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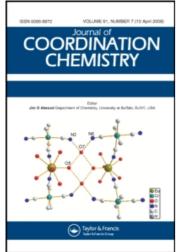
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Zhi-Li Fanga; Qi-Xiang Nieb

<sup>a</sup> School of Basic Science, East China Jiaotong University, Nanchang 330013, P.R. China <sup>b</sup> School of Civil Engineering and Architecture, East China Jiaotong University, Nanchang 330013, P.R. China

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# Zinc(II) and cadmium(II) complexes of Schiff bases derived from amino acids and pyridine-3-carboxaldehyde: synthesis, crystal structures, and fluorescence

ZHI-LI FANG† and QI-XIANG NIE\*‡

†School of Basic Science, East China Jiaotong University, Nanchang 330013, P.R. China ‡School of Civil Engineering and Architecture, East China Jiaotong University, Nanchang 330013, P.R. China

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Two d<sup>10</sup> Schiff-base complexes,  $Zn_2(L_1)_2(H_2O)_6 \cdot SO_4$  (1) and  $Zd(L_2)_2(H_2O)_4$  (2) [HL<sub>1</sub> = 3-((pyrid-3-yl)-methylene)aminobenzoic acid;  $Zd(H_2O)_4$  (2) [HL<sub>1</sub> = 3-((pyrid-3-yl)-methylene)aminobenzoic acid], have been synthesized and structurally characterized by elemental analyses, FT-IR spectra, and thermal studies, as well as single crystal X-ray diffraction. Complex 1 is a dinuclear macrocyclic structure with 22-membered rings and is assembled into a 3-D sandwich supramolecular network motif through H-bonding interactions; 2 is a mononuclear structure and is interlinked through H-bonding and  $Zd(H_2O)_4$  supramolecular network. Furthermore, fluorescent properties of the two complexes are also reported.

Keywords: Schiff base; H-bonding and  $\pi \cdots \pi$  stacking interactions; Crystal structures; Fluorescence

#### 1. Introduction

The design and construction of metal coordination polymers based on metal ions and multifunctional bridging ligands are of interest due to their intriguing topologies and potential applications as functional materials [1–9]. The design and synthesis of metal Schiff-base complexes through appropriate Schiff-base bridging ligands that are capable of binding to metal ions are well-known examples in heterogeneous catalysis, biological activity, magnetism, and photochemistry [10–17]. Through rational design, various structures have already been obtained, such as helical-chain complexes [18, 19], macrocycle complexes [20–23], and interpenetrated complexes [24]. It has been thought that novel properties and potential applications would emerge from unusual molecular structures and much effort toward the synthesis of unusual complexes with useful physical–chemical properties and intriguing structural topologies has been made [25–27].

<sup>\*</sup>Corresponding author. Email: nieqixiang@126.com

Aromatic amino acids and pyridine-3-carboxaldehyde can be condensed into Schiff-base ligands after the dehydration of an amino and a carbonyl by nucleophilic addition, and used to construct unusual metal coordination polymers based on the following considerations: (i) the Schiff base is a multifunctional ligand with both pyridine and carboxylate available to coordinate with metal ions; (ii) the designed Schiff-base ligand through the adjusting sites of pyridine and carboxylate can be optimized to construct different metal coordination polymers. In addition, d<sup>10</sup> metal ions can tolerate various coordination numbers and geometries and can also exhibit excellent luminescent properties when bound to functional ligands [28, 29].

In this report, we synthesized two new d<sup>10</sup> Schiff-base complexes,  $Z_1(L_1)_2(H_2O)_6$  SO<sub>4</sub> (1) and  $Z_1(L_2)_2(H_2O)_4$  (2) [HL<sub>1</sub>=3-((pyrid-3-yl)-methylene)aminobenzoic acid; HL<sub>2</sub>=4-((pyrid-3-yl)-methylene)aminobenzoic acid]. Both complexes were structurally characterized by elemental analyses, thermal analyses, infrared (IR) spectra, and single crystal X-ray diffraction. Single crystal X-ray structure analysis revealed that 1 is a dinuclear macrocyclic structure, forming a 3-D supramolecular network *via* H-bonding, while 2 is a mononuclear structure which is assembled into a 3-D supramolecular network motif *via* H-bonding and  $\pi \cdots \pi$  stacking interactions. Both complexes exhibit intense fluorescent emissions at room temperature.

### 2. Experimental

#### 2.1. Materials and physical measurements

All materials and reagents were obtained commercially and used without purification. Elemental (C, H, N) analyses were performed on a Perkin-Elmer 240 element analyzer. FT-IR spectra were recorded from KBr pellets at 4000–400 cm<sup>-1</sup> on a Nicolet 5DX spectrometer. Thermogravimetric analyses (TGA) were carried out on a Perkin-Elmer TGA 7 TG analyzer with a heating rate of 10°C min<sup>-1</sup> from room temperature to 700°C under nitrogen. Fluorescence spectra were recorded with an F-2500 FL Spectrophotometer analyzer.

#### 2.2. Synthesis of 1 and 2

 $Zn_2(L_1)_2(H_2O)_6 \cdot SO_4$  (1). The Schiff base 3-((pyrid-3-yl)-methylene)aminobenzoic acid (HL<sub>1</sub>) was synthesized by the condensation of 1 mmol pyridine-3-carboxaldehyde and 1 mmol 3-aminobenzoic acid in an anhydrous methanol solution. Complex 1 was synthesized from the reaction of 1 mmol HL<sub>1</sub> and 1 mmol  $ZnSO_4 \cdot 7H_2O$  in 75% methanol: water (3:1; v/v) mixture. Colorless block single crystals were obtained at room temperature by slow evaporation of the solvent over a period of several days. Anal. Calcd (%) for  $C_{26}H_{30}Zn_2N_4O_{14}S$ : C, 39.73; H, 3.82; N, 7.13. Found (%): C, 39.71; H, 3.79; N, 7.15. (KBr pellet) (cm<sup>-1</sup>): 651, 698, 769, 827, 885, 933, 1031, 1053, 1091, 1151, 1394, 1438, 1473, 1541, 1591, 1606, 1631, 3091, 3109, 3412.

 $Cd(L_2)_2(H_2O)_4$  (2). The Schiff base 4-((pyrid-3-yl)-methylene)aminobenzoic acid (HL<sub>2</sub>) was prepared by the condensation of 1 mmol pyridine-3-carboxaldehyde and 1 mmol 4-aminobenzoic acid in an anhydrous methanol solution. Complex 2 was synthesized from the reaction of 1 mmol HL<sub>2</sub> and 1 mmol  $Cd(NO_3)_2 \cdot 4H_2O$  in 75%

Table 1. Crystallographic data and structure refinement summary for 1 and 2.

	1	2
Empirical formula	$C_{26}H_{30}Zn_2N_4O_{14}S$	$C_{26}H_{32}CdN_4O_8$
Formula weight	785.34	634.91
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	$P2_I/c$
Unit cell dimensions (Å, °)	,	•
a	33.6305(1)	13.6111(1)
b	11.9798(4)	7.1768(1)
С	7.6933(2)	13.7381(1)
α	90.00	90.00
β	97.5400(10)	110.387(1)
γ	90.00	90.00
Volume ( $\mathring{A}^3$ ), Z	3072.73(16), 4	1257.93(2), 2
F (000)	1608	644
Calculated density (mg cm <sup>-3</sup> )	1.698	1.676
Absorption coefficient (mm <sup>-1</sup> )	1.706	0.927
Goodness-of-fit on $F^2$	1.031	1.017
Crystal size (mm <sup>3</sup> )	$0.21 \times 0.17 \times 0.12$	$0.20 \times 0.17 \times 0.15$
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0.0406, wR_2 = 0.0948$	$R_1 = 0.0198, wR_2 = 0.0479$
R indices (all data)	$R_1 = 0.0600, \ wR_2 = 0.1055$	$R_1 = 0.0273, \ wR_2 = 0.0515$

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

methanol: water (3:1; v/v). Two weeks later suitable single crystals of **2** were obtained at room temperature by slow evaporation of the solvent. Anal. Calcd (%) for  $C_{26}H_{32}CdN_4O_8$ : C, 49.14; H, 5.04; N, 8.82. Found (%): C, 49.11; H, 5.01; N, 8.84. (KBr pellet) (cm<sup>-1</sup>): 597, 696, 790, 891, 979, 1053, 1205, 1300, 1334, 1361, 1408, 1429, 1494, 1519, 1593, 2318, 2351, 2476, 2547, 2839, 2970, 3030, 3064, 3128, 3145, 3203.

## 2.3. X-ray crystallography

Single crystal X-ray diffraction data collections for 1 and 2 were performed on a Bruker Apex II CCD diffractometer operating at  $50\,\mathrm{kV}$  and  $30\,\mathrm{mA}$  using Mo-K $\alpha$  radiation ( $\lambda = 0.71073\,\mathrm{Å}$ ) at 293 K. Data collection and reduction were performed using SMART and SAINT software [30]. A multi-scan absorption correction was applied using SADABS [30]. The two structures were solved by direct methods and refined by full-matrix least squares on  $F^2$  using the SHELXTL program package [30]. Hydrogens were located from difference Fourier maps and a riding mode. The crystal parameters and details of the data collection and refinement are given in table 1. Selected bond lengths and angles are given in table 2. Hydrogen bonds are given in table 3.

#### 3. Results and discussion

#### 3.1. Synthesis and characterization

The synthesis of 1 and 2 is described in the experimental section. In IR spectra, a strong peak at 1591 cm<sup>-1</sup> for 1 and 1593 cm<sup>-1</sup> for 2 are attributed to C=N stretch. The features at 1541, 1394 cm<sup>-1</sup> for 1 and 1519, 1361 cm<sup>-1</sup> for 2 are associated with the

Table 2.	Selected bond	distances (Å)	and angles (°)	for <b>1</b> and <b>2</b> .
----------	---------------	---------------	----------------	-----------------------------

1			
Zn(1)–O(1)	2.201(3)	O(3w)-Zn(1)-O(1w)	176.9(1)
Zn(1)–O(2)	2.186(3)	O(2w)-Zn(1)-O(2)	155.1(1)
Zn(1)-O(1w)	2.138(3)	$N(1)^{i}$ – $Zn(1)$ – $O(2)$	98.0(1)
Zn(1)–O(2w)	2.024(3)	O(3w)-Zn(1)-O(2)	93.1(1)
Zn(1)-O(3w)	2.099(3)	O(1w)-Zn(1)-O(2)	89.6(1)
$Zn(1)-N(1)^{i}$	2.072(4)	O(2w)-Zn(1)-O(1)	95.6(1)
$O(2w)-Zn(1)-N(1)^{i}$	106.7(1)	$N(1)^{i}$ – $Zn(1)$ – $O(1)$	157.7(1)
O(2w)-Zn(1)-O(3w)	89.5(1)	O(3w)-Zn(1)-O(1)	91.4(1)
$N(1)^{i}$ – $Zn(1)$ – $O(3w)$	89.2(1)	O(2w)-Zn(1)-O(1)	91.3(1)
O(2w)-Zn(1)-O(1w)	88.7(1)	O(2)-Zn(1)-O(1)	59.7(1)
$N(1)^{i}$ – $Zn(1)$ – $O(1w)$	89.0(1)		
2			
Cd(1)-O(2w)	2.2871(15)	$N(1)-Cd(1)-N(1)^{i}$	180.0
Cd(1)=N(1)	2.3185(16)	$O(2w)^{i}$ - $Cd(1)$ - $O(1w)$	94.32(6)
Cd(1)-O(1W)	2.3384(16)	O(2w)- $Cd(1)$ - $O(1w)$	85.68(6)
O(2w)i-Cd(1)-O(2w)	180.0	N(1)– $Cd(1)$ – $O(1w)$	88.17(6)
$O(2w)^{i}$ - $Cd(1)$ - $N(1)$	87.69(6)	$N(1)^{i}$ -Cd(1)-O(1w)	91.79(6)
O(2w)-Cd(1)-N(1)	92.33(5)		. ,

Symmetry codes 1:  $^{i}$  -x + 1/2, -y + 3/2, -z; 2:  $^{i}$  -x + 1, -y, -z + 1.

Table 3. Hydrogen bond data for 1 and 2.

D–H···A	d(D-H) (Å)	$d(H \cdots A) (\mathring{A})$	$d(D \cdots A) (\mathring{A})$	∠(DHA) (°)
1				
$O(1w)-H(1w)\cdots O(3)^{i}$	0.82(1)	2.02(1)	2.808(1)	160(1)
$O(1w)-H(1w)\cdots O(5)^{ii}$	0.82(1)	1.91(1)	2.703(1)	163(4)
$O(1w)-H(2w)\cdots O(1)^{iii}$	0.81(1)	2.01(4)	2.810(5)	169(4)
$O(2w)$ - $H(3w) \cdots O(4)^i$	0.82(1)	2.10(1)	2.864(1)	154(2)
$O(2w)-H(3w)\cdots O(6)^{ii}$	0.82(1)	2.05(2)	2.724(2)	139(2)
$O(2w)$ - $H(4w) \cdots O(4)^{iv}$	0.78(1)	2.13(1)	2.851(1)	155(1)
$O(2w)$ - $H(4w) \cdots O(4)^v$	0.78(1)	1.96(2)	2.731(2)	169(2)
$O(2w)-H(5w)\cdots O(1)^{iii}$	0.82(1)	2.00(4)	2.753(2)	152(2)
$O(3w)-H(6w)\cdots O(6)^{vi}$	0.78(1)	2.03(2)	2.802(2)	168(2)
$O(3w)-H(6w)\cdots O(5)^{vii}$	0.78(1)	2.03(2)	2.688(2)	141(1)
2				
$O(1w)-H(1w)\cdots O(2)^{i}$	0.82(2)	1.99(2)	2.804(2)	173(2)
$O(1w)-H(2w)\cdots O(2)^{ii}$	0.82(2)	2.03(2)	2.836(2)	170(2)
$O(2w)-H(3w)\cdots O(2)^{iii}$	0.81(2)	1.98(2)	2.765(2)	163(2)
$O(2w)-H(4w)\cdots O(1)^{ii}$	0.82(2)	1.88(2)	2.689(2)	173(2)

Symmetry transformations used to generate equivalent atoms for 1:  ${}^{1}1/2-x$ , 1/2-y, 2-z;  ${}^{ii}1/2+x$ , 1/2-y, 1/2+z;  ${}^{ii}x$ , -y, 1/2+z;  ${}^{iv}1/2-x$ , 1/2-y, 2-z;  ${}^{v}1/2+x$ , 1/2-y, 1/2+z;  ${}^{vi}1/2-x$ , 1/2-y, 1/2+z;  ${}^{ii}1/2+x$ , 1/2-y, 1/2+z; 1/2+x, 1/2-y, 1/2

asymmetric (COO) and symmetric (COO) stretching. A broad band at 3412 and  $3203 \, \text{cm}^{-1}$  for 1 and 2, respectively, may be assigned to  $\nu$ (O–H) of water.

#### 3.2. Crystal structures

**3.2.1.** The structure of 1. Single crystal X-ray structure analysis shows that 1 is a dinuclear macrocyclic structure, belonging to a monoclinic crystal system space

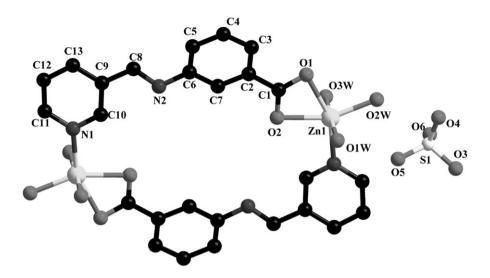


Figure 1. View of the asymmetric unit of 1. All hydrogens were omitted for clarity.

group C2/c. An ORTEP view of the asymmetric unit of 1 is shown in figure 1. In the dinuclear structure, each Zn(II) is in a distorted octahedral coordination geometry, defined by one pyridyl nitrogen (Zn-N=2.072(4) Å), two carboxyl oxygens and three aqua ligands  $[Zn-O=2.024(3)\sim 2.201(3) \text{ Å}]$  [31]. The Schiff-base ligand has an imine bond length of 1.260(5) Å, clearly indicating almost double-bond character [32]. Two symmetrically related  $L_1$  ligands interlink two Zn(II) ions to generate a 22-membered macrocyclic host unit with a  $Zn\cdots Zn$  separation of 10.6(1) Å. The pyridyl and phenyl moieties of  $L_1$  are non-coplanar with a dihedral angle of  $22.67(3)^\circ$ . Opposite corners of the host unit, made up of  $Zn(OH_2)_3$ , play a crucial role in assembly of the structure of 1. The corners act as both H-bonding donors and acceptors (table 3), resulting in a smooth layer in the bc-plane. The H-bonding acceptors of independent  $SO_4^{2-}$  situate in the middle of the layers, interconnecting adjacent layers via intermolecular H-bonding interactions to form a 3-D sandwich supramolecular network (figure 2).

3.2.2. The structure of 2. Single crystal X-ray structure analysis shows that 2 is mononuclear, crystallizing in a monoclinic crystal system space group  $P2_I/c$ . An ORTEP view of the asymmetric unit of 2 is shown in figure 3. The Cd(II) lies on an inversion center and is coordinated by two nitrogens from two L<sub>2</sub> ligands and four waters in a distorted octahedral geometry. The distances [Cd-O=2.288(1)-2.340(2) Å; Cd-N=2.318(2) Å) fall in normal ranges [33]. The bond distance of the imine (1.266(3) Å) confirms the double-bonded character of the Schiff-base ligand. The dihedral angle between pyridyl and phenyl of L<sub>2</sub> is 32.96°. In 2, the deprotonated L<sub>2</sub> is an H-bond acceptor, while in the central unit, Cd(OH<sub>2</sub>)<sub>4</sub> is a H-bond donor; as a result, a 3-D supramolecular network is generated (figure 4). The network is further stabilized by  $\pi$ - $\pi$  stacking interactions. The centroid-centroid distance and dihedral

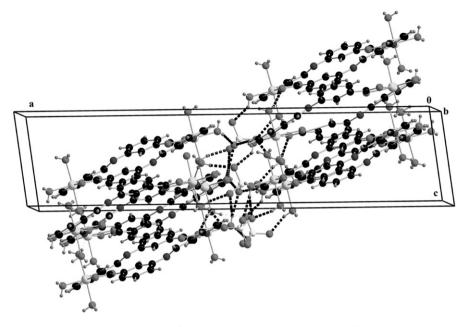


Figure 2. Packing diagram of 1. The hydrogen bonds are indicated by dashed lines.

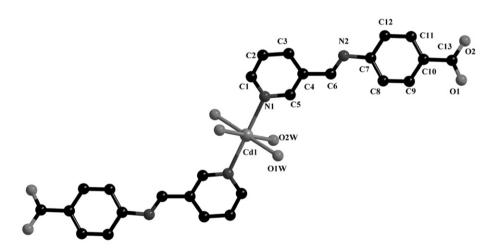


Figure 3. View of the asymmetric unit of 2. All hydrogens were omitted for clarity.

angle between pyridyl plane and phenyl plane are  $3.635\,\text{Å}$  and  $4.08^\circ$ , respectively, indicating a normal  $\pi\cdots\pi$  contact [34].

## 3.3. Thermal and luminescent properties

For 1, TGA studies indicated first weight loss (13.7%) in the range of 61–150°C, attributed to the loss of six molecules of water (Calcd 13.8%). In the range 150–323°C,

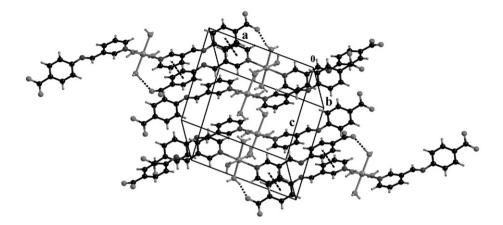


Figure 4. Packing diagram of 2. The hydrogen bonds and  $\pi$ - $\pi$  stacking interactions are indicated by dashed lines.

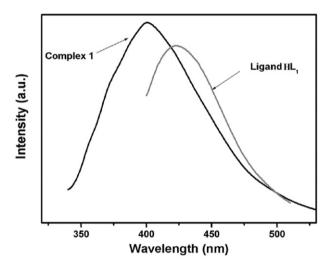


Figure 5. Solid state fluorescent emission spectra of  $HL_1$  and 1.

little weight is lost indicating a stable framework. The second weight loss of 64.9% (Calcd 65.4%) from 323 to 623°C corresponds to the loss of the Schiff base. The final residual weight is 21.0% (Calcd 20.8%) corresponding to ZnO.

For **2**, the first weight loss (11.5%) from 89°C to 132°C corresponds to the loss of four molecules of water (Calcd 11.3%). A stable framework exists over 298°C. The second weight loss of 67.9% between 298°C and 548°C is attributed to the loss of the Schiff base (Calcd 68.4%). Upon further heating, the mass is unchanging with a weight of 20.1%, corresponding to CdO (Calcd 20.2%) (Supplementary material).

Owing to the excellent luminescent properties of  $d^{10}$  metal ions, the solid state photoluminescence of  $HL_1$ ,  $HL_2$ , 1, and 2 were investigated at room temperature. Upon excitation at 305 nm, the emission spectrum of 1 shows a broad emission with

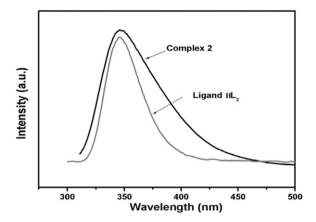


Figure 6. Solid state fluorescent emission spectra of HL<sub>2</sub> and 2.

the maximum located at 402 nm, whereas free  $HL_1$  displays a typical ligand-based fluorescent emission (422 nm) as evident by the obvious vibronic structure (figure 5). Therefore, we tentatively ascribe the fluorescence to metal-perturbed, ligand-centered transitions, probably mixed with some ligand to metal charge transfer character [35–37]. Upon excitation at 285 nm, the maximal emission of 2 at 346 nm (figure 6) is essentially the same as that of free  $HL_2$ ; therefore, we believe that the observed luminescence of 2 can be ascribed to ligand-based  $\pi \cdots \pi^*$  transitions.

#### 4. Conclusion

We report two new d<sup>10</sup> metal Schiff-base complexes exhibiting excellent photoluminescence at room temperature. Characterization of the two complexes was by elemental analyses, FT-IR, thermal studies, and X-ray structural analyses. Use of this successful synthetic approach provides us with a potential route to other metal Schiff-base complexes with interesting structures, topologies, and properties.

#### Supplementary material

Crystallographic data for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 686227 and No. 686228. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336 033, E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk), or is also be available from the author on request.

## Acknowledgment

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