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 [M2LCl4] and [M2L2Cl4] 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 [M2L(PPh3)2Cl2] were M= Co(II) and Ni(II) were also prepared in 1:2 (complex: PPh3) 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 PPh3 adducts were 1:2 electrolytes. Magnetic moments and electronic spectra indicate that the complexes have either tetrahedral or octahedral geometry while the complex Ni2LCl4 has square planer configuration.

Keywords: Schiff base, Isophthalaldehyde complexes, 2-amino-5-nitropyridine.
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 [Cu2LX2] derived from the condensation of 1,3-diaminopropane and benzene-1,3-dicarboxaldehyde in the presence of CuX(X=Cl-,Br-,I-) salt has been prepared and the single X-ray diffraction technique indicates that each Cu(I) ion coordinates with N2X2 donor sets having distorted tetrahedral geometry (Khandar et al., 2010).

Phesico-chemical studies of the complex [Cu2L(SCN)2] derived from the condensation of 1,3-dicarboxaldehyde with 1,3-diaminopropane in the presence of Cu(CH3CN)4ClO4 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 Brucker FT-IR spectrophotometer (400-4000 cm⁻¹) using KBr disc. Electronic spectra were recorded in DMF or DMSO for (10⁻³M) solutions on Shimadzu UV⁄Vis recording UV-160 spectrophotometer at 25°C using 1cm quartz cell. Molar conductance was measured for 10⁻³M 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 Brucker 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) isophthalidene (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 1 h, 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 [M₂LCl₄] and [M₂L₂Cl₄] 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 100 ml round bottom flask. A solution of (0.002 mole) of metal salt [MnCl₂.4H₂O(0.39g), CoCl₂.6H₂O(0.48g), NiCl₂.6H₂O (0.48g) or CuCl₂.2H₂O(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 [M₂(PPh₃)₂Cl₂]Cl₂ 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) | $\Lambda_m$ cm$^{-1}$ ohm$^{-1}$ cm$^2$mol$^{-1}$ |
|-----|-----------|--------------------|---------|------------------|------------------|-----------------------------------------------|
| 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   | [Co$_2$Cl$_4$] | Yellow         | 254     | 14.11(14.01)     | 10.93(10.41)     | 10.79                                          |
| 6   | [Ni$_2$Cl$_4$] | Yellowish green | 251     | 14.01(14.90)     | 11.60(11.33)     | 17.13                                          |
| 7   | [Cu$_2$Cl$_4$] | Green          | 242     | 13.88(13.67)     | 12.43(11.84)     | 16.21                                          |
| 8   | [Co$_2$(PPh$_3$)$_2$Cl$_2$]Cl$_2$ | Dark green | 222     | 12.22(12.09)     | 10.16(10.06)     | 74.01*                                         |
| 9   | [Ni$_2$(PPh$_3$)$_2$Cl$_2$] | Brown       | 244     | 12.23(11.99)     | 10.12(9.88)      | 73.32*                                         |

$\Lambda_m$ = 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 $\nu$(C=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 $\nu$(C=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$_{\text{ring}}$ 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 $\nu$(M-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=Nimin) | ν (C=Nring) | δ(Preim) | ν (M-N) |
| L   | C₁₈H₁₂N₆O₄ | 1633_S | 1593_m | 638_S | ---- |
| 1   | [Mn₂LCl₄] | 1645_S | 1614_S | 660 | 438_m,492_m |
| 2   | [Co₂LCl₄] | 1645_S | 1612_S | 661_S | 442_m,474_m |
| 3   | [Ni₂LCl₄] | 1644_S | 1612_m | 663_S | 420_m,445_m |
| 4   | [Cu₂LCl₄] | 1635_S | 1600_m | 655_S | 432_w,462_w |
| 5   | [Mn₂L₂Cl₄] | 1651_S | --- | 665_S | 438_m,444_m |
| 6   | [Co₂L₂Cl₄] | 1649_S | 1593_S | 638_m | 418_m |
| 7   | [Ni₂L₂Cl₄] | 1647_S | --- | 650_S | 432_m,452w |
| 8   | [Cu₂L₂Cl₄] | 1639_m | 1614_m | 648_S | 432_w,459_w |
| 9   | [Co₂L(PPh₃)₂Cl₂] | 1652_m | 1618_S | 650_S | 450_m,492_m |
| 10  | [Ni₂L(PPh₃)₂Cl₂] | 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 π → π* and n → π* 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 6A₁g towards the quartet terms ⁴T₁g(G), ⁴Eg(E), ⁴T₂g(G), ⁴T₁g(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 ν₃, ⁴A₂(F) → 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 ν₁ and ν₂ 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 ¹A₁g → ¹A₂g and ¹A₁g(F) → ¹B₁g 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 ³A₂g(F) → ³T₂g(F), ³A₂g(F) → ³T₁g(F) and ³A₂g(F) → ³T₁g(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 $\nu_3$ at 11037 cm\(^{-1}\) due to $^3T_1(F) \rightarrow ^3T_1(p)$ transition in tetrahedral geometry (Patil, 2007), the other two bands $\nu_1$ and $\nu_2$ 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 | $\mu_{\text{eff}}$ (B.M) | Assignment | Band maxima $\lambda_\nu$ (cm\(^{-1}\)) | Struct. |
|-----|-----------|---------------------------|------------|----------------------------------------|---------|
| L   | C\(_{18}\)H\(_{12}\)N\(_{6}\)O\(_{4}\) | --- | $n \rightarrow \pi^*$ | 27777 |         |
| L   | C\(_{18}\)H\(_{12}\)N\(_{6}\)O\(_{4}\) | --- | $\pi \rightarrow \pi^*$ | 34722 |         |
| 1   | [Mn\(_2\)LCl\(_4\)] | 5.75 | --- | --- | Td |
| 2   | [Co\(_2\)LCl\(_4\)] | 4.32 | $^4A_2(F) \rightarrow ^4T_1(p)$ | 14830,16441 | Td |
| 3   | [Ni\(_2\)LCl\(_4\)] | Dia | $^1A_1g \rightarrow ^1A_2g$ | 16025 | Sq.Pl. |
|     |             |             | $^1A_1g \rightarrow ^1B_1g$ | 24875 |         |
| 4   | [Cu\(_2\)LCl\(_4\)] | 1.69 | $^2T_2 \rightarrow ^2E$ | 10504 | Td |
| 5   | [Mn\(_2\)LCl\(_4\)] | 5.85 | --- | --- | Oh |
| 6   | [Co\(_2\)LCl\(_4\)] | 4.51 | $^4A_2(F) \rightarrow ^4T_1(p)$ | 14832,16450 | Td |
| 7   | [Ni\(_2\)LCl\(_4\)] | 3.09 | $^3A_2g(F) \rightarrow ^3T_2g(F)$ | 10080 | Oh |
|     |             |             | $^3A_2g(F) \rightarrow ^3T_1g(F)$ | 14534 |         |
|     |             |             | $^3A_2g(F) \rightarrow ^3T_1g(F)$ | 24509 |         |
| 8   | [Cu\(_2\)LCl\(_4\)] | 1.81 | $^2E_g \rightarrow ^2T_{2g}$ | 12482 | Oh |
| 9   | [Co\(_2\)L(PPh\(_3\))\(_2\)Cl\(_2\)] Cl\(_2\) | 4.38 | $^4A_2(F) \rightarrow ^4T_1(p)$ | 14740,16286 | Td |
| 10  | [Ni\(_2\)L(PPh\(_3\))\(_2\)Cl\(_2\)] 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 tetradeptate 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).
[M₂LCl₄]
Complexes (1,2,4)
M=Mn(II), Co(II), Ni(II) and Cu(II)

[Co₂L₂Cl₄]
Complex (6)

[M₂L₂Cl₄]
Complexes (5,7,8)
M=Mn(II), Ni(II) and Cu(II)

[M₂L₃(PPh₃)₂Cl₂]Cl₂
Complexes (9, 10)
M=Co(II) and Ni(II)

Fig. 1: Suggested structure for complexes

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