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### Two cadmium(II) 1-D coordination polymers, $\{[\text{Cd}_2(\mu\text{-Cl})_4\text{Cl}_2(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot (\text{H-aql})_2\}$ and $[\text{Cd}(\mu\text{-Cl})_2(\text{aql})]$ : synthesis, crystal structures and fluorescent properties

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## Two cadmium(II) 1-D coordination polymers, $\{[\text{Cd}_2(\mu\text{-Cl})_4\text{Cl}_2(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot (\text{H-aql})_2\}_n$ and $[\text{Cd}(\mu\text{-Cl})_2(\text{aql})]_n$ : synthesis, crystal structures and fluorescent properties

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Two Cd(II) 1-D chain coordination polymers with Cd–Cl–Cd–Cl 1-D chains as bases, one connected to protonated 8-aminoquinoline by intermolecular hydrogen bonds  $\{[\text{Cd}_2(\mu\text{-Cl})_4(\text{Cl})_2(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot (\text{H-aql})_2\}_n$  (**1**) (aql = 8-aminoquinoline) and the other to 8-aminoquinoline  $[\text{Cd}(\mu\text{-Cl})_2(\text{aql})]_n$  (**2**), have been synthesized and structurally characterized by single-crystal X-ray analysis. **1** belongs to the monoclinic system with space group  $P2(1)/m$  and **2** belongs to the monoclinic system with space group  $C2/c$ . Fluorescent properties of aql, **1**, and **2** showed that the fluorescence spectrum ( $\lambda_{\text{max}} = 386$  nm) of **1** is mainly of aql  $\pi\pi^*$  character. The fluorescence spectrum of **2** which emits blue-green light ( $\lambda_{\text{max}} = 497$  nm) mainly involves a metal-perturbed intraligand  $\pi\pi^*$  transition from strong covalent bonds. Thermal stabilities of **1** and **2** have also been studied.

**Keywords:** Cadmium complex; 8-Aminoquinoline; One-dimensional chain; Fluorescent property

### 1. Introduction

Infinite 1-D chains are of interest because they can be used as non-linear optical [1], magnetic [2, 3], and luminescent materials [4], and the structural units can be further expanded to 2-D layers or 3-D networks either by covalent bonds [5, 6] or by hydrogen bonds and  $\pi\text{-}\pi$  interactions [3, 4, 7–10]. A large number of papers have reported optical properties and crystal structures of infinite 1-D chains which are organized by organic bridging ligands such as bipyridine compounds or dicarboxylic acids [11–14]. However, only a few examples of 1-D Cd(II) coordination polymers containing chloride-bridging Cd–Cl inorganic chains and their photophysical properties have been studied [15–17].

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Thus, constructing new frameworks of this type and studying the luminescent properties stemming from their special structures to obtain potential fluorescent materials is a challenge.

For this goal, we synthesized a multifunctional ligand 8-aminoquinoline dihydrochloride in which two nitrogen atoms are protonated. This compound not only can coordinate to metal ions to form metal-organic networks after deprotonating but also can provide extra chlorides to coordinate metal ions as bridges under appropriate circumstances. Two different Cd(II) salts,  $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$  and  $\text{Cd}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$ , were selected to self-assemble different 1-D Cd-Cl chains. The first cadmium salt contains sufficient chloride ions to form Cd-Cl chains. The second one, however, with higher pH, deprotonates 8-aminoquinoline prior to coordination to Cd(II). By this way, we have synthesized two 1-D coordination polymers of different structures which possess aql molecules or protonated aql molecules and Cd-Cl chains with different connection between aql or H-aql and Cd-Cl chains (covalent bonds and hydrogen bonds). Structures were studied by X-ray single-crystal structural analysis. Furthermore, we studied fluorescent properties of the two compounds in order to study the effect of structure on the fluorescence. Finally, their thermal stabilities under argon were examined.

## 2. Experimental

### 2.1. Materials and measurements

8-Aminoquinoline dihydrochloride ( $\text{C}_9\text{H}_8\text{N}_2 \cdot 2\text{HCl}$ ) was synthesized by neutralization to pH=3 with concentrate hydrochloride in benzene, precipitation and drying in air [Anal. Calcd% (found%) for  $\text{C}_9\text{H}_{10}\text{N}_2\text{Cl}_2$ : C, 49.79 (50.02); H, 4.64 (4.56); N, 12.90 (13.16)]. Other compounds were used as purchased from creditable companies.

IR spectra were recorded on a FTS-40 infrared spectrophotometer using KBr discs in the  $4000\text{--}400\text{ cm}^{-1}$  region. Fluorescent properties were determined by a Hitachi-F4500 spectrofluorometer. Thermogravimetric analysis was performed under argon at a heating rate of  $10^\circ\text{C min}^{-1}$  on a NETZSCH STA409 PC instrument, in the temperature range  $25\text{--}750^\circ\text{C}$ . Elemental analysis was performed on a Flash EA 1112 analyzer.

### 2.2. Synthesis of $\{[\text{Cd}_2(\mu\text{-Cl})_4(\text{Cl})_2(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot (\text{H-aql})_2\}_n$ (1)

8-Aminoquinoline dihydrochloride (0.022 g, 0.1 mmol) was dissolved in 10 mL methanol. A solution of 15 mL methanol containing  $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$  (0.023 g, 0.1 mmol) was added dropwise to the above solution with stirring for 15 min and filtered. The filtrate was allowed to slowly evaporate at ambient temperature. Yellow needles were obtained from the filtrate after one month, collected by filtration, washed using methanol and dried in air. Yield: 40%. Anal. Calcd% (found%) for  $\text{C}_{19}\text{H}_{24}\text{Cd}_2\text{Cl}_6\text{N}_4\text{O}_2$ : C, 29.33 (29.45); H, 3.11 (3.07); N, 7.20 (7.09). IR (KBr,  $\text{cm}^{-1}$ ): 3473m, 3350vs, 2784w, 1649m, 1564w, 1494w, 1420w, 1384w, 1312w, 829s, 765m, 524w.

### 2.3. Synthesis of $[Cd(\mu-Cl)_2(aql)]_n$ (**2**)

The same synthetic procedure as for **1** was used except that  $CdCl_2 \cdot 2.5H_2O$  was replaced with  $Cd(OOCCH_3)_2 \cdot 2H_2O$  (0.027 g, 0.1 mmol), obtaining colorless needles. Yield: 45%. Anal. Calcd% (found%) for  $C_9H_8CdCl_2N_2$ : C, 33.01 (32.70); H, 2.46 (2.36); N, 8.55 (8.37). IR (KBr,  $cm^{-1}$ ): 3351w, 3254vs, 1623w, 1591w, 1503vs, 1470m, 1397s, 1321w, 1091w, 1070m, 830s, 784s, 716w.

### 2.4. Crystal structural determinations

Single crystals of  $0.17 \times 0.07 \times 0.04$  mm dimensions for **1** and  $0.19 \times 0.09 \times 0.08$  mm<sup>3</sup> for **2** were mounted on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite crystal and incident beam monochromator using Mo-K $\alpha$  radiation ( $\lambda = 0.71073$  Å). A total of 8189 reflections were measured over the ranges  $2.47^\circ \leq \theta \leq 27.50^\circ$ ,  $-9 \leq h \leq 9$ ,  $-13 \leq k \leq 24$ ,  $-11 \leq l \leq 12$ , yielding 3052 unique reflections for **1**, a total of 3944 reflections were measured over the ranges  $3.55^\circ \leq \theta \leq 25.47^\circ$ ,  $-23 \leq h \leq 23$ ,  $-14 \leq k \leq 20$ ,  $-8 \leq l \leq 7$ , yielding 2055 unique reflections for **2**. The structures were solved by direct methods. Non-hydrogen atoms were located by direct phase determination and subjected to anisotropic refinement. The hydrogen atoms were located in their calculated positions and refined using a riding model (the hydrogen atoms of amine group were located using difference Fourier method). Disordered methanol was treated by setting the occupations of three methyl hydrogen atoms to 50%. The full-matrix least-squares calculations on  $F^2$  were applied to the final refinement using the SHELX-97 program [18]. The refinement converged at  $R_1 = 0.0229$  and  $wR_2 = 0.0501$  values for reflections with  $I > 2\sigma(I)$  for **1**, and  $R_1 = 0.0593$  and  $wR_2 = 0.1456$  values for reflections with  $I > 2\sigma(I)$  for **2**. Details of the crystal structure determinations of **1** and **2**

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

	<b>1</b>	<b>2</b>
Empirical formula	$C_{19}H_{24}Cd_2Cl_6N_4O_2$	$C_9H_8CdCl_2N_2$
Formula weight	777.92	327.48
Crystal system	Monoclinic	Monoclinic
Space group	$P2(1)/m$	$C2/c$
$a$ (Å)	7.6012(6)	19.567(8)
$b$ (Å)	18.5360(16)	16.952(7)
$c$ (Å)	9.5177(8)	7.294(3)
$\alpha$	90.00	90.00
$\beta$	104.3480(10)	105.258(7)
$\gamma$	90.00	90.00
$V$ (Å <sup>3</sup> )	1299.18(19)	2334.2(18)
$D_c$ (Mg m <sup>3</sup> )	1.357	1.864
$Z$	2	8
$\mu$ (mm <sup>-1</sup> )	2.279	2.291
Reflections collected/unique	8189/3052	3944/2055
Data/restraints/parameters	3052/4/170	2055/0/128
$R_1$	0.0229	0.0593
$wR_2$	0.0501	0.1456
Goodness-of-fit on $F^2$	1.055	1.025
$\Delta\rho_{\min}$ and $\Delta\rho_{\max}$ (e Å <sup>-3</sup> )	0.424 and -0.357	2.018 <sup>a</sup> and -1.415 <sup>b</sup>

<sup>a</sup>A distance of 0.97 Å away from Cd(1), <sup>b</sup>A distance of 0.98 Å away from Cd(1).

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

<b>1</b>		<b>2</b>	
Cd(1)–O(2)	2.336(3)	Cd(1)–N(1)	2.319(6)
Cd(1)–Cl(2)	2.5342(9)	Cd(1)–N(2)	2.343(6)
Cd(1)–Cl(3)	2.6233(6)	Cd(1)–Cl(1)	2.556(2)
Cd(1)–Cl(3)#1	2.6233(6)	Cd(1)–Cl(2)	2.5819(17)
Cd(1)–Cl(1)#1	2.6839(6)	Cd(1)–Cl(3)	2.585(2)
Cd(1)–Cl(1)	2.6839(6)	Cl(2)–Cd(1)#1	2.5819(17)
Cd(2)–O(1)	2.382(3)	Cl(3)–Cd(1)#2	2.585(2)
Cd(2)–Cl(4)	2.5579(9)	N(2)–C(1)	1.453(8)
Cd(2)–Cl(3)	2.6112(6)		
Cd(2)–Cl(3)#1	2.6112(6)	N(1)–Cd(1)–N(2)	73.3(2)
Cd(2)–Cl(1)#2	2.6602(6)	N(1)–Cd(1)–Cl(1)	160.73(15)
Cd(2)–Cl(1)#3	2.6602(6)	N(2)–Cd(1)–Cl(1)	98.43(15)
Cl(1)–Cd(2)#4	2.6603(6)	N(1)–Cd(1)–Cl(2)	99.08(14)
N(1)–C(1)	1.382(3)	N(2)–Cd(1)–Cl(2)	96.09(14)
		Cl(1)–Cd(1)–Cl(2)	99.04(5)
O(2)–Cd(1)–Cl(2)	176.12(7)	N(1)–Cd(1)–Cl(3)	94.49(15)
O(2)–Cd(1)–Cl(3)	87.34(5)	N(2)–Cd(1)–Cl(3)	166.45(13)
Cl(2)–Cd(1)–Cl(3)	95.45(2)	Cl(1)–Cd(1)–Cl(3)	91.59(6)
O(2)–Cd(1)–Cl(3)#1	87.34(5)	Cl(2)–Cd(1)–Cl(3)	91.28(7)
Cl(2)–Cd(1)–Cl(3)#1	95.45(2)		
Cl(3)–Cd(1)–Cl(3)#1	87.94(3)		
O(2)–Cd(1)–Cl(1)#1	82.63(5)		
Cl(2)–Cd(1)–Cl(1)#1	94.50(2)		
Cl(3)–Cd(1)–Cl(1)#1	169.84(2)		
Cl(3)#1–Cd(1)–Cl(1)#1	93.21(2)		
O(2)–Cd(1)–Cl(1)	82.63(5)		
Cl(2)–Cd(1)–Cl(1)	94.50(2)		
Cl(3)–Cd(1)–Cl(1)	93.21(2)		
Cl(3)#1–Cd(1)–Cl(1)	169.84(2)		
Cl(1)#1–Cd(1)–Cl(1)	83.91(3)		
O(1)–Cd(2)–Cl(4)	176.46(8)		
O(1)–Cd(2)–Cl(3)	84.26(5)		
Cl(4)–Cd(2)–Cl(3)	98.26(2)		

Symmetry transformations used to generate equivalent atoms for **1**: #1  $x, -y+1/2, z$  #2  $x+1, y, z$  #3  $x+1, -y+1/2, z$  #4  $x-1, y, z$ .

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

and their selected bond distances and angles are summarized in tables 1, 2 and 3. Full atomic data are available as a file in CIF format.

### 3. Results and discussion

#### 3.1. Crystal structures of the two compounds

The crystal structure of **1** belongs to the monoclinic system with space group  $P2(1)/m$ . Figure 1 shows that there are two coordination environments for Cd(II). Two Cd ions are bridged by two Cl ions and each Cd ion is coordinated to one terminal  $\text{Cl}^-$ . However, one Cd ion is coordinated to one water but the other is coordinated to methanol. These distinct Cd ions have distorted octahedral symmetry with four bridging Cl in the equatorial plane, one  $\text{Cl}^-$  and one water or methanol in the

Table 3. Hydrogen bond distances (Å) and angles (°) for **1**.

D–H...A	d (D–H)	d (H...A)	d (D...A)	∠(D–H...A)
N(1)–H(1A)...Cl(4)#5	0.86	2.61	3.442(2)	163.9
N(1)–H(1B)...O(1)#6	0.86	2.60	3.103(3)	118.6
N(1)–H(1B)...Cl(1)#7	0.86	2.82	3.656(3)	163.1
O(2)–H(1E)...Cl(4)#4	0.794(19)	2.26(2)	3.054(3)	178(6)
O(1)–H(2W)...Cl(2)	0.846(10)	2.312(10)	3.158(3)	177(4)
O(1)–H(1W)...N(1)#8	0.839(10)	2.471(8)	3.103(3)	132.8(5)
O(1)–H(1W)...N(1)#6	0.839(10)	2.471(8)	3.103(3)	132.8(5)

Symmetry transformations used to generate equivalent atoms: #1  $x, -y+1/2, z$  #2  $x+1, y, z$  #3  $x+1, -y+1/2, z$ . #4  $x-1, y, z$  #5  $-x+1, -y+1, -z+2$  #6  $-x+1, -y+1, -z+1$ . #7  $-x, -y+1, -z+1$  #8  $-x+1, y-1/2, -z+1$ .

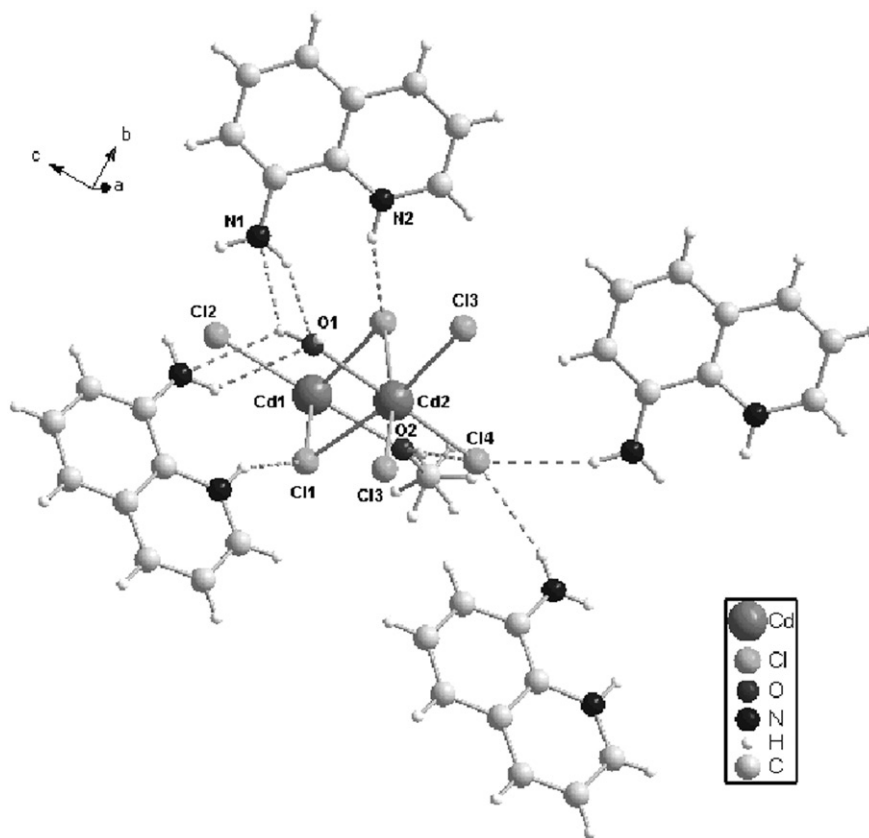


Figure 1. The molecular structure and hydrogen bonds of **1** (interrupted line: hydrogen bonds).

axial positions. The bond lengths between bridging Cl ions and Cd ions (from 2.6112(6) Å to 2.6839(6) Å) are longer than those for monodentate Cl<sup>-</sup> (from 2.5342(9) Å to 2.5579(9) Å). Each structural unit contains two aql molecules, which are protonated and do not participate in coordination.

There are four types of hydrogen bonds: N–H...Cl, N–H...O, O–H...Cl, O–H...N (D–H...A); O–H...Cl is intramolecular and the others are intermolecular

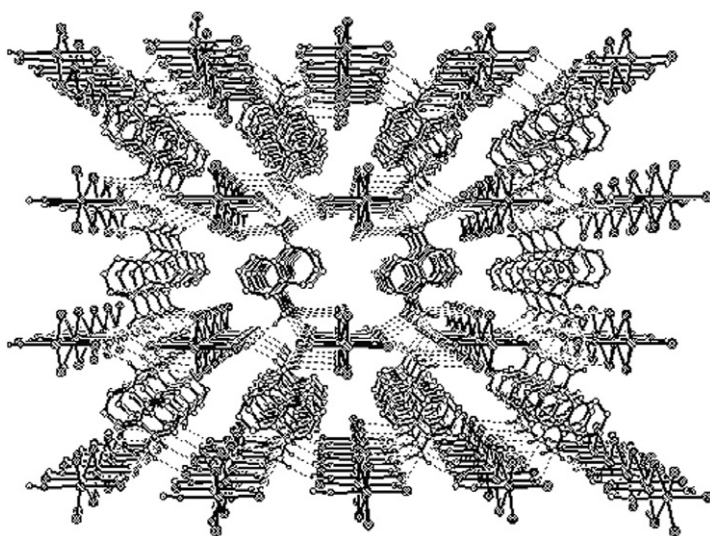


Figure 2. The  $\pi$ - $\pi$  interactions and packing of **1**.

hydrogen bonds. Both N atoms of the amino group of H-aql and O atoms of coordinating water molecules are either donors or acceptors in hydrogen bonds (shown as table 3). The lengths of hydrogen bonds vary from 2.26 Å to 2.82 Å. There are  $\pi$ - $\pi$  stacking interactions between neighboring aql rings around the infinite 1-D chains with centroid to centroid distance of 3.813(2) Å and dihedral angle 2.104(1)° or centroid to centroid distance 3.842(2) Å and dihedral angle 2.104(1)° (shown as figure 2).

Inorganic Cd-Cl 1-D chains with negative charges are constructed by bridging Cl ions coordinating to Cd ions along the *a* axis. Protonated aql molecules with positive charges link into pseudo organic infinite 1-D chains along the *a* axis by  $\pi$ - $\pi$  stacking interactions. The negative 1-D chains are linked with the positive ones by hydrogen bonds between O atoms of coordinating water and methanol molecules, bridging Cl ions and protonated N atoms of aql molecules. These 1-D chains expand into 3-D supramolecules along the *b* and *c* axes by intermolecular hydrogen bonds and  $\pi$ - $\pi$  stacking interactions (shown as figure 2).

The crystal structure of **2** is monoclinic with space group  $C2/c$ . Three  $\mu$ -Cl ions, one terminal Cl ion and two N atoms of one aql molecule are coordinated to Cd. Two  $\mu$ -Cl ions and two N atoms of one aql molecule occupy the equatorial plane and two  $\mu$ -Cl ions are apical to form distorted octahedral geometry.

Two Cd ions were alternately bridged by one Cl<sup>-</sup> and by three Cl<sup>-</sup>s forming a 1-D infinite chain. In the infinite chain, aql coordinates Cd by two N atoms (shown as figure 3). Two neighboring infinite 1-D chains penetrate into each other by aql rings with two types of  $\pi$ - $\pi$  stacking interactions, one centroid to centroid distance is 3.78(1) Å with dihedral angle 2.558(8)°, another centroid to centroid distance is 3.98(1) Å with dihedral angle 0° (shown as figures 3 and 4). The  $\pi$ - $\pi$  stacking interactions contribute to construction of a pseudo 2-D layer for **2** (shown as figure 4).

Comparing structures of the two compounds, it can obviously be seen that the major difference between **1** and **2** is two N atoms of aql coordinating to Cd in **2** while not in **1**. Another difference is that protonated ligands with positive charges are components in **1**.



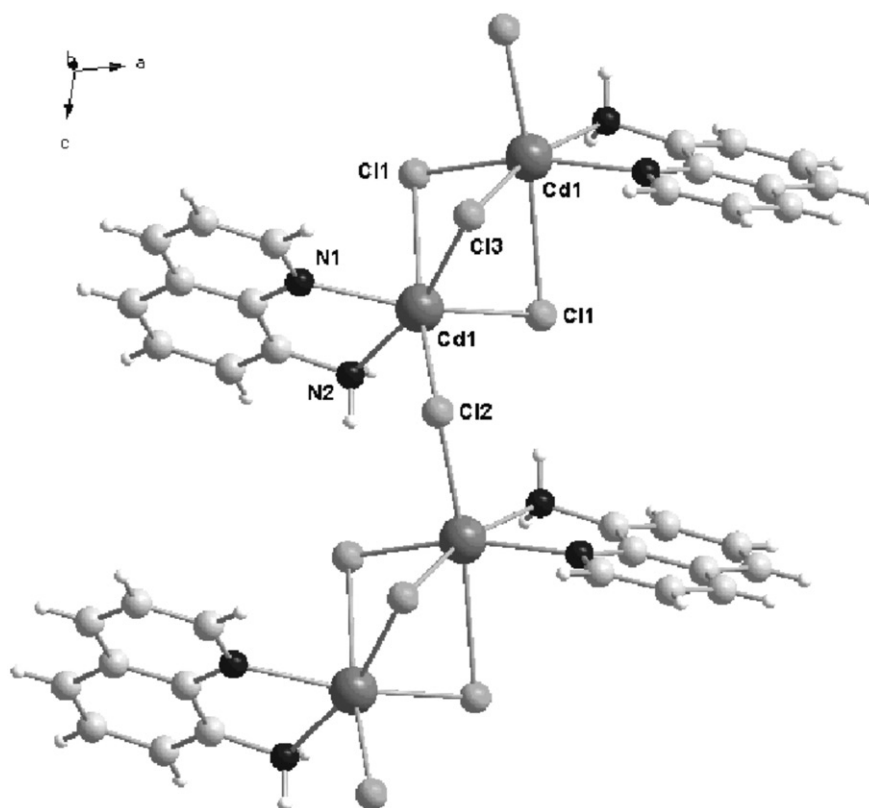


Figure 3. The molecular structure of **2**.

Adequate Cl ions may prevent ligands from coordinating to Cd ions resulting in non-coordinating protonated ligands functioning as counterions to the negatively-charged Cd–Cl chains when **1** self-assembles. For **2**, however, neutral ligands are coordinated to Cd(II) of neutral Cd–Cl chains after deprotonating in a higher pH solution from the presence of  $\text{Cd}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$ . Therefore, the bond length between coordinating N atom in amino group of aql in **2** and C atom in the ring of aql tends to be longer than that in the **1** (**2**,  $\text{N}(2)\text{--C}(1) = 1.453(8) \text{ \AA}$ ; **1**,  $\text{N}(1)\text{--C}(1) = 1.382(3) \text{ \AA}$ ).

### 3.2. Fluorescence spectra

Solid fluorescence spectra of the two complexes and the ligand aql have been determined at room temperature (emission spectral graphs shown as figure 5). There are almost the same emission peaks and spectral graph of **1** and aql which are very different from **2** (the emission maximum wavelength at 386 nm for **1**, 497 nm for **2**, 391 nm for aql) in the same excitation (243 nm). Weak effects of hydrogen bonds between 1-D infinite chains and aql cause little shift of the emission strength and maximum wavelength. Strong covalent bonds between Cd ion and nitrogen atoms of aql and deprotonation of H-aql made the emission maximum of **2** change to



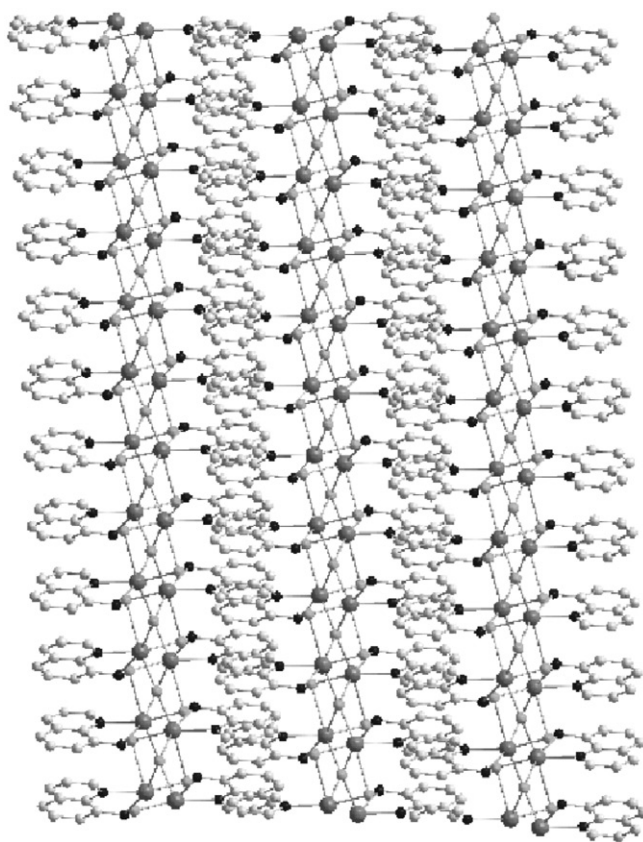


Figure 4. The  $\pi$ - $\pi$  interactions and pseudo 2-D layer of **2**.

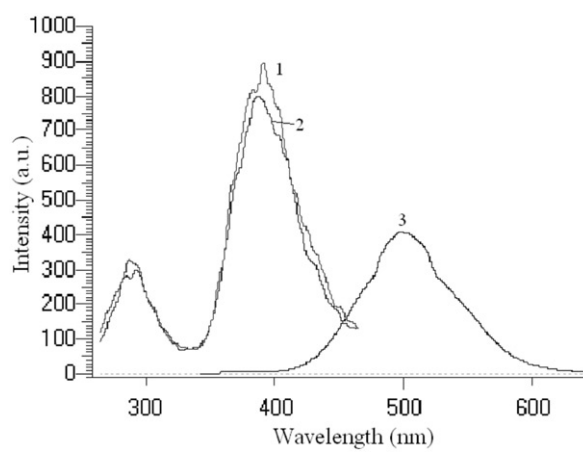


Figure 5. The emission spectra of both complexes and the ligand (1: ligand; 2: complex **1**; 3: complex **2**).

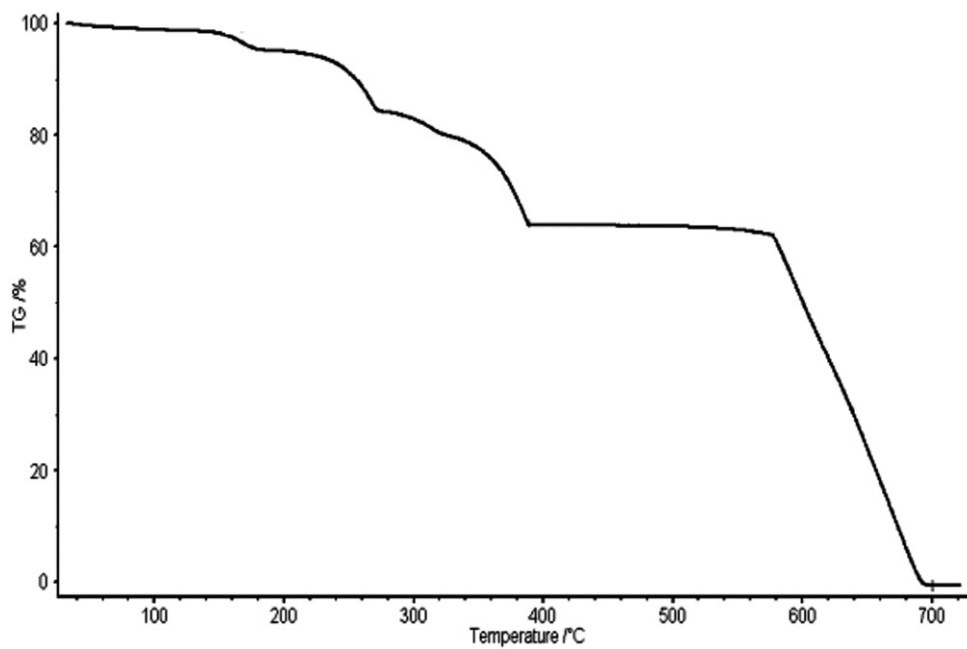


Figure 6. TG curve of 1.

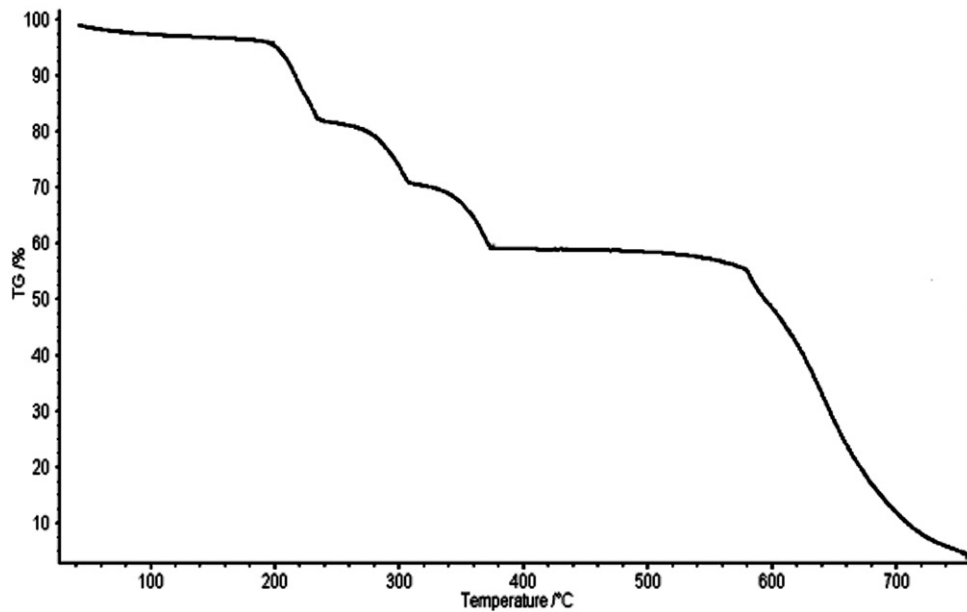


Figure 7. TG curve of 2.

blue-green spectra; the fluorescence emission intensity of **2** becomes lower than that of **1** and aql for the same reason.

Like literature reports about the fluorescent properties of Cd(II) or other d<sup>10</sup> metal complexes [19–21], the fluorescence spectrum of **1** can be assigned to ligand aql  $\pi\pi^*$  transition, but the fluorescent character of **2** mainly involves a metal-perturbed intraligand  $\pi\pi^*$  transition. For **2**, deprotonation of the ligand as well as coordination to Cd(II) could lower the HOMO-LUMO energy gap of the complex and then result in the large red-shift of the emission energy from the free ligand to the complex [22–24].

### 3.3. Thermal analysis

Thermal decompositions of **1** and **2** under argon were carried out at a heating rate of 10°C min<sup>-1</sup> (figures 6 and 7). The starting point of decomposition of **1** is about 120°C, lower than **2**, with one water and one methanol lost between 140°C to 180°C. Compound **2** is thermally stable to 200°C in argon.

## 4. Conclusion

We have synthesized and structurally characterized two new 1-D coordination polymers and analyzed the effect of structure on fluorescent properties. The different fluorescent properties of the two compounds come from the different connecting modes between inorganic 1-D chains and organic compounds with aromatic rings, though there are almost the same units of Cd–Cl–Cd–Cl 1-D chains and aql in the two compounds. Strong covalent bonds change the emission spectra; **2** exhibits intense blue-green fluorescent emission, good thermal stability, and thus may be utilized as a fluorescent material.

## Supplementary material

Crystallographic data for the structures reported in this article have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC 605408 for **1** and no. CCDC 606766 for **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].

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