Synthesis and Spectral Studies of Some Metal Complexes with Bis[O,O-2,3;O,O-5,6(Chlorocarboxylic Methyliden)] L-Ascorbic Acid

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Abstract

The reaction of L-ascorbic acid with the tirchloroacetic acid in the presence of potassium hydroxide gave new product Bis[O,O-2,3;O,O-5,6(chlorocarboxylicmethyliden)]L-ascorbic acid (H_2L) which was isolated and characterized by ^1H,^{13}C-NMR, elemental analysis (C,H,N), UV-Visible and Fourier Transform Infrared (FTIR). The complexes of the ligand (H_2L) with metal ions, M^{+2}= (Cu, Co, Ni, Cd and Hg) were synthesized and characterized by FTIR, UV-Visible, molar conductance, atomic absorption, magnetic susceptibility and the molar ratio. The analysis evidence showed the binding of the metal ions with (H_2L) through the bicarboxylato group manner resulting in six-coordinated metal ion. The TLC for (H_2L) and complexes showed one spot for each indicating the purity of these compounds.

Key words: Synthesis; L-ascorbic acid; Complexes; Analysis

Introduction

L-Ascorbic acid (vitamin c) is an important molecule in both chemistry and biology, and its complexes with metals are of particular interest in both of these areas[1-3]. This vitamin is present in various foods, particularly of plant origin, that are several orders of magnitude higher than those of other vitamins.[4]

Musa-etal, [5-7] synthesized derivatives of L-ascorbic acid such as 5,6-O-isopropylidene-2,3-(2ˉ-X,1carboxyl) deoxy-L-ascorbic acid, where X = H, Cl., 1,2-dihydroxyl ethyl-1-(2-mercaptophenyl)-5-(2-mercaptophenyl)-2,5-dihhydro-1H-pyrrol-3,4-diol. Finally 5,6-O, 2,3-O L-ascorbic acid were substituted by acetic acid.

L-ascorbic acid derivatives of transition and alkali metal ions complexes were also synthesized and characterized.

Experimental part

Instruments, materials and methods

All chemicals were purchased from BDH, and used without further purifications. FTIR spectra were recorded in KBr on Shimadzu- spectrophotometer in the range of (4000-400)cm^{-1}. Electronic spectra in distilled water were recorded using the UV-visible spectrophotometer type Shimadzu in the range of (200-1100)nm with quartz cell of (1cm) path leangth. Melting points where measured with an electrothermal Stuart apparatus, model SMP30. Electrical conductivity measurements of the complexes were recorded at (25^{o}C) for 10^{-3} mol.L^{-1} solution of the samples in distilled water using Ltd 4071 digital conductivity meter. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 300-MHz spectrometer in DMSO-d_6. Chemical shifts are in ppm relative to internal Me_4Si 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 college of science. While Hg metal is determined using Biotech Eng. Management Co. Ltd. (UK), University of Mustansiriyah, College of Science. Magnetic susceptibility values were obtained at room temperature using the Gouy method, Johnson Mattey, England, were performed at Al-Nahrain University. Thin Layer Chromatography (TLC): the (TLC) was performed on aluminum plates coated with silica gel (Fluka), and detected by iodine.

Synthesis of ligand (H_2L)

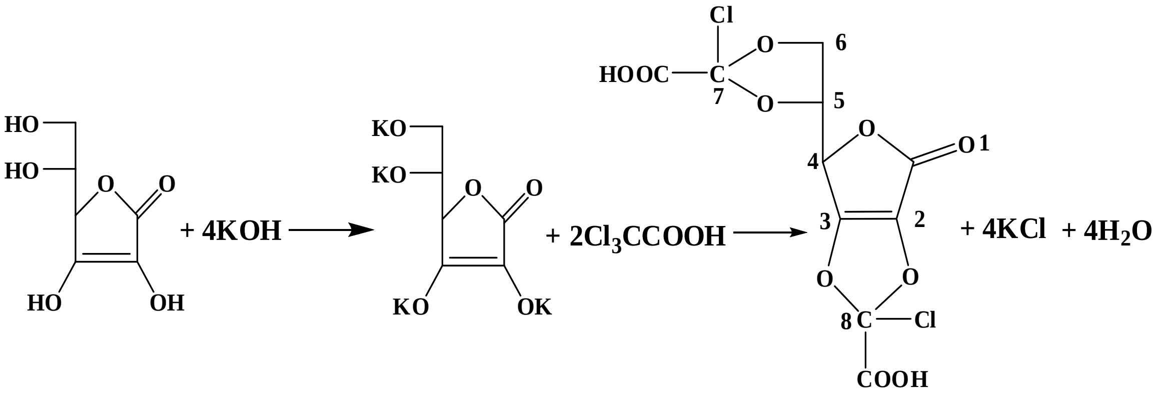
L-ascorbic acid (0.18gm, 1m mole) was dissolved in the mixture of (15ml ethanol + 5ml water). Potassium hydroxide (0.23gm, 4m mole)in ethanol (10ml) solution was added. The solution was stirring for 30 minutes. The trichloroacetic acid (0.32gm, 2m mole) was added drop wise, stirring was continued for one hour and left to evaporate slowly. The precipitate was recrystallized from ethanol giving a yellow precipitate melting point (138C°), yield 68%.

Synthesis of complexes (M^{II} = Cu, Co, Ni, Cd, Hg)

To a solution of the (H_2L) (0.35gm, 1m mole) in mixture of (15ml ethanol + 5ml water) was added, a solution of 2m mole of metal chloride in 20ml ethanol 0,34gm $CuCl_2.2H_2O$, 0.24gm $NiCl_2.6H_2O$, 0.48gm $CoCl_2.6H_2O$, 0.46gm $CdCl_2.H_2O$, 0.54gm $HgCl_2$. The solutions were stirring for one hour and left them to evaporate slowly to bring down the complexes. The complexes were washed from mixture of ethanol + water (4:1). The isolated complexes are colored solids, stable in air and insoluble in common organic solvents but completely soluble in water, ethanol, DMSO and DMF. Some physical properties for all synthesized ligand (H_2L) and its complexes are shown in Table 1.

Results and discussion

Bis[O,O-2,3;O,O-5,6(chlorocarboxylic methyliden)]L-ascorbic acid (H_2L) was synthesized in a good yield by the reaction of L-ascorbic acid with trichloroacetic acid in the ratio (1:2) in presence of four mole of potassium hydroxide (Scheme 1).



Scheme (1) The reaction of L-ascorbic acid with trichloroacetic acid in base media

FT-IR spectral analysis

The I.R spectrum of L-ascorbic acid as starting material is compared with the new ligand (H_2L) spectrum, the results are summarized in table (2). L-ascorbic acid exhibits bands at 3525, 3410, 3313 and 3213 cm^{-1} caused by υ(OH) positions (C-5,6,2,3) respectively, these are disappeared in the spectrum of the new ligand accompanied by the appearance of three bands in the 3380 (broad), 1566 (asym.), 1417 (sym.) are due to carboxylate group. (C-1=O) stretching vibration appeared at (1718 cm^{-1}). The bands at (1678,1647) cm^{-1} in starting L-ascorbic acid are due to the υ(C=C), υ(C=O) appeared as broad band centre at 1660 cm^{-1} in the new ligand.

The bands located at (1566,1417) cm^{-1} which were assigned to υ(C=O) stretching vibration for (COOH) in free ligand (H_2L), were shifted to lower frequency and appeared at [1400,1344] cm^{-1} in (Cu), [1420,1344] cm^{-1} in (Co), [1445,1384] cm^{-1} in (Ni), [1415,1361] cm^{-1} in (Cd), [1425,1361] cm^{-1} in (Hg). Each two bands were assignable to asymmetric and symmetric stretching frequencies of the carboxylate ion with average separation $\Delta\upsilon coo^{-}$; 56, 76, 61, 54, 64) cm^{-1} respectively indicating the deprotonation of the carboxylic proton and suggests that coordination occurs through the carboxylate ion as a bidentates bonding nature[7-9]. New bands appeared in the range 428-482cm^{-1} in the all complexes assignable to υ(M-O) vibrations[10]. A band due to υ(C=O) of the lacton ring appeared as a shoulder within the range (1730-1735) cm^{-1} in all complexes. A strong broad absorption band appeared around (3566-3433) cm^{-1} associated with water molecules in these metal complexes. Coordinated H_2O appeared at range (825-844) cm^{-1} in all complexes[11].

NMR spectrum for the ligand (H_2L)

^{1}H-NMR spectrum of the ligand (L) (Figure 1)in DMSO-d_6 exhibited at δ(3.019-3.518) and (4.436) ppm are due to (CH_2-6) and (CH-5) respectively, (CH-4) lactone appeared at δ(5.135) ppm. The OH- carboxylic showed broad center at 8.281 ppm.

The ^{13}C-NMR of the ligand (L) (Figure 2) showed the following data.

- The signal at δ=177 ppm is due to carboxylic acid
- The signal at δ=163 ppm is due to C=O lactone
- The signals at δ=(125, 128, 79 and 60) 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}H,^{13}C-NMR previous obtained on derivatives of L-ascorbic acid[12,13].

Spectral studies

The electronic absorption bands as well as the magnetic moment values are summarized in Table 3. The UV-Visible spectrum of the ligand (H_2L) showed one absorption at (40000 cm^{-1}) is due to $\pi-\pi^*$ transition[14].

The electronic spectrum of Co-complex in water solution exhibited two bands appeared at (15873 cm^{-1}) and (19230 cm^{-1}) were assigned to the ${}^4T_1g\rightarrow{}^4A_2g$ (υ_2) and ${}^4T_1g\rightarrow{}^4T_1g(p)$ (υ_3) transitions respectively of octahedral geometry[15]. From the ratio of (υ_3)/(υ_2) (1.21), the value of $B_{complex}$ (872.1) as well as position of υ_1 (8284 cm^{-1}) were calculated by using Tanaba-Sugano diagram for d^7 configuration of the octahedral configuration geometry[16]. The value of β (0.89) indicates some covalent character. The conductivity measurement indicates that the complex is non-ionic.

Spectrum of Ni(II) complex showed three bands in the visible region at (24390 cm^{-1}) ${}^3A_2g\rightarrow{}^3T_1g_{(P)}$ (υ_3), (14556 cm^{-1}) ${}^3A_2g\rightarrow{}^3T_1g_{(F)}$ (υ_2) and the last one is at (9259 cm^{-1}) ${}^3A_2g\rightarrow{}^3T_2g$, ($\upsilon_1$). The ratio of υ_2/υ_1, (1.57) was applied on Tanaba-Sugano diagram for d^8 octahedral complexes[17,18], $B_{complex}$ and β, 10Dq(υ_1) were calculated theoretically. The conductivity showed that the Ni(II)-complex was non-electrolyte.

The spectrum of Cu(II) complex showed broad band at (12391cm^{-1}) assigned to ${}^2Eg\rightarrow{}^2T_2g$ transition which refers to Jahn-Teller distortion of octahedral geometry[19]. The conductivity measurement of the complex indicates that the complex is non-electrolyte.

The spectra of Cd(II), Hg(II) complexes gave no bands in the visible region, only bands assigned to charge transfer transitions (39682cm^{-1}) Cd(II), (34482cm^{-1}) Hg(II) complexes were observed, compared with free ligand showed one band at (40000cm^{-1}, 250nm) confirms the complexes formation[20]. The conductivity measurements of the two complexes indicate that the complexes are non-electrolyte.

Magnetic studies

The magnetic moment values at (294K) of the $[M_2LCl_4].XH_2O$ M^{2+} = Cu, Ni, Co Table 3 show values (0.78, 1.57, 2.22)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[21-22].

Molar ratio

The complexes of the ligand (H_2L) 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[23]. A series of solutions were prepared having a constant concentration (C) 10^{-3} M of the hydrated metal salts and the ligand (H_2L). the (M:L) ratio was determined from the relationship between the absorption of the observed light and mole ratio (M:L) found to be (2:1). The results of complexes formation in solution are shown in Table (4), these data are compatible with the results obtained by atomic absorption for determination metal analysis in the complexes.

Thin layer chromatography (TLC)

The solution of ligand (H_2L) and its complexes in water as solvent appeared in one spot, this is confidence that all these compounds are pure and have one isomer. Table (1) shows the R_f for complexes and ligand (H_2L).

Conclusion

The ligand (H_2L) acts as a tetra-dentate dianion with two metal ions coordinate with each carboxylate group in an octahedral geometry. Figure (3)

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Table No. (1): Physical properties, analytical data for synthesized ligand (H_2L) and its complexes

Empirical formula	Color	m.p. °C	Yield (%)	Found(Calc.)(%)			R_f
				C	H	M(II)	
Ligand(L) $C_{10}H_6O_{10}Cl_2$	Yellow	138	68	33.1 (33.6)	1.47 (1.69)	–	0.62
$[Cu_2(C_{10}H_4O_{10}Cl_4.6H_2O)].5H_2O$	Brown	187	71	–	–	16.68 (16.91)	0.55
$[Co_2(C_{10}H_4O_{10}Cl_4.6H_2O)].2H_2O$	Red	122	65	–	–	16.96 (17.15)	0.48
$[Ni_2(C_{10}H_4O_{10}Cl_4.6H_2O)].3H_2O$	Green	118	74	–	–	16.43 (16.64)	0.50
$[Cd_2(C_{10}H_4O_{10}Cl_4.6H_2O)].2H_2O$	Yellow	167	77	–	–	28.81 (28.46)	0.43
$[Hg_2(C_{10}H_4O_{10}Cl_4.6H_2O)].H_2O$	white	173	58	–	–	42.21 (42.07)	0.37

Table No. (2): Assignments of the IR spectral bands of L-ascorbic acid, ligand(H_2L) and its complexes(cm^{-1})

Empirical formula	υ_{OH}	$\upsilon_{C=O}$	$\upsilon_{C=C}$ $\upsilon_{C=O}$	$\upsilon_{as(COO)^-}$	$\upsilon_{s(COO)^-}$	Coord. H_2O	υ_{M-O}
L-ascorbic acid $C_6H_8O_6$	3525,s 3410,s 3313,s 3213,s	1720	1678 1647	–	–	–	–
	υ_{COOH}						
Ligand(L) $C_{10}H_6O_{10}Cl_2$	3380,br	1718	1660,br	1566	1417	–	–
$[Cu_2(C_{10}H_4O_{10}Cl_4.6H_2O)]5H_2O$	3342,m	1743	1651,m	1400	1344	839,m	428
$[Co_2(C_{10}H_4O_{10}Cl_4.6H_2O)]2H_2O$	3448,br	1735	1640,m	1420	1344	844,s	476
$[Ni_2(C_{10}H_4O_{10}Cl_4.6H_2O)]3H_2O$	3377,br	1730	1654,m	1445	1384	840,m	420
$[Cd_2(C_{10}H_4O_{10}Cl_4.6H_2O)]2H_2O$	3473,br	1715	1662	1415	1361	829,s	425
$[Hg_2(C_{10}H_4O_{10}Cl_4.6H_2O)]H_2O$	3520,m	1720	1670	1425	1361	835,m	482

Table No. (3): Magnetic moments and electronic spectral bands (cm^{-1}) of the complexes.

V(ml)	L-Cu (λ=800)	L-Co (λ=510)	L-Ni (λ=740)	L-Cd (λ=340)	L-Hg (λ=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

Table No. (4) Molar ratio data for H_2L-complexes

Complex	μ_{eff} (B.M.)	Band position cm^{-1}	Assignments	$B_{complex}$	β	10Dq (υ_1) theoretical cm^{-1}	Λ_m Ω^{-1} cm^2mol^{-1}
L-Co(II)	2.25	19230 υ_3 15873 υ_2	$^4T_1g\rightarrow{}^4T_1g(p)$ $^4T_1g\rightarrow{}^4A_2g$	872.1	0.89	8284	11.93
L-Ni(II)	1.85	24390 υ_3 14556 υ_2 9259 υ_1	$^3A_2g\rightarrow{}^3T_1g_{(P)}$ $^3A_2g\rightarrow{}^3T_1g_{(F)}$ $^3A_2g\rightarrow{}^3T_2g$	649.2	0.63	10006	7.31
L-Cu(II)	0.82	12391	$^2Eg\rightarrow{}^2T_2g$	–	–	–	9.13
L-Cd(II)	–	39682	ILCT	–	–	–	11.40
L-Hg(II)	–	34482	ILCT	–	–	–	6.37

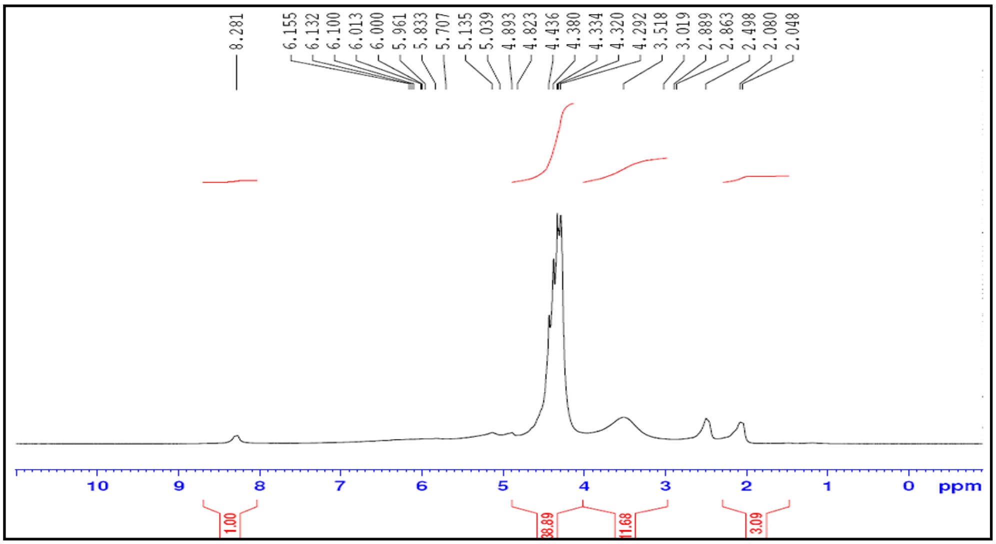

Figure (1): ^{1}H-NMR spectrum of the ligand

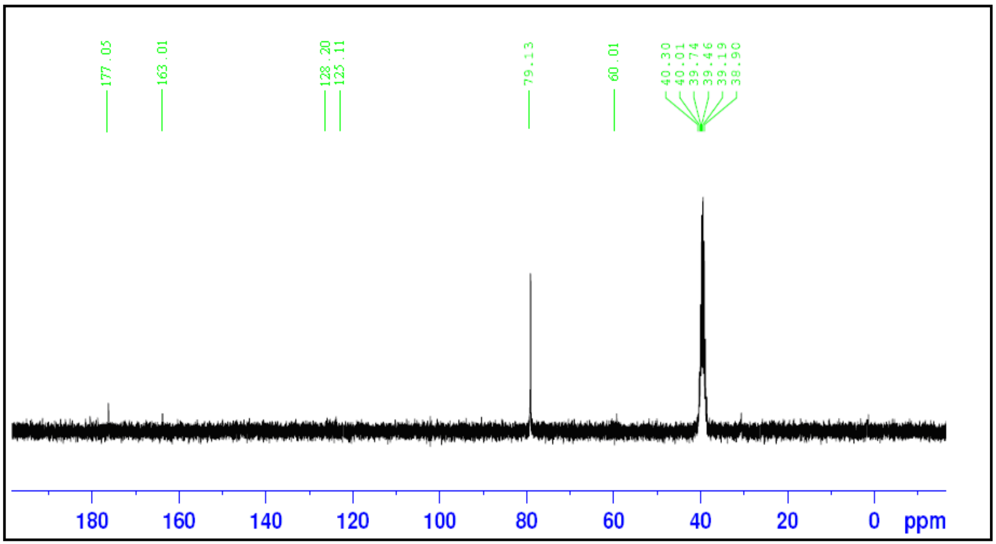

Figure (2): ^{13}C-NMR spectrum of the ligand

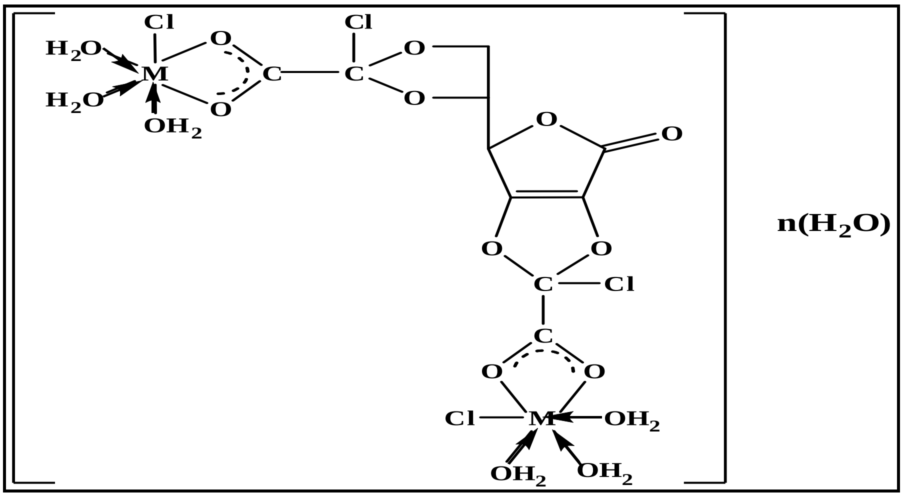

Figure (3): Suggested structure of $[M_2LCl_4.6H_2O]_nH_2O$

M = Cu(II), Hg(II) n = $2H_2O$, Co(II), Ni(II) n = $4H_2O$, Cd(II), n = H_2O

تحضير ودراسة طيفية لبعض المعقدات الفلزية مع ثنائي-6,5-O,O;3,2-O,O] (كلورو كاربوكسلك مثيليدين)] L- حامض الاسكوربك

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الخلاصة

أعطى تفاعل L–حامض الاسكوربك مع ثلاثي كلورو حامض الخليك بوجود هيدروكسيد البوتاسيوم ناتجاً جديداً هو ثنائي[6,5-O,O;3,2-O,O (كلورو كاربوكسلك مثيليدين)] L- حامض الاسكوربك (L)، فصل الناتج و شخص بوساطة التقنيات الاتية تحليل العناصر (H,C,N)، و الاشعة تحت الحمراء، و الاشعة فوق البنفسجية-المرئية، وطيف الرنين النووي المغناطيسي – البروتون H^1 و الكاربون C^{13} .

باستخدامه حضرت وشخصت معقدات املاح بعض ايونات العناصر الثنائية التكافؤ (Co, Ni, Cu, Cd و Hg) باستخدام تقنيات الاشعة تحت الحمراء، والاشعة فوق البفسجية-المرئية ، والتوصيلية الكهربائية ، والامتصاصية الذرية، والحساسية المغناطيسية ، والنسبة المولية. واستنتج من التحاليل ان تناسق الايونات الفلزية الثنائية التكافؤ مع اللكند (H_2L) ثم من خلال مجموعتي الكربوكسيل معطياً ايون الفلز سداسي التناسق، أظهرت كرومتوكرافيا الطبقة الرقيقة (TLC) ان اللكند (L) ومعقداته الفلزية هي مركبات نقية.

الكلمات المفتاحية : تحضير، L- حامض الاسكوربك، معقدات، تحليل