were calculated by applying Dq/B on Tanaba-Sugano diagram for d<sup>7</sup> configuration of the octahedral configuration geometry[18,19]. The value of  $\beta$  (0.91) indicates some covalent character. Spectrum of Ni(II) complex showed three bands in the visible region at (26109 cm<sup>-1</sup>, 383 nm)  ${}^3A_{2g} \rightarrow {}^3T_{1g}$  ( $\nu_3$ ), (14662 cm<sup>-1</sup>, 682 nm)  ${}^3A_{2g} \rightarrow {}^1E_g$  ( $\nu_2$ ) and last one is at (9523 cm<sup>-1</sup>, 1050 nm)  ${}^3A_{2g} \rightarrow {}^3T_{2g}$ , ( $\nu_1$ ). The ratio of  $\nu_2/\nu_1$ , (1.54) was applied on Tanaba-Sugano diagram for d<sup>8</sup> octahedral complexes[20], B<sub>complex</sub> and  $\beta$ , 10Dq( $\nu_1$ ) were calculated theoretically.

$$\nu_2/\nu_1 = 1.54$$

$$Dq/B = 1.55$$

$$\frac{\nu_3}{B} = 37.57 \Rightarrow B = \frac{26109}{37.57} = 694.8$$

$$\frac{Dq}{694.8} = 1.55 \Rightarrow Dq = 1077$$

$$\beta = \frac{B_{\text{complex}}}{B_{\text{free ion(Ni)}}} = \frac{694.8}{1030} = 0.67$$

$$\Delta_o(10Dq)\nu_1 = 18 \times 694.8 = 12506 \text{ cm}^{-1}$$

The spectrum of Cu(II) complex (Figure 6) showed broad band at (12484cm<sup>-1</sup>, 801nm) assigned to  ${}^2E_g \rightarrow {}^2T_{2g}$  transition which refers to Jahn-Teller distortion of octahedral geometry[21]. The spectra of Cd(II), Hg(II) complexes gave no bands in the visible region, only bands assigned to intraligand  $\pi-\pi^*$  transition (39840cm<sup>-1</sup>, 251nm) Cd(II), (35714cm<sup>-1</sup>, 280nm) Hg(II) complexes were observed, compared with free ligand showed one band at (40160cm<sup>-1</sup>, 249nm) confirms the complexes formation[22].

### 3.4. Magnetic studies

The magnetic moment values at (294K) of the [M<sub>2</sub>LCl<sub>2</sub>].XH<sub>2</sub>O M<sup>2+</sup> = Cu, Ni, Co Table 3 show values (0.82, 1.85, 2.25)B.M respectively which are lower than the total spin-only values indicating a high spin octahedral geometry around metal ion. The lowering of these magnetic moments indicates a dominate antiferromagnetic interaction in all complexes. This may due to the fact that the Syn-Syn carboxylate provide a small metal-metal distance and results in a good overlap of the magnetic orbitals, an antiferromagnetic coupling is always induced[23,24].

### 3.5. Molar Ratio

The complexes of the ligand (L) with metal ions [Co(II), Ni(II), Cu(II), Cd(II), Hg(II)] were studied in solution using water as solvent, in order to determine (M:L) ratio in the prepared complexes, following molar ratio method[25]. A series of solutions were prepared having a constant concentration (C) 10<sup>-3</sup> 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 (L:M) found to be (1:2). The results of complexes formation in solution are shown in Table (4), (Figure 7)

these data are compatible with the results obtained by atomic absorption for determination metal analysis in the complexes.

### 3.6. Thin Layer Chromatography (TLC)

The solution of Liand (L) and its complexes in water as solvent appeared in one spot, this is confidence all these compounds are purely and have one isomer. Table (1) show the  $R_f$  for complexes and ligand (L).

## 4. Conclusion

From these data, the ligand (L) acts as a tetra-dentate monoanion in its metal complexes and coordinated through oxygen atoms of carboxylate group in an octahedral geometry. Figure (8)

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**Table 1** Physical properties, analytical data for synthesized ligand (L) and its complexes

Empirical formula	color	m.p. °C	$\Lambda_m$ ( $\Omega^{-1}$ $\text{cm}^2\text{mol}^{-1}$ )	Found(Calc.)(%)			$R_f$
				C	H	M(II)	
(1) Ligand(L) $\text{C}_{10}\text{H}_8\text{O}_{10}$	Brown	164	—	40.8 (41.6)	3.03 (2.78)	—	0.5
(2) $[\text{Cu}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]2\text{H}_2\text{O}$	Deep green	197	12.5	—	—	20.48 (20.78)	0.48
(3) $[\text{Co}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]4\text{H}_2\text{O}$	Red brown	253	18.3	—	—	18.23 (18.49)	0.41
(4) $[\text{Ni}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]4\text{H}_2\text{O}$	Green	226	14.6	—	—	17.97 (18.36)	0.34
(5) $[\text{Cd}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]\text{H}_2\text{O}$	Deep brown	272	15.2	—	—	32.95 (32.56)	0.46
(6) $[\text{Hg}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]2\text{H}_2\text{O}$	Brown	230	23.3	—	—	44.87 (45.26)	0.28

**Table 2** Assignments of the IR spectral bands of L-ascorbic acid, ligand(L) and its complexes( $\text{cm}^{-1}$ )

Empirical formula	$\nu_{\text{OH}}$	$\nu_{\text{C=O}}$	$\nu_{\text{C=C}}$ $\nu_{\text{C=O}}$	$\nu_{\text{as(COO)-}}$	$\nu_{\text{s(COO)-}}$	Coord. $\text{H}_2\text{O}$	$\nu_{\text{M-O}}$
(1) L-ascorbic acid	3525,s 3410,s 3313,s 3213,s	1720	1678 1647	—	—	—	—
(2) Ligand(L) $\text{C}_{10}\text{H}_8\text{O}_{10}$	3371,br	1720	1630,br	1551	1371	—	—
(3) $[\text{Cu}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]2\text{H}_2\text{O}$	3342,m	1725	1616,m	1410	1320	821,m	511
(4) $[\text{Co}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]4\text{H}_2\text{O}$	3448,br	1745	1635,m	1392	1319	810,m	462
(5) $[\text{Ni}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]4\text{H}_2\text{O}$	3377,br	1738	1635,m	1440	1384	825,s	420
(6) $[\text{Cd}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]\text{H}_2\text{O}$	3473,br	1710	1625	1410	1311	821,s	425
(7) $[\text{Hg}_2(\text{C}_{10}\text{H}_{18}\text{O}_{15}\text{Cl}_2)]2\text{H}_2\text{O}$	3520,m	1716	1624	1400	1320	823,w	430

s= strong, m= medium, w= weak, br= brud

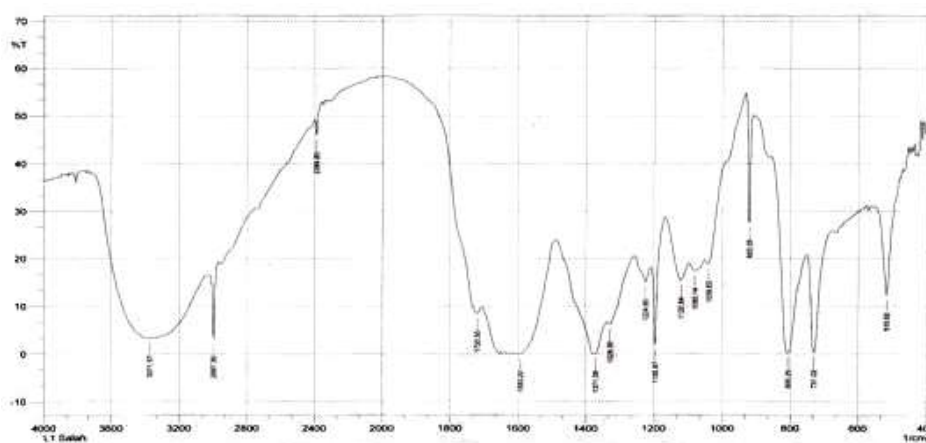
**Table 3** Magnetic moments and electronic spectral bands ( $\text{cm}^{-1}$ ) of the M(II) complexes.

Complex	$\mu_{\text{eff}}$ (B.M.)	Band position $\text{cm}^{-1}$	Assignments	$B_{\text{complex}}$	$\beta$	$10Dq$ $\text{cm}^{-1}$
Co(II) complex	2.25	16129 19607	${}^4\text{T}_{1g} \rightarrow {}^4\text{A}_{2g}$ ${}^4\text{T}_{1g} \rightarrow {}^4\text{T}_{1g}(\text{p})$	889.17	0.91	8774
Ni(II) complex	1.85	26109 14662 9523	${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g}$ ${}^3\text{A}_{2g} \rightarrow {}^1\text{E}_g$ ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{2g}$	694.8	0.67	12506
Cu(II) complex	0.82	12484	${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}$	—	—	—
Cd(II) complex	—	39840	$\pi-\pi^*$	—	—	—
Hg(II) complex	—	35714	$\pi-\pi^*$	—	—	—

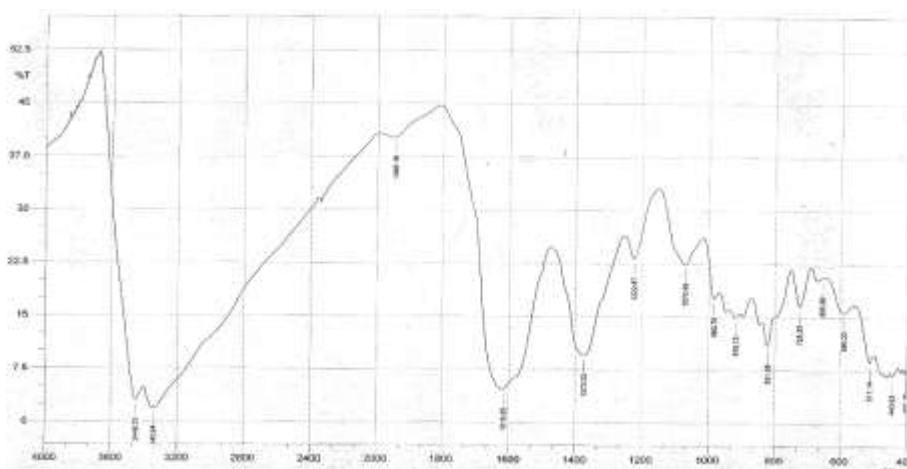
**Table 4** Molar ratio data for L-complexes

V(mL)	L-Cu ( $\lambda=800$ )	L-Co ( $\lambda=510$ )	L-Ni ( $\lambda=740$ )	L-Cd ( $\lambda=340$ )	L-Hg ( $\lambda=280$ )
(1) 0.5	0.11	1.32	1.75	0.33	0.56
(2) 1	0.17	1.94	1.95	0.56	1.23
(3) 1.5	0.23	2.76	2.87	0.98	1.42
(4) 2	0.29	3.44	3.51	1.12	1.85
(5) 2.5	0.31	3.68	3.72	1.25	1.94
(6) 3	0.23	3.91	3.98	1.38	2.37
(7) 3.5	0.33	4.21	4.19	1.61	2.55
(8) 4	0.35	4.50	4.48	1.95	2.86
(9) 4.5	0.37	4.63	4.70	2.33	3.12
(10) 5	0.39	4.79	4.89	2.34	3.11

**Fig. 1** I.R. spectrum of the ligand (L)



**Fig. 2** I.R. spectrum of the Cu(II) complex



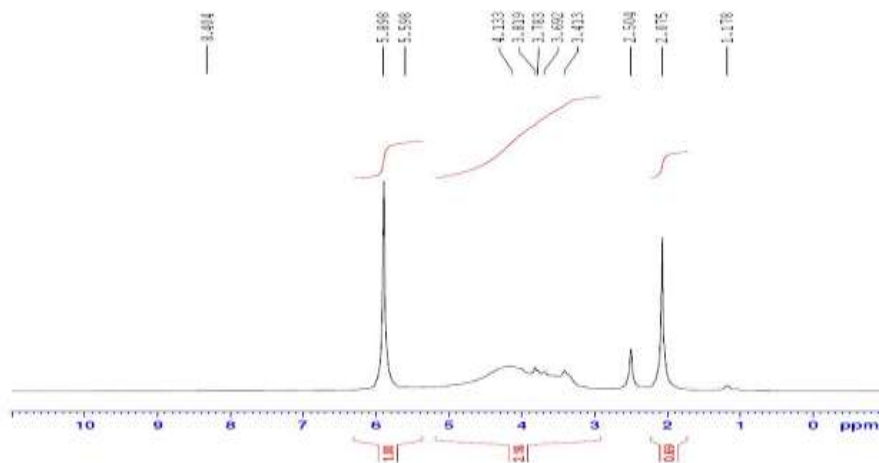
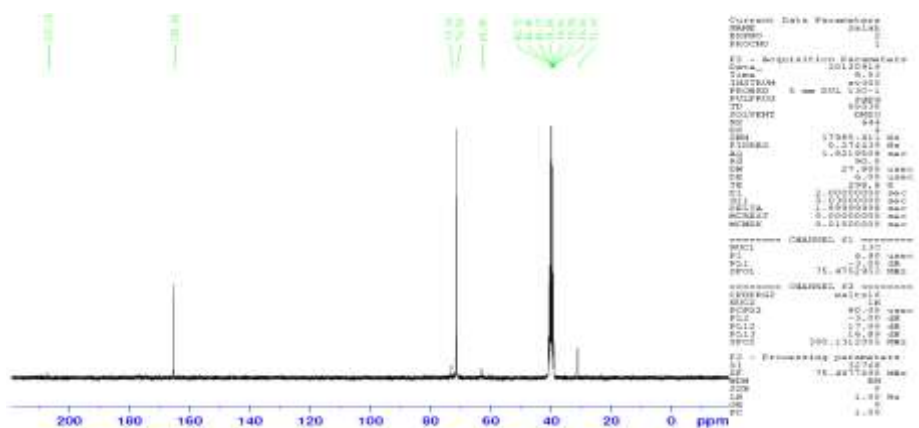
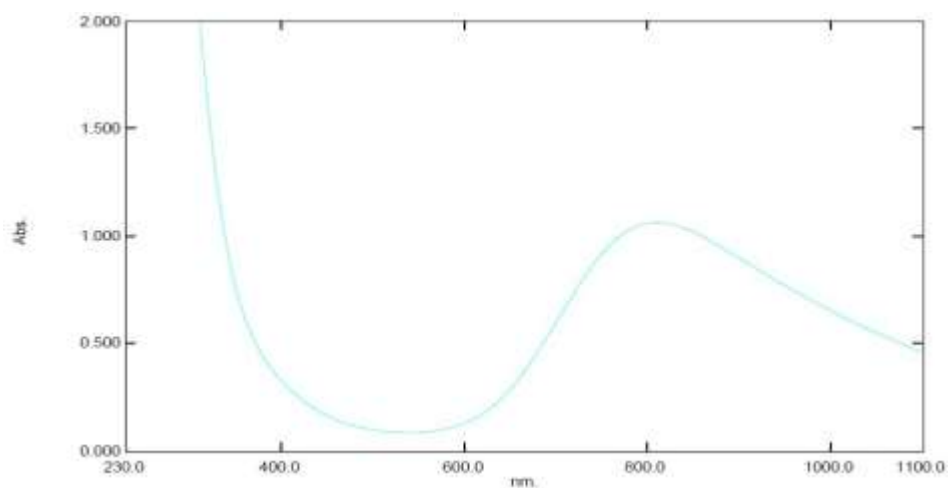


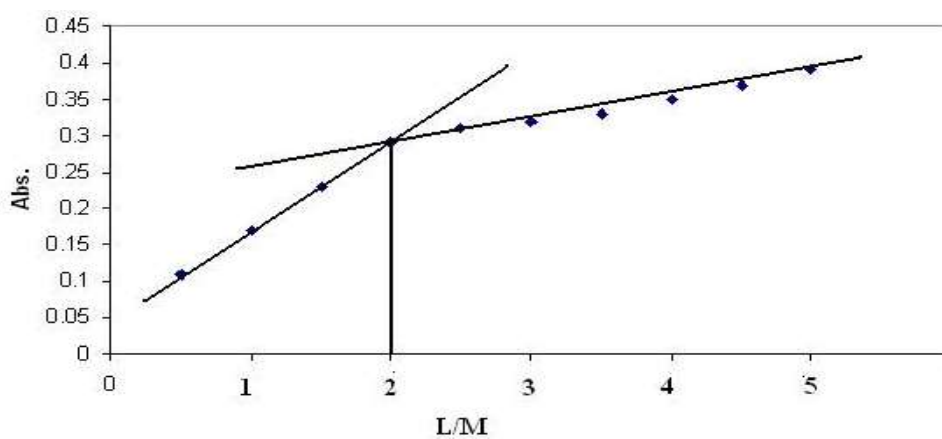
Fig. 3 <sup>1</sup>H-NMR spectrum of the ligand (L)



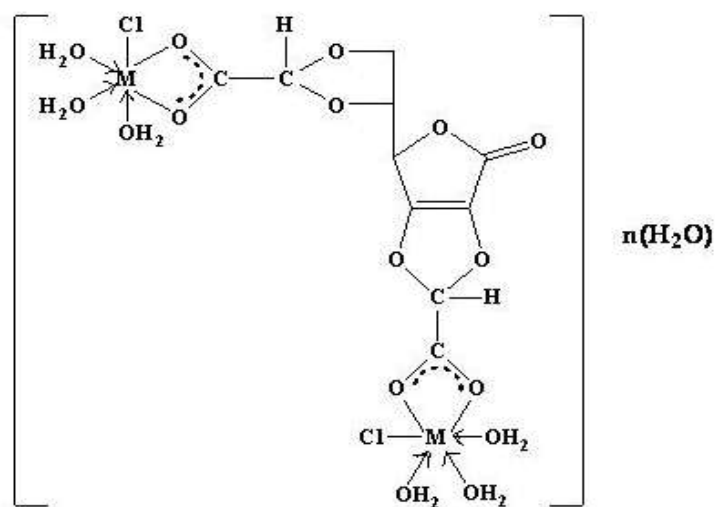




**Fig. 6** UV-visible spectrum of the Cu(II) complex



**Fig. 7** Molar ratio curve of Cu(II) complex in water solution at  $\lambda=800$



**Fig. 8** Suggested structure of  $[M_2LCl_2]_nH_2O$   
 M = Cu(II), Hg(II)  $n = 2H_2O$ , Co(II), Ni(II)  $n = 4H_2O$ , Cd(II),  $n = H_2O$