

Synthesis and Characterization of New Metal Complexes of α -Aminonitriles Derived from P- Toluidine and Aromatic Aldehydes

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Abstract:

New metal complexes of some transition metal ions Co(II), Cu(II), Cd(II) and Zn(II) were prepared by their reaction with previously prepared ligands HL_I = (P-methyl anilino) phenyl acetonitrile and HL_{II} = (P-methyl anilino) -P- chloro phenyl acetonitrile. The two ligands were prepared by Strecker's procedure which include the reaction of p- toluidine with benzaldehyde and P- chlorobenzaldehyde respectively. Structures were proposed depending on atomic absorption, i.r. and u.v. visible spectra in addition to magnetic susceptibility and electrical conductivity measurements.

Key words: complexes of α – Aminonitriles.

Introduction:

Aminonitrile compounds are well known to be biologically active species such as pharmaceutical interest and biocatalysis in industrial synthesis [1-3]. Beside that they were used as synthetic activating transcription for the explanation of biological reaction [4] and identified as biologically inhibitors [5-8]. Furthermore aminonitrile is potentially a chelating ligand [9,10]. These α -amino nitriles were synthesized by a modified Strecker's procedure [11,12], all above gave us the motives to synthesis and characterization of a new series of transition metal ions Co(II), Cu(II), Cd(II) and Zn(II) complexes by their reaction with two α -aminonitriles HL_I and HL_{II} which were previously prepared [13].

In this work α -aminonitriles coordination could occur through the nitrogen of α -amino group and the nitrogen of C \equiv N group, no evidence for such behavior is found. The

coordination chemistry of these ligands with metal ions was studied.

Materials and Methods:

Melting points (uncorrected) were obtained by using Gallenkamp MF-600-010 F melting point apparatus. Infrared spectra of metal complexes were recorded as CsI discs on SHIMADZU FTIR-8400S, Fourier Transform, Infrared Spectrophotometer. Electronic Spectra of the two ligands and their metal complexes in DMF were recorded on U.V. – visible Spectrophotometer Shimadzu U.V.-160 A. Magnetic susceptibility (μ_{eff} B.M) of metal complexes were measured at room temperature by Faraday method using Balance, Magnetic susceptibility, Model MSB-MK-1. Metal contents determined by using Varian – AA775, Atomic Absorption Spectrophotometer and Perkin – Elmer 5000 Atomic Absorption Spectrophotometer. Electrical conductivities of metal complexes at room temperature were

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measured by using Hunts Capacitors Trade Mark British .

Metal salts $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 99% (Fluka); $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ 99% (Merck) ; $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ 98.5% and $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ 99% (BDH) , ethanol absolute 99% (Fluka) were used as received from suppliers. Dimethyl formamide (DMF) was dried and distilled prior to use [14] .

Preparation of metal complexes ;

Ethanolic solution of metal salts $\text{Co}(\text{II})$, $\text{Cu}(\text{II})$, $\text{Cd}(\text{II})$ and $\text{Zn}(\text{II})$ (1m mole) were added to an ethanolic solution of ligand (2 mmole) with continuous stirring. The precipitation took place immediately. Each mixture heated under reflux for 30 minutes to

complete precipitation except the complex of C_4 which was precipitated immediately without heating. The products were filtered off, washed with ethanol and dried under vacuum.

Results and Discussion:

a) Physical data and atomic absorption :

Table (1) describes the physical properties of metal complexes, the suggested molecular formula were supported by atomic absorption , spectral studies and electrical conductivity measurements. Atomic absorption of some complexes showed less agreeable results because of technical errors in the instrument.

Table (1): Molecular formula , physical properties and atomic absorption of metal complexes for HL_I and HL_{II} .

symbol molecular formula	colour	M.P ^o c	Yield %	M% Found (calc.)
HL_I $\text{C}_{15}\text{H}_{14}\text{N}_2$	Yellow	102-103	90	-
C_1 $[\text{Co}(\text{HL}_I)_2] \cdot (\text{NO}_3)_2 \cdot \text{H}_2\text{O}$	Yellow	250 decomp.	42.61	9.13 (9.13)
C_2 $[\text{Cu}(\text{L}_I)_2] \cdot 0.2\text{H}_2\text{O}$	Brown	260-261	33.52	12.87 (12.45)
C_3 $[\text{Cd}(\text{HL}_I)_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$	Pale yellow	263	45.41	17.00 (17.31)
HL_{II} $\text{C}_{15}\text{H}_{13}\text{N}_2\text{Cl}$	Yellow	86-87	92	-
C_4 $[\text{Co L}_{II} \text{NO}_3(\text{H}_2\text{O})_3] \cdot 2.2\text{H}_2\text{O}$	Violet	230	34.22	12.56 (12.51)
C_5 $[\text{Cu L}_{II}\text{NO}_3(\text{H}_2\text{O})_2] \cdot 0.2\text{H}_2\text{O}$	greenish brown	> 260 decomp.	55.31	15.32 (15.10)
C_6 $[\text{Cd}(\text{L}_{II})_2(\text{H}_2\text{O})_2] \cdot 3\text{H}_2\text{O} \cdot \text{EtOH}$	White	250	60.01	14.35 (14.80)
C_7 $[\text{Zn L}_{II} \text{OAc} (\text{H}_2\text{O})_3] \text{H}_2\text{O}$	Off white	> 260 decomp.	42.5	14.70 (14.47)

b) Infrared spectra :

Important characteristic stretching frequencies of the ligands and their metal complexes are listed in table (2) and their spectra are shown in figure (1). The most important stretching modes exhibited by HL_I and HL_{II} are represented by α -amino and nitrile group. Bands related to stretching vibrations of aromatic and aliphatic C-H appeared at (3213-3032) and (2975-2800) cm^{-1} respectively [15-17]. The bands related to δ_{CH_3} and $\nu_{\text{C-N}}$

vibrations appeared at (1450-1358) and (1125-1088) cm^{-1} respectively [16,17]. The bands related to $\text{C}\equiv\text{N}$ stretching vibrations of free ligands HL_I and HL_{II} appeared at (2220) cm^{-1} [13,18] shifted to higher frequencies in complexes C_3 and C_7 as a result of coordination with metal ions through the lone pair electrons of nitrogen atom[10],while the spectra of complexes C_2 and C_4 showed shifts in $\text{C}\equiv\text{N}$ stretching vibrations to lower frequencies which indicates the linkage of $\text{C}\equiv\text{N}$ from

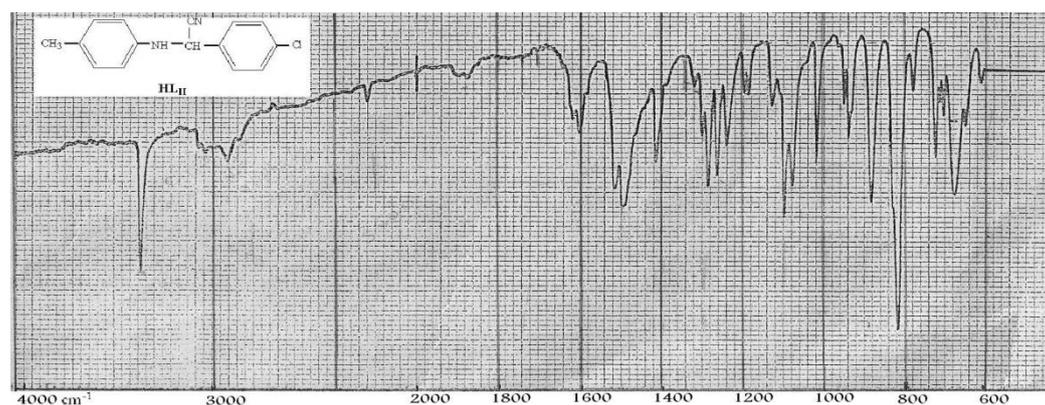
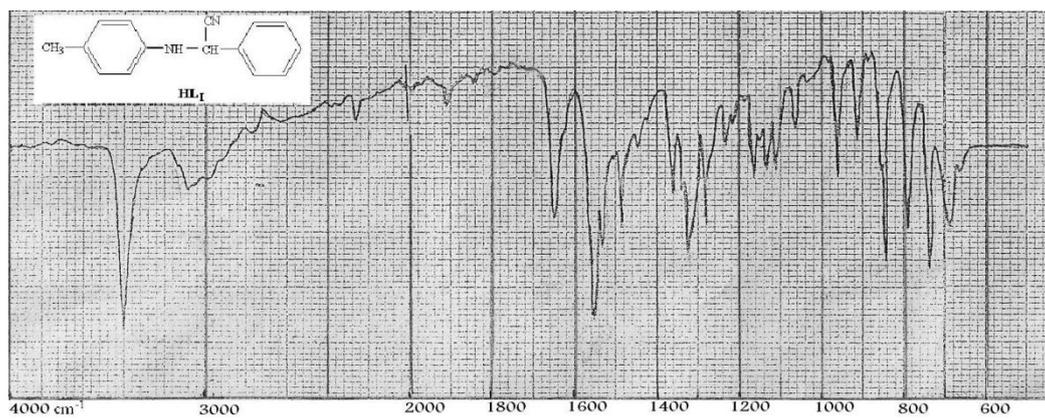
nitrogen atom, the decreases are attributed to metal $d\pi$ to ligand $p\pi^*$ back-bonding [10,19]. But the increase of $\nu_{C\equiv N}$ attributed to presence of π -acceptor ligands in to complexes C_3 and C_7 should decrease the back-bonding of electrons from the metal into the nitrile ligand [10]. The spectra of complexes C_1 , C_5 and C_6 showed splitting or broadening of $C\equiv N$ which assigned to coordination of $C\equiv N$ with metal ions[19]. The i.r spectra of complexes C_1 and C_3 showed shifts in stretching vibrations of ν_{N-H} and δ_{N-H} ($1582-1513$) cm^{-1} , but the complexes C_2 , C_4 , C_5 , C_6 and C_7 showed disappearance the bands related to ν_{N-H} and δ_{N-H} . In both cases the ligands coordinated with metal ions through the nitrogen atom of α -amino group [15]. The spectra of complexes $C_1 - C_7$ exhibited additional bands related to lattice and coordinate water vibrations [15,20]. The spectra of C_1 complex of Co(II) showed a broad band related to mixing vibrations of ν_{N-H} and lattice water. The spectra of the complex C_6

showed bands related to lattice ethanol appeared at 3538 , 1600 and 1050 cm^{-1} attributed to ν_{OH} , δ_{OH} and ν_{C-O} respectively [16,17]. Nitrate ions exhibited vibration modes related mainly to ionic behaviors in the complex Co(II) C_1 , while monodentate in the complexes Cd(II) C_3 and Co(II) C_4 and bidentate (chelating) bonding in the complex Cu(II) C_5 [20]. The vibrational of nitrate ions in the spectra of these complexes showed ionic nitrate vibrations > monodentate vibrations > bidentate bonding vibrations. Acetate ion in the complex Zn(II) C_7 showed monodentate behaviors [20,21]. Further bands appeared at lower frequencies which are assigned to $M-N\equiv C$, $M-N$ and $M-O$ stretching vibration [20,21-23]. Generally the shifts of $C\equiv N$ and $N-H$ stretching frequencies in all the metal complexes are believed that chelation of the metal ions in their α -amino nitrile's complexes occurs through N (nitrile) and N (amino).

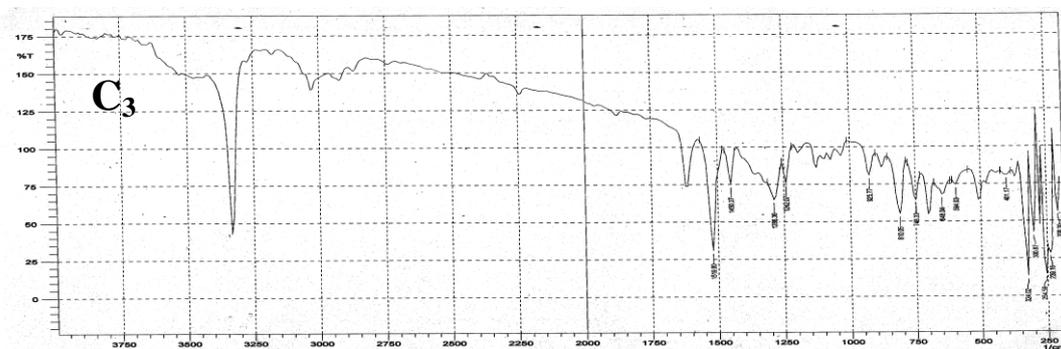
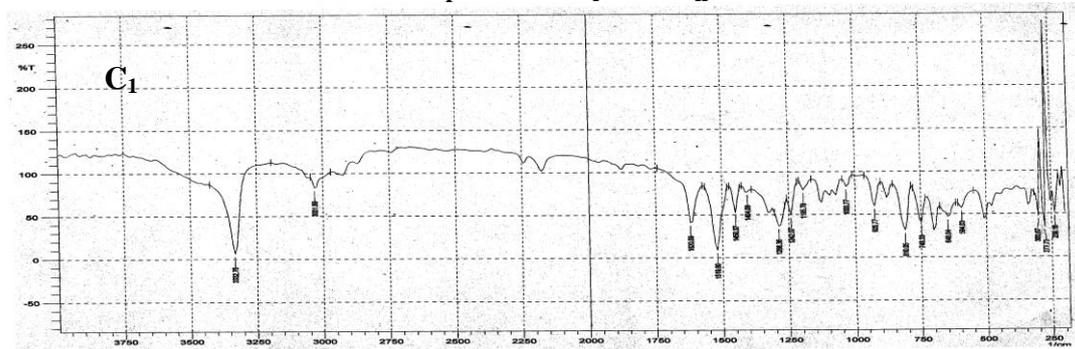
Table (2): Characteristic stretching vibrations $\nu(cm^{-1})$ of i.r spectra for ligands and their metal complexes .

Symbol	ν_{N-H}	$\nu_{C\equiv N}$	ν_{H_2O} Lattice (coordinate)	$\nu_{M-N\equiv C}$	ν_{M-N}	ν_{M-O}	ν_{NO_3} and others	ν_{C-H} out of plane
HL _I	3400	2220	-	-	-	-	-	840 790 740
C_1 Co(II)	3332	2236 2188	(3650-3450)sh.	400	(300) ^b	-	$\begin{pmatrix} 1775 \\ 1033 \\ 700 \end{pmatrix}^e$	860 810 748
C_2 Cu(II)	-	2168	(3448)br.	462	(340) ^a	-	-	802
C_3 Cd(II)	3350	2240	(3710-3520)br.	324	(277) ^b	(300) ^c	$\begin{pmatrix} 1350-1313 \\ 1020 \\ 700 \end{pmatrix}^f$	870 810 750
HL _{II}	3360	2220	-	-	-	-	-	820 780 720
C_4 Co(II)	-	2176	3448- 3386 (610)	332	(309) ^a	(278) ^c (447) ^d	$\begin{pmatrix} 1513-1413 \\ 1050 \\ 950 \end{pmatrix}^f$	825 700
C_5 Cu(II)	-	2168 2137	3440- 3217 $\begin{pmatrix} 675 \\ 625 \end{pmatrix}$	417	(313) ^a	(486) ^d $\begin{pmatrix} 285 \\ 255 \end{pmatrix}^c$	$\begin{pmatrix} 1380 \\ 1312 \\ 1013 \\ 950 \\ 713 \end{pmatrix}^g$	826 772 713
C_6 Cd(II)	-	2400 2075	3488	350	-	(450) ^d	-	813
C_7 Zn(II)	-	2225	3463 (613)	462	(310) ^a	(340) ^d (400) ^h	$\begin{pmatrix} 1600 \\ 1375 \end{pmatrix}^*$	875

Where : a = ν_{M-N} , b = ν_{M-NH} , c = ν_{M-ONO_2} , d = ν_{M-OH_2} , e = free ion of NO_3^- , f = monodentate of NO_3^- ; g = bidentate of NO_3^- ; h = ν_{M-AOC} and * = monodentate of acetat



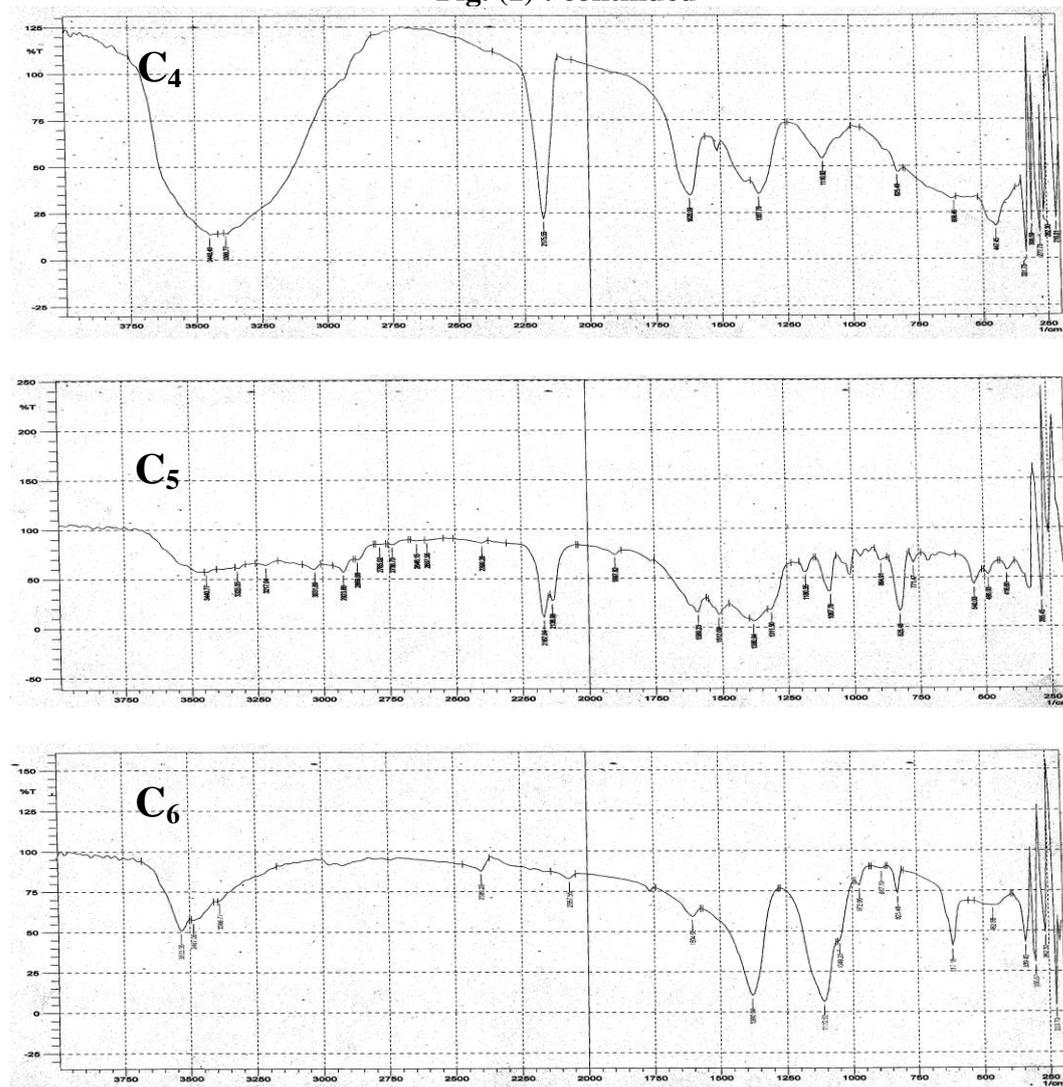
I.R spectra of HL_I and HL_{II}



FTIR Spectra of HL_I complexes C₁ of Co(II) and C₃ of Cd(II).

Fig. (1)

Fig. (1) : continued

FTIR Spectra of HL_{II} complexes C₄ of Co(II) ; C₅ of Cu(II) and C₆ of Cd(II)

c) Electronic Spectra , magnetic susceptibility and conductivity measurement :

The ligands HL_I and HL_{II} exhibited two bands in DMF, the first is sharp high intensity band appeared at (32051 and 32851)cm⁻¹ for HL_I and HL_{II} and the second is shoulder appeared at (29709 and 28813)cm⁻¹ respectively. The two bands are attributed to $\pi \rightarrow \pi^*$ transitions of aromatic ring and to conjugated of nitrile group (10, 16, 17). Bands related to $n \rightarrow \pi^*$ transition appeared at (29269 and 26562)cm⁻¹ for HL_I and HL_{II} respectively this transition may be masked by $\pi \rightarrow \pi^*$ bands (17).

Complexation of HL_I and HL_{II} with metal ions caused shifts with appearance of new bands in the visible and near i.r regions. These bands are attributed to M-L charge transfer and to ligand field transitions (24,25). Table (3) shows bands of maximum absorptions of HL_I , HL_{II} and their metal complexes figure (2).

i) Cobalt (II) Complexes C₁ and C₄ :

The spectra of complex C₁ showed a single band observed at (10101)cm⁻¹ and multiplet of three bands appeared at (12625, 14925 and 18518)cm⁻¹. The first band was attributed to ν_2 while the average value of the multiplet (15356)cm⁻¹ was assigned to ν_3 . In complex C₄ showed

two bands attributed to ν_2 and ν_3 . The transition of these bands for C_1 and C_4 are given in table (3) which exhibited the transition of tetrahedral Co(II) and octahedral Co(II) geometry respectively (24,25). Crystal field parameters (\bar{B} , Dq/\bar{B} , $10Dq$ and β) in Co(II) complexes and positions of absent bands were calculated by applying band ratios and energies of absorption bands on Tanabe Saugano diagrams. The value of β indicated covalent character (24,25). Magnetic moment of C_1 ($\mu_{\text{eff}}=3.98$ B.M) and C_4 ($\mu_{\text{eff}}=4.74$ B.M) agree with those of high spin tetrahedral and high spin octahedral Co(II) complexes respectively (24,25).

ii) Copper (II) complexes C_2 and C_5 :

The spectra of the complex C_2 showed two bands which exhibit the transition of square planar Cu(II) complexes (24,26), the band appeared at $(27275) \text{ cm}^{-1}$ in C_2 is due to charge transfer $L \rightarrow M$ transition (24). In spectra of complex C_5 showed two bands were attributed to spin allowed transitions of Jahn Teller tetragonally distorted octahedral Cu(II) complexes (24,25). Magnetic moment of C_2 was 1.83 B.M indicating a paramagnetic character which refers to square planar

geometry (27), while the magnetic moment of C_5 was 2.35 B.M, the high value was attributed to spin orbital coupling (25).

iii) Cadmium (II) complexes C_3 and C_6 :

The spectra of Cd(II) complexes showed no transition bands in the visible region except that of C.T transition which is quite familiar with d^{10} configuration where no d-d transition is involved (24,25). Complexation caused bathochromic shifts of HL_I and HL_{II} and these shifts are given in table (3). The solid complexes are diamagnetic materials.

vi) Zinc (II) complex C_7 :

The zinc (II) complex showed no ligand field transition for zinc ion. These bands resulted from either intraligand or charge transfer transition (24,25), which refers to a similar behavior to that of Cd(II) complexes. The solid complex are diamagnetic also.

Conductivity measurements of complexes in DMF ($10^{-3}M$) showed that they were electrolytes with ionic ratio of 1:2 for complexes C_1 , while the complexes $C_2 - C_7$ were found to be non electrolytic (28).

Table (3) : Electronic spectra data , electrical conductivities (DMF $10^{-3}M$) and suggested geometries for metal complexes of HL_I and HL_{II} .

Symbol	Maximum absorption ν max(cm^{-1})	Band assignment	Dq/\bar{B}	\bar{B} (cm^{-1})	B	$10 Dq$ (cm^{-1})	ν_2/ν_1	Molar conductivity in DMF $S.\text{mol}^{-1}.\text{cm}^2$	μ_{eff} B.M	Suggested structure
HL_I	ν_1 27909 ν_2 29269 ν_3 32051	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	-	-	-	-	-	-	-	-
C_1 Co(II)	ν_1 6048 cal ν_2 10101 ν_3 15357(avr.)	${}^4A_{2g} \rightarrow {}^4T_{2g}$ (F) ${}^4A_{2g} \rightarrow {}^4T_{1g}$ (F) ${}^4A_{2g} \rightarrow {}^4T_{1g}$ (P)	1.23	488	0.5	5996	1.67	163	3.98	Tetrahedral
C_2 Cu(II)	ν_1 13423 ν_2 18868 ν_3 27275	${}^2B_{1g} \rightarrow {}^2A_{1g}$ ${}^2B_{1g} \rightarrow {}^2B_{2g}$ $L \rightarrow M$ (C.T)	-	-	-	-	-	30	1.83	Square planar
C_3 Cd(II)	ν_1 29270 ν_2 31580	$L \rightarrow M$ (C.T) Intraligand $\pi \rightarrow \pi^*$	-	-	-	-	-	25	Diamagnetic	Octahedral
HL_{II}	ν_1 26562 ν_2 28813 ν_3 32851	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	-	-	-	-	-	-	-	-
C_4 Co(II)	ν_1 6045(cal.) ν_2 13484 ν_3 16667	${}^4T_{1g} \rightarrow {}^4T_{2g}$ (F) ${}^4T_{1g} \rightarrow {}^4A_{2g}$ (F) ${}^4T_{1g} \rightarrow {}^4T_{1g}$ (P)	0.65	801	0.83	5207	2.23	20	4.74	Octahedral
C_5 Cu(II)	ν_1 12376 ν_2 17392	${}^2B_{1g} \rightarrow {}^2A_{1g}$ ${}^2B_{1g} \rightarrow {}^2B_{2g}$	-	-	-	-	-	46	2.35	Octahedral
C_6 Cd(II)	ν_1 27700 ν_2 32894 ν_3 36101	$L \rightarrow M$ (C.T) Intraligand $\pi \rightarrow \pi^*$	-	-	-	-	-	29.7	Diamagnetic	Octahedral
C_7 Zn(II)	ν_1 22642 ν_2 28573 ν_3 32894	$L \rightarrow M$ (C.T) Intraligand $\pi \rightarrow \pi^*$	-	-	-	-	-	23	Diamagnetic	Octahedral

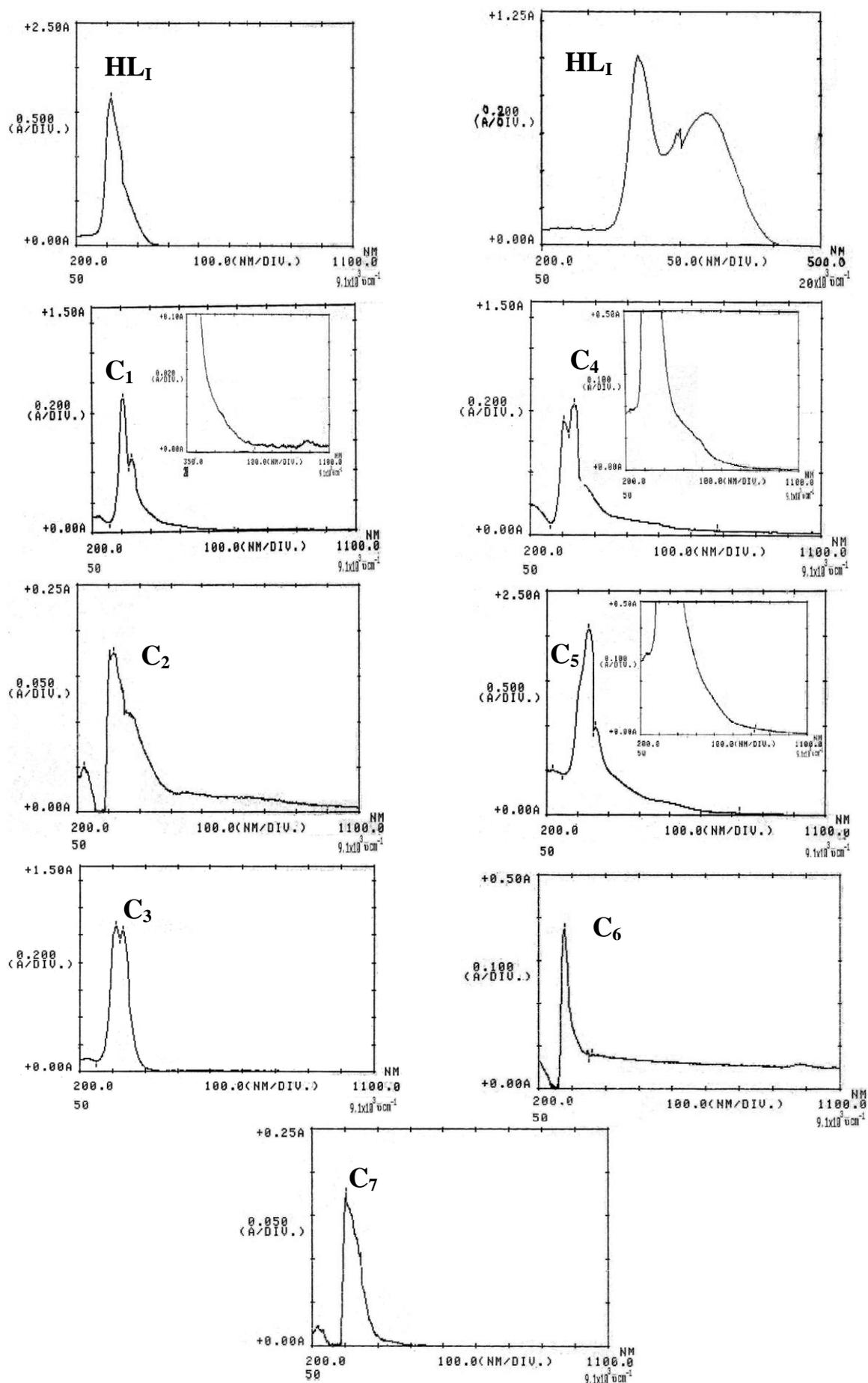
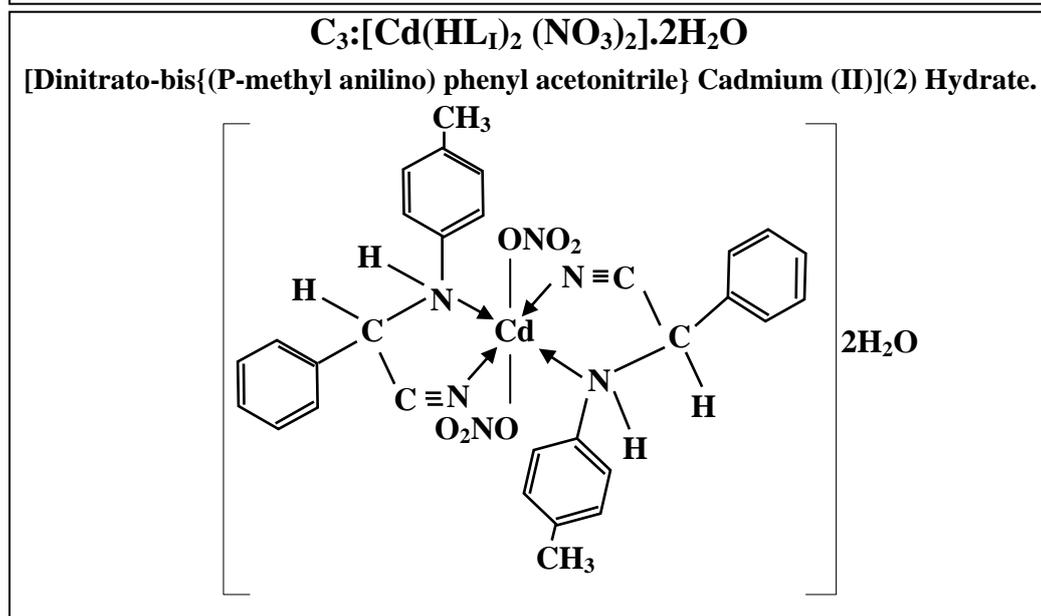
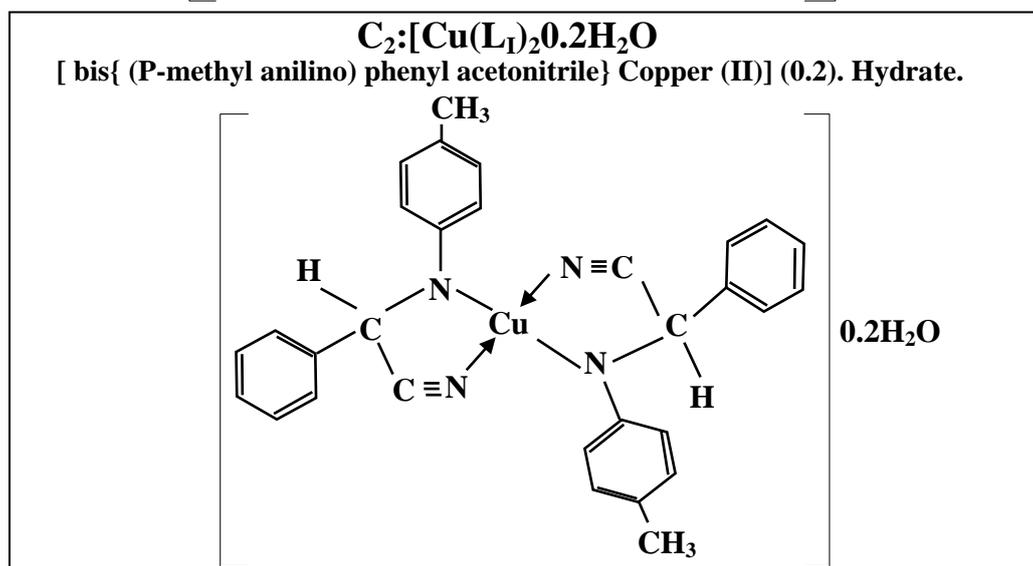
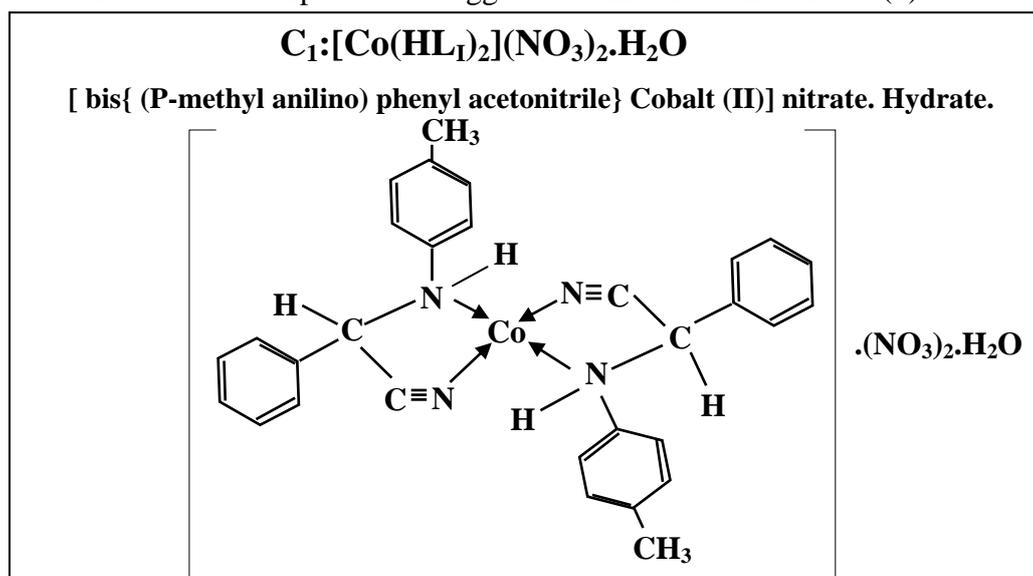
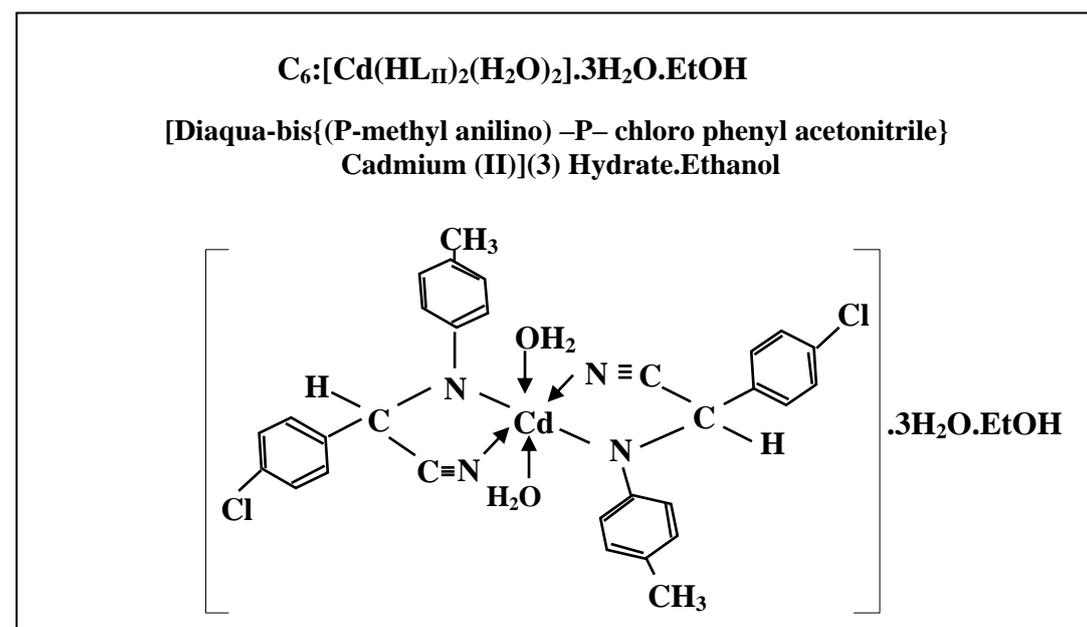
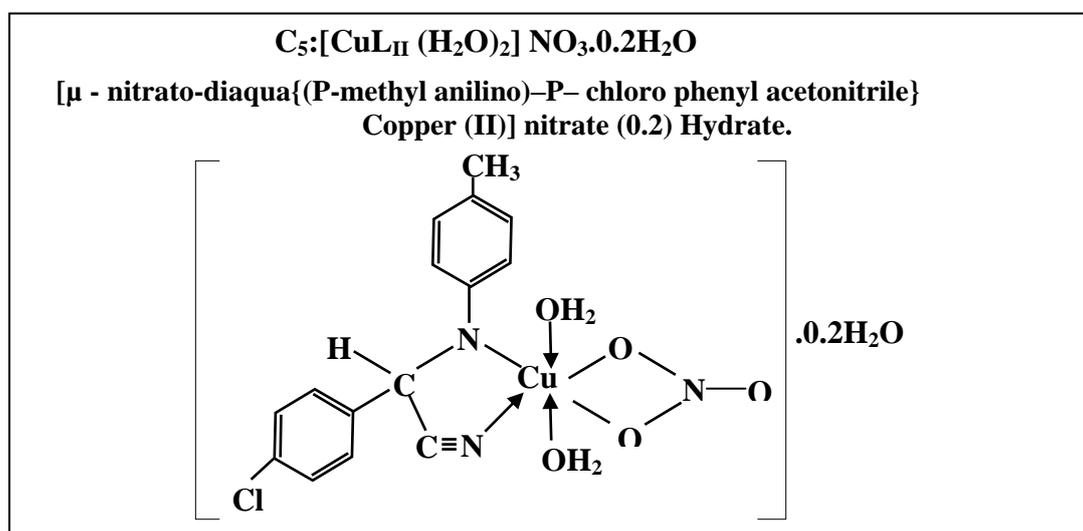
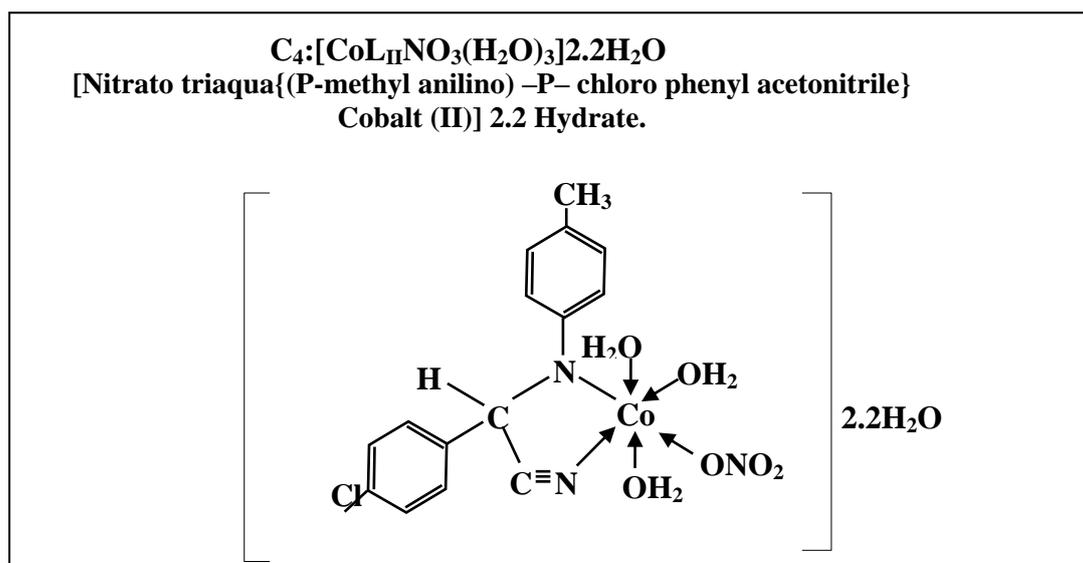
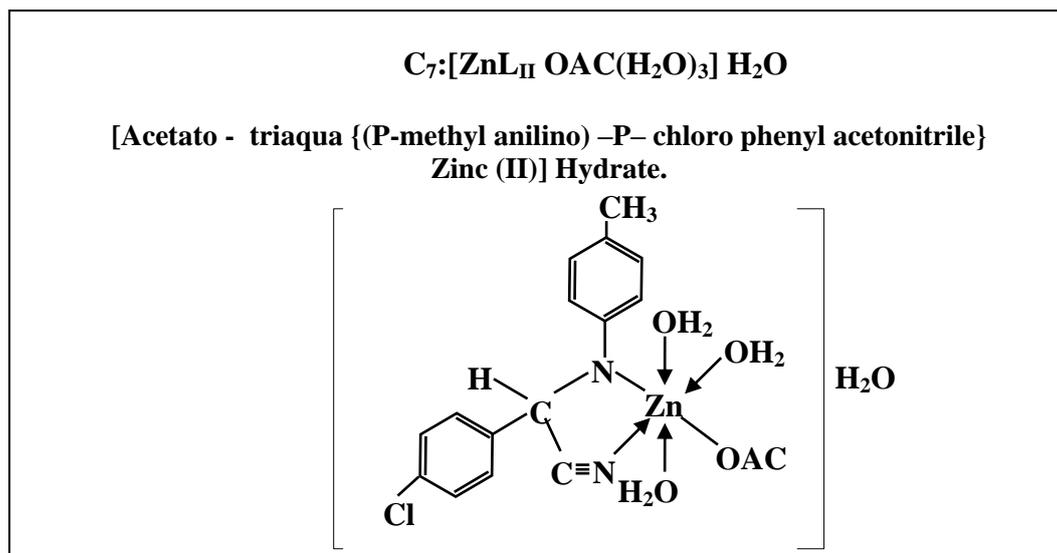


Fig. (2) : Electronic spectra of HL_I and their metal complexes (C₁, C₄) of Co(II) ; (C₂, C₅) of Cu(II) ; (C₃, C₆) of Cd(II) and C₇ of Zn(II) .

According to the observation obtained from atomic absorption and i.r spectra the structures of complexes are suggested as illustrated in scheme (1).







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تحضير وتشخيص معقدات فلزية جديدة لألفا – امينو نتريل مشتقة من باراتولودين مع الالديهيدات الأروماتية

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الخلاصة :

حضرت المعقدات الفلزية الجديدة لبعض ايونات العناصر الانتقالية Cd(II) ، Cu(II) ، Co(II) و Zn(II) من تفاعلها مع ليكاندين محضرين سابقاً HL_1 (بارا-مثيل انيلينو) فنيل اسيتونتريل و HL_{II} (بارا-مثيل انيلينو) – بارا-كلوروفنيل اسيتونتريل. حضر الليكاندان بطريقة ستريكر من مفاعلة بارا-تولودين مع البنزالديهيد وبارا-كلورو بنزالديهيد على التوالي. شخصت تراكيب المعقدات الفلزية الجديدة بوساطة الامتصاص الذري اللهيبي ، اطياف الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية فضلاً عن قياسات الحساسية المغناطيسية والتوصيلية الكهربائية للمعقدات الفلزية.