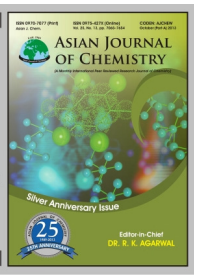
Asian Journal of Chemistry; Vol. 25, No. 14 (2013), 7867-7870

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.14663



Synthesis and Crystal Structures of One-Dimensional Chain Copper(II) Complexes of Two Phenol-Containing Schiff-Base Ligands

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(*Received*: 11 October 2012; *Accepted*: 22 July 2013) AJC-13829

Two copper(II) complexes of the tridentate Schiff-base ligands $(L^1)^-$ and $(L^2)^-$, $[CuL^1(\mu\text{-Br})]_\infty$ (**1**) and $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**), have been synthesized and characterized. The ligands $[(L^1)^-$ and $(L^2)^-$ are the deprotonated forms of 2-[(2-morpholin-4-ylethylimino)methyl]phenol (HL^1) and 2-[(2-piperidin-1-ylethylimino)methyl]phenol (HL^2), respectively]. X-Ray structure determinations carried out on complexes **1** and **2** reveal that both complexes are mono(μ-bromo)-bridged (for **1**) or mono($\mu_{1,3}$-azido)-bridged (for **2**) square pyramidal copper(II) one-dimensional chain polymeric complexes.

Key Words: Schiff-base, Copper(II) complexes, Tridentate ligand, Phenol, Thermal analysis.

INTRODUCTION

Over the years much attention has been paid to the polynuclear metal complexes due to their potential applications in the areas such as metalloenzyme models[1, 2], catalysts[3-6], magnetic materials[7-9] and photoelectronic devices[10-12]. A lot of different types of polynucleating ligands and their metal complexes have been reported[13-15]. Of these ligands, the Schiff-bases containing phenol units attracted particular attention. This is because the phenol group has many useful electronic and structural characteristics, such as the bridging capability of the phenol oxygen atom and the easy fuctionalization and great synthetic flexibility for the benzene ring present[16]. Many dinuclear and polynuclear complexes with phenol-containing Schiff-base ligands have been reported, which exhibit interesting properties[16-18]. For polynuclear complexes, their catalytic, biological and magnetic properties usually rely on the unsaturated coordination environments of the metal ions and the distance between the adjacent metal ions. We to report here the synthesis, crystal structures and properties of two new one-dimensional chain copper(II) complexes of $(L^1)^-$ and $(L^2)^-$ in the forms of $[CuL^1(\mu\text{-Br})]_\infty$ (**1**) and $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**) [$(L^1)^-$ and $(L^2)^-$ are the deprotonated forms of 2-[(2-morpholin-4-ylethylimino)methyl]phenol (HL^1) and 2-[(2-piperidin-1-ylethylimino)methyl]phenol (HL^2), respectively]. X-Ray structure determinations carried out on complexes **1** and **2** reveal that both complexes are mono(μ-bromo)-bridged (for **1**) or mono($\mu_{1,3}$-azido)-bridged (for **2**) square pyramidal copper(II) one-dimensional chain polymeric complexes.

EXPERIMENTAL

All chemicals and solvents were of reagent grade and were used as received. ^{1}H NMR spectra were recorded on a Bruker AVANCE 400 MHz spectrometer with tetramethylsilane as an internal reference. Infrared spectra were obtained on a Nicolet Avatar 360 FT-IR spectrometer as pressed KBr discs. Thermogravimetric measurements were carried out on a NETZSCH STA 449F3 thermal analyzer from room temperature to 1000 ºC in air with a heating rate of 10 ºC min^{-1}.

Synthesis of ligands HL^1 and HL^2: Ligands HL^1 and HL^2 were synthesized by condensation of salicylaldehyde (0.612 g, 5.0 mmol) with N-(2-aminoethyl)morpholine (0.650 g, 5.4 mmol) and N-(2-aminoethyl)piperidine (0.641 g, 5 mmol), respectively, in ethanol (30 mL) at room temperature. After rotary evaporation of the solvent and drying *in vacuo*, HL^1 was given as a yellow oil in 98 % yield and HL^2 as an orange oil in 97 % yield[19,20]. ^{1}H NMR data ($CDCl_3$, ppm): For HL^1: δ = 2.56 (t, 4H), 2.72 (t, 2H), 3.75 (m, 6H), 6.90 (t, 1H), 6.98 (d, 1H), 7.27 (m, 1H), 7.32 (m, 1H), 8.38 (s, 1H), 13.42 (s, 1H). For HL^2: δ = 1.46 (m, 2H), 1.62 (m, 4H), 2.50 (s, 4H), 2.68 (t, 2H), 6.88 (t, 1H), 6.97 (d, 1H), 7.30 (m, 2H), 8.37 (s, 1H), 13.51 (s,1H).

Synthesis of $[CuL^1(\mu\text{-Br})]_\infty$ (1): To a solution of ligand HL^1 (0.106 g, 0.45 mmol) in methanol (5 mL) was added

triethylamine (0.046 g, 0.45 mmol) in methanol (2 mL). To this resulting solution was added dropwise a deep yellow solution of copper(II) bromide (0.106 g, 0.45 mmol) in methanol (2 mL), giving a green solution. The resulting solution was stirred overnight and then evaporated slowly for 5 days, and finally green crystals formed. The crystals were collected by filtration, washed with methanol and dried *in vacuo* (0.019 g, yield: 11 %). Single crystals suitable for X-ray determination were obtained by vapour diffusion of diethyl ether into a methanol solution of complex **1**.

Synthesis of $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (2): Caution! Azide compounds are potentially explosive and should be handled with care and in small quantities. In particular, copper(II) azide is explosive and in those reactions where an excess of copper(II) salt is used, followed by addition of azide ions, an excess of azide should be avoided.

To a solution of ligand HL^2 (0.116 g, 0.50 mmol) in methanol (3 mL) were added triethylamine (0.051 g, 0.50 mmol) in methanol (3 mL) and sodium azide (0.032 g, 0.50 mmol) in methanol (3 mL). To this resulting solution was added dropwise a light blue solution of copper(II) tetrafluoroborate tetrahydrate (0.156 g, 0.51 mmol) in methanol (3 mL), leading to the formation of a precipitate. The resulting mixture was stirred overnight after which the violet solid was filtered off, washed with methanol and dried *in vacuo* (0.110 g, yield: 66 %). Single crystals suitable for X-ray determination were obtained by slow evaporation of a methanol solution of complex **2**.

X-Ray crystallography: All X-ray data were collected on a Bruker Smart Apex II CCD diffractometer using graphite-monochromated MoK_α radiation ($\lambda = 0.071073$ nm). The data were reduced using the SAINT program and semi-empirical absorption corrections (SADABS) were applied. The crystal structures were solved by direct methods and refined on F^2 by full-matrix least-squares methods with the SHELXTL-97 program[21]. All non-hydrogen atoms were made anisotropic. Hydrogen atoms were placed at calculated positions and rode on the atoms to which they are attached (including their isotropic thermal parameters which were equal to 1.2 times the equivalent isotropic displacement parameter for the attached non-hydrogen atom). Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (reference numbers CCDC 927118 and 927119). A summary of the key crystallographic information is given in Table-1.

TABLE-1
CRYSTALLOGRAPHIC DATA FOR **1** AND **2**

	1	**2**
Empirical formula	$C_{13}H_{17}BrN_2O_2Cu$	$C_{28}H_{38}N_{10}O_2Cu_2$
Formula weight	376.74	673.76
Temperature	295(2) K	296(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Monoclinic	Tetragonal
Space group	P2(1)/c	P4(3)2(1)2
Unit cell dimensions	a = 7.4413(15) Å b = 22.370(5) Å c = 8.6975(17) Å β = 105.60(3)°	a = 11.1663(5) Å b = 11.1663(5) Å c = 48.724(5) Å
Volume	1394.5(5) $Å^3$	6075.3(7) $Å^3$
Z, Calculated density	4, 1.794 Mg/m^3	8, 1.473 Mg/m^3
Absorption coefficient	4.430 mm^{-1}	1.444 mm^{-1}
$F_{(000)}$	756	2800
θ range for data collection	1.82 to 25.00°	1.67 to 25.00°
Limiting indices	-8 ≤ h ≤ 8, -25 ≤ k ≤ 26, -6 ≤ l ≤10	-12 ≤ h ≤ 13, -13 ≤ k ≤ 12, -57 ≤ l ≤ 57
Reflections collected/unique	7680/2446 [R_{int} = 0.0444]	42944/5349 [R(int) = 0.0725]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	2446/0/172	5349/0/380
Goodness-of-fit on F^2	1.021	1.147
Final R indices [I > 2σ(I)]	R_1 = 0.0319, wR_2 = 0.0631	R_1 = 0.0510, wR_2 = 0.1129
R indices (all data)	R_1 = 0.0531, wR_2 = 0.0694	R_1 = 0.0690, wR_2 = 0.1200
Largest diff. peak and hole	0.375 and -0.396 e $Å^{-3}$	0.893 and - 0.482 e $Å^{-3}$

RESULTS AND DISCUSSION

The copper(II) complexes of the deprotonated tridentate ligands $(L^1)^-$ and $(L^2)^-$, $[CuL^1(\mu\text{-Br})]_\infty$ (**1**) and $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**), were synthesized in methanol by mixing HL^1/HL^2, triethylamine, the appropriate copper(II) salt and sodium azide (for **2**, as the desired co-ligand). The X-ray crystal structure determinations reveal that complexes **1** and **2** are mono(μ-bromo)-bridged (for **1**) or mono($\mu_{1,3}$-azido)-bridged (for **2**) copper(II) 1D chain complexes (Figs. 1 and 2).

In the infrared spectra of complexes **1** and **2**, the imine bands occur at 1637 and 1638 cm^{-1}, respectively. For **2** there is also a strong and sharp band at 2044 cm^{-1} which is due to the asymmetric stretching vibration of the azide group. These values are similar to those reported for closely related structurally characterized copper(II) 1D chain complexes[20].

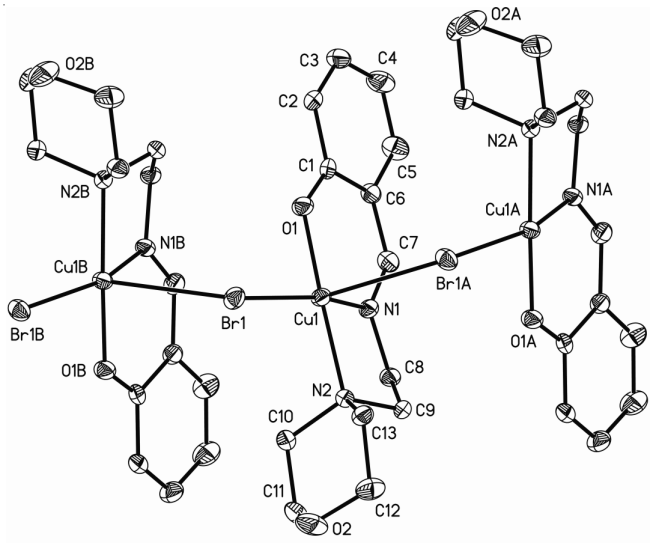

Fig. 1. Perspective view of the polymeric chain structure of the complex $[CuL^1(\mu\text{-Br})]_\infty$ (**1**). Thermal ellipsoids are drawn at the 30 % probability level. Hydrogen atoms have been omitted for clarity. Symmetry operations A is x, 0.5-y, z-0.5; B is x, 0.5-y, z + 0.5

X-Ray crystal structures of complexes 1 and 2: Complexes **1** and **2** are isostructural. The crystal structures of **1** and **2** are shown in Figs. 1 and 2, respectively and the selected bond lengths and angles are listed in Tables 2 and 3. Both structures consist of neutral $[CuL^1Br]$ (**1**) or $[CuL^2N_3]$ (**2**) units. These units are singly bridged by the bromide ions (**1**) or azide ions (**2**), forming polymeric 1D chain structures $[CuL^1(\mu\text{-Br})]_\infty$

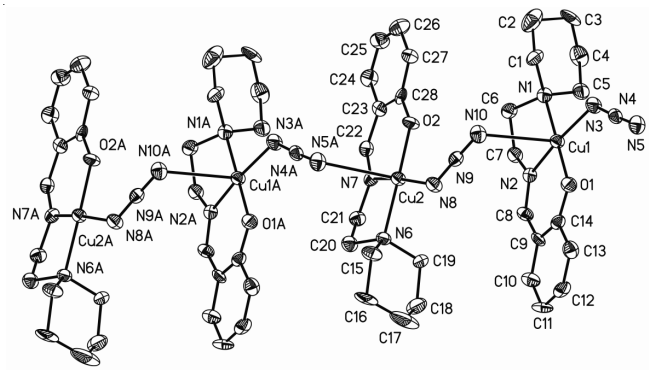

Fig. 2. Perspective view of the polymeric chain structure of the complex $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**). Thermal ellipsoids are drawn at the 30 % probability level. Hydrogen atoms have been omitted for clarity. Symmetry operations A is x, y-1, z

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (º) FOR COMPLEX **1**

Cu(1)-O(1)	1.901(2)	Cu(1)-N(1)	1.950(3)
Cu(1)-N(2)	2.115(3)	Cu(1)-Br(1)	2.4225(8)
Cu(1)-Br(1B)	2.9977(9)	Cu(1)···Cu(1A)	4.4313 (8)
O(1)-Cu(1)-N(1)	92.18(11)	O(1)-Cu(1)-N(2)	176.59(11)
N(1)-Cu(1)-N(2)	84.42(12)	O(1)-Cu(1)-Br(1)	90.34(7)
N(1)-Cu(1)-Br(1)	162.10(10)	N(2)-Cu(1)-Br(1)	92.98(8)
Br(1)-Cu(1)-Br(1A)	106.52(4)	Br(1A)-Cu(1)-O(1)	90.87(8)
Br(1A)-Cu(1)-N(1)	91.17(10)	Br(1A)-Cu(1)-N(2)	88.94(10)
Cu(1)-Br(1)-Cu(1B)	109.22(4)	–	–

Symmetry transformations used to generate equivalent atoms: A is x, 0.5-y, z-0.5 and B is x, 0.5-y, 0.5 + z.

TABLE-3
SELECTED BOND LENGTHS (Å) AND ANGLES (º) FOR COMPLEX **2**

Cu(1)-O(1)	1.904(5)	Cu(1)-N(1)	2.108(6)
Cu(1)-N(2)	1.921(5)	Cu(1)-N(3)	1.973(6)
Cu(1)-N(10)	2.788(6)	Cu(2)-O(2)	1.894(4)
Cu(2)-N(6)	2.044(5)	Cu(2)-N(7)	1.939(5)
Cu(2)-N(8)	1.975(6)	Cu(2)-N(5A)	2.830(6)
Cu(1)···Cu(2)	5.5823 (13)	Cu(2)···Cu(1A)	5.6694 (13)
O(1)-Cu(1)-N(2)	93.9(2)	O(1)-Cu(1)-N(3)	90.8(2)
N(2)-Cu(1)-N(3)	168.1(3)	O(1)-Cu(1)-N(1)	177.4(2)
N(1)-Cu(1)-N(2)	83.9(2)	N(1)-Cu(1)-N(3)	91.1(2)
O(1)-Cu(1)-N(10)	92.5(2)	N(1)-Cu(1)-N(10)	88.5(2)
N(2)-Cu(1)-N(10)	80.2(2)	N(3)-Cu(1)-N(10)	110.6(2)
N(4)-N(3)-Cu(1)	124.2(5)	N(9)-N(10)-Cu(1)	114.6(4)
O(2)-Cu(2)-N(7)	93.4(2)	O(2)-Cu(2)-N(8)	90.3(2)
N(7)-Cu(2)-N(8)	168.9(2)	O(2)-Cu(2)-N(6)	178.8(2)
N(6)-Cu(2)-N(7)	85.8(2)	N(6)-Cu(2)-N(8)	90.7(2)
O(2)-Cu(2)-N(5A)	91.53(19)	N(5A)-Cu(2)-N(6)	87.5(2)
N(5A)-Cu2-N(7)	78.7(2)	N(5A)-Cu(2)-N(8)	111.7(2)
N(9)-N(8)-Cu(2)	123.1(4)	N4-N5-Cu(2A)	115.3(5)
N(8)-N(9)-N(10)	177.9(7)	N(3)-N(4)-N(5)	176.8(7)

Symmetry transformations used to generate equivalent atoms: A is x, y-1, z.

(**1**) and $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**). For **2** two crystallographically independent consecutive $[CuL^2N_3]$ units are present in the chain structure. The azide ions bridge these repeating double units in an end-to-end fashion.

In both cases the arrangement of donor atoms around copper(II) centre can be described as a distorted square pyramid according to the Addison τ values of 0.24 for **1** and 0.16 for **2**[22]. Each copper ion is coordinated to two nitrogen atoms and one oxygen atom of the deprotonated tridentate ligand $(L^1)^-$ or $(L^2)^-$ and two bromide ions (**1**) or two nitrogen atoms from two bridging azide ions (**2**), giving an N_2OBr_2 (**1**) or N_4O (**2**) coordination environment overall. The basal coordination sites are occupied by the three donor atoms of the Schiff-base ligand and the bromine atom Br(1) for **1** or one nitrogen atom [N(3) or N(8)] of the azide ligand for **2**, while the apical position is occupied by the bromine atom [Br(1A)] for **1** or one nitrogen atom [N(10) or N(5A)] of the azide ligand of the next repeating unit. The apical Cu-Br(1A) (**1**) or Cu-N(azide) [Cu(1)-N(10), Cu(2)-N(5A)] (**2**) bond is significantly longer than the corresponding basal Cu-Br(1) (**1**) or Cu-N [Cu(1)-N(3), Cu(2)-N(8)] (**2**) bond (Tables 2 and 3), a feature which appears frequently in square-pyramidal copper(II) complexes[23-26]. The copper atom is located 0.1682(15) Å for **1** or 0.1063(29) and 0.0828(29) for **2** out of the basal plane towards the apical ligand. In the coordination polyhedron, the average value of the Br(apical)-Cu(1)-atom(basal) angles in 1 is 94.4º, while the corresponding value of the N(apical)-Cu(1)-atom(basal) or N(apical)-Cu(2)-atom(basal) angles in **2** is 93º or 92.4º. In addition the average value of the *cis* basal angles in **1** is 90º, while the corresponding value of the *cis* basal angles in **2** is 89.9º or 90.0º. So these average angle values in **1** are very close to those observed in **2**. The bond lengths and angles in **1** and **2** compare well with the values reported for related copper(II) complexes[19,20,24,27-30].

Mono(μ-bromo)-bridged copper(II) chain complexes are scarce. Only a few structurally characterized complexes have been reported to date[24,27,28,31]. The polymeric 1D chain structure is the most unusual feature of the crystal structure for complex **1**. The basal and apical Cu-Br bond lengths in complex **1** are close to those observed in these reported chain examples (2.382-2.469 Å for basal Cu-Br bond and 2.856-3.027 Å for apical Cu-Br bond). In **1** the Cu-Br-Cu angle is 109.22(4)º, which lies in the reported range 102.2-134.0º. Correspondingly, the intrachain minimum Cu···Cu separation, 4.431 Å, is in the literature range 4.189-4.851 Å.

In the 1D chain structure of complex **2** the basal Cu-N(azide) bond lengths [1.973(6) Å for Cu(1)-N(3), 1.975(6) Å for Cu(2)-N(8)] are close to those observed in the reported chain examples[20,29,32-34]. However, the apical Cu-N(azide) bond lengths [2.788(6) Å for Cu(1)-N(10) and 2.830(6) Å for Cu(2)-N(5A)] are significantly longer than literature values (2.355-2.621 Å) for the related chain structures. This indicates that $[CuL^2N_3]$ units are weakly linked through the end-to-end azide bridges, resulting in a 1D chain polymer. The intrachain minimum Cu···Cu separations, 5.5823(13) Å for Cu(1)-Cu(2) and 5.6694(13) Å for Cu(1)-Cu(2A), are similar to the literature values reported for related singly azide-bridged copper(II) chain structures.

Thermal stability: Thermogravimetric analyses were carried out on complexes **1** and **2** from room temperature to 1000 ºC in air with a heating rate of 10 ºC min^{-1}. The TGA curves are shown in Fig. 3. The TGA curve for complex **1** shows that **1** decomposed in four steps. The first step in the temperature range of 29-320 ºC accompanied with weight loss of 21.12 % (calcd. 21.21 %). This may be attributed to the loss of a bromide ion. The second step took place within the temperature range 320-450 ºC with weight loss of 10.87 %

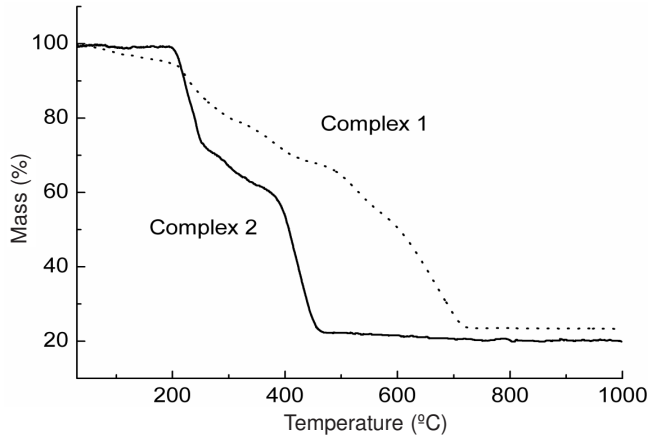

Fig. 3. TGA curves of complexes **1** and **2**

(calcd. 10.90 %), which may correspond to the decomposition of the linker between the benzene and morpholine rings and the loss of CH and CH_2CH_2 groups. The next two steps, which occurred within the temperature range 450-710 ºC, may involve the decomposition of the benzene and morpholine rings with weight loss of 43.01 % (calcd. 43.06 %). The final residue was calculated to be CuO with found weight loss of 23.66 % (calcd. 21.12 %).

The TGA curve for complex **2** shows that **2** is stable up to 200 ºC. It then decomposed in three steps. The first step in the temperature range of 200-255 ºC occurred with weight loss of 27.00 % (calcd. 26.45 %), which may be attributed to the loss of C_6H_4CH moiety. The second step took place within the temperature 255-375 ºC with weight loss of 13 % (calcd. 12.47 %). This is probably due to the loss of an azide ion. The third step, which occurred in the temperature range 375-475 ºC, may involve the loss of N-(2-aminoethyl)piperidine moiety $[C_5H_{10}N(CH_2)_2N]$ with observed weight loss of 37.77 % (calcd. 37.46 %). The final residue was calculated to be CuO with found weight loss of 22.23 % (calcd. 23.61 %).

Conclusion

Two copper(II) complexes of phenol-containing tridentate Schiff base ligands $(L^1)^-$ and $(L^2)^-$ in the forms of $[CuL^1(\mu\text{-Br})]_\infty$ (**1**) and $[CuL^2(\mu_{1,3}\text{-}N_3)]_\infty$ (**2**) have been synthesized and characterized. Single crystal X-ray structure determinations carried out on **1** and **2** reveal that both complexes are mono(μ-bromo)-bridged (for **1**) or mono($\mu_{1,3}$-azido)-bridged (for **2**) square pyramidal copper(II) one-dimensional chain polymeric complexes. Thermogravimetric analysis results show that complex **2** is more stable than complex **1**.

ACKNOWLEDGEMENTS

The authors acknowledged the financial support for this work by the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry, Huai'an Industry-University-Institute Cooperation Foundation (HAC201014) and Jiangsu Key Laboratory for the Chemistry of Low-Dimensional Materials (JSKC12108).

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