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 includ 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 pharmaccutical interest biocatyalysis and in industrial synthesis [1-3]. Beside that they were synthetic activating used as transcription for the explanation of biological reaction [4] and identified as biologically inhibitors [5-8]. Furthure more aminonitrile is potentially a chelating ligand [9,10]. These ∞ 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 ∞ -aminonitriles HL_{I} and HL_{II} which were previously prepared [13].

In this work α -aminonitriles coordination could occur through the nitrogen of ∞ -amino group and the nitrogen of C=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 melting F point apparatus. Infrared spectra of metal complexes were recorded as CsI discs on SHIMADZU FTIR-8400S, Fourier Transform, Infrared Spectro photometer. 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 $(\mu_{eff} B.M)$ 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 $Co(NO_3)_2.6H_2O$ 99% (Fluka); $Cu(NO_3)_2.3H_2O$ 99% (Merck); $Zn(OAC)_2.2H_2O$ 98.5% and $Cd(NO_3)_2.4H_2O$ 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 Co(II), Cu(II), Cd(II) and Zn(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 vaccum.

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°c	Yield %	M% Found (calc.)	
$\begin{array}{c} HL_{I}\\ C_{15}H_{14}N_{2} \end{array}$	Yellow	102-103	90	-	
C ₁ [Co(HL ₁) ₂].(NO ₃) ₂ .H ₂ O	Yellow	250 decopm.	42.61	9.13 (9.13)	
C_2 [Cu(L _I) ₂].0.2H ₂ O	Brown	260-261	33.52	12.87 (12.45)	
C_3 [Cd(HL ₁) ₂ (NO ₃) ₂].2H ₂ O	Pale yellow	263	45.41	17.00 (17.31)	
$\begin{array}{c} HL_{II}\\ C_{15}H_{13}N_2Cl \end{array}$	Yellow	86-87	92	-	
C ₄ [Co L _{II} NO ₃ (H ₂ O) ₃]2.2H ₂ O	Violet	230	34.22	12.56 (12.51)	
C ₅ [Cu L _{II} NO ₃ (H ₂ O) ₂].0.2H ₂ O	greenish brown	> 260 decomp.	55.31	15.32 (15.10)	
C_{6} [Cd(L _{II}) ₂ (H ₂ O) ₂].3H ₂ O.EtOH	White	250	60.01	14.35 (14.80)	
C ₇ [Zn L _{II} OAC (H ₂ O) ₃] H ₂ 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 ∞ -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_{CH3} and ν_{C-N} vibrations appeared at (1450-1358) and (1125-1088) cm⁻¹ respectively [16,17]. The bands related to C=N stretching vibrations of free ligands HL_I and HL_{II} appeared at (2220)cm⁻¹[13,18] shifted to higher frequencies in complexes C₃ and C₇ as a result of coordination with metal ions through the lone pair electrons of nitrogen atom[10],while the spectra of complexes C₂ and C₄ showed shifts in C=N stretching vibrations to lower frequencies which indicates the linkage of C=N from

nitrogen atom, the decreases are attributed to metal $d\pi$ to ligand $p\pi^*$ back-bonding [10,19]. But the increase of $v_{C=N}$ attributed to presence of π acceptor ligands in to complexes C_3 and C₇ should decrease the backbonding of electrons from the metal into the nitrile ligand [10]. The spectra of complexes C_1 , C_5 and C_6 showed splitting or broading of C=N which assigned to coordination of C=N with metal ions[19]. The i.r spectra of complexes C₁ and C₃ showed shifts in stretching vibrations of υ_{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 v_{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 v_{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 v_{OH} , δ_{OH} and v_{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)C₄ and bidentate (chelating) bonding in complex Cu(II) C₅[20].The 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=C, M-N and Mstretching vibration [20,21-23]. 0 Generally the shifts of C≡N and N-H stretching frequencies in all the metal complexes are believed that chelation of the metal ions in their ∞ -amino nitrile's complexes occurs through N (nitrile) and N (amino).

Symbol	$\upsilon_{\text{N-H}}$	υ _{C=N}	v _{H20} Lattice (coordinate)	υ _{M-N∎C}	$\upsilon_{M\text{-}N}$	$\upsilon_{M\text{-}O}$	v _{NO3} and others	γс-н out of plane
HL	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	-	i.	-	-	-	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	$ \begin{array}{c} 3440 \\ 3217 \\ (675 \\ 625 \end{array} $	417	(313) ^a	$(486)^d \\ \begin{pmatrix} 285 \\ 255 \end{pmatrix}^c$	$\begin{pmatrix} 1380\\ 1312\\ 1013\\ 950\\ 713 \end{pmatrix}^{s}$	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

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

Where : $a = v_{M-N}$, $b = v_{M-NH}$, $c = v_{M-ONO2}$, $d = v_{M-OH2}$, e = free ion of NO₃⁻, f = monodentate of NO₃⁻; g = bidentate of NO₃⁻; h= v_{M-AOC} and * = monodentate of acetat

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Fig. (1)



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 C_1 and C_4 :

The spectra of complex C_1 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 v_2 while the average value of the multiplet (15356)cm⁻¹ was assigned to v_3 . In complex C_4 showed two bands attributed to v_2 and v_3 . The transition of these bands for C₁ and C₄ are given in table (3) which exhibited the transition of tetrahedral Co(II) and octahedral Co(II)geometry respectively Crystal field parameters (24,25). $(\overline{B}, Dq/\overline{B}, 10Dq \text{ and } \beta)$ 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 (µ_{eff}=3.98 B.M) and C_4 (µeff=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 C₂ and C₅:

The spectra of the complexes C_2 and C_3 . 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) 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 C₃ and C₆:

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 C₇ :

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).

Symbol	Maximum absorption ນ max(cm ⁻¹⁾	Band assignment	Dq/ \overline{B}	$\overline{B}_{(cm^{-1})}$	В	10 Dq (cm ⁻ ¹)	υ ₂ / υ 1	Molar conductivity in DMF S.mol ⁻¹ .cm ²	μ _{eff} B.M	Suggested structure
HL	υ ₁ 27909 υ ₂ 29269 υ ₃ 32051	$\begin{array}{c} n \rightarrow \pi^{*} \\ \pi \rightarrow \pi^{*} \\ \pi \rightarrow \pi^{*} \end{array}$	-	-	-	-	-	-	-	-
C ₁ Co(II)	$v_1 6048 \text{ cal}$ $v_2 10101$ $v_3 15357(avr.)$	${}^{4}A_{2}g \rightarrow {}^{4}T_{2}g (F)$ ${}^{4}A_{2}g \rightarrow {}^{4}T_{1}g (F)$ ${}^{4}A_{2}g \rightarrow {}^{4}T_{1}g (P)$	1.23	488	0.5	5996	1.67	163	3.98	Tetrahedral
C ₂ Cu(II)	υ ₁ 13423 υ ₂ 18868 υ ₃ 27275	$\label{eq:B1g} \begin{split} ^2B_1g &\to {}^2A_1g \\ ^2B_1g &\to {}^2B_2g \\ L &\to M \ (C.T) \end{split}$	-	-	-	-	-	30	1.83	Square planar
C ₃ Cd(II)	$ \begin{array}{c} \upsilon_{1} \ 29270 \\ \upsilon_{2} \ 31580 \end{array} $	$L \rightarrow M (C.T)$ Intraligand $\pi \rightarrow \pi^*$	-	-	-	-	-	25	Diamagnetic	Octahedral
HL_{II}	$v_1 26562 v_2 28813 v_3 32851$	$\begin{array}{l} n \rightarrow \pi^{*} \\ \pi \rightarrow \pi^{*} \\ \pi \rightarrow \pi^{*} \end{array}$	-	-	-	-	-	-	-	-
C ₄ Co(II)	$v_1 6045(cal.)$ $v_2 13484$ $v_3 16667$	${}^{4}T_{1}g \rightarrow {}^{4}T_{2}g (F)$ ${}^{4}T_{1}g \rightarrow {}^{4}A_{2}g (F)$ ${}^{4}T_{1}g \rightarrow {}^{4}T_{1}g (P)$	0.65	801	0.83	5207	2.23	20	4.74	Octahedral
C ₅ Cu(II)	υ ₁ 12376 υ ₂ 17392	$^{2}B_{1}g \rightarrow ^{2}A_{1}g$ $^{2}B_{1}g \rightarrow ^{2}B_{2}g$	-	-	-	-	-	46	2.35	Octahedral
C ₆ Cd(II)	$\begin{array}{c} \upsilon_{1} 27700 \\ \upsilon_{2} 32894 \\ \upsilon_{3} 36101 \end{array}$	$L \rightarrow M (C.T)$ Intraligand $\pi \rightarrow \pi^*$	-	-	-	_	-	29.7	Diamagnetic	Octahedral
C ₇ Zn(II)	$\left[\begin{array}{c} \upsilon_{1} & 22642 \\ \upsilon_{2} & 28573 \\ \upsilon_{3} & 32894 \end{array}\right]$	$ \begin{array}{c} L \rightarrow M \ (C.T) \\ Intraligand \\ \pi \rightarrow \pi^* \end{array} $						23	Diamagnetic	Octahedral

Table (3) : Electronic spectra data , electrical conductivities (DMF 10^{-3} M) andsuggested geometries for metal complexes of HLI and HLII.



Cu(II); (C₃,C₆) of Cd(II) and C₇ of Zn(II).

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







C₆:[Cd(HL_{II})₂(H₂O)₂].3H₂O.EtOH

[Diaqua-bis{(P-methyl anilino) –P– chloro phenyl acetonitrile} Cadmium (II)](3) Hydrate.Ethanol





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

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الخلاصة:

حضرت المعقدات الفلزية الجديدة لبعض ايونات العناصر الانتقالية (Co(II) ، Co(II) ، Co(II) ، العناصر الانتقالية (Co(II) ، Co(II) ، الرار و و (Zn(II) من تفاعلها مع ليكاندين محضرين سابقاً HL= (بارا مثيل انيلينو) فنيل اسيتونتريل وHLI= (بارا مثيل انيلينو) – بارا - كلوروفنيل اسيتونتريل. حضر الليكاندان بطريقة ستريكر من مفاعلة بارا تولودين مع البنز الديهايد وبارا -كلورو بنز الديهايد على التوالي. شخصت تراكيب المعقدات الفلزية الجديدة بوساطة المتصاص الذري اللهبي ، اطياف الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية فضلاً عن قياسات الحساسية المغناطيسية والتوصيلية الكهربائية للمعقدات الفلزية.