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Chloro-bridged complexes of copper(II) and manganese(II) derived from unsymmetric bidentate ligands: synthesis, crystal structure and characterization

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Two new usymmetric bidentate Schiff-base ligands (2-pyridyl-2-furylmethyl)imine (L1) and (2-pyridyl-phenylmethyl)imine (L2) were prepared. The crystal structures of two chloro-bridged complexes $[Cu_2(\mu-Cl)_2(L1)_2Cl_2]$ (1) and $[Mn (\mu-Cl)_2(L2)]$ (2) derived from the each ligand have been confirmed by single-crystal X-ray diffraction analysis. The complexes were characterized by IR, elemental analysis and spectroscopic methods. In complex 1, the two copper atoms are five-coordinate involving a square-pyramidal geometry having a N₂Cl₃ donor set with the two chlorine atoms bridging the two copper atoms. In complex 2, the manganese atoms are both six-coordinate. In contrast to 1, all chlorine atoms in 2 are bridging chlorides and link adjacent manganese atoms together forming 1-D infinite chains.

Keywords: Chloro-bridged; Crystal structure; Bidentate Schiff-base ligand; Copper(II) complex; Manganese(II) complex

1. Introduction

Attention has been directed to polynuclear complexes of transition metals with small anions (like chlorine or hydroxo) as bridging groups. Many of these polynuclear complexes are of interest because these complexes provide opportunities for crossing boundaries both within and between the fields of metallobiochemistry [1–2], materials science [3] and theoretical chemistry [4]. Among a variety of transition metal complexes, those containing chloro-bridges are of particular interest because they might represent valuable model compounds for the active site of a number of metalloenzymes and also for their interesting magnetic properties [1].

Unsymmetrical Schiff-base ligands have been investigated to synthesize model complexes for bioinorganic chemistry [5–9], catalysis [10–14], metalloproteins and medical chemistry [15]. Series of chloro-bridged transition metal complexes derived

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from Schiff-base ligands have been increasingly investigated [16–18]. The coordination chemistry of these complexes play an important role with respect to their properties [19, 20]. In this paper, two new unsymmetric Schiff-base ligands (2-pyridyl-2-furylmethyl)imine (L1) and (2-pyridyl-phenylmethyl)imine (L2) have been synthesized. The spectral properties and the crystal structure of complexes 1 and 2 derived from the two ligands are described.

2. Experimental

2. 1 Main reagents and apparatus

All reagents and solvents were of analytical or reagent grade, purchased commercially and used without further purification.

The C, H and N analyses were taken with a Perkin-Elmer model 2400 element analyzer. IR spectra were recorded on an EQUINOX 55 IR spectrophotometer by using KBr pellets. The UV-visible absorption spectra measurements were made on a UV-1700 spectrophotometer in the range of 200–800 nm.

2.2. Synthesis

2.2.1. Synthesis of Schiff-base ligand (2-pyridyl-2-furylmethyl)mine (L1). 2-Furylmethylamine (0.9705 g, 10.00 mmol) in methanol (8 mL) was added to a solution of an equimolar amount of 2-pyridinecarboxaldehyde (1.071 g, 10.00 mmol) in methanol (6 mL), and the mixed solution turned light yellow immediately. After stirring at room temperature for 12 h, the solvent was then removed by rotary evaporation to obtain a yellow hygroscopic liquid. The ligand was then used for metalation without further purification [21, 22].

2.2.2. Synthesis of Schiff base (2-pyridyl-phenylmethyl)imine (L2). This ligand was prepared in a manner similar to L1. L2 was also a yellow hygroscopic liquid.

2.2.3. Synthesis of $[Cu_2(\mu-Cl)_2(L1)_2Cl_2]$ (1). L1 (0.0603 g, 0.300 mmol) was dissolved in diethyl ether, and CuCl₂·2H₂O (0.0872 g, 0.300 mmol) in methanol. Using the method of three-layer diffusion, light-green crystals (m.p. > 573 K) suitable for X-ray diffraction formed after approximately 3 weeks in 70% yield. Anal. Calcd for Cu₂Cl₄C₂₂H₂₀N₄O₂ (%): C, 39.87; H, 3.35; N, 11.62. Found: C, 40.11; H, 3.21; N, 11.49.

2.2.4. Synthesis of $[Mn(\mu-Cl)_2(L2)]$ (2). A 7 mL ethanolic solution of $MnCl_2 \cdot 2H_2O$ (0.0511 g, 0.300 mmol) was added dropwise to a 6 mL ethanolic solution of L2 (0.0603 g, 0.700 mmol). The mixed solution turned orange at once.

Stirring at room temperature for 16 h, the resulting solution was filtered. Light-brown crystals (m.p. > 573 K) suitable for X-ray diffraction were grown about one week later by direct diffusion of diethyl ether into the resulting solution. The yield was 43%.

	Complex 1	Complex 2
Empirical formula	Cu2Cl4C22H20N4O2	MnCl ₂ C ₁₃ H ₁₂ N ₂
Formula weight	637.89	338.09
Temperature (K)	296(2)	296(2)
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	P2(1)/c
a (Å)	8.051(7)	11.127(7)
b (Å)	8.570(8)	18.313(12)
c (Å)	10.069(9)	6.925(4)
α (°)	77.570(2)	90
β (°)	72.119(2)	106.8360(10)
γΘ	70.2350(10)	90
$V(\text{\AA}^3)$	617.1(10)	1350.5(15)
Z	2	4
Radiation (λ, \dot{A})	Mo-Ka (0.71073)	Mo-Ka (0.71073)
$D_{\text{Calcd}} (\text{Mg cm}^{-3})$	1.726	1.663
Absorption coefficient (mm^{-1})	2.185	1.364
Crystal size (mm ³)	$0.34 \times 0.20 \times 0.05$	$0.38 \times 0.28 \times 0.10$
θ range for data collection (°)	2.14-25.09	1.91-25.10
Reflections collected	3114	6728
Independent reflections (R_{int})	2151(0.0190)	2406(0.0222)
Goodness-of-fit on F^2	1.078	1.040
Final R indices	$R_1 = 0.0360, wR_2 = 0.0903$	$R_1 = 0.0263, wR_2 = 0.0645$
R indices (all data)	$R_1 = 0.0547, wR_2 = 0.0952$	$R_1 = 0.0400, wR_2 = 0.0684$
Largest difference in peak and hole $(e \text{ Å}^{-3})$	0.366 and -0.403	0.244 and -0.285

Table 1. Crystal data of 1 and 2.

Anal. Calcd for MnCl₂C₁₃H₁₂N₂ (%): C, 48.48; H, 3.76; N, 8.70. Found: C, 48.01; H, 3.29; N, 8.92.

2.3. Crystal structure measurement

The data of complexes 1 and 2 were collected on a Bruker Smart-1000CCD diffractometer using graphite-monochromated Mo-K α radiaton (λ 0.71073 Å). Intensity measurements were performed from a sealed tube and a monocapillary collimator. SMART was used for preliminary determination of the cell constants and data collection control. The determination of integral intensities and global cell refinement were performed with the Bruker SAINT software package using a narrow-frame integrative algorithm. The structure was solved by direct methods (SHELXTL-97) and refined by full-matrix least-squares on F^2 . The final refinement included anisotropic displacement parameters for all atoms and a secondary extinction parameter. Crystallographic details are listed in table 1.

3. Discussion

3.1. Description of the crystal structure

3.1.1. Description of the crystal structure of complex 1. Selected bond lengths and angles of 1 are listed in table 2. The structural diagram and the crystal packing diagram along the *c* axis of complex 1 are depicted in figures 1 and 2, respectively.

Bond length			
Cu(1) - N(1)	2.033(3)	Cu(1)– $Cl(1A)$	2.2698(10)
Cu(1) - N(2)	2.043(3)	Cu(1)-Cl(2)	2.2522(11)
Cu(1)-Cl(1)	2.6497(10)	N(1)-C(5)	1.3550(4)
Bond angle			
N(1)-Cu(1)-N(2)	79.83(12)	N(2)-Cu(1)-Cl(2)	160.85(9)
N(1)-Cu(1)-Cl(2)	92.34(9)	N(1)-Cu(1)-Cl(1)	92.87(8)
N(2)-Cu(1)-Cl(1)	91.70(8)	Cl(2)-Cu(1)-Cl(1)	106.22(4)
N(1)-Cu(1)-Cl(1A)	172.95(9)	Cu(1)- $Cl(1)$ - $Cu(1A)$	90.65(3)

Table 2. Selected bond lengths ($\times 10^{-1}$ nm) and bond angles (°) of 1.



Figure 1. Perspective view of molecule for the 1.



Figure 2. Packing diagram of the 1 along the *c* axis.

The X-ray structure of 1 shows that both copper atoms have the same coordination. Each copper atom is defined by the nitrogen atom N(1) of the pyridine ring, N(2) which belongs to imino group, and three chlorine atoms. Each copper uses one chlorine to link to the other copper atom, and the other chlorine atom is non-bridging. The Cu(1), Cu(1A), Cl(1) and Cl(1A) atoms are coplanar. When $\iota = 1.0$ for the regular trigonal bipyramidal (RTB) and $\iota = 0.0$ for a regular square-based pyramidal (RSBP) stereochemistry [23], in complex 1, ι value of 0.19 [24], so both coppers in the N₂Cl₃ environment have a distorted square-pyramidal configuration (figure 1). The N(1), N(2), Cl(2) and Cl(1A) make up the basal plane, with Cl(1) as the vertex. The Cu resides in the N₂Cl₂ plane. The N(1)–Cu(1)–N(2) and N(1A)–Cu(1A)–N(2A) bond angles are 79.83°, the same and smaller than 90°, due to the strain caused by the three-bond ligand bite [25]. The sum of the inplane angles around both coppers is 359.1°.

The average bond length of Cu–N (2.038 Å) is slightly shorter than the three Cu–Cl (2.391 Å) bonds (table 2). Furthermore, the two ligands L1 are in the 'trans' position [26]. Complex 1 is stabilized by the interaction of C–H····Cl bond and forms many "grids" along c axis (figure 2).

Many dinuclear $Cu_2N_4Cl_4$ complexes are known in which two chlorides doubly bridge the coppers. Several of these complexes also have a distorted square-pyramidal geometry around the copper ions, in which the apical position of one copper is occupied by the chloride equatorial to the other copper [27–29]. The reported Cu–/N distances vary from 1.98 to 2.12 Å, the in-plane Cu–Cl distances vary from 2.19 to 2.31 Å, the axial Cu–Cl distances vary from 2.63 to 2.95 Å, the Cu–Cu distances vary from 3.42 to 3.74 Å and Cu–Cl–Cu angles vary from 87.3 to 94.88° [25]. The distances and angles in complex 1 lie between these values.

3.1.2. Description of the crystal structure of 2. Selected bond lengths and angles of 2 are listed in table 3. The coordination involves an infinite 1D chain along the *c* axis as shown in figures 3 and 4. The packing diagram along the *a* axis is represented in figure 5. In contrast to 1, as shown in figure 3, the coordination sphere of each six-coordinate manganese consists of one pyridine nitrogen atom, one nitrogen imine and four chlorine atoms from three different manganese atoms. The geometry of the manganese can best be described as octahedral. The N(1A), N(2A), Cl(2) and Cl(1B) are nearly coplanar. There are four bridging chlorides and two Mn–N bonds for each manganese atom. Bond lengths of Mn(1A)–N(2A), Mn(1A)–N(1A), Mn(1A)–Cl(1B), Mn(1A)–Cl(2), Mn(1A)–Cl(1A) and Mn(1A)–Cl(2A) (2.2802, 2.2807, 2.4748, 2.4621,

Table 3. Selected bond lengths ($\times 10^{-1}$ nm) and bond angles (°) of 2.

Bond length			
Mn(1A)– $Cl(1A)$	2.6914(6)	Mn(1A)-N(1A)	2.2807(15)
Cl(1B)-Mn(1A)	2.4748(6)	Mn(1)-Cl(2)	2.4616(6)
Mn(1A)-Cl(2A)	2.5723(6)	Mn(1A)-N(1A)	2.2807(15)
Mn(1A)-N(2A)	2.2802(16)	Mn(1A)-Cl(2)	2.4621(6)
Bond angle			
N(2A)-Mn(1A)-N(1A)	72.96(6)	N(2)-Mn(1)-Cl(2)	88.52(4)
N(1A)-Mn(1A)-Cl(2A)	95.01(4)	N(2)-Mn(1)-Cl(1)	86.33(4)
N(1)-Mn(1)-Cl(1)	80.39(4)	Cl(2A)-Mn(1A)-Cl(1A)	173.921(18)
Mn(1A)-Cl(1A)-Mn(1)	92.918(18)	N(2A)-Mn(1A)-Cl(2)	95.22(4)



Figure 3. The intact coordinated mode of manganese.



Figure 4. Infinite 1D chain of 2 along c axis.



Figure 5. Packing diagram of **2** along *a* axis.

2.5723 and 2.6914 Å) differ from each other (table 3). Bond lengths of Mn(1A)–Cl(1A) and Mn(1A)–Cl(2A) are longer than other bonds and the bond angle of Cl(1A)–Mn(1A)–Cl(2A)(173.921°) is the largest, so the Cl(2A), Mn(1A), Cl(1A) atoms compose the upright axis in the octahedron. The Mn₂Cl₂ diamond cores, which join related molecules, are strictly planar, as is also observed in the dimanganese(II) chloride complexes $[Mn_2(\mu-Cl)_2(bpea)_2Cl_2]$ (bpea = N,N-bis(2-pyridylmethyl)ethylamine) [30], $[Mn(\mu-Cl)_2(bpy)]_n$ (bpy = 2,20-bipyridine) [31] and $[Mn(\mu-Cl)_2(phen)]_n$ [32].

Compared with 1, the chlorine atoms connect with two other adjacent manganese atoms in 2, so the $[Mn(\mu-Cl)_2-(phen)]_n$ units which are similar to complexes [30–32] cited previously are linked by double μ_2 -bridging chlorides to form one-dimensional zigzag chains that run along the *c* axis of the crystal cell, as shown in figure 4. There may, however, be some weaker C–H···Cl interactions, as the C(11)–Cl(2) and C(4)–Cl(1) bond distances are found to be 3.582(3) and 3.732(6) Å, respectively, in complexes 1 and 2. With similar hydrogen bonding interactions, complexes 1 and 2 extend in space, forming three-dimensional structures (figures 2 and 5).

3.2. IR spectra

The IR spectrum of **1** is as follows: the absorption peak of $v_{C=N}$ is at 1598 cm⁻¹ [33, 34]; the absorption peak of δ_{CH2} is at 1445 cm⁻¹; the absorption peak of v_{CO-C} is at 1019 cm⁻¹; and the absorption peak of v_{C-H} in the furan ring is at 3121 cm⁻¹. The IR spectrum of **2** is as follows: the absorption peak of $v_{C=N}$ is at 1594 cm⁻¹; the absorption peaks of δ_{Ar-H} are at 699 and 776 cm⁻¹; and the absorption peak of δ_{CH2} is at 1442 cm⁻¹. For complexes **1** and **2**, there are strong absorption bands at 1600 cm⁻¹ which can be assigned to the C=N stretching vibration, indicating the formation of the Schiff base products [33, 34].

3.3. UV-visible spectra

The UV spectra of L1, L2 and complexes 1, 2 were all measured in anhydrous ethanol; the data are shown in table 4.

In the UV spectrum of L1, there are two peaks: at 232 nm attributed to the π - π * transitions of the benzene rings, and the latter peak attributed to the C=N imine group is observed at 272 nm [34]. In 1, the two peaks, which belong to ligand L1 and become broader, are all red-shifted to 247 and 328 nm, respectively, as a result of coordination. The third absorption peak at 736 nm is ascribed to the d-d transition of Cu(II).

The spectra of L2 has two peaks at 236 and 273 nm, also attributed to the π - π * and C=N imine group transitions. Similar to complex 1, the two peaks are red-shifted to

Compound	Formulas	⁷ ormulas $\lambda \max nm^{-1}$		
L1	$C_{11}H_{10}N_2O$	232	272	
Complex 1	$Cu_2Cl_4C_{22}H_{20}N_4O_2$	247	328	736
L2	$C_{13}H_{12}N_2$	236	273	-
Complex 2	$MnCl_2C_{13}H_{12}N_2$	242	326	-

Table 4. The UV-vis data of the ligands and complexes.

242 and 326 nm in **2**. The $Mn^{2+} d-d$ transition is not observed in the visible region, due to the $3d^5$ electronic configuration of Mn^{2+} [35].

There are no transitions observed in the visible region for the Schiff base ligands L1 and L2. The absorption peaks in 1 and 2 are all red-shifted and broader than the peaks in the ligands due to formation of M(II)–N(M = Cu, Mn) bonds which affect the π – π conjugative system [35].

4. Conclusion

In this study, we have synthesized and structurally characterized two new copper(II) and manganese(II) complexes with unsymmetrical Schiff-base binucleating ligands L1 and L2. The reported complexes show bridging chlorides between copper and manganese centers respectively. Complex 1 is binuclear, and 2 is a polymer. Further studies on other derivatives (e.g., adding different groups such as hydroxyl and carboxyl in ligands) and their metal complexes (e.g., complex of Cu(II) with L2 and complex of Mn(II) with L1 etc.) are underway in our laboratory.

Supplementary material

Crystallographic data for structures reported in the article have been deposited with the Cambridge Crystallographic Data Centre (CCDC) as supplementary material, CCDC-612855 & 612856, for complexes 1 and 2 respectively. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: (44)1223-336-033; Email: deposit@ccdc.cam.ac.uk or www: http// www.ccdc.cam.ac.uk).

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