

Synthesis and Characterization of Mn(II), Co(II), Ni(II) and Cu(II) Complexes with New Schiff Base Derived from Isophthalaldehyde and 2-amino-5-nitropyridine

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ABSTRACT

A series of new complexes of the type $[M_2LCl_4]$ and $[M_2L_2Cl_4]$ where $M=Mn(II)$, $Co(II)$, $Ni(II)$ and $Cu(II)$, $L=\{1,3-Bis(2-amino-5-nitropyridine)isophthylidene\}$ have been prepared. Adduct complexes of the type $[M_2L(PPh_3)_2Cl_2]Cl_2$ were $M= Co(II)$ and $Ni(II)$ were also prepared in 1:2 (complex: PPh_3) molar ratio. The complexes were characterized by molar conductance, magnetic moment, and FT-IR, UV-visible spectral studies and metal content analysis. Conductivity data showed that the complexes were non-electrolytes and the PPh_3 adducts were 1:2 electrolytes. Magnetic moments and electronic spectra indicate that the complexes have either tetrahedral or octahedral geometry while the complex Ni_2LCl_4 has square planer configuration.

Keywords: Schiff base, Isophthalaldehyde complexes, 2-amino-5-nitropyridine.

Cu(II) Ni(II) Co(II) Mn(II)

-5- -2

$[M_2L_2Cl_4]$ $[M_2LCl_4]$
{ (-5 -2) -3 1 } =L Cu(II) Ni(II),Co(II),Mn(II)=M
(L:M) 2:2 1:2 Ni(II),Co(II)=M $[M_2L(PPh_3)_2Cl_2]$
. (:PPh3) 1:2

C.H.N

(1:2)



$$-5 \quad -2 \quad :$$

INTRODUCTION

The chemistry of transition metal complexes with Schiff base ligands still attracting increasing attention by chemists and has undergone a spectacular growth during the last few years due to their involvement in catalytic processes and discovery of proteins and enzymes which required two or more metal ions for their activity (Patil, 2007).

Schiff bases have a remarkable property of forming binuclear complexes and serve as excellent chelating ligands (Patil, 2007 ; Guidote *et al.*, 2001; Zeyrek *et al.*, 2005; Brandt *et al.*, 2007). Schiff base metal complexes have broad applications and have been used as analytical reagents (Krishnan *et al.*, 2002), catalysts (Wu *et al.*, 2001) and showed anti-tumor (Phatak *et al.*, 2000), anti-viral (Karthikeyan *et al.*, 2006), anti-fungal (Wadher *et al.*, 2009) and anti-bacterial (Padmaja *et al.*, 2011) activities.

Schiff base complex of the type $[\text{Cu}_2\text{LX}_2]$ derived from the condensation of 1,3-diaminopropane and benzene-1,3-dicarboxaldehyde in the presence of CuX ($\text{X}=\text{Cl}^-, \text{Br}^-, \text{I}^-$) salt has been prepared and the single X-ray diffraction technique indicates that each Cu(I) ion coordinates with N_2X_2 donor sets having distorted tetrahedral geometry (Khandar *et al.*, 2010).

Phesico-chemical studies of the complex $[\text{Cu}_2\text{L}(\text{SCN})_2]$ derived from the condensation of 1,3-dicarboxaldehyde with 1,3-diaminopropane in the presence of $\text{Cu}(\text{CH}_3\text{CN})_4\text{ClO}_4$ and KSCN salt indicate that each metal ion has distorted trigonal planer geometry (Khandar *et al.*, 2011). Therefore, it was of interest to investigate the coordination behavior of the new Schiff base derived from isophthalaldehyde with some divalent transition metal chlorides.

EXPERIMENTAL

All chemicals used were of reagent grade purchased from B.D.H or Fluka companies, used as supplied.

Melting point or decomposition temperature were determined on electrothermal 9300 melting point apparatus. IR spectra were recorded on Tensor 27 Bruker FT-IR spectrophotometer ($400\text{-}4000\text{ cm}^{-1}$) using KBr disc. Electronic spectra were recorded in DMF or DMSO for (10^{-3}M) solutions on Shimadzu UV/Vis recording UV-160 spectrophotometer at 25°C using 1cm quartz cell. Molar conductance was measured for 10^{-3}M solution in DMF or DMSO using a conductivity meter model PCM3-Jenway. The magnetic susceptibility measurements of the complexes were carried out at 25°C on the solid state, by Faraday' method using Bruker BM6 apparatus. An elemental analysis of the ligand was performed on Perkin Elmer 2400 analyzer at Al-al-Bayt University (Jordan) Metal content of complexes was determined spectro- photometrically using Shimadzu AA670 atomic absorption spectrophotometer, chloride was determined using the precipitation method (Vogel, 1967).

Synthesis of 1,3-Bis(2-amino-5-nitropyridine) isophthylidene(L).

A clear solution of 2-amino-5-nitropyridine (2.78 g, 0.02 mole) in methanol (10 ml) was added slowly (dropwise) to a solution of isophthaldehyde (1.34 g, 0.01 mole) in methanol (10 ml), and the mixture stirred for about 30 min. The solution was then refluxed for 1h, after cooling, a yellow precipitate was formed filtered off, washed with cold methanol and recrystallized from methanol then dried under vacuum. Elemental analysis, (C:57.44(57.01), H:3.21(3.68), N:22.33(22.12) calc. (found)%.

Preparation of $[M_2LCl_4]$ and $[M_2L_2Cl_4]$ complexes

M=Mn(II),Co(II),Ni(II) and Cu(II)

The ligand L (0.37g, 0.001 mole or 0.75g, 0.002 mole) was dissolved in 10 ml methanol in a 100ml round bottom flask. A solution of (0.002 mole) of metal salt [$MnCl_2 \cdot 4H_2O$ (0.39g), $CoCl_2 \cdot 6H_2O$ (0.48g), $NiCl_2 \cdot 6H_2O$ (0.48g) or $CuCl_2 \cdot 2H_2O$ (0.34g)] in 10 ml absolute ethanol was added drop wise for 10 min. with continuous stirring at room temp. The mixture was refluxed for (1-2) h, and the precipitate appeared within 15 min, after cooling the precipitate filtered off, washed with hot methanol and diethylether then dried under vacuum.

Preparation of $[M_2(PPh_3)_2Cl_2]Cl_2$ complexes.

M=Co(II),Ni(II)

The adduct compounds were prepared by adding (0.53 g, 0.002 mole) of triphenylphosphine in (10ml) methanol to (0.001 mole) of complex (2 or 3) in (10 ml) DMF. After reflux with stirring for (2-3)h the precipitate formed was filtered off, washed with diethylether and dried.

RESULTS AND DISCUSSION

All complexes prepared in this paper are quite stable in dry air and decomposed above 200°C. They are insoluble in most organic solvent but soluble in DMF while adduct complexes soluble in DMSO.

Some physical properties of the ligand and its complexes are listed in Table (1). The electrical conductivity of complexes (1-8) in 10^{-3} M DMF solution are in the range (15.12-17.13) indicating the neutral nature of the complexes (Padmaja *et al.*, 2011; Jayaseelan *et al.*, 2011). The conductivity of the adduct complexes (9,10) in 10^{-3} M DMSO solution are (74.01) and (73.32) respectively indicating (1:2) electrolyte (Geary,1971). This is consistent with the stoichiometry assumed for complexes on the basis of the analytical data.

Table 1: Some physical properties and analytical data of the ligand and its complexes

NO.	Complexes	Color	d. (°C)	Cl% Calc. (found)	M% Calc. (found)	Λ_m ohm ⁻¹ cm ² mol ⁻¹
L	C ₁₈ H ₁₂ N ₆ O ₄	Yellow	200	---	---	---
1	[Mn ₂ LCl ₄]	Pale Yellow	278	22.58(22.21)	17.49(17.01)	15.12
2	[Co ₂ LCl ₄]	Green	283	22.29(22.10)	18.53(18.20)	17.10
3	[Ni ₂ LCl ₄]	Yellowish green	279	22.31(22.11)	18.47(18.12)	15.20
4	[Cu ₂ LCl ₄]	Green	239	21.97(21.67)	19.69(19.30)	17.12
5	[Mn ₂ L ₂ Cl ₄]	Yellow	254	14.11(14.01)	10.93(10.41)	10.79
6	[Co ₂ L ₂ Cl ₄]	Dark green	257	14.01(14.89)	11.64(11.32)	17.13
7	[Ni ₂ L ₂ Cl ₄]	Yellowish green	251	14.01(14.90)	11.60(11.33)	15.31
8	[Cu ₂ L ₂ Cl ₄]	Green	242	13.88(13.67)	12.43(11.84)	16.21
9	[Co ₂ L(PPh ₃) ₂ Cl ₂] ₂ Cl ₂	Dark green	222	12.22(12.09)	10.16(10.06)	74.01*
10	[Ni ₂ L(PPh ₃) ₂ Cl ₂] ₂ Cl ₂	Brown	244	12.23(11.99)	10.12(9.88)	73.32*

d = decomposition temperature

* = in (DMSO) solution

FT-IR spectra

The most important IR assignment of ligand and its complexes are listed in Table (2). The ligand show an intense band due to $\nu(\text{C}=\text{N})$ of azomethine group at 1633 cm⁻¹ consistent with the iminic absorption of free Schiff bases (Jayaseelan *et al.*, 2011). In all complexes this band undergoes a shift to higher frequencies and was observed at (1635-1653 cm⁻¹) (Ali *et al.*, 2001), this may be due to the increasing bond order of the C=N double bond on coordination with the metal ions as a result of electron donating of the other attached groups or due to the strain that occurred on coordination (Dayagi and Degai, 1970). The band at 1593 cm⁻¹ which assigned to $\nu(\text{C}=\text{N})$ in pyridine ring of the ligand was shifted to higher frequencies and appear at (1600-1618 cm⁻¹) in the spectra of the complexes. This suggests that the coordination takes place also through the nitrogen of the pyridine ring (Reddy *et al.*, 2005; Srivastava *et al.*, 2010). Complex (6) shows no change in the frequency of this band which indicates that the N_{ring} isn't shared in coordination.

The pyridine in-plane deformation mode observed at 638 cm⁻¹ in the spectrum of the ligand was shifted to the region (645-665 cm⁻¹) in the spectra of all complexes suggesting coordination of the heterocyclic nitrogen atom (Nakamoto, 1997) except for complex (6), this band doesn't show any shift which indicates there is no coordination with the nitrogen of the pyridine ring.

The ligand coordination is substantiated by a new band appearing at the ranges (418-496 cm⁻¹) for the complexes, these are mainly attributed to $\nu(\text{M}-\text{N})$ band, (Zeyrek *et al.*, 2005).

Table 2: Characteristic Infrared spectral data of the ligand and its complexes

No	Complexes	IR spectral bands (cm ⁻¹)			
		ν (C=N _{imin})	ν (C=N _{ring})	δ (Py _{ring})	ν (M-N)
L	C ₁₈ H ₁₂ N ₆ O ₄	1633 _s	1593 _m	638 _s	----
1	[Mn ₂ LCl ₄]	1645 _s	1614 _s	660	438 _m , 492 _m
2	[Co ₂ LCl ₄]	1645 _s	1612 _s	661 _s	442 _m , 474 _m
3	[Ni ₂ LCl ₄]	1644 _s	1612 _m	663 _s	420 _m , 445 _m
4	[Cu ₂ LCl ₄]	1635 _s	1600 _m	655 _s	432 _w , 462 _w
5	[Mn ₂ L ₂ Cl ₄]	1651 _s	---	665 _s	438 _m , 444 _m
6	[Co ₂ L ₂ Cl ₄]	1649 _s	1593 _s	638 _m	418 _m
7	[Ni ₂ L ₂ Cl ₄]	1647 _s	---	650 _s	432 _m , 452 _w
8	[Cu ₂ L ₂ Cl ₄]	1639 _m	1614 _m	648 _s	432 _m , 459 _w
9	[Co ₂ L(PPh ₃) ₂ Cl ₂] Cl ₂	1652 _m	1618 _s	650 _s	450 _m , 492 _m
10	[Ni ₂ L(PPh ₃) ₂ Cl ₂] Cl ₂	1653 _m	1600 _m	645 _m	436 _w , 451 _w

s=strong ,m= medium, w=weak

Electronic spectra and magnetism

The magnetic moments of the complexes and the electronic spectral data at 25°C of the ligand and its complexes are listed in Table (3). The electronic spectra of the ligand L in DMF show intense bands at 34722 cm⁻¹ and 27777 cm⁻¹ is attributed to benzene $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition of the non-bonding electrons present on the nitrogen of the azomethin group, these transitions are also found in the spectra of the complexes but their shifting, confirmed the coordination of the ligand to the metal ion (Zeyrek *et al.*, 2005).

The Mn(II) complexes (1,5) show a magnetic moments (5.75 and 5.85 B.M) which are appreciably close to the calculated spin-only value for five unpaired electrons, and reveal a high spin state. The electronic spectra of Mn(II) complexes show no prominent absorption that could be assigned for d-d transition. The d-d transition from ⁶A_{1g} towards the quartet terms ⁴T_{1g}(G), ⁴E_g(E), ⁴T_{2g}(G), ⁴T_{1g}(G) exhibit very weak intensity and are concealed by the intraligand transitions, and in tetrahedral environment these transitions are still spin-forbidden but no longer parity forbidden, these transitions are ~100 times stronger, therefore, the structures of the prepared Mn(II) complexes (1,5) were proposed depending on the data of other measurements namely metal content and IR spectra (Cotton *et al.*, 1999).

The magnetic moment values of Co(II) complexes (2,6,9) were in the range (4.32-4.51 B.M) indicating a tetrahedral geometry (Patil, 2007), their electronic spectra exhibit one transition ν_3 , ⁴A₂(F) \rightarrow ⁴T₁(p) consisting of two humps located at (14740-14832 cm⁻¹) and (16286-16450 cm⁻¹), the splitting of this band is expected to be due to the distortion of Jahn-Teller type of the tetrahedral structure in the excited state (Lei *et al.*, 2010), the other two bands ν_1 and ν_2 are below the spectrophotometer limits used.

The magnetic measurements of dinuclear Ni(II) complexes show that complex (3) is diamagnetic and its electronic spectra show two absorption bands at 16025 cm⁻¹ and 24875 cm⁻¹ assigned to ¹A_{1g} \rightarrow ¹A_{2g} and ¹A_{1g}(F) \rightarrow ¹B_{1g} transitions, respectively. This indicates a square planer geometry around Ni(II) ion (Raman *et al.*, 2007). The magnetic moment of complex (7) was found to be 3.09 B.M and the electronic spectra of this complex show three bands at 10080, 14534 and 24509 cm⁻¹ assigned to ³A_{2g}(F) \rightarrow ³T_{2g}(F), ³A_{2g}(F) \rightarrow ³T_{1g}(F) and ³A_{2g}(F) \rightarrow ³T_{1g}(p) transition, respectively, which indicate an

octahedral geometry in this complex (Jayaseelan *et al.*, 2011). The magnetic moment value of complex (10) is found to be 3.88 B.M and its electronic spectra show a band ν_3 at 11037 cm^{-1} due to ${}^3T_1(F) \rightarrow {}^3T_1(p)$ transition in tetrahedral geometry (Patil, 2007), the other two bands ν_1 and ν_2 are located in the lower field of the spectra, below the spectrophotometer limits used.

The obtained magnetic moment values per Cu(II) ion for complexes (4 and 8) were 1.69 and 1.81 B.M these values correspond to the presence of one unpaired electron for Cu(II) (Karaböcek *et al.*, 2007).

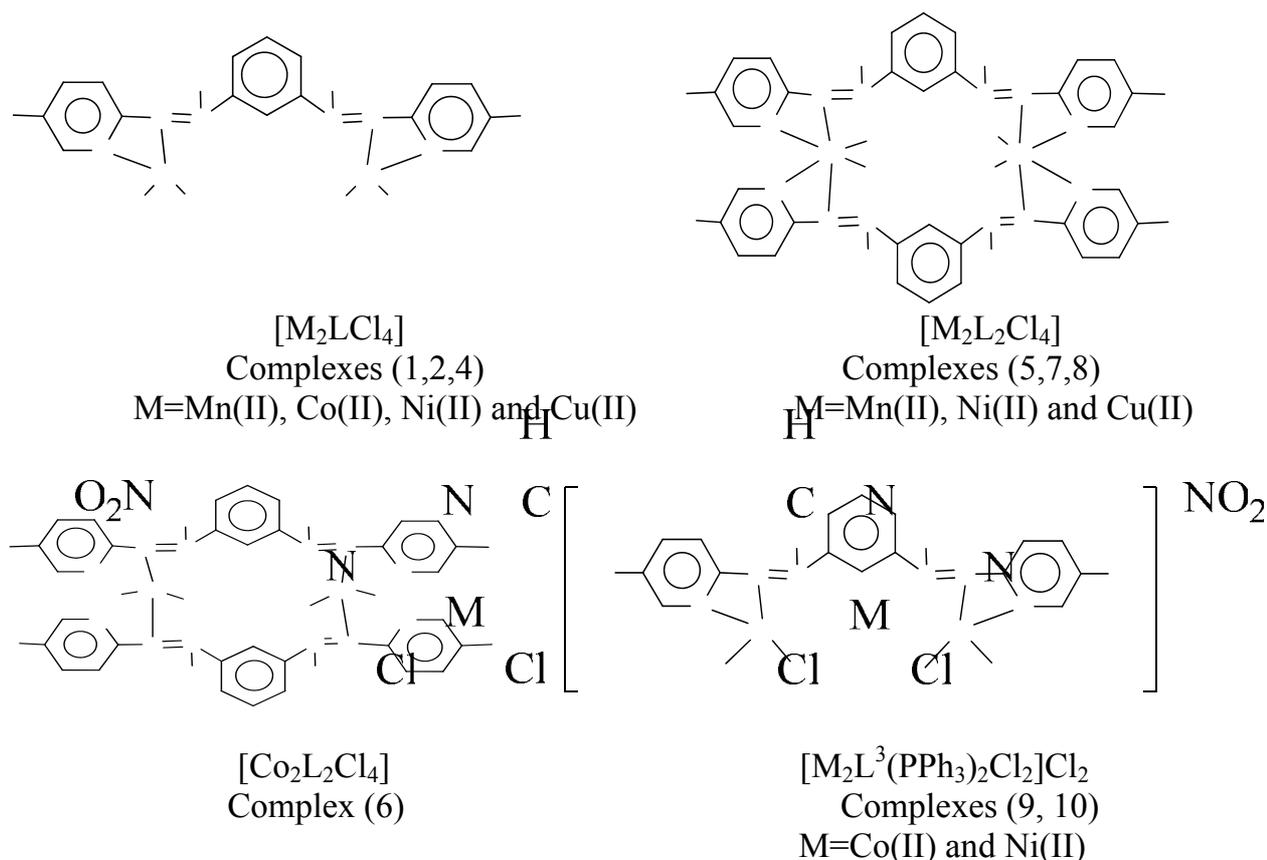
The electronic spectra of Cu(II) complex (4) showed a broad band at 10504 cm^{-1} attributed to ${}^2T_2 \rightarrow {}^2E$ transition which is comparable with complexes having a tetrahedral structure (Turan and Sekerci, 2009), while complex (8) showed a broad absorption band at 12482 cm^{-1} arises due to the d-d transition ${}^2E_g \rightarrow {}^2T_{2g}$ suggest that the Cu (II) ion exhibits an octahedral geometry (Jayaseelan *et al.*, 2010).

Table 3: Magnetic moments per metal ion and the electronic spectral data of the ligand and it's complexes

No.	Complexes	μ_{eff} (B.M)	Assignment	Band maxima λ (cm^{-1})	Struct.
L	$C_{18}H_{12}N_6O_4$	---	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	27777 34722	
1	$[Mn_2LCl_4]$	5.75	-----	-----	Td
2	$[Co_2LCl_4]$	4.32	${}^4A_2(F) \rightarrow {}^4T_1(p)$	14830,16441	Td
3	$[Ni_2LCl_4]$	Dia	${}^1A_{1g} \rightarrow {}^1A_{2g}$ ${}^1A_{1g} \rightarrow {}^1B_{1g}$	16025 24875	Sq.Pl.
4	$[Cu_2LCl_4]$	1.69	${}^2T_2 \rightarrow {}^2E$	10504	Td
5	$[Mn_2L_2Cl_4]$	5.85	-----	-----	Oh
6	$[Co_2L_2Cl_4]$	4.51	${}^4A_2(F) \rightarrow {}^4T_1(p)$	14832,16450	Td
7	$[Ni_2L_2Cl_4]$	3.09	${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$	10080 14534 24509	Oh
8	$[Cu_2L_2Cl_4]$	1.81	${}^2E_g \rightarrow {}^2T_{2g}$	12482	Oh
9	$[Co_2L(PPh_3)_2Cl_2] Cl_2$	4.38	${}^4A_2(F) \rightarrow {}^4T_1(p)$	14740,16286	Td
10	$[Ni_2L(PPh_3)_2Cl_2] Cl_2$	3.88	${}^3T_1(F) \rightarrow {}^3T_1(p)$	11037	Td

CONCLUSION

The ligand and its complexes were prepared and characterized by physio-chemical methods. The molar conductance of binuclear complexes $M=Mn(II)$, $Co(II)$, $Ni(II)$ and $Cu(II)$ show a non-electrolyt nature while the adduct compounds have 1:2 electrolyte. The spectroscopic data of metal complexes in which the coordination occur through the nitrogen of azomethin group and the nitrogen of the pyridine ring indicate that the Schiff base acts as tetradentate and acts as bidentate through azomethine groups only for complex (6). Hence, all complexes have a tetrahedral or octahedral structure except for complex (3) which has a square planer structure (Fig. 1).


Fig. 1: Suggested structure for complexes
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