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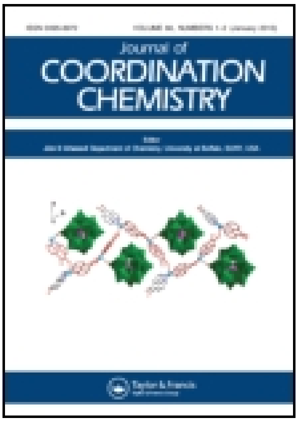

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Structural diversity and solid-state properties of Co^{II} and Zn^{II} coordination complexes with 5-aminonicotinate through metal direction

Pei Yang[a], Zhi-Wei Ge[a], Peng-Wen Liu[a] & Jing Chen[a]

[a] College of Chemistry, Tianjin Key Laboratory of Structure and Performance for Functional Molecules, MOE Key Laboratory of Inorganic-Organic Hybrid Functional Material Chemistry, Tianjin Normal University, Tianjin, P.R. China

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PEI YANG, ZHI-WEI GE, PENG-WEN LIU and JING CHEN*

College of Chemistry, Tianjin Key Laboratory of Structure and Performance for Functional Molecules, MOE Key Laboratory of Inorganic-Organic Hybrid Functional Material Chemistry, Tianjin Normal University, Tianjin, P.R. China

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Zinc and cobalt 5-aminonicotinate (5-AN^-) complexes, $Co(5\text{-}AN)_2(H_2O)_4$ (**1**) and $\{[Zn(5\text{-}AN)_2](H_2O)\}_n$ (**2**), have been hydrothermally synthesized and structurally characterized. Single-crystal X-ray diffraction results indicate that coordination geometries are different (octahedral for Co^{II} and tetrahedral for Zn^{II}) and 5-AN^- adopts distinct binding modes (terminal in **1** and bridging in **2**), forming a simple mononuclear coordination motif for **1** and a 2-D (4,4) coordination layer for **2**. The higher-dimensional supramolecular architectures for both complexes are constructed via hydrogen bonding. Both complexes have been characterized by IR, microanalysis, and powder X-ray diffraction techniques and, their thermal stability and fluorescence have also been investigated.

Keywords: Metal-directed assembly; Hydrogen bonding; 5-Aminonicotinate

1. Introduction

Assembly of metallosupramolecular systems has developed rapidly because of their fascinating structures and functions such as adsorption, ion exchange, catalysis, luminescence, and magnetism [1–5]. Intensive studies have focused on their structure–function correlation. An effective strategy for constructing such architectures is to employ ligands as subunits to link metal centers to obtain self-assembled complexes. Other physical or chemical factors will also affect the coordination assembly, such as solvent, counterion, template, temperature, pH [6–10]. Generally, the tectonic ligands containing pyridyl and/or carboxyl groups have proven to be the most effective ligands in structural regulation of coordination polymers due to their rich coordination modes [11–15]. Carboxylates may display unidentate, chelating and bridging coordination, and pyridyl are good candidates to coordinate transition metal ions. Non-covalent cooperative forces such as hydrogen bonding and aromatic stacking are also significant to extend the structure to form higher-dimensional structures [16, 17].

Nicotinic acid and its derivatives have been used in the assembly of crystal materials [18, 19]. In this context, 5-aminonicotinic acid (5-HAN) was chosen to construct crystalline materials, with multiple potential coordination sites containing pyridyl and carboxyl groups

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*Corresponding author. Email: hxxychj@mail.tjnu.edu.cn

as well as a significant substituent effect of amino group. The 5-HAN ligand has various coordination modes and may serve as terminal or bridging building blocks, hence leading to discrete or higher-dimensional coordination frameworks. The amino and carboxyl groups of 5-HAN can provide hydrogen-bonding sites to extend the coordination entities to high-dimensional hydrogen-bonding architectures.

Two distinct structures, $Co(5\text{-}AN)_2(H_2O)_4$ (**1**) and $\{[Zn(5\text{-}AN)_2](H_2O)\}_n$ (**2**), were obtained under similar hydrothermal conditions (mononuclear for **1** and 2-D layer for **2**). Both complexes have been characterized by IR, microanalysis, and powder X-ray diffraction (PXRD) techniques. Solid-state properties such as thermal stability of **1** and **2** and luminescence of **2** have also been explored.

2. Experimental

2.1. *Materials and methods*

All reagents and solvents were commercially available and used as received. Elemental analyses of carbon, hydrogen, and nitrogen were carried out with a CE-440 (Leeman labs) analyzer. FTIR spectra (KBr pellets) were taken on an AVATAR-370 (Nicolet) spectrometer. PXRD patterns were recorded on a Rigaku D/Max-2500 diffractometer at 40 kV and 100 mA for a Cu-target tube (λ = 1.5406 Å). The calculated PXRD patterns were obtained from the single-crystal diffraction data using PLATON [20]. Thermogravimetric analysis (TGA) experiments were performed on a TGA Q500 thermal analyzer from 25 to 600 °C (heating rate: 10 °C min^{-1}) under N_2. Fluorescence spectra of the solid samples were measured on a Cary Eclipse spectrofluorimeter (Varian) at room temperature.

2.2. *Preparation of the complexes*

2.2.1. Synthesis of $Co(5\text{-}AN)_2(H_2O)_4$ (1). A mixture of 5-HAN (13.8 mg, 0.1 mM) and $Co(OAc)_2{\cdot}4H_2O$ (24.9 mg, 0.1 mM) in water (10 mL) was sealed in a Teflon-lined stainless steel vessel (20 mL), which was heated to 100 °C for 24 h and then gradually cooled to room temperature at 5 °C h^{-1}. Red block single crystals of **1** were obtained in 43% yield (8.7 mg, based on 5-HAN). Anal. Calcd for $C_{12}H_{18}CoN_4O_8$ (**1**): C, 35.57; H, 4.48; N, 13.83%. Found: C, 35.49; H, 4.42; N, 13.79%. IR (cm^{-1}): 3372 b, 1649 m, 1589 s, 1570 w, 1382 *vs*. 1330 *vs*. 1279 s, 1166 w, 1130 w, 1023 s, 881 w, 794 s, 696 s, 619 w, 572 w.

2.2.2. Synthesis of $\{[Zn(5\text{-}AN)_2](H_2O)\}_n$ (2). The same synthetic method as that for **1** was used except that $Co(OAc)_2{\cdot}4H_2O$ was replaced by $Zn(OAc)_2{\cdot}2H_2O$ (21.9 mg, 0.1 mM), affording colorless block single crystals of **2** in 39% yield (6.9 mg, based on 5-HAN). Anal. Calcd for $C_{12}H_{12}ZnN_4O_5$ (**2**): C, 40.30; H, 3.38; N, 15.67%. Found: C, 40.26; H, 3.34; N, 15.69%. IR (cm^{-1}): 3413 b, 3341 b, 1639 m, 1598 w, 1480 *vs*. 1446 s, 1377 *vs*. 1334 s, 1298 m, 1169 m, 1133 m, 1024 s, 884 s, 821 s, 784 s, 720 s, 694 w, 600 w, 471 w.

2.3. *X-ray crystallography*

X-ray single-crystal diffraction data for **1** and **2** were collected on a Bruker Apex II CCD diffractometer at ambient temperature with Mo K_α radiation (λ = 0.71073 Å). There was no

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

	1	**2**
Chemical formula	$C_{12}H_{18}CoN_4O_8$	$C_{12}H_{12}ZnN_4O_5$
Formula mass	405.23	357.63
Crystal system	Triclinic	Orthorhombic
Space group	*P-1*	*Ibca*
a/Å	7.0969(7)	14.1929(9)
b/Å	7.6731(8)	14.4100(9)
c/Å	8.7447(14)	27.4173(17)
α/°	94.989(2)	90.00
β/°	110.972(2)	90.00
γ/°	117.0960(10)	90.00
Unit cell volume/Å^3	377.52(8)	5607.4(6)
Temperature/K	173(2)	296(2)
No. of formula units per unit cell, Z	1	16
No. of reflections measured	1924	13,577
No. of independent reflections	1308	2487
R_{int}	0.0148	0.0247
Final R_1 values ($I > 2\sigma(I)$)	0.0271	0.0285
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.0685	0.0743
Final R_1 values (all data)	0.0282	0.0373
Final $wR(F^2)$ values (all data)	0.0695	0.0815
Goodness of fit on F^2	1.070	1.060

evidence of crystal decay during data collection. Semiempirical absorption corrections were applied (SADABS) and SAINT was used for integration of the diffraction profiles [21]. Both structures were solved by direct methods using SHELXS of the SHELXTL package and refined with SHELXL [22, 23]. The final refinements were performed by full-matrix least-squares methods on F^2 with anisotropic thermal parameters for all non-H atoms. Generally, C-bound hydrogens were placed geometrically and refined as riding, whereas O- and N-bound hydrogens were first determined in difference Fourier maps and then fixed in calculated positions. In **2**, the lattice waters of O5 and O6 were assigned to 1/2 occupancy to obtain the appropriate thermal parameters. Further crystallographic details are summarized in table 1 and selected bond lengths and angles are shown in table 2.

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

1			
Co1–O4	2.087(2)	Co1–N1	2.123(2)
Co1–O3	2.174(1)		
O4–Co1–N1	92.43(6)	O4–Co1–O3	95.08(6)
N1–Co1–O3	87.32(6)		
2			
Zn1–O4A	1.931(2)	Zn1–O1	1.960(2)
Zn1–N1B	2.018(2)	Zn1–N3	2.032(2)
O4A–Zn1–O1	108.0(1)	O4A–Zn1–N1B	117.5(1)
O1–Zn1–N1B	112.7(1)	O4A–Zn1–N3	114.2(1)
O1–Zn1–N3	94.8(1)	N1B–Zn1–N3	107.4(1)

Symmetry codes for **2**: A = $x-1/2, -y, z$; B = $-x + 1/2, y-1/2, z$.

3. Results and discussion

3.1. *Description of crystal structure of 1*

Single-crystal X-ray diffraction analysis indicates that **1** is mononuclear. As shown in figure 1(a), Co^{II}, lying on an inversion center, shows an octahedral environment to coordinate two nitrogens from two 5-AN^- with Co–N distance of 2.123(2) Å, as well as four oxygens from water with Co–O distances of 2.087(1) and 2.174(1) Å. 5-AN^- adopts the κ_N coordination mode with the pyridine bonding to Co^{II} to afford a mononuclear coordination motif. The amino and carboxylate groups of 5-AN^- can behave as the hydrogen-bonding acceptor and donor to result in a higher-dimensional supramolecular system. As depicted in figure 1(b), the amino group is involved in N2–H2A···O3 and N2–H2B···O1 hydrogen bonds with water ligand and carboxylate of 5-AN^- to interconnect adjacent mononuclear molecules to afford a 1-D chain. Such 1-D chains are further interlinked via O3–H3A···O2 and O4–H4A···O1 hydrogen bonds between the coordinated water and carboxylate of 5-AN^- to generate a 2-D layer network (figure 1(c) and table 3). As shown in figure 1(d), such 2-D layers are further extended to afford the resultant 3-D supramolecular architecture via O3–H3B···O2 and O4–H4B···O2 interactions involving carboxylate and coordinated water.

3.2. *Description of crystal structure of 2*

By introducing Zn^{II} into the assembling system instead of Co^{II}, a 2-D polymeric layer of **2** is afforded. The asymmetric unit of **2** consists of one Zn^{II}, two 5-AN^- ligands, and half-occupied lattice waters (figure 2(a)). Each Zn^{II} is four-coordinate to two carboxylate oxygens from two 5-AN^- ligands (Zn–O: 1.931(2) and 1.960(2) Å) and two pyridines from two 5-AN^- ligands (Zn–N: 2.018(2) and 2.032(2) Å), forming a distorted tetrahedral geometry. 5-AN^- features κ_{N,COO^-} coordination with monodentate carboxylate and pyridine. Adjacent Zn^{II} centers are interconnected by pairs of bridging 5-AN^- ligands resulting in a 2-D (4,4) coordination network (figure 2(b)), with neighboring Co···Co lengths of 7.206(1) and 7.160(1) Å. Uncoordinated O2 and O3 of carboxylate are involved in H-bonding interactions with amino groups of 5-AN^- (N2–H2B···O2 and N4–H4B···O3) to result in the final 3-D supramolecular architecture (figure 2(c) and table 3).

3.3. *Comparison of the structure features for 1 and 2*

Complexes **1** and **2** were obtained by assembling 5-HAN with $Co(OAc)_2{\cdot}4H_2O$ and $Zn(OAc)_2{\cdot}2H_2O$, showing mononuclear and 2-D layered structures, respectively. This difference may be attributed to different coordination of the metal cations and different binding of 5-AN^-. The Co^{II} in **1** is six-coordinate in a distorted octahedral geometry, whereas the coordination sphere around Zn^{II} in **2** is a distorted tetrahedral geometry. 5-AN^- adopts κ_N coordination in **1**, whereas another mode of κ_{N,COO^-} can be found in **2**. Both complexes are extended to 3-D supramolecular networks via hydrogen-bonding interactions. For **1**, multiple hydrogen bonds involving amino group, carboxylate group of 5-AN^-, and coordinated waters via N–H···O and O–H···O link the mononuclear structure to form the 3-D supramolecular architecture. With regard to **2**, adjacent 2-D layers are connected to afford the 3-D network via N–H···O hydrogen bonds involving the amino and carboxylate groups.

Structural comparison with the previous results has also been made. For Co^{II} compounds based on other pyridine carboxylates, the Co^{II} is six-coordinate in most cases but may

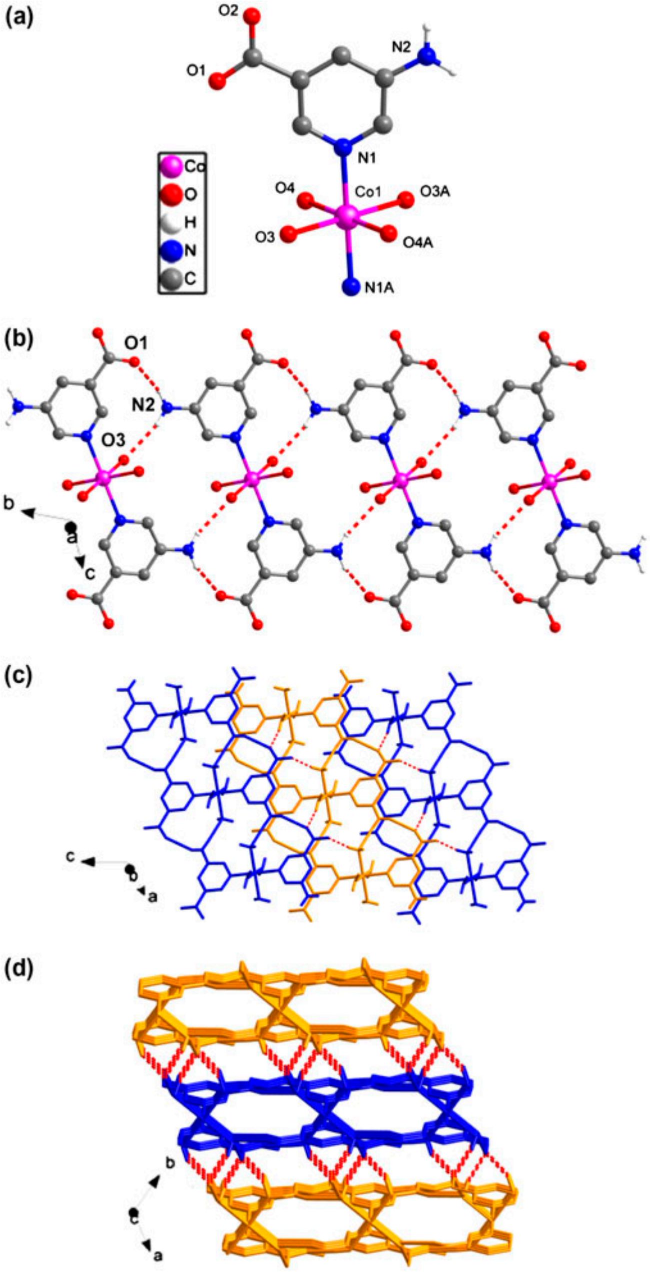

Figure 1. Crystal structure of **1**. (a) View of the asymmetric coordination unit and coordination sphere of Co(II). (b) 1-D chain showing N–H···O hydrogen-bonding interactions. (c) 2-D hydrogen-bonding network constructed via O3–H3A···O2 and O4–H4A···O1 (adjacent chains are represented in different colors and hydrogen bonds are shown as red dashed lines). (d) 3-D hydrogen-bonded network constructed via O3–H3B···O2 and O4–H4B···O2 interactions (adjacent layers are represented in different colors and hydrogen bonds are shown as red dashed lines) (see http://dx.doi.org/10.1080/00958972.2013.834334 for color version).

Table 3. H-bonding geometries (Å, °) for **1** and **2**.

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	D–H···A
N2–H2A•••O3^{a}	0.88	2.27	3.129(2)	166
N2–H2B•••O1^{a}	0.88	2.32	3.117(3)	150
O3–H3A•••O2^{b}	0.84	1.94	2.758(3)	163
O3–H3B•••O2^{c}	0.84	1.99	2.800(2)	162
O4–H4A•••O1^{d}	0.84	1.79	2.617(2)	168
O4–H4B•••O2^{e}	0.84	2.00	2.792(3)	157
N2–H2A•••O6^{a}	0.86	2.52	3.280(8)	147
N2–H2B•••O2^{b}	0.86	2.16	2.924(3)	148
N4–H4A•••O5^{c}	0.86	2.19	2.959(6)	149
N4–H4B•••O3^{d}	0.86	2.30	3.014(3)	141

Symmetry codes for **1**: a = $x + 1, y + 1, z$; b = $x, y, z + 1$; c = $-x, -y + 2, -z$; d = $-x, -y + 1, -z$; e = $x + 1, y, z + 1$; for **2**: a = $x - 1/2, -y + 1, z$; b = $x, y + 1/2, -z + 1$; c = $x + 1/2, y, -z + 1/2$; d = $x - 1/2, y, -z + 1/2$.

represent different octahedral environments. For example, as reported by Zhao and Li [24], in $[Co_{1.5}L(H_2O)_4]_n$ (L = 4-hydroxypyridine-2,6-dicarboxylic acid), the octahedral environment of Co^{II} is defined by six oxygens, different to that in **1**, resulting in a different 1-D stair-chain structure. Chen *et al.* [25] have recently reported another Co^{II} complex $[Co(L)(OH)_2]\cdot 2H_2O$ (L = 1,1′-(1,4-butanediyl)bis(3-carboxyl-2-oxidopyridinium)), the Co^{II} center also represents a different octahedral environment defined by six oxygens. The resultant 1-D "Z" coordination chain is also different to that in **1**. Most Co^{II} ions take typical octahedral geometries in complexes based on many other ligands [26–28]. With regard to the Zn^{II} compounds, the Zn^{II} centers generally adopt octahedral or trigonal-bipyramidal geometries in complexes based on other pyridine carboxylates, such as $[Zn(L)(OH)_2]\cdot 4H_2O$ or $[Zn(L)(H_2O)]$ (L = 1,1′-(1,4-butanediyl)bis(3-carboxyl-2-oxidopyridinium)) [25] and $\{[Zn_2(HCAM)(H_2CAM)_2]\cdot(4,4'\text{-bipyridine})\cdot 3.5H_2O\}_n$ (H_3CAM = 2,6-dicarboxy-4-hydroxypyridine) [29]. The Zn^{II} in **2** takes the uncommon tetrahedral coordination different to that in previously reported complexes. Some similar ligands, such as nicotinic acid or isonicotinic acid, have also been frequently used to assemble with other metal salts, such as Ln^{III} and Cd^{II} salts [30, 31]. Different to Co^{II}/Zn^{II} complexes, the carboxylates in these Ln^{III} and Cd^{II} complexes take various monodentate, bidentate chelating/bridging coordination modes, and the pyridine N atoms are not coordinated (the carboxyl group of 5-HAN is monodentate in **1** and uncoordinated in **2**. The pyridine of 5-HAN in **1** and **2** participates in coordination). So, the different coordination tendency of metal salts and ligands results in distinct coordination structures.

3.4. *IR, PXRD, TGA, and fluorescence*

In IR spectra of **1** and **2**, the absence of absorption at 1650–1700 cm^{-1} confirms a deprotonation of carboxylate. The phase purities of the complexes were identified by PXRD patterns, which are in agreement with the correspondingly simulated ones (see figure S1). TGA experiments were performed to investigate thermal stabilities. The TGA curves of **1** and **2** are shown in figure S2. For **1**, weight loss (18.8%) from room temperature to 151 °C corresponds to loss of four guest waters (calculated: 17.8%). The residual framework starts to decompose beyond 272 °C with a series of complicated weight loss steps and does not stop until heating ends at 600 °C. For **2**, weight loss starts from the room temperature and does not stop until heating ends at 600 °C. Solid-state photoluminescence spectra for **2** were

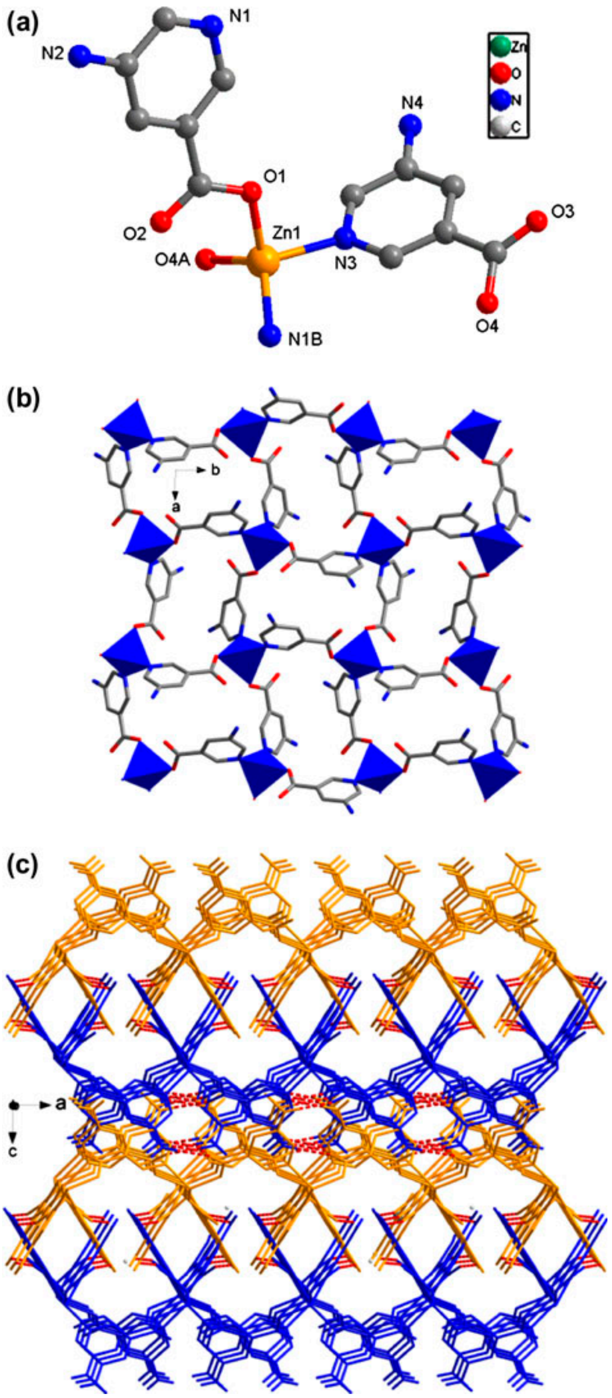

Figure 2. Crystal structure of **2**. (a) View of the asymmetric unit and the coordination environment of Zn(II). (b) 2-D coordination motif linked by bridging 5-AN$^-$. (c) 3-D supramolecular network constructed via N2–H2A···O6 and N2–H2B···O2 hydrogen-bonding interactions (adjacent layers are represented in different colors and hydrogen bonds are shown as red dashed lines) (see http://dx.doi.org/10.1080/00958972.2013.834334 for color version).

measured at room temperature (see figure S3). Upon excitation at 290 nm for **2**, the maximal emissions appear at 397 nm. No obvious fluorescent emissions for 5-HAN can be found under similar conditions. Thus, the photoluminescent mechanism of **2** can be ascribed to metal-ligand (5-HAN) interactions.

4. Conclusion

Two coordination complexes were synthesized under similar hydrothermal conditions, assembled by 5-HAN with $Co(OAc)_2 \cdot 4H_2O$ or $Zn(OAc)_2 \cdot 2H_2O$. The deprotonated 5-AN^- in **1** and **2** coordinates differently to construct the complexes. Amino and carboxylate groups of 5-HAN provide stable donors or acceptors of hydrogen bonding to afford higher-dimensional networks. The distinct coordination and extended network structures of these complexes can be ascribed to the coordination tendency of Co^{II} or Zn^{II} as well as coordination modes of 5-HAN. This motivates us to further explore the metal-directed assembly of metal–organic architectures.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, 947523 and 947524 for **1** and **2**. Copies of this information may be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK]. Fax: C44 1223 336 066; E-mail: deposit@ccdc.cam.ac.uk.

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