Synthesis, structural characterization and substituent effects of two copper(II) complexes with benzaldehyde ortho-oxime ligands

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Received: 31 December 2011/Accepted: 30 January 2012/Published online: 22 February 2012 © Springer Science+Business Media B.V. 2012

Abstract Two new Cu(II) complexes, $[Cu(L^1)_2]$ (1) and $[Cu(L^2)_2]$ (2) (HL¹ = (*E*)-3-bromo-5-chloro-2-hydroxy benzaldehyde *O*-methyl oxime; $HL^2 = (E)$ -3-bromo-5chloro-2-hydroxy benzaldehyde O-ethyl oxime), have been synthesized and characterized by physicochemical and spectroscopic methods. X-ray crystallographic analyses show that complexes 1 and 2 have similar structures, consisting of one Cu(II) atom and two L⁻ units. In both complexes, the Cu(II) atom, lying on an inversion center, is four-coordinated in a trans-CuN₂O₂ square-planar geometry by two phenolate O and two oxime N atoms from two symmetry-related N.O-bidentate oxime ligands. Moreover, both complexes form an infinite three-dimensional supramolecular structure involving intermolecular C-H--Br hydrogen bonds and $\pi \cdots \pi$ stacking interactions between the metal chelate rings and aromatic rings. Substituent effects in the two complexes are discussed.

Introduction

Oximes have long been used as chelating ligands in the synthesis of transition metal complexes due to their ease of preparation and structural variety. Those derived from

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salicylaldehyde and various amines have been used to synthesize many complexes of copper(II) and nickel(II) [1, 2]. In recent years, there has been enhanced interest in the synthesis and characterization of such complexes due to their interesting properties [3-5], and they have been increasingly used in analytical chemistry [6], supramolecular chemistry [7–9], extraction metallurgy [10], medicine [11], insecticide [12], anti-virus [13] and other applications. Herein, two new Cu(II) complexes with mono-oxime ligands, $[Cu(L^{1})_{2}]$ (1) (HL¹ = (*E*)-3-bromo-5-dichoro-2hydroxy benzaldehyde *O*-methyl oxime) and $[Cu(L^2)_2]$ (2) $(HL^2 = (E)-3$ -bromo-5-dichoro-2-hydroxy benzaldehyde O-ethyl oxime), have been synthesized and characterized by elemental analyses, IR spectra and UV-Vis spectra. In addition, X-ray crystallographic analyses reveal that the structures of complexes 1 and 2 are very similar. They are both mononuclear, and all the Cu(II) atoms are fourcoordinated. However, the bond distances and angles vary due to the different substituents in the ligands.

Experimental

Methoxyamine and ethoxyamine were purchased from Alfa Aesar and used without further purification. 3-Bromo-5chloro-2-hydroxybenzaldehyde and the other reagents and solvents were analytical grade from Tianjin Chemical Reagent Factory. Elemental analyses for Cu were obtained using an IRIS ER/S·WP-1 ICP atomic emission spectrometer. C, H and N analyses were obtained using a GmbH VarioEL V3.00 automatic elemental analysis instrument. IR spectra were recorded on a VERTEX70 FTIR spectrophotometer, with samples prepared as KBr (500–4000 cm⁻¹) or CsI (100-500 cm⁻¹) pellets. UV–Vis absorption spectra were recorded on a Shimadzu UV-2550 spectrometer. X-ray

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single-crystal structures determinations were carried out on a Bruker Smart APEX CCD diffractometer.

Synthesis of HL¹

To a stirred solution of 3-bromo-5-chloro-2-hydroxybenzaldehyde (0.2355 g, 1 mmol) in hot EtOH (5 ml) was added an EtOH (3 ml) solution of methoxyamine (0.0835 g, 1 mmol). After stirring the reaction mixture at 338 K for 10 h, the solvent was removed under reduced pressure and the residue was recrystallized from ethanol to give the title compound. Yield 0.138 g (52%). mp. 362–365 K. Anal. Calcd for C₈H₇BrClNO₂: C, 36.3; H, 2.7; N, 5.3. Found: C, 36.4; H, 2.6; N, 5.2.

Synthesis of HL²

To a stirred solution of 3-bromo-5-chloro-2-hydroxybenzaldehyde (0.2355 g, 1 mmol) in hot EtOH (4 ml) was added an EtOH solution (3 ml) of ethoxyamine (0.0976 g, 1 mmol). After stirring the reaction mixture at 338 K for 10 h, the solvent was removed under reduced pressure and the residue was recrystallized from ethanol to give the title compound. Yield 0.156 g (56%). mp. 340–341 K. Anal. Calcd for C₉H₉BrClNO₂: C, 38.8; H, 3.3; N, 5.0. Found: C, 38.9; H, 3.5; N, 4.9.

Synthesis of complex 1

A solution of copper(II) acetate monohydrate (0.0022 g, 0.01 mmol) in MeOH (3 ml) was added dropwise to a solution of HL¹ (0.0028 g, 0.01 mmol) in dichloromethane (5 ml) at room temperature. The color of the mixture turned pale yellow immediately. Upon standing at room temperature for 2 weeks, the solvent partially evaporated and yellow prismatic single crystals suitable for X-ray crystallographic analysis were obtained. Yield 0.0021 g (35%). Anal. Calcd for C₁₆H₁₂Br₂Cl₂CuN₂O₄ (%): C, 32.5; H, 2.1; N, 4.7; Cu, 10.8. Found: C, 32.6; H, 2.1; N, 4.6; Cu, 10.9.

Synthesis of complex 2

A solution of copper(II) acetate monohydrate (0.0034 g, 0.02 mmol) in MeOH (3 ml) was added dropwise to a solution of HL² (0.0047 g, 0.02 mmol) in dichloromethane (3 ml) at room temperature. The color of the mixture turned pale yellow immediately. Upon standing at room temperature for 2 weeks, the solvent partially evaporated and yellow prismatic single crystals suitable for X-ray crystallographic analysis were obtained. Yield 0.0031 g (27%). Anal. Calcd. for $C_{18}H_{16}Br_2Cl_2CuN_2O_4$ (%): C, 35.0; H, 2.6; N, 4.5; Cu, 10.3. Found: C, 34.9; H, 2.7; N, 4.4; Cu, 10.4.

Crystal structure determinations

Single crystals of complexes **1** and **2** with approximate dimensions of $0.38 \times 0.12 \times 0.06$ mm and $0.21 \times 0.18 \times 0.07$ mm were mounted on a Bruker Smart 1000 CCD area detector. The diffraction data were collected using graphite monochromated Mo K α radiation ($\lambda = 0.071073$ nm) at 298(2) K. The LP factor and semi-empirical absorption corrections were applied to the intensity data. The structures were solved using the program SHELXS-97 [14] and refined by full-matrix least-squares methods on F^2 using SHELXL-97 [14]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically. Details of the data collection and refinements of complexes **1** and **2** are given in Table 1.

Results and discussion

The most important FTIR bands for HL¹, HL² and complexes 1 and 2 are given in Table 2. The O-H stretching frequency of oxime-type ligands is generally expected in the 3300–3800 cm^{-1} region; the bands for HL¹ and HL² are observed at 3420 cm⁻¹ and 3411 cm⁻¹, respectively, due to the internal hydrogen bond $OH \cdots N=C$ [15]. The free ligands HL¹ and HL² exhibit characteristic C=N stretching frequencies at 1608 and 1602 cm⁻¹, while the C=N stretching frequencies of complexes 1 and 2 were both observed at 1606 cm⁻¹. Hence, the C=N stretching frequencies are slightly shifted upon complexation [16–18]. The Ar–O stretching frequency occurs at 1213 cm^{-1} for HL^{1} , and at 1219 cm⁻¹ for complex 1; this band occurs at 1221 cm^{-1} for HL² and at 1211 cm^{-1} for complex 2. Hence, the Ar-O stretching frequency is shifted by ca. 7 cm^{-1} and 9 cm^{-1} , respectively.

The far-infrared spectra of complexes **1** and **2** were also obtained in the region 500–100 cm⁻¹ in order to observe the Cu–O and Cu–N stretching frequencies. The FTIR spectrum of complex **1** showed the v(Cu-N) and v(Cu-O) absorption frequencies at 459 cm⁻¹ and 420 cm⁻¹ (469 cm⁻¹ and 421 cm⁻¹ for complex **2**) [19], respectively. These assignments are consistent with the literature [20–23].

The UV–Vis absorption spectra of the free ligands HL¹, HL² and their corresponding Cu(II) complexes were determined in 5×10^{-5} mol L⁻¹ DMF solution. The absorption spectrum of HL¹ shows a peak at 317 nm assigned to a π – π^* transition, while equivalent band is observed at 315 nm in the spectrum of complex **1**. The absorption spectrum of HL² shows a peak at 315 nm assigned to a π – π^* transition [24, 25], while the equivalent peak is at 319 nm in the spectrum of complex **2**. Hence, the shift upon complexation is hypsochromic in complex **1** and bathochromic in complex **2**, perhaps due to the different

Table 1 Crystal data and structure refinement paramet for complexes 1 and 2

Table 1 Crystal data and structure refinement parameters	Compounds	Complex 1	Complex 2
for complexes 1 and 2	Empirical formula	C ₁₆ H ₁₂ Br ₂ Cl ₂ CuN ₂ O ₄	$C_{18}H_{16}Br_2Cl_2CuN_2O_4$
	Formula weight	590.54	618.59
	Temperature (K)	298(2)	298(2)
	Wavelength (Å)	0.71073	0.71073
	Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, P-1
	Unit cell dimensions (Å, °)		
	a	4.0174(5)	8.017(1)
	b	13.820(1)	8.058(1)
	С	17.321(2)	8.837(1)
	α	90	106.067(2)
	β	92.509(1)	100.989(1)
	γ	90	100.165(1)
	Volume (Å ³)	960.8(2)	522.4(1)
	Z, Calculated density (Mg/m ³)	2, 2.041	1, 1.966
	Absorption coefficient (mm ⁻¹)	5.599	5.153
	F(000)	574	303
	Crystal size (mm)	$0.38\times0.12\times0.06$	$0.21 \times 0.18 \times 0.07$
	θ range for data collection (°)	1.89–25.01	2.48-25.02
	Limiting indices	-4,4/-16,16/-20,14	-7,9/-9,7/-10, 10
	Reflections collected/unique	4547/1669 [$R_{\rm int} = 0.0441$]	2693/1817 [$R_{\rm int} = 0.0190$]
	Completeness to θ	98.8% ($\theta = 25.01$)	98.1% ($\theta = 25.02$)
	Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
	Max. and min. transmission	0.7299 and 0.2248	0.7143 and 0.4108
	Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares
	Data/restraints/parameters	1669/0/125	1817/0/134
	Goodness-of-fit on F ²	1.113	1.006
w = 1/2	Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0624, wR_2 = 0.1496^{a}$	$R_1 = 0.0326, wR_2 = 0.0736^{\rm b}$
$[\sigma^2(F_0^2) + (0.0689P)^2 + 5.0095P]$	R indices (all data)	$R_1 = 0.0736, wR_2 = 0.1544$	$R_1 = 0.0469, wR_2 = 0.0789$
$w = 1/[\sigma^2(F_0^2) + (0.0395P)^2],$ where $P = (F_0^2 + 2F_0^2)/3$	Largest diff. peak and hole $(e.A^{-3})$	1.294 and -0.850	0.434 and -0.414

Table 2 Main IR bands for the ligands and their complexes (cm^{-1})

Compounds	$v_{\rm (O-H)}$	v _(C=N)	$v_{(Ar-O)}$	v _(Cu–N)	v _(Cu-O)
HL^1	3420	1608	1213	-	-
Complex 1	-	1606	1219	459	420
HL^2	3411	1602	1221	-	-
Complex 2	-	1606	1211	469	421

substituent effects. In addition, a new peak was observed at 377 or 371 nm in the absorption spectra of complexes 1 and 2, respectively.

Description of the crystal structures

ORTEP representations of complexes 1 and 2 are shown in Figs. 1 and 2. Selected bond distances and angles are given in Tables 3 and 4.

X-ray crystal structure determination reveals that complex 1 crystallizes in the monoclinic system and $P2_1/c$ space group, whereas complex 2 crystallizes in the triclinic system and P-1 space group. Both the compounds can be described as centrosymmetric mononuclear Cu(II) complexes, in which the Cu(II) center, lying on the inversion center, is four-coordinated in a trans-CuN₂O₂ square-planar geometry, with two phenolate O and two oxime N atoms from two N,O-bidentate oxime $(L^1)^-$ or $(L^2)^-$ ligands.

As shown in Fig. 3, the crystal structure of complex 1 is stabilized by intermolecular C1-H1C…Br1 hydrogen bonds (Table 5) with a graph-motif R_2^2 [18], which link neighbouring molecules into extended chains, forming an infinite three-dimensional supramolecular network. Meanwhile, intermolecular C8-H8...Br1 hydrogen bonds and $\pi \dots \pi$ stacking interactions between the metal chelate ring and aromatic ring provide further stabilization (Fig. 4 and Table 6). In Figs. 5 and 6, in complex 2, there exist intermolecular C7-H7…Br1 and C3-H3…O1 hydrogen bonds both with graph-motifs R_2^2 [8] (Table 5) and $\pi \cdots \pi$ stacking interactions (Table 6), forming an infinite threedimensional supramolecular structure [26-29].



Fig. 1 ORTEP view of the crystal structure of complex 1 with thermal ellipsoids drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity



Fig. 2 ORTEP view of the crystal structure of complex 2 with thermal ellipsoids drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity

Table 3 Selected bond lengths (Å) and bond angles (°) for complex 1

Symmetry codes: (I) -1+X, Y, Z; (II) 2-X, 1-Y, 1-Z. DCC = distance between ring centroids; α = dihedral angle between planes I and J; β = Angle Cg(I)…Cg(J) vector and normal to plane I. Cg(I)-perp = perpendicular distance of Cg(I) from ring J; Cg(J)-perp = perpendicular distance of Cg(J) from ring I. Cg1 and Cg3 for complex 1 are the centroids of atoms Cu1\O2\C4\C3\C2\N1 and C3-C8, respectively; Cg1 and Cg3 for complex 2 are the centroids of atoms Cu1\O2\C5\C4\C3\N1 and C4-C9, respectively.

Substituent effects

The overall structures of the two complexes are found to be identical except that the oxime portion differs by a single -CH₂- group. In both cases, two imino N and two phenolic O atoms form a square plane around copper, with the Cu-N bonds being slightly longer than the corresponding Cu-O bonds. The elongation of the Cu-N bonds probably due to the weakening of the coordination abilities of the nitrogen atoms by the larger electronegativity of oxime oxygen atoms. Similar elongation has been observed in Cu(II) complexes with Salen-type ligands [30, 31]. However, due to the presence of an extra $-CH_2$ - group in the HL² ligand, the Cu-N bond (2.010 Å) of complex **1** is longer than that Cu-N (1.992 Å) of complex 2, and the Cu-O bond (1.913 Å) of complex **1** is also longer than that Cu–O (1.878 Å) of complex 2. Meanwhile, the angle N1-Cu1-O2 (89.62°) in complex 1 is smaller than equivalent angle (90.02°) in complex 2. The substituent effects also give rise to the variations in IR and UV-Vis spectra in complexes 1 and 2.

Bond distances					
Cu(1)–O(2) ^{#1}	1.914(5)	N(1)–C(2)	1.273(1)	C(3)–C(4)	1.424(1)
Cu(1)–O(2)	1.914(5)	N(1)–O(1)	1.429(8)	C(4)–C(5)	1.404(1)
Cu(1)–N(1)	2.01(6)	O(1)–C(1)	1.431(1)	C(5)–C(6)	1.391(1)
Cu(1)-N(1) ^{#1}	2.01(6)	O(2)–C(4)	1.305(9)	C(6)–C(7)	1.377(1)
Br(1)–C(5)	1.899(8)	C(2)–C(3)	1.453(1)	C(7)–C(8)	1.37(1)
Cl(1)–C(7)	1.764(8)	C(3)–C(8)	1.418(1)		
Bond angles					
O(2)-Cu(1)-O(2) ^{#1}	180.000	N(1)-O(1)-C(1)	109.5(6)	C(6)-C(5)-C(4)	123.4(7)
O(2)-Cu(1)-N(1)	89.6(2)	C(4)–O(2)–Cu(1)	131.7(5)	C(6)–C(5)–Br(1)	117.9(6)
O(2) ^{#1} -Cu(1)-N(1)	90.4(2)	N(1)-C(2)-C(3)	125.4(7)	C(4)-C(5)-Br(1)	118.7(6)
O(2)-Cu(1)-N(1) ^{#1}	90.4(2)	C(8)–C(3)–C(4)	120.9(7)	C(7)-C(6)-C(5)	119.1(7)
$O(2)^{\#1}$ -Cu(1)-N(1) $^{\#1}$	89.6(2)	C(8)–C(3)–C(2)	116.7(7)	C(8)-C(7)-C(6)	121.0(8)
N(1)-Cu(1)-N(1) ^{#1}	180.000	C(4)–C(3)–C(2)	122.4(7)	C(8)–C(7)–Cl(1)	119.5(6)
C(2)–N(1)–O(1)	111.2(6)	O(2)–C(4)–C(5)	121.3(7)	C(6)–C(7)–Cl(1)	119.5(6)
C(2)–N(1)–Cu(1)	127.6(5)	O(2)–C(4)–C(3)	123.1(6)	C(7)–C(8)–C(3)	119.9(7)
O(1)-N(1)-Cu(1)	121.1(5)	C(5)-C(4)-C(3)	115.5(7)		

Symmetry transformations used to generate equivalent atoms: $^{\#1}$ -x+1, -y+1, -z+1 for complex 1

Table 4 Selected bond lengths (Å) and bond angles (°) for complex 2

Bond distances					
Cu(1)–O(2)	1.878(3)	N(1)-C(3)	1.286(5)	C(4)–C(5)	1.403(5)
Cu(1)-O(2) ^{#2}	1.878(3)	N(1)–O(1)	1.423(4)	C(4)–C(9)	1.415(5)
Cu(1)-N(1) ^{#2}	1.992(3)	O(1)–C(1)	1.435(5)	C(5)–C(6)	1.409(5)
Cu(1)–N(1)	1.992(3)	O(2)–C(5)	1.308(5)	C(6)–C(7)	1.375(5)
Br(1)–C(6)	1.886(4)	C(1)–C(2)	1.510(5)	C(7)–C(8)	1.373(5)
Cl(1)–C(8)	1.749(4)	C(3)–C(4)	1.427(5)	C(8)–C(9)	1.366(5)
Bond angles					
O(2) ^{#2} -Cu(1)-O(2)	180.00	N(1)-O(1)-C(1)	109.9(3)	C(4)-C(5)-C(6)	116.3(4)
O(2) ^{#2} -Cu(1)-N(1) ^{#2}	90.02(1)	C(5)–O(2)–Cu(1)	131.1(2)	C(7)-C(6)-C(5)	122.7(4)
O(2)-Cu(1)-N(1) ^{#2}	89.8(1)	O(1)-C(1)-C(2)	107.4(3)	C(7)–C(6)–Br(1)	118.7(3)
O(2) ^{#2} -Cu(1)-N(1)	89.8(1)	N(1)-C(3)-C(4)	124.7(4)	C(5)–C(6)–Br(1)	118.6(3)
O(2)-Cu(1)-N(1)	90.2(1)	C(5)-C(4)-C(9)	120.5(3)	C(8)-C(7)-C(6)	119.7(4)
N(1) ^{#2} -Cu(1)-N(1)	180.0	C(5)–C(4)–C(3)	122.6(4)	C(9)-C(8)-C(7)	120.5(4)
C(3)–N(1)–O(1)	109.6(3)	C(9)–C(4)–C(3)	116.9(4)	C(9)–C(8)–Cl(1)	121.2(3)
C(3)–N(1)–Cu(1)	127.1(3)	O(2)–C(5)–C(4)	123.9(3)	C(7)–C(8)–Cl(1)	118.3(3)
O(1)–N(1)–Cu(1)	122.6(2)	O(2)-C(5)–C(6)	119.8(3)	C(8)-C(9)-C(4)	120.3(4)

Symmetry transformations used to generate equivalent atoms: $^{#2}$ -x+1, -y+1, -z+1 for complex 2

Fig. 3 Part of the supramolecular structure containing hydrogen bonds for complex 1



	D–H…A	d(D–H)	$d(H \cdots A)$	∠DHA	$d(D \cdots A)$	Symmetric code
Complex 1	C8-H8···Br1	0.931	3.049	114.9	3.540(3)	2-x, -1/2+y, 1/2-z
	C1-H1C…Br1	0.960	3.015	158.2	3.923(3)	2-x, 1-y, 1-z
Complex 2	C7-H7···Br1	0.930	3.037	139.5	3.793(3)	2-x, -y, 1-z
	C3-H3····O1	0.930	2.600	125	3.221(5)	1-x, -y, 1-z

Table 5 Putative hydrogen bonds [Å, °] in complexes 1 and 2



Fig. 4 Diagram showing $\pi \cdots \pi$ stacking interaction for complex **1**



Fig. 6 Diagram showing $\pi \cdots \pi$ stacking interactions of complex 2

Table 6 Putative π - π stacking interactions [Å, °] for complexes 1 and 2

Ring (I)	Ring (J)	α	β	DCC	Cg(I)-perp	Cg(J)-perp	Slippage
Complex 1	C. al	2.5(2)	20.22	2.0(4(4)	2 427 (2)	2 401(2)	
Complex 2	Cgs	2.5(3)	30.33	3.964(4)	3.427 (3)	-3.421(3)	-
Cg1	Cg3 ^{II}	3.4(2)	22.51	3.769(2)	3.401 (1)	-3.482(2)	-
Cg3	Cg3 ^{II}	0	21.65	3.702(2)	3.441(2)	3.441(2)	1.366

Fig. 5 Part of the 3D supramolecular structure showing hydrogen bonds of complex 2



Conclusion

In conclusion, we have reported two new Cu(II) complexes, $[Cu(L^1)_2]$ and $[Cu(L^2)_2]$ with salicyl mono-oxime ligands. The crystal structures of the two complexes have indicated that complexes **1** and **2** have similar structures, consisting of one Cu(II) atom and two L⁻ units. In both complexes, the Cu(II) atom, lying on an inversion center, is four-coordinated in a trans-CuN₂O₂ square-planar geometry by two phenolate O and two oxime N atoms from two symmetry-related N,O-bidentate oxime ligands. Moreover, both complexes form an infinite three-dimensional supramolecular structure involving intermolecular C–H…Br hydrogen bonds and $\pi \dots \pi$ stacking interactions between metal chelate rings and aromatic rings. The abnormalities of IR and UV–Vis spectra in the two complexes may be caused by the substituent effect.

Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as a supplementary publication (no. CCDC-757709 for complex **1** and CCDC-757705 for complex **2**.). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk). These data can be also obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html

Acknowledgments This work was supported by the Foundation of the Education Department of Gansu Province (No. 0904-11) and the 'Jing Lan' Talent Engineering Funds of Lanzhou Jiaotong University, which are gratefully acknowledged.

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