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# Molecular Crystals and Liquid Crystals

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## Synthesis and Structure of Three Novel Complexes Based on 4-Chlorophenyloxyacetic Acid

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### Synthesis and Structure of Three Novel Complexes Based on 4-Chlorophenyloxyacetic Acid

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Three new metal–organic complexes,  $[CoL_2(H_2O)_4]$  **1**,  $[CdL_2(H_2O)_2]$  **2**, and  $[CuL_2(H_2O)_2]$  **3** [HL = 4-chlorophenyloxyacetic acid], have been synthesized and characterized by IR spectra, elemental analysis, fluorescence spectra, and single-crystal X-ray diffraction. Compounds**1**,**2**, and**3**are all zero-dimensional dimers. The carboxy $late ligand exhibits different coordination modes in all compounds. These compounds exhibit interesting supramolecular architecture according to <math>O-H\cdots O$  and  $C-H\cdots Cl$  interactions. Compounds **1**, **2**, and **3** develop to different framework though they all are from same ligands, which will be helpful to design of different materials.

[Supplemental materials are available for this article. Go to the publisher's online edition of *Molecular Crystals and Liquid Crystals* to view the free supplemental file: CCDC 902676, 902674, 902675 contains the supplementary crystallographic data for this article. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/ retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: C44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk/.]

Keywords 4-Chlorophenyloxyacetic acid; complex; dimer

#### Introduction

Halogen phenoxyl carboxylates have been widely used as excellent pesticides and as a plant auxin in agriculture in recent decades [1–4]. However, it can also pollute our environment and hurt the health of animals and humans [5]. In order to decrease these bad effects, many researches focused on the degradation of the pesticide. Coordinating halogen phenoxyl carboxylates with metal ion is a good way. In addition, the self-assembly of metal ions and halogen phenoxyl carboxylate ligands have made progress by detailed studies of their structural characteristics, such as diverse coordination modes and conformations, and their potential applications as catalysts, luminescent, sorption, magnetic materials, and biological reagents [6–10]. As bridging ligands, halogen phenoxyl carboxylates are of huge interest in the construction of polymeric coordination architectures, not only because of the fact that these polymers have a wide range of structural diversities and potential applications

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as porous materials and magnetic materials, but also because these ligands exhibit rich coordination chemistry. It is well known that many of the most interesting metal–organic polymers with one-, two-, and three-dimensional networks have been engineered using anionic and carboxylate ligands. The title ligand, (4-chlorophenyloxy)acetic acid, is a typical carboxylate ligand because it possesses one flexible acetate. In supermolecular construction, hydrogen bonding and the  $\pi$ - $\pi$  stacking interactions are usually very important, which make the structure more stable.

However, no literatures related to the titled complexes have been published. Herein, we report the synthesis and structure of three new complexes:  $[CoL_2(H_2O)_4]$  **1**,  $[CdL_2(H_2O)_2]$  **2**, and  $[CuL_2(H_2O)_2]$  **3** [HL = 4-chlorophenyloxyacetic acid]. In addition, the function of water cluster existing in these complexes was also investigated. The work will contribute to the supermolecular construction of different framework with the same ligands.

#### Experimental Section

#### Materials and Instrumentation

All reagents and solvents were used directly as supplied commercially without further purification. Elemental analysis for C and H was carried out on a Perkin–Elmer 2400 II elemental analyzer. The FT-IR spectrum was obtained on a PE spectrum one FT-IR spectrometer Fourier transform infrared spectroscopy in the 4000–400 cm<sup>-1</sup> regions, using KBr pellets. Excitation and emission spectra were acquired on a Perkin–Elmer instruments LS55 spectrofluorometer.

#### Synthesis of Complex 1

To a solution of HL (93 mg, 0.5 mmol) in 5 mL methanol and 15 mL H<sub>2</sub>O, the solution of sodium hydroxide was added dropwise with stirring to adjust its pH to 6. And 10 mL aqueous solution of CoCl<sub>2</sub>·6H<sub>2</sub>O (118.5 mg, 0.5 mmol) was subsequently added. The mixture in autoclave was reacted for three days at 130°C, then cooled to room temperature. A week later, X-ray quality red block-shaped single crystals were obtained. The crystals were isolated, washed with carbinol, and dried at room temperature (Yield: 65% based on Co). Calcd. For C<sub>16</sub>H<sub>20</sub>Cl<sub>2</sub>CoO<sub>10</sub>: C 38.24, H 3.98%. Found: C 38.18, H 3.91%. IR (KBr, cm<sup>-1</sup>): 3454 (s), 1659 (w), 1610 (s), 1492 (s), 1426 (s), 1217 (m), 821 (w), 725 (m).

#### Synthesis of Complex 2

The synthesis of **2** was similar to that of **1** except that metallic moiety is  $CdCl_2$  (91.5 mg, 0.5 mmol). A week later, colorless block-shaped single crystals were obtained. The crystals were isolated, washed with ethanol, and dried at room temperature (Yield: 70% based on Cd). Calcd.For  $C_{16}H_{16}CdCl_2O_8$ : C 36.99, H 3.82%. Found: C 36.93, H 3.74%. IR (KBr, cm<sup>-1</sup>): 3557 (m), 1625(w), 1595(w), 1490 (s), 1428 (s), 1235 (s), 819 (s), 706 (m).

#### Synthesis of Complex 3

The synthesis of **3** was similar to that of **1** except that metallic moiety is  $CuSO_4 \cdot 5H_2O$  (125 mg, 0.5 mmol). A week later, blue block-shaped single crystals were obtained. The crystals were isolated, washed with ethanol, and dried at room temperature (Yield: 72% based on Cu). Calcd.For C<sub>16</sub>H<sub>16</sub>Cl<sub>2</sub>CuO<sub>8</sub>: C 40.85, H 3.40%. Found: C 40.78, H 3.36%.

IR (KBr, cm<sup>-1</sup>): 3428 (s), 1658(w), 1594 (w), 1492 (m), 1426 (w), 1237 (m), 821 (w), 704 (m).

#### X-ray Data Collection and Structure Refinement

Crystallographic data of complexes were collected on a Bruker SMART CCD diffractometer with graphite monochromated Mo-Ka radiation ( $\lambda = 0.71073$  Å) at T = 296 K. Absorption corrections were applied by using the multi-scan program [11]. The structure was solved by direct methods and successive Fourier difference syntheses (SHELXS-97),

Compounds	1	2	3
Empirical formula	C <sub>16</sub> H <sub>20</sub> Cl <sub>2</sub> CoO <sub>10</sub>	$C_{16}H_{16}CdCl_2O_8$	C <sub>16</sub> H <sub>16</sub> Cl <sub>2</sub> CuO <sub>8</sub>
Formula weight	502.15	519.59	470.74
Crystal system	Triclinic	Monoclinic	Triclinic
Space group	<i>P</i> -1	C2	<i>P</i> -1
<i>a</i> (Å)	4.923(2)	11.805(5)	5.1160(19)
<i>b</i> (Å)	5.789(2)	5.485(2)	7.060(3)
<i>c</i> (Å)	18.276(7)	14.948(6)	13.253(5)
$\alpha$ (°)	96.997(5)	90	81.801(4)
$\beta$ (°)	90.252(5)	101.211(5)	86.325(5)
$\gamma$ (°)	97.047(5)	90	76.315(4)
V (Å <sup>3</sup> )	513.0(3)	949.4(7)	460.1(3)
Z	1	2	1
$D_c (Mg/m^3)$	1.626	1.817	1.699
$\mu (\text{mm}^{-1})$	1.148	1.472	1.519
<i>F</i> (000)	257	516	239
Crystal size (mm)	$0.36 \times 0.34 \times 0.33$	$0.38\times0.35\times0.32$	$0.36 \times 0.33 \times 0.32$
$\theta$ range	1.12-28.32	1.39-28.28	1.55-28.61
Reflections collected	3315	3116	3053
Independent reflections	2279 [ $R(int) =$	1952 [ $R(int) =$	2070 [R(int) =
	0.0267]	0.0309]	0.0199]
Completeness to $\theta = 25.00$	0.984	0.995	0.985
Absorption correction	Multi-scan	Multi-scan	Multi-scan
Max. and min. transmission	0.7032 and 0.6827	0.6501 and 0.6046	0.6421 and 0.6108
Data/restraints/parameters	2279/0/133	1952/1/123	2070/0/124
Goodness of fit on F	1.054	1.058	1.139
<i>R</i> indices $[I > 2\sigma(I)]$	R1 = 0.0413,	R1 = 0.0350,	R1 = 0.0392,
	wR2 = 0.1222	wR2 = 0.0913	wR2 = 0.1037
R indices (all data)	R1 = 0.0502,	R1 = 0.0355,	R1 = 0.0510,
	wR2 = 0.1358	wR2 = 0.0916	wR2 = 0.1358
Largest diff. features $(e Å^{-3})$	0.437 and -0.737	0.869 and -0.420	0.426 and -0.438

Table 1. Experimental data for Complexes 1, 2, and 3

D−H…A	<i>D</i> —Н	H···A	D····A	D—H···A	Symmetry codes
			Complex	: 1	
O4-H4AO2	0.850	1.888	2.679	154.22	-x, -y + 1, -z + 2
O4-H4BO5	0.850	2.090	2.884	155.25	x + 1, y, z
O5-H5AO2	0.849	1.896	2.724	164.51	x, y + 1, z
O5-H5BO3	0.850	2.042	2.872	164.95	x – 1, y, z
			Complex	2	
O4-H4C…O2	0.850	1.849	2.697	176.13	x, y – 1, z
O4-H4DO3	0.850	1.855	2.704	176.27	x + 1/2, y - 1/2, z
			Complex	3	
O7-H7EO3	0.850	1.877	2.722	172.83	x – 1, y, z
O7−H7F…O2	0.850	1.818	2.663	172.79	-x + 2, -y + 1, -z + 1

Table 2. Hydrogen-bond geometry (Å)

and anisotropic thermal parameters for all nonhydrogen atoms were refined by full-matrix least-squares procedure against  $F^2$  (SHELXL-97) [12]. All nonhydrogen atoms were refined anisotropically. Hydrogen atoms were set in calculated positions and refined by a riding mode, with a common thermal parameter. H atoms for H<sub>2</sub>O molecules were located in different synthesis and refined isotropically. The crystallographic data and experimental details for the structure analysis are summarized in Table 1, the hydrogen-bonds of the three complexes are listed in Table 2, and the selected bond lengths (Å) and bond angles (å) for the three complexes are shown in Table 3.

#### **Results and Discussion**

#### Crystal Structure Descriptions

Single crystal X-ray analysis reveals that **1** and **3** crystallize in the triclinic *P*-1 space group, while **2** belongs to the monoclinic system with space group *C*2. The structure of **1** can be assigned as a dimer; the Co1 coordinate with two carboxyl oxygen atoms from two L ligands and four oxygen atoms from water molecules (Fig. 1). The two L ligands nearly locate on a same plane in which Co1, O3, and O4 lay, and Co1–O5 in its vertical plane. In the asymmetric unit of **1**, there are a half of Co(II) ion, one L ligand, and two water molecules. The Co1–O3, Co1–O4, and Co1–O5 bond lengths are 2.084(5), 2.073(7), and 2.105(5)Å, respectively. Two H<sub>2</sub>O are created by symmetry operation of the other two. These four water molecules develop the dimer in two direction and make it a 2D layer through the typical hydrogen bonds O–H…O (Fig. 2). Since any kinds of  $\pi$ - $\pi$  stacking interactions can not occur in this structure, what makes the complex to be a 3D supermolecule? It is the C–H…Cl interactions that link the 2D layers together to be a 3D structure (Fig. 3): in these interactions the distance of C–H…Cl is only 3.037(2)Å.

X-ray diffraction analysis for Complex 2 confirms that the asymmetric unit contains one L ligand, a half of cadmium ion, and one water molecule. Each cadmium (II) center adopts a distorted octahedral coordination, and its six hybrid orbits are coordinated by two oxygen atoms from two water molecules and four oxygen atoms from two carboxyl of two L ligands (Fig. 4). Contrary to Complex 1, the carboxyl of Complex 2 shows bidentate



**Figure 1.** The coordination environment of **1**. Thermal ellipsoids are shown at 30% probability. All H atoms are omitted for clarity.



Со Сі О Н С

Figure 2. The 2D layer of Complex 1 via H-bonding. Unnecessary atoms are omitted for clarity.



**Figure 3.** A part of 3D structure of Complex 1 constructed by O–H…O and C–H…Cl interactions. Unnecessary atoms are omitted for clarity.



**Figure 4.** The coordination environment of **2**. Thermal ellipsoids are shown at 30% probability. All H atoms are omitted for clarity.

Complex 1						
Co(1)-O(4)#1	2.0727(19)	Co(1)-O(3)	2.084(2)			
Co(1)-O(4)	2.0727(19)	Co(1)-O(5)#1	2.105(2)			
Co(1)-O(3)#1	2.084(2)	Co(1)-O(5)	2.105(2)			
O(4)#1-Co(1)-O(4)	180.0(2)	O(4)-Co(1)-O(5)#1	90.58(8)			
O(4)#1-Co(1)-O(3)#1	88.39(8)	O(3)#1-Co(1)-O(5)#1	91.87(8)			
O(4)-Co(1)-O(3)#1	91.61(8)	O(3)-Co(1)-O(5)#1	88.13(8)			
O(3)#1-Co(1)-O(3)	180.00(7)	O(3)#1-Co(1)-O(5)	88.13(8)			
O(4)#1-Co(1)-O(5)#1	89.42(8)	O(3)-Co(1)-O(5)	91.87(8)			
	Comp	lex 2				
Cd(1)-O(4)#1	2.197(4)	Cd(1)-O(2)#1	2.349(4)			
Cd(1)-O(4)	2.197(4)	Cd(1)-O(3)	2.370(4)			
Cd(1)-O(2)	2.349(4)	Cd(1)-O(3)#1	2.370(4)			
O(4)#1-Cd(1)-O(4)	92.8(2)	O(2)-Cd(1)-O(3)	55.82(11)			
O(4)#1-Cd(1)-O(2)	142.94(13)	O(2)#1-Cd(1)-O(3)	90.52(13)			
O(4)-Cd(1)-O(2)	101.21(16)	O(4)#1-Cd(1)-O(3)#1	124.42(14)			
O(2)-Cd(1)-O(2)#1	87.99(18)	O(2)#1-Cd(1)-O(3)#1	55.82(11)			
O(4)#1-Cd(1)-O(3)	87.99(13)	O(3)-Cd(1)-O(3)#1	134.81(19)			
O(4)-Cd(1)-O(3(CLR))	124.42(14)					
	Comp	lex 3				
Cu(1)-O(7)	1.947(2)	Cu(1)-O(3)	1.952(2)			
Cu(1)-O(7)#1	1.947(2)	Cu(1)-O(1)#1	2.401(3)			
Cu(1)-O(3)#1	1.952(2)	Cu(1)-O(1)	2.401(3)			
O(7)-Cu(1)-O(7)#1	180.00(17)	O(7)#1-Cu(1)-O(1)#1	88.24(11)			
O(7)-Cu(1)-O(3)#1	90.20(11)	O(3)#1-Cu(1)-O(1)#1	74.52(9)			
O(7)#1-Cu(1)-O(3)#1	89.80(11)	O(3)-Cu(1)-O(1)#1	105.48(9)			
O(3)#1-Cu(1)-O(3)	180.00(7)	O(3)#1-Co(1)-O(5)	88.13(8)			
O(7)-Cu(1)-O(1)#1	91.76(11)	O(3)#1-Cu(1)-O(1)	105.48(9)			

Table 3. The selected bond lengths (Å) and bond angles (å) for Complexes 1, 2, and 3

*Note.* Symmetry transformations used to generate equivalent atoms: #1 for Complex 1: -x, -y + 1, -z + 2; #1 for Complex 2: -x + 1, y, -z; #1 for Complex 3: -x + 2, -y, -z + 1.

coordination, in which two L ligands that link the Cd1 ion make the complex a V shape. The dihedral angle about the two benzene rings is  $84.65(4)^{\circ}$  and the Cd1–O2 and Cd1–O3 bond lengths are 2.349(4) and 2.371(4), respectively. Fig. 5 shows the mode that hydrogen bonds O4–H4C…O2 and O4–H4D…O3 connect adjacent dimer to form a 2D layer. Here, an interesting phenomenon is that the the distance of C–H…Cl in **2** is 3.037(2)Å, absolutely the same with **1**. This interaction builds these 2D layers to be a 3D network.

As shown in Fig. 6 for Complex 3, the coordination mode is different from that of 1 and 2. One O atom of the carboxyl and one O atom from the phenoxy work together to be a bidentate coordination. This kind of coordination constructs the Complex 3 to be a zigzag dimer. Two water molecules coordinate with the center Cu(II) ion of the zigzag dimer, which link the other dimer to be a 2D layer according to O7-H7E...O3 and O7-H7F...O2 hydrogen bonds. In this new 2D layer, all Cl atoms arrange on the surface of layer. So, the C-H...Cl interactions can be created during the adjacent layers, and help the





Figure 5. The 2D layer of Complex 2 linked by hydrogen bonding.



**Figure 6.** The coordination environment of **3**. Thermal ellipsoids are shown at 30% probability. All H atoms are omitted for clarity.



**Figure 7.** The 3D supermolecule network of Complex **3** via O–H…O and C–H…Cl interactions. Unnecessary atoms are omitted for clarity.

structure become a stable 3D supermolecule network (Fig. 7). This new network offers the supermolecule more stable 2D and 3D structures.

#### **IR** Spectroscopy

The IR spectrum of Complexes 1, 2, and 3, respectively, exhibit two peaks at 1659 and  $1426 \text{ cm}^{-1}$ , 1625 and  $1428 \text{ cm}^{-1}$ , and 1658 and  $1426 \text{ cm}^{-1}$ , which are assigned to the  $\nu_{\text{COO}-}$  stretching vibration and indicate that in complexes these carboxylate groups undoubtedly coordinate with center metal because of their large red-shift comparing with that of the free L ligand. The skeleton vibration bands of the phenyl ring appear at 1610 and 1492 in 1, 1595 and 1490 in 2, and 1594 and 1492 in 3. Furthermore, the band at around 821 cm<sup>-1</sup> in all these complexes are attributable to the  $\gamma(= C-H)$  of 1, 4-substituted phenyl ring, and the peak at around 706 cm<sup>-1</sup> can be attributed to the  $\gamma(-CH 2-)$  of the ligand.

#### **Fluorescence Emission Properties**

The photoluminescence behavior of the free L ligand and its corresponding cobalt(II), cadmium(II), and copper(II) compounds was studied in the solution of methanol at room temperature. The excitation and emission spectra of L, **1**, **2**, and **3** are in Fig. 8. Upon photoexcitation at 288 nm, the free L exhibits fluorescence emission at 310 nm. Upon excitation at 260 nm, the Compound **1** shows a similar emission peak at 314 nm. Compound **2** exhibits emission around 313 nm upon excitation at 262 nm. Upon photoexcitation at



Figure 8. Photoluminescent spectrum of Complexes 1, 2, and 3 in the solution of methanol at room temperature.

286 nm, Compound **3** shows a similar but broad emission peak at 312 nm. The emission band for Compounds **1**, **2**, and **3** are similar to that found for the free L ligand in terms of position and band shape. Therefore, the luminescence behaviors in **1**, **2**, and **3** may be attributed to the intraligand  $(\pi - \pi^*)$  transition; the expected metal-to-ligand charge transfer (MLCT) or ligand-to-metal transfer (LMCT) is not observed.

#### Conclusion

Complexes of  $[CoL_2(H_2O)_4]$  **1**,  $[CdL_2(H_2O)_2]$  **2**, and  $[CuL_2(H_2O)_2]$  **3** were synthesized, and they were characterized by elemental analysis, single-crystal X-ray diffraction techniques, X-ray powder diffraction, and photoluminescent spectrum. The results show that the water cluster in these complexes play an important role in developing the structure to be a stable 2D layer by their hydrogen bonds. The successful preparation of these complexes confirmed that the hydrogen bonds of water cluster are important factors in influencing the architecture of metal-L complexes. In addition, all the three complexes exhibit fluorescence property.

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