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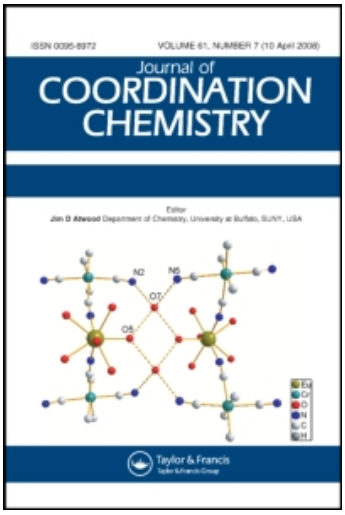

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Syntheses and crystal structures of two Schiff-base copper(II) complexes with antibacterial activities

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Syntheses and crystal structures of two Schiff-base copper(II) complexes with antibacterial activities

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Two structurally similar trinuclear complexes, $[Cu(Cu(\mu\text{-}Cl)_2L1)_2]$ (**1**) and $[Cu(Cu(\mu\text{-}Cl)_2L2)_2]$ (**2**) (HL1 = 4-chloro-2-[(2-morpholin-4-ylethylimino)methyl]phenol, HL2 = 1-[(2-piperidin-1ylethylimino)methyl]naphthalen-2-ol), have been synthesized and structurally characterized. Both complexes are bridged trinuclear compounds. The central Cu in each complex is in an octahedral environment with two phenolate and four bridging chlorides. The symmetry-related terminal Cu in each complex is square pyramidal with one phenolate oxygen, one imine nitrogen and one amine nitrogen of the Schiff-base ligand, one Cl^- in the basal plane, and one bridging Cl^- in the apical position. The complexes and Schiff bases were tested *in vitro* for their antibacterial activities.

Keywords: Schiff base; Copper; Synthesis; Crystal structure; Antibacterial

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1. Introduction

Schiff bases are readily synthesized by the condensation of carbonyl compounds with primary amines [1, 2], and have been widely investigated for their antibacterial and antitumor activities [3–5]. Metal complexes of Schiff bases play an important role in catalysis and enzymatic reactions, magnetism, molecular architectures [6–8], and also exhibit interesting biological activities [9–11].

The Schiff bases 4-chloro-2-[(2-morpholin-4-ylethylimino)methyl]phenol (HL1) and 1-[(2-piperidin-1-ylethylimino)methyl]naphthalen-2-ol (HL2) are tridentate ligands. Complexes with HL1 have never been reported, and only two mononuclear nickel(II) complexes with HL2 have been reported [12, 13]. Chloride is a good bridging group and polynuclear complexes with chloride bridges have been reported [14–16]. In this article, two structurally similar chloride-bridged trinuclear copper(II) complexes, $[Cu(Cu(\mu\text{-}Cl)_2L1)_2]$ (**1**) and $[Cu(Cu(\mu\text{-}Cl)_2L2)_2]$ (**2**), have been synthesized and structurally characterized. The antibacterial activities against *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas fluorescens* were evaluated for the compounds (scheme 1).

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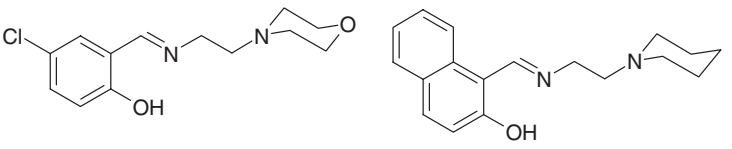

HL1 HL2

Scheme 1. Structure of ligands HL1 and HL2.

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2. Experimental

2.1. *Materials and measurements*

5-Chloro-2-hydroxybenzaldehyde, 2-hydroxy-1-naphthyaldehyde, 4-(2-aminoethyl)-morpholine, and 4-(2-aminoethyl)piperidine of AR grade were obtained from Aldrich and used as received. Elemental analyses were performed using a Perkin-Elmer 240C analytical instrument (USA). Infrared spectra were recorded on a Nicolet 5DX FT-IR spectrophotometer (USA) with KBr pellets.

2.2. *Synthesis of HL1 and HL2*

HL1 was prepared by refluxing 5-chloro-2-hydroxybenzaldehyde (156.6 mg, 1.0 mmol) and 4-(2-aminoethyl)morpholine (130.2 mg, 1.0 mmol) in 30 mL of methanol for 30 min. The clear yellow solution was evaporated to give yellow powder, which was washed three times with methanol and dried in air. Yield: 95%. Anal. Calcd for $C_{13}H_{17}ClN_2O_2$ (%): C, 58.1; H, 6.4; N, 10.4. Found (%): C, 58.5; H, 6.2; N, 10.7.

HL2 was prepared by a similar procedure as that for HL1, with 2-hydroxy-1-naphthyaldehyde (172.2 mg, 1.0 mmol) and 4-(2-aminoethyl)piperidine (128.2 mg, 1.0 mmol). Yield: 93%. Anal. Calcd for $C_{18}H_{22}N_2O$ (%): C, 76.6; H, 7.8; N, 9.9. Found (%): C, 77.0; H, 7.9; N, 9.6.

2.3. *Synthesis of [Cu(Cu(μ-Cl)$_2$L1)$_2$] (1)*

To a solution of copper(II) chloride (35.0 mg, 0.2 mmol) in methanol (10 mL), a solution of HL1 (26.8 mg, 0.1 mmol) in methanol (5 mL) was added with stirring. The mixture was stirred for 30 min and filtered. The filtrate was kept undisturbed at room temperature for 5 days. Deep blue crystals of **1** suitable for X-ray diffraction were obtained on slow evaporation of the solvent. Crystals were isolated by filtration and dried in air; 67% yield with respect to HL1. Anal. Calcd for $C_{26}H_{32}Cl_6Cu_3N_4O_4$ (%): C, 36.0; H, 3.7; N, 6.5. Found (%): C, 36.7; H, 3.9; N, 6.3.

2.4. *Synthesis of [Cu(Cu(μ-Cl)$_2$L2)$_2$] (2)*

Complex **2** was synthesized and crystallized by the same procedure as for **1**, with HL1 replaced by HL2 (28.2 mg, 0.1 mmol); yield 73% with respect to the HL2. Anal. Calcd for $C_{36}H_{42}Cl_4Cu_3N_4O_2$ (%): C, 48.3; H, 4.7; N, 6.3. Found (%): C, 48.8; H, 4.6; N, 6.7.

Table 1. Crystallographic and experimental data for complexes **1** and **2**.

	1	**2**
Chemical formula	$C_{26}H_{32}Cl_6Cu_3N_4O_4$	$C_{36}H_{42}Cl_4Cu_3N_4O_2$
Formula weight	867.9	895.2
Temperature (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Color; shape	Blue; block	Blue; block
Crystal size (mm^3)	$0.23 \times 0.22 \times 0.20$	$0.32 \times 0.30 \times 0.30$
Crystal system	Orthorhombic	Monoclinic
Space group	*Pbca*	$P2_1/c$
Unit cell dimensions (Å, °)		
a	9.078(2)	14.150(2)
b	16.076(2)	9.381(1)
c	21.600(3)	14.161(2)
β	90	106.06(3)
Volume ($Å^3$), *Z*	3152.3(9), 4	1806.4(5), 2
Calculated density ($g\,cm^{-3}$)	1.829	1.646
Absorption coefficient (mm^{-1})	2.555	2.085
θ range for data collection (°)	1.89–27.00	1.50–27.49
F(000)	1748	914
Max. and min. transmission	0.591 and 0.629	0.555 and 0.574
No. of measured reflections	18,229	14,949
No. of unique reflections	3434	4094
No. of observed reflections	2707	3407
Parameters/restraints	196/0	223/0
R_{int}	0.0440	0.0352
Goodness-of-fit on F^2	1.021	0.918
Final *R* indices $[I > 2\sigma(I)]$[a]	$R_1 = 0.0303$, $wR_2 = 0.0682$	$R_1 = 0.0367$, $wR_2 = 0.917$
R indices (all data)[a]	$R_1 = 0.0451$, $wR_2 = 0.0749$	$R_1 = 0.0464$, $wR_2 = 0.0975$

[a] $R_1 = \sum|F_0| - |F_c|/\sum|F_0|$, $wR_2 = [\sum w(F_0^2 - F_c^2)^2/\sum w(F_0^2)^2]^{1/2}$.

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2.5. *X-ray crystallography*

Suitable high-quality single crystals of **1** and **2** were selected and mounted on a Bruker Smart 1000 CCD area-detector diffractometer (Germany) with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Diffraction data for both complexes were collected by ω scan at 298(2) K. Data reduction and cell refinement were performed by the SMART and SAINT programs [17]. Empirical absorption correction was applied using SADABS [18]. The structures were solved by direct methods and refined with full-matrix least-squares using the SHELXL-97 package [19]. Non-H atoms in the structures were subjected to refined anisotropic refinement. Hydrogens were located geometrically and treated with the riding mode. Crystallographic data and experimental details for the complexes are summarized in table 1, and selected bond lengths and angles are listed in table 2.

2.6. *Antibacterial test*

The antibacterial activities of HL1, HL2, and both complexes were tested *in vitro* against *B. subtilis*, *S. aureus*, *E. coli*, and *P. fluorescens* using MH medium (Mueller–Hinton medium: casein hydrolysate 17.5 g, soluble starch 1.5 g, beef extract 1000 mL). The minimum inhibitory concentrations (MICs) of the test compounds were determined by a calorimetric method using the dye MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) [20]. A solution of the compound ($50\,\mu g\,mL^{-1}$) in

Table 2. Selected bond lengths (Å) and angles (°) for complexes **1** and **2**.

1			
Cu1–N1	1.944(2)	Cu1–N2	2.045(2)
Cu1–O1	1.978(2)	Cu1–Cl2	2.275(1)
Cu1–Cl3	2.683(1)	Cu2–O1	1.958(2)
Cu2–Cl2	2.858(1)	Cu2–Cl3	2.329(1)
N1–Cu1–O1	90.2(1)	N1–Cu1–N2	85.5(1)
O1–Cu1–N2	171.9(1)	N1–Cu1–Cl2	155.7(1)
O1–Cu1–Cl2	89.8(1)	N2–Cu1–Cl2	97.0(1)
N1–Cu1–Cl3	111.5(1)	O1–Cu1–Cl3	76.0(1)
N2–Cu1–Cl3	99.3(1)	Cl2–Cu1–Cl3	92.0(1)
O1–Cu2–O1A	180	O1–Cu2–Cl3A	94.5(1)
O1–Cu2–Cl3	85.5(1)	Cl3–Cu2–Cl3A	180
O1–Cu2–Cl2	74.7(1)	O1–Cu2–Cl2A	105.3(1)
Cl2–Cu2–Cl3	86.6(1)	Cl2–Cu2–Cl2A	180
Cl2–Cu2–Cl3A	93.4(1)		
2			
Cu1–N1	1.927(2)	Cu1–N2	2.041(2)
Cu1–O1	1.969(2)	Cu1–Cl1	2.273(1)
Cu1–Cl2	2.668(1)	Cu2–O1	2.003(2)
Cu2–Cl1	2.823(1)	Cu2–Cl2	2.285(1)
N1–Cu1–O1	90.2(1)	N1–Cu1–N2	85.2(1)
O1–Cu1–N2	174.1(1)	N1–Cu1–Cl1	164.4(1)
O1–Cu1–Cl1	88.9(1)	N2–Cu1–Cl1	96.5(1)
N1–Cu1–Cl2	101.2(1)	O1–Cu1–Cl2	76.9(1)
N2–Cu1–Cl2	100.4(1)	Cl1–Cu1–Cl2	93.7(1)
O1–Cu2–O1A	180	O1–Cu2–Cl2A	93.9(1)
O1–Cu2–Cl2	86.1(1)	Cl2–Cu2–Cl2A	180
Cl1–Cu2–Cl2	89.5(1)	Cl1–Cu2–O1	74.0(1)
Cl1–Cu2–Cl1A	180	Cl1–Cu2–Cl2A	90.5(1)
Cl1–Cu2–O1A	106.0(1)		

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DMSO was prepared, and graded quantities of the test compounds were incorporated in a specified quantity of sterilized liquid MH medium. A specified quantity of the medium containing the compound was poured into microtitration plates. Suspension of the microorganism was prepared to contain about 10^5 colony forming units (cfu) mL^{-1} and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37°C for 24 h. After the MICs were visually determined on each of the microtitration plates, 50 µL of Phosphate Buffered Saline (PBS 0.01 mol L^{-1}, pH 7.4: $Na_2HPO_4 \cdot 12H_2O$ 2.9 g, KH_2PO_4 0.2 g, NaCl 8.0 g, KCl 0.2 g, distilled water 1000 mL) containing 2 mg of MTT was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed and 100 µL of isopropyl alcohol containing 5% 1.0 mol L^{-1} HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a microplate reader at 550 nm. The observed MICs are presented in table 3.

3. Results and discussion

3.1. *Crystal structure description*

Figures 1 and 2 give perspective views of **1** and **2**, respectively. Both complexes are chloride and phenolate-bridged centrosymmetric trinuclear copper(II) compounds with

Table 3. Antibacterial activities.

	MIC ($\mu g\,mL^{-1}$)			
	B. subtilis	*E. coli*	*P. fluorescens*	*S. aureus*
HL1	63.1	27.6	45.5	25.2
HL2	>100	68.3	>100	12.0
1	23.0	3.2	12.0	4.3
2	31.8	5.4	23.3	1.7
Penicillin	1.3	>100	>100	2.1

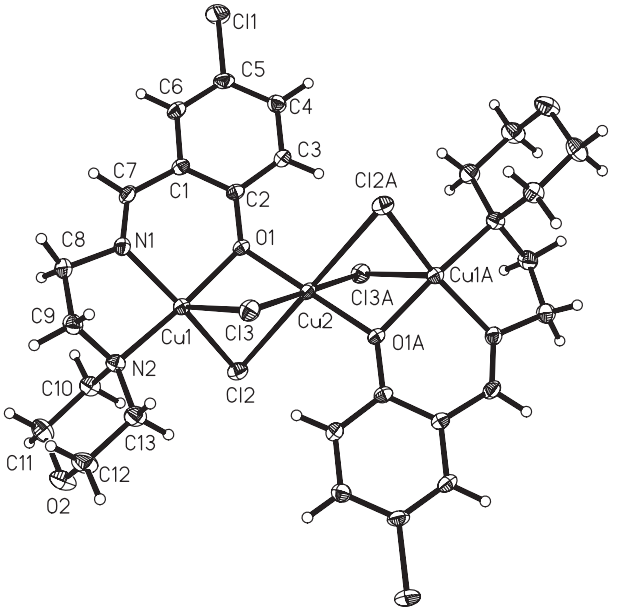

Figure 1. Molecular structure of **1**. Displacement is drawn at the 30% probability level. Atoms labeled with the suffix A or unlabeled are at the symmetry position $1-x, -y, 1-z$.

inversion centers at the central Cu. The Cu···Cu distances are 2.943(1) Å in **1** and 2.911(1) Å in **2**, comparable with those found in similar chloride-bridged copper(II) complexes [21, 22].

In each complex, coordination around the inversion-related terminal Cu is square pyramidal. The equatorial plane of each square pyramid is defined by the N_2O donor set of the Schiff-base ligand and a terminal chloride, Cl2 for **1** and Cl1 for **2**. The apical position of each square pyramid is occupied by bridging chloride, Cl3 for **1** and Cl2 for **2**. The substantial distortion of each square pyramid is revealed by bond angles between apical and equatorial donors, which show deviation from the ideal 90° angle in a regular square pyramid. Cu1 is displaced out of the least-squares plane defined by the four equatorial donors in the direction of the apical position by 0.170(2) Å for **1** and 0.114(2) Å for **2**.

Cu2 in each complex, lying on the inversion center, is six-coordinate octahedral, with two phenolate oxygens and two bridging chlorides defining the basal plane, and another

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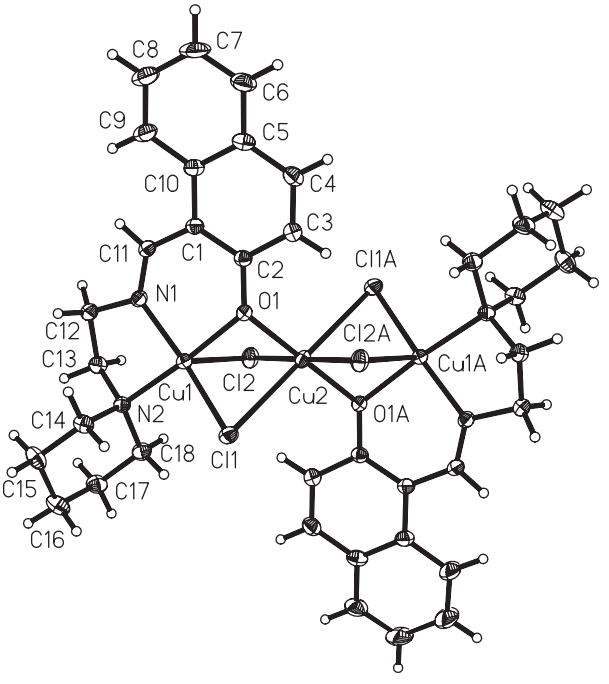

Figure 2. Molecular structure of **2**. Displacement is drawn at the 30% probability level. Atoms labeled with the suffix A or unlabeled are at the symmetry position $1-x$, $1-y$, $-z$.

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two chlorides occupying axial positions. Distortion of the octahedron lies in the bond lengths and angles among the axial and basal bonds. The axial Cu–Cl bonds (2.858(1) Å for **1** and 2.823(1) Å for **2**) are much longer than those in the basal plane (2.329(1) Å for **1** and 2.285(1) Å for **2**), caused by Jahn–Teller distortion.

The Cu1–O1–Cu2, Cu1–Cl2–Cu2, and Cu1–Cl3–Cu2 bridging angles in **1** are 96.8(1), 68.9(1), and 71.5(1)°, respectively. The Cu1–O1–Cu2, Cu1–Cl1–Cu2, and Cu1–Cl2–Cu2 bridging angles in **2** are 94.2(1), 68.7(1), and 71.5(1)°, respectively. The Cu–N(iminic) bond lengths are shorter than the Cu–N(aminic) bonds, which have been widely observed in similar complexes [15, 23]. The Cu–Cl bond lengths in both complexes are comparable with those reported in chloride-bridged polynuclear copper(II) complexes [24, 25].

In the crystal structure of **1**, molecules are linked through weak intermolecular C–H $\cdots$ Cl hydrogen bonds, forming layers parallel to the *ab* plane, while in the crystal structure of **2** there are no obvious short contacts. The difference between the molecular packing of the two complexes may be caused by the larger steric effects of the naphthyl rings in **2** than those of the chloride-substituted benzene rings in **1**.

3.2. *IR spectra*

The weak and broad band in the region 3300–3400 cm^{-1} is due to stretching of the phenolic OH groups of the Schiff bases; these bands are absent in the spectra of both complexes indicating the coordination of the phenolate oxygen of the Schiff bases

to copper. The phenolic ν(C–O) in the spectra of the Schiff bases are observed as strong bands at 1200 cm^{-1}. However, in the spectra of both complexes, bands appear at 1180 cm^{-1}, confirming the deprotonation and coordination of the phenolate oxygen to copper. The strong bands at 1632 cm^{-1} in the spectrum of HL1 and 1635 cm^{-1} in the spectrum of HL2 are due to the azomethine; these bands shift to lower frequencies (1613 cm^{-1} for **1** and 1615 cm^{-1} for **2**), indicating the coordination of azomethine nitrogens to copper. Weak bands at 342 cm^{-1} for **1** and 343 cm^{-1} for **2** are attributed to Cu–Cl vibrations.

3.3. *Antibacterial activities*

The Schiff bases and the two complexes were screened *in vitro* for antibacterial activities against *B. subtilis*, *S. aureus*, *E. coli*, and *P. fluorescens* by the MTT method. The MICs of the compounds against the bacteria are presented in table 3. Penicillin was used as a reference.

HL1 shows antibacterial activities against the four bacteria, and HL2 shows antibacterial activities against *E. coli* and *S. aureus*, but no activity against *B. subtilis* and *P. fluorescens*. Complexes **1** and **2** show stronger activities against the bacteria than the corresponding Schiff bases. Complex **1** shows stronger activities against *B. subtilis*, *E. coli*, and *P. fluorescens*, but shows weaker activity against *S. aureus* than **2**. Antibacterial activities against *E. coli*, *P. fluorescens*, and *S. aureus* of the two complexes are superior to the Penicillin. The trends in the present work are in accord with those in the literature with Schiff-base complexes having stronger antibacterial activities than the Schiff base [26–28].

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4. Conclusion

Two chloride-bridged centrosymmetric trinuclear copper(II) complexes have been synthesized and structurally characterized. The Schiff bases, 4-chloro-2-[(2-morpholin-4-ylethylimino)methyl]phenol and 1-[(2-piperidin-1-ylethylimino)methyl]naphthalen-2-ol, coordinate to copper through phenolate oxygen, imine nitrogen, and amine nitrogen. The chlorides are bridging groups. The steric effects of the chloride-substituted benzene ring and the naphthyl ring influence the molecular packing modes. The antibacterial activities of the Schiff bases and the complexes were tested. The results indicate that the complexes are potential antibacterial materials.

Supplementary material

CCDC reference numbers 737473 for **1** and 737474 for **2** contain the supplementary crystallographic data for this article. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk, or from Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; Email: deposit@ccdc.cam.ac.uk.

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