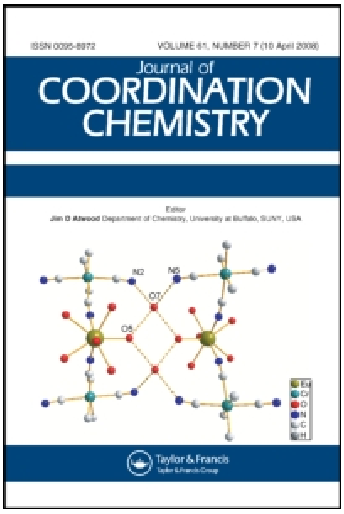

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Syntheses, crystal structures and fluorescent properties of two 4,4′-dipyridylsulfide based zinc(II) and cadmium(II) helical complexes

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Two allomeric helical coordination polymers, $\{[Zn(4,4'\text{-dps})_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ (**1**) and $\{[Cd(4,4'\text{-dps})_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ (**2**) (4,4′-dps = 4,4′-dipyridylsulfide, H_3SSA = 5-sulfosalicylic acid), have been synthesized by similar experimental methods and characterized by elemental analysis, single-crystal X-ray diffraction, and fluorescence spectra. Both crystallize in monoclinic system, space group P_21/n, with $a = 11.7947(13)$ Å, $b = 13.7475(15)$ Å, $c = 20.183(2)$ Å, and $Z = 4$ for **1** and $a = 11.940(7)$ Å, $b = 14.068(8)$ Å, $c = 20.323(12)$ Å, and $Z = 4$ for **2**. In both complexes, the metal (Zn^{II} for **1** and Cd^{II} for **2**) are six-coordinate with a N_3O_3 donor set in a distorted octahedron. Half of the 4,4′-dps are μ_2-bridging, linking adjacent metal centers and forming P_21/n dimensional helical structures along the *b*-axis. Fluorescence measurements show that **1** and **2** have medium fluorescent emission at 402 and 405 nm, respectively.

Keywords: Helix; Coordination polymer; 4,4′-Dipyridylsulfide; Crystal structure; Fluorescence measurements

1. Introduction

Use of bipyridyl-based bridging ligands and transition metal centers in preparation of coordination compounds have aroused interest for intriguing structures and potential in magnetism [1], non-linear optics [2], catalysis [3], etc. 4,4′-Dipyridylsulfide (4,4′-dps) possesses a magic angle (C–S–C ≈ 100°) and conformational non-rigidity so it has flexibility compared with linear rigid ligands such as simple 4,4′-bipyridine analogs. A number of metal complexes derived from 4,4′-dps have been reported, silver(I) complexes exhibiting yellow luminescence [4], interpenetrating (4,4) nets [5], and anion exchangeability [6]; copper(I) compounds showing 2-fold interpenetrating structures [7, 8], third order non-linear optical [NLO] properties [9], supramolecular isomerism

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phenomenon [10]; and nickel(II) complex displaying two structural phases [11]. Morever, 4,4′-dps has shown five coordination modes, non-coordinate [12–14], monodentate [15], μ_2-bridging [5, 6, 9, 10], monodentate and μ_2-bridging together [16], μ_2, and μ_3 together [4]. In this article, we describe the syntheses, crystal structures, and fluorescent properties of two allomeric helical coordination polymers, $\{[Zn(4,4'\text{-}dps)_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ (**1**) and $\{[Cd(4,4'\text{-}dps)_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ (**2**).

2. Experimental

2.1. *Materials and methods*

All chemicals were of reagent grade obtained from commercial sources and used without purification. The C, H, N, and S elemental analyses were preformed on an Elementar Vario EL elemental analyzer. The fluorescence spectrum was measured on an F-4500 FL-Fluorescence Spectrophotometer. The wavelength was scanned from 380–600 nm at 20 nm s^{-1}. Compounds **1** and **2** and free 4,4′-dps ligand were of the same concentration in DMF; data were collected at room temperature.

2.2. *Synthesis of 1*

To a stirred solution of 4,4′-dps (0.5 mmol, 0.094 g) and H_3SSA (0.5 mmol, 0.109 g) in 20 mL (v/v, 1 : 1) DMF: H_2O solution, solid $ZnCl_2$ (0.5 mmol, 0.682 g) was added. The mixture was treated with NaOH (0.5 mol L^{-1}) to a pH of 5.0, stirred at 343 K for 5 h, and filtered. One week later, colorless strip-shaped crystals were grown from the filtrate by slow evaporation. Yield: 120 mg (32%). Anal. Calcd for $C_{27}H_{30}N_4O_{11}S_3Zn$ (%): C, 43.31; H, 4.01; N, 7.48; S, 12.83. Found: C, 43.36; H, 4.05; N, 7.45; S, 12.88.

2.3. *Synthesis of 2*

To a stirred solution of 4,4′-dps (0.5 mmol, 0.094 g) and H_3SSA (0.5 mmol, 0.109 g) in 20 mL (v/v, 1 : 1) DMF: H_2O solution, solid $CdCl_2\cdot 2.5H_2O$ (0.5 mmol, 0.114 g) was added. The mixture was treated with NaOH (0.5 mol L^{-1}) to a pH of 6.0, stirred at 343 K for 4 h, and filtered. One week later, colorless claviform-shaped crystals were grown from the filtrate by slow evaporation. Yield: 142 mg (35%). Anal. Calcd for $C_{27}H_{30}CdN_4O_{11}S_3$ (%): C, 40.75; H, 3.77; N, 7.04; S, 12.07. Found: C, 40.71; H, 3.72; N, 7.09; S, 12.03.

2.4. *X-ray crystallographic studies*

The single crystal X-ray diffraction data for **1** and **2** were collected on a Bruker APEX-II CCDC area detector diffractometer using graphite-monochromated Mo-$K\alpha$ radiation ($\lambda = 0.71073$ Å). Cell constants and orientation matrix for data collection were obtained from least-squares refinement by using setting angles in the range 2.43–25.50 for **1** and 2.39–25.50 for **2**, respectively. Absorption corrections were applied using SADABS [17]. The structures were solved by direct methods [18] and refined

Table 1. Crystal data and structure refinement parameters for **1** and **2**.

Complex	**1**	**2**
Empirical formula	$C_{27}H_{30}N_4O_{11}S_3Zn$	$C_{27}H_{30}CdN_4O_{11}S_3$
Formula weight	748.10	795.13
Temperature (K)	291(2)	291(2)
Crystal system	Monoclinic	Monoclinic
Space group	P_21/n	$P\ 21/n$
Unit cell dimensions (Å, °)		
a	11.7947(13)	11.940(7)
b	13.7475(15)	14.068(8)
c	20.183(2)	20.323(12)
α	90	90
β	95.7580(10)	95.137(9)
γ	90	90
V (Å^3)	3256.0(6)	3400(4)
Z	4	4
D_{Calcd}(g cm^{-3})	1.526	1.553
Absorption coefficient (mm^{-1})	1.010	0.887
F(000)	1544	1616
Crystal size (mm^3)	0.36 × 0.26 × 0.09	0.37 × 0.27 × 0.23
θ-range (°)	2.43–25.50	2.39–25.50
Limiting indices $h/k/l$ (max, min)	−14, 13/−16, 16/−24, 24	−14, 14/−17, 17/−24, 24
Reflections collected	21,403	24,410
Independent reflections (R_{int})	6057 (0.0448)	6166 (0.1265)
Completeness to $\theta = 25.50$ (%)	99.9	97.4
Max. and min. transmission	0.9155 and 0.7150	0.8206 and 0.7360
Data/restraints/parameters	6057/0/416	6166/0/416
Goodness-of-fit on F^2	1.033	1.016
R indices [$I > 2\sigma(I)$]	$R_1 = 0.0446$, $wR_2 = 0.1053$	$R_1 = 0.0759$, $wR_2 = 0.1732$
R indices (all data)	$R_1 = 0.0722$, $wR_2 = 0.1206$	$R_1 = 0.1459$, $wR_2 = 0.2180$
Largest difference peak and hole (e Å^{-3})	0.740 and −0.343	1.294 and −0.691

$w(\mathbf{1}) = 1/[\sigma^2(F_o)^2 + (0.0548\,P)^2 + 1.4852P]$, $P = (F_o^2+2F_c^2)/3$.
$w(\mathbf{2}) = 1/[\sigma^2(F_o)^2 + (0.1158\,P)^2 + 0.0000P]$, $P = (F_o^2+2F_c^2)/3$.

by full-matrix least-squares on F^2 using SHELXL97 software [19]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were generated geometrically and treated by a mixture of independent and constrained refinement. The crystallographic data for **1** and **2** are listed in table 1, selected bond lengths (Å) and angles (°) are shown in table 2 and details of the hydrogen bonds are listed in table 3.

3. Results and discussion

3.1. *Structural description of* $\{[Zn(4,4'\text{-dps})_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ *(1)*

Single crystal X-ray diffraction analysis reveals that **1** is a zinc helical coordination polymer and the asymmetric unit contains a Zn^{II}, two 4,4′-dps, a deprotonated HSSA dianionic ligand, two coordinated, and three uncoordinated water molecules. The Zn^{II} ion is six-coordinate in a distorted octahedron (figure 1 and table 2) formed by two coordinated water molecules [Zn1–O4 = 2.166(2) Å, Zn1–O5 = 2.124(2) Å], one carboxylate of HSSA dianion [Zn1–O7 = 2.093(2) Å], one N atom from monodentate

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

Complex **1**			
Zn(1)–O(7)	2.093(2)	Zn(1)–O(5)	2.124(2)
Zn(1)–N(3)	2.149(3)	Zn(1)–O(4)	2.166(2)
Zn(1)–N(1)	2.194(3)	Zn(1)–N(4)^{a}	2.212(3)
O(7)–Zn(1)–O(5)	80.32(9)	O(7)–Zn(1)–N(3)	172.62(10)
O(5)–Zn(1)–N(3)	93.81(10)	O(7)–Zn(1)–O(4)	89.92(9)
O(5)–Zn(1)–O(4)	169.62(10)	N(3)–Zn(1)–O(4)	96.19(10)
O(7)–Zn(1)–N(1)	90.75(11)	O(5)–Zn(1)–N(1)	92.83(10)
N(3)–Zn(1)–N(1)	94.03(12)	O(4)–Zn(1)–N(1)	83.69(10)
(7)–Zn(1)–N(4)^{a}	86.51(10)	O(5)–Zn(1)–N(4)^{a}	93.64(10)
N(3)–Zn(1)–N(4)^{a}	89.42(11)	O(4)–Zn(1)–N(4)^{a}	89.26(10)
N(1)–Zn(1)–N(4)^{a}	172.44(11)		
Complex **2**			
Cd(1)–O(6)	2.257(6)	Cd(1)–O(4)	2.310(6)
Cd(1)–N(3)	2.316(7)	Cd(1)–N(1)	2.337(8)
Cd(1)–O(5)	2.351(6)	Cd(1)–N(4)^{b}	2.373(8)
O(6)–Cd(1)–O(4)	79.2(2)	O(6)–Cd(1)–N(3)	171.3(2)
O(4)–Cd(1)–N(3)	94.4(2)	O(6)–Cd(1)–N(1)	91.6(2)
O(4)–Cd(1)–N(1)	92.9(2)	N(3)–Cd(1)–N(1)	94.7(3)
O(6)–Cd(1)–O(5)	88.6(2)	O(4)–Cd(1)–O(5)	167.0(2)
N(3)–Cd(1)–O(5)	98.2(2)	N(1)–Cd(1)–O(5)	82.8(2)
O(6)–Cd(1)–N(4)^{b}	85.3(2)	O(4)–Cd(1)–N(4)^{b}	94.5(3)
N(3)–Cd(1)–N(4)^{b}	89.2(3)	N(1)–Cd(1)–N(4)^{b}	171.3(2)
O(5)–Cd(1)–N(4)^{b}	89.0(2)		

Symmetry codes: $^{a}-x+3/2$, y + 1/2, $-z+1/22$. $^{b}-x+5/2$, $y-1/2$, $-z+1/2$.

Table 3. Hydrogen bonds (Å, °) for **1** and **2**.

Donor–H···Acceptor	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
Complex **1**				
O(8)–H(8)···O(6)	0.82	1.82	2.545(4)	146.8
O(5)–H(9W)···O(1)^{a}	0.83	2.09	2.835(4)	149.6
O(5)–H(10W)···O(2)^{b}	0.83	1.88	2.699(4)	170.0
O(4)–H(8W)···O(6)	0.82	1.89	2.684(3)	161.3
O(4)–H(7W)···O(10)^{c}	0.83	1.87	2.684(3)	168.6
O(3)–H(6W)···O(9)^{c}	0.84	2.35	3.020(5)	137.2
O(3)–H(5W)···O(8)^{d}	0.84	2.15	2.904(4)	149.0
O(2)–H(4W)···N(2)^{e}	0.83	2.20	2.853(5)	135.5
O(2)–H(3W)···O(9)^{f}	0.85	1.93	2.782(4)	179.3
O(1)–H(2W)···O(11)^{c}	0.83	2.08	2.870(4)	158.7
O(1)–H(1W)···O(11)^{g}	0.82	2.07	2.884(5)	170.2
Complex **2**				
O(8)–H(8)···O(7)	0.82	1.83	2.552(9)	146.8
O(5)–H(10W)···O(10)^{h}	0.83	1.89	2.702(9)	166.2
O(5)–H(9W)···O(7)	0.83	2.03	2.740(8)	143.9
O(4)–H(8W)···O(3)^{a}	0.83	2.06	2.854(9)	161.5
O(4)–H(7W)···O(2)^{i}	0.83	1.92	2.705(10)	158.0
O(3)–H(6W)···O(11)^{j}	0.83	2.20	2.878(11)	138.0
O(3)–H(5W)···O(11)^{a}	0.85	2.04	2.890(10)	179.0
O(2)–H(4W)···N(2)^{k}	0.83	2.38	2.863(13)	117.6
O(2)–H(3W)···O(9)	0.83	1.98	2.779(10)	163.2
O(1)–H(2W)···O(8)^{c}	0.88	2.01	2.881(11)	170.0
O(1)–H(1W)···O(9)	0.83	2.22	3.041(12)	170.4

Notes: Symmetry codes: $^{a}-x+1, -y+1, -z+1$; $^{b}x, y, z-1$; $^{c}-x+1/2, y-1/2, -z+1/2$; $^{d}x+1, y, z$; $^{e}-x+1, -y, -z+1$; $^{f}-x, -y+1, -z+1$; $^{g}x+1, y, z+1$; $^{h}-x+3/2, y+1/2, -z+1/2$; $^{i}-x+1, -y+1, -z$; $^{j}x, y, z+1$; $^{k}x-1, y-1, z$.

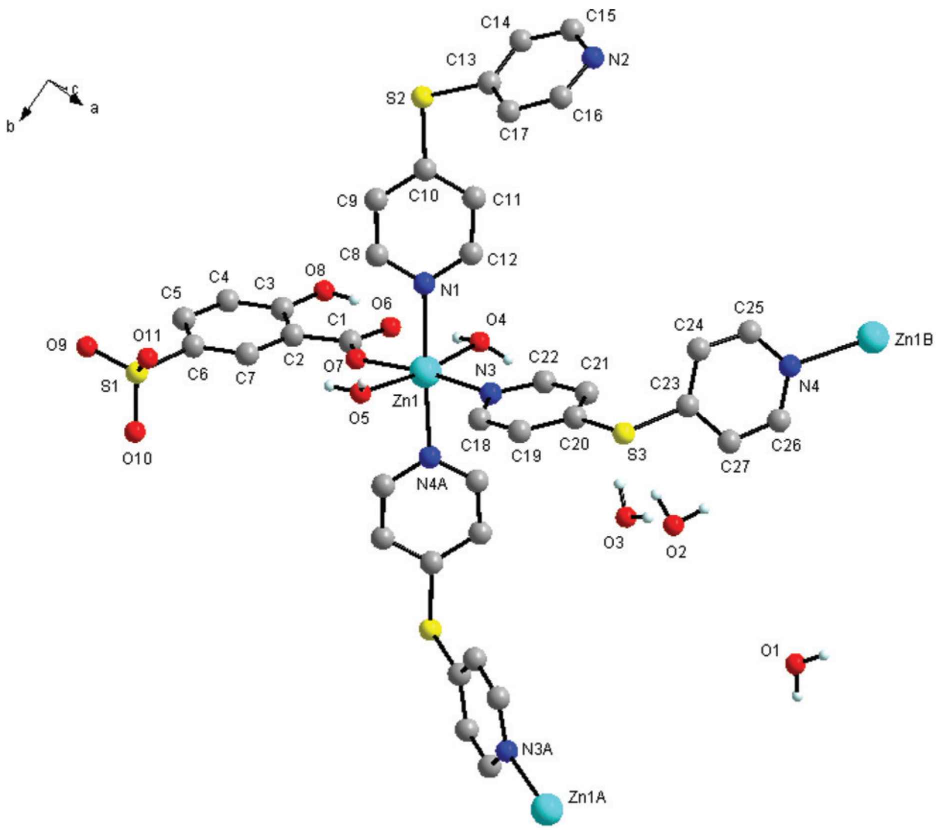

Figure 1. The coordination environment of Zn^{II} ion in **1**. H atoms on C atoms have been omitted [symmetry codes: A: $1.5-x$, $0.5+y$, $0.5-z$; B: $1.5-x$, $-0.5+y$, $0.5-z$].

4,4′-dps ligand [Zn1–N1 = 2.194(3) Å], and another two N atoms from another two μ_2-bridging 4,4′-dps [Zn1–N3 = 2.149(3) Å, Zn1–N4A = 2.212(3) Å] (symmetry codes for A: $1.5-x$, $0.5+y$, $0.5-z$). N3, O4, O5, and O7 atoms define the equatorial plane while N1, N4A occupy axial positions. Zn1 deviates from the least-squares plane of the equatorial plane N3/O4/O5/O7 by 0.0024 Å towards N1. Half of the 4,4′-dps are monodentate and the other half are μ_2-bridging. Through the μ_2-bridging adjacent Zn^{II} centers are joined to form a 1-D helical structure (figures 2a and b) along the *b*-axis in the monoclinic unit cell, with Zn···Zn1A ($1.5-x$, $0.5+y$, $0.5-z$) or Zn···Zn1B ($1.5-x$, $-0.5+y$, $0.5-z$) distance of 10.9718 (10) Å. The helical pitch is 13.7475(16) Å.

The phenolic hydroxyl and carboxyl of HSSA dianion are involved in intramolecular hydrogen bonding (figures 3a and b, and table 3). Together with the other O–H···O intermolecular hydrogen bonds with participation of water molecules, the helical structure is further stabilized.

3.2. *Structural description of* $\{[Cd(4,4'\text{-dps})_2(HSSA)(H_2O)_2]\cdot 3(H_2O)\}_n$ *(2)*

Complex **2** is an allomer to **1** and the asymmetric unit consists of Cd^{II}, two 4,4′-dps, a deprotonated HSSA dianion, two coordinated, and three uncoordinated

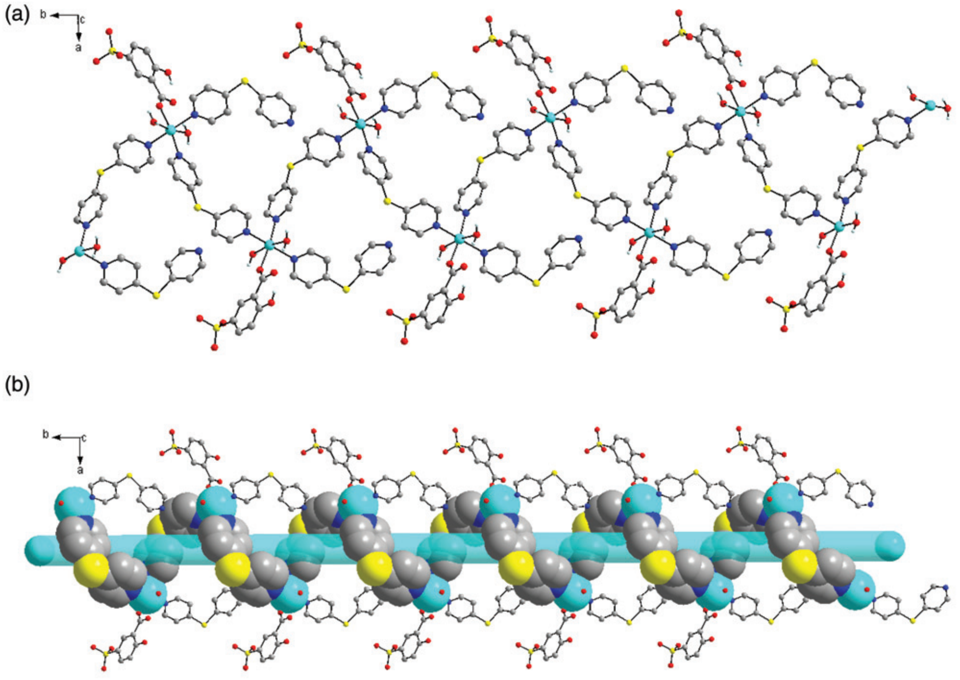

Figure 2. (a) The helical structure for **1** along *b*-axis. Uncoordinate water molecules and H atoms on C atoms have been omitted. (b) The space filled diagram of the helical structure for **1** along *b*-axis. Uncoordinate water molecules and H atoms have been omitted.

water molecules. The Cd^{II} is bonded to two water molecules [Cd1–O4 = 2.310(6) Å, Cd1–O5 = 2.351(6) Å], one carboxylate of HSSA [Cd1–O6 = 2.257(6) Å], one nitrogen from monodentate 4,4′-dps [Cd1–N1 = 2.337(8) Å], and another two nitrogens from another two μ_2-bridging 4,4′-dps [Cd1–N3 = 2.316(7) Å, Cd1–N4A = 2.373(8) Å] (symmetry codes for A: $2.5-x$, $-0.5+y$, $0.5-z$) in a distorted octahedron (figure 4 and table 2). N3, O4, O5, and O6 atoms define the equatorial plane and N1 and N4A are axial. Cd1 deviates from the least-squares plane of N3/O4/O5/O6 by 0.0048 Å towards N1. Half of the 4,4′-dps are monodentate and the other half are μ_2-bridging, connecting neighboring Cd^{II} centers to form a 1-D helical structure along the *b*-axis, with Cd···Cd1A ($2.5-x$, $-0.5+y$, $0.5-z$) or Cd···Cd1B ($2.5-x$, $0.5+y$, $0.5-z$) distance of 11.2231(46) Å. The helical pitch is 14.0680(8) Å.

The phenolic hydroxyl and carboxyl of HSSA dianion take part in intramolecular hydrogen bonding interactions (table 3). Together with the other O–H···O intermolecular hydrogen bonds with water molecules, the helical structure is further stabilized.

The helical structures of **1** and **2** are nearly the same with a reported Ni^{II} complex "Poly [[diaqua(μ_2-4,4′-dipyridylsulfide-κ^2N:N′)(4,4′-dipyridylsulfide-κN)(2-hydroxy-5-sulfonatobenzoato-κO^1)nickel(II)]dihydrate]" [16]. The difference is that for the latter there are two uncoordinated water molecules and the helical pitch is 13.9441(14) Å, which is in the range of that of **1** and **2**; their geometric parameters agree well.

Comparing allomeric helical **1** with **2**, the helical pitch is gradually increasing for the same main group element from zinc to cadmium.

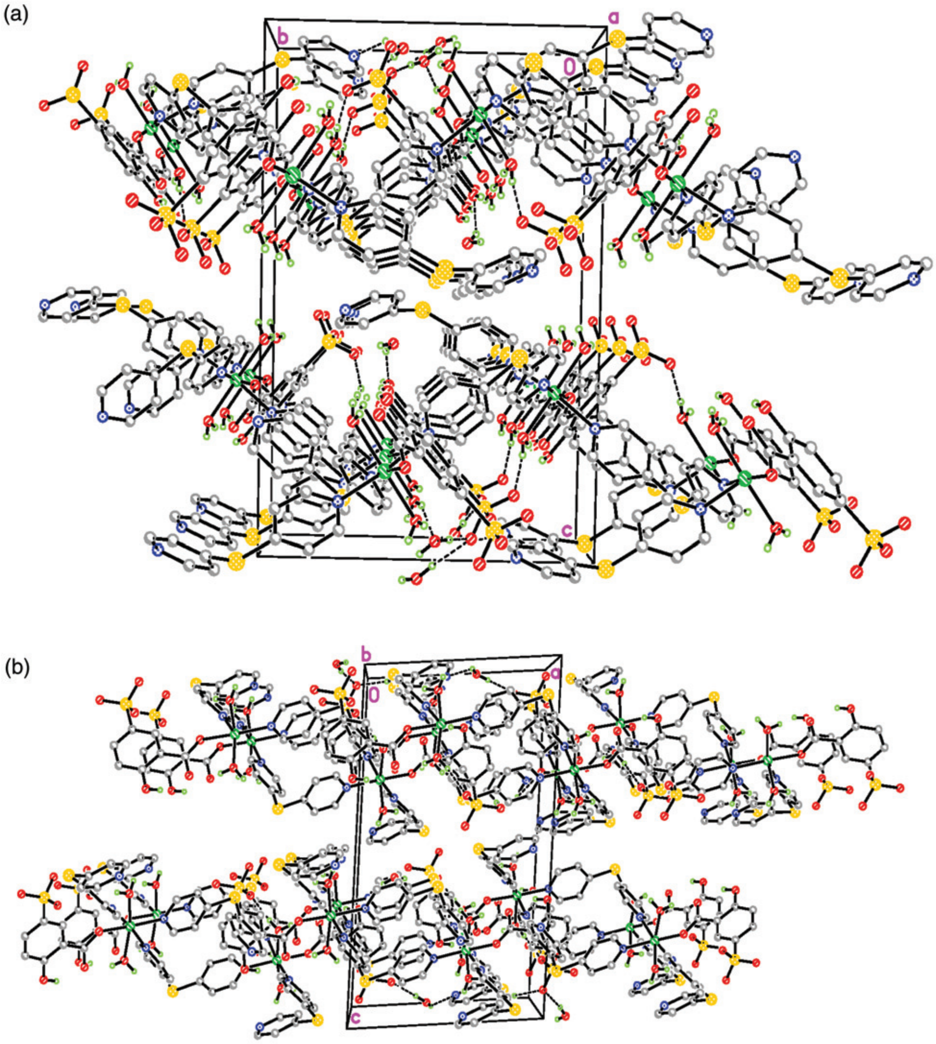

Figure 3. Hydrogen bonds in **1**, shown as dashed lines. H atoms bound to C atoms have been omitted. Hydrogen bonds projected on (a) the bc plane (b) the ac plane.

3.3. *Fluorescence spectrum*

Measurements of the photoluminescence spectra of **1** show a blue fluorescence emission at 402 nm upon excitation at 290 nm; **2** emits blue fluorescent band at 405 nm when excited at 288 nm. Comparably, free 4,4′-dps displays an emission band centered at about 415.2 nm when excited at 290 nm (figure 5). Compared with free 4,4′-dps, the fluorescence of **1** and **2** are intensified, blue shifting 13 nm for **1** and 10 nm for **2**; the fluorescence may be attributed to the metal-to-ligand charge transfer [MLCT] [20–24]. Thus, they may be better candidates for light emitting materials than free 4,4′-dps.

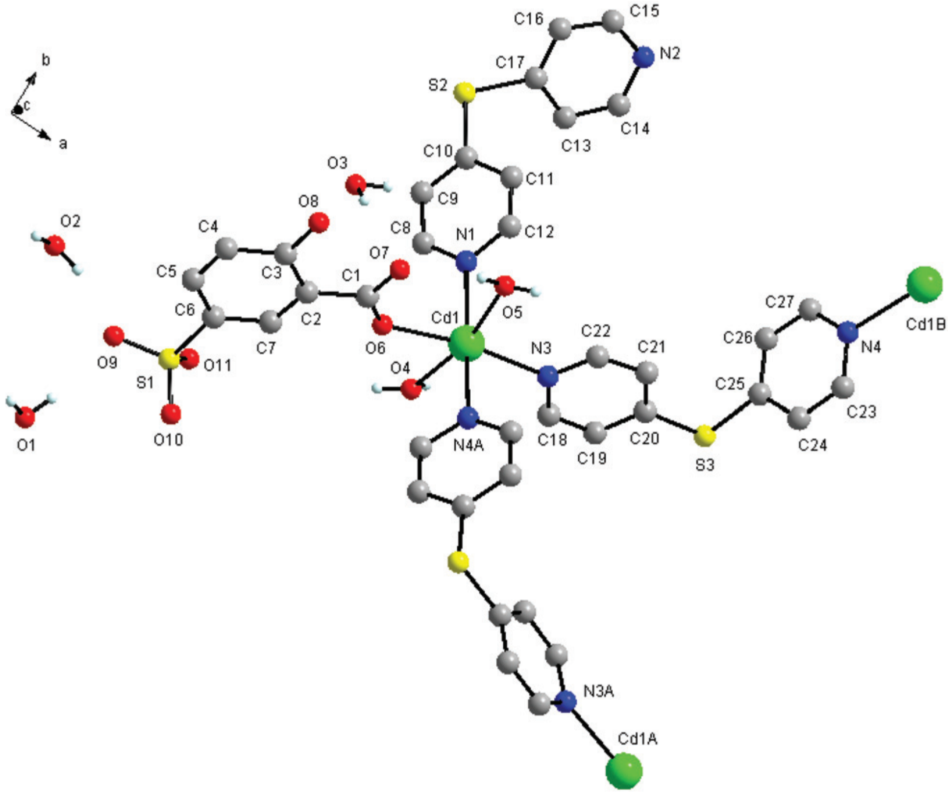

Figure 4. The coordination environment of Cd^{II} ion in **2**. H atoms on C atoms have been omitted [symmetry codes: A: $2.5-x$, $-0.5+y$, $0.5-z$; B: $2.5-x$, $0.5+y$, $0.5-z$].

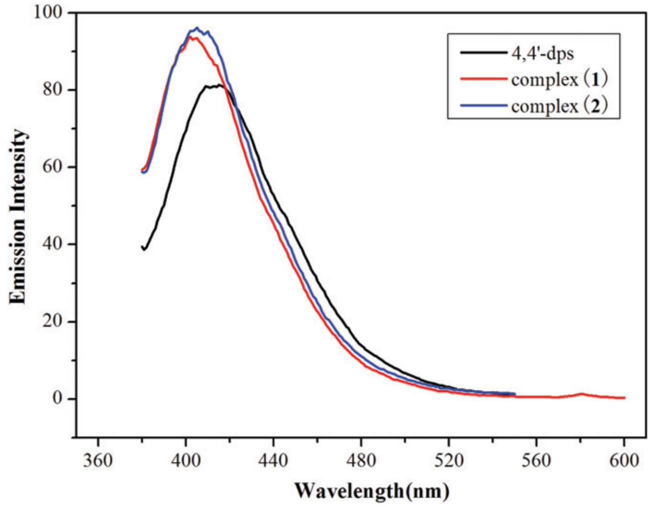

Figure 5. The emission spectra of free 4,4′-dps ($\lambda_{ex} = 290\,nm$) and complexes **1** ($\lambda_{ex} = 290\,nm$) and **2** ($\lambda_{ex} = 288\,nm$) in DMF solution at room temperature.

4. Conclusions

We described two allomeric helical coordination polymers, $\{[\mathrm{Zn}(4,4'\text{-dps})_2(\mathrm{HSSA})(\mathrm{H_2O})_2]\cdot 3(\mathrm{H_2O})\}_n$ (**1**) and $\{[\mathrm{Cd}(4,4'\text{-dps})_2(\mathrm{HSSA})(\mathrm{H_2O})_2]\cdot 3(\mathrm{H_2O})\}_n$ (**2**). The center metal ions ($\mathrm{Zn^{II}}$ for **1** and $\mathrm{Cd^{II}}$ for **2**) are six-coordinate with a N_3O_3 donor set in a distorted octahedral environment. The μ_2-bridging 4,4′-dps ligands link adjacent metal centers forming two similar 1-D helical structures along the *b*-axis. Fluorescence measurements show that **1** and **2** fluoresce at 402 and 405 nm and might be candidates for light emitting materials.

Supplementary material

CCDC-668092 (**1**) and CCDC-668088 (**2**) contains the supplementary crystallographic data for this article. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, CB2 1EZ, UK; Telephone: (44) 01223 762910; Facsimile: (44) 01223 336033; Email: deposit@ccdc.cam.ac.uk].

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