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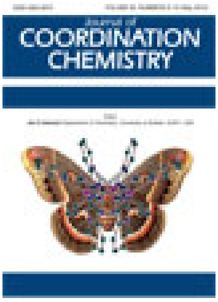

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Syntheses, structures, and luminescence of three anion-dependent cadmium coordination polymers

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Syntheses, structures, and luminescence of three anion-dependent cadmium coordination polymers

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Self-assembly of the flexible ligand 1,3,5-tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene (ttmb) with Cd(II) salts gave three coordination polymers $\{[Cd(ttmb)_2](SO_4)(H_2O)_{16}\}_n$ (**1**), $\{[Cd(ttmb)_2(H_2O)_2](NO_3)_2(H_2O)_6\}_n$ (**2**), and $\{[Cd(ttmb)_2(H_2O)_2](ClO_4)_2(H_2O)_7\}_n$ (**3**). Complex **1** has a (3,6)-connected $CdCl_2$-type 2-D network. However, **2** and **3** show the 2-D (4,4) network and 1-D double chain, respectively. Complexes **1**, **2**, and **3** exhibit luminescent emission maxima at 306, 339, and 298 nm, respectively, in the solid state at room temperature.

Keywords: Cadmium MOFs; $CdCl_2$-type network; Anion-dependent network; 2-D network and double chain; Luminescence; 1,3,5-Tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene

1. Introduction

Metal–organic frameworks (MOFs) have attracted attention because of potential applications as functional materials, architectures, and intriguing topologies [1–6]. A universal strategy for construction of MOFs is appropriate choice of metal and ligands. However, because of the presence of various subtle interactions in the assembly process and the difficulty of predicting compositions or structures of the products, it is still a challenging issue in synthetic chemistry [7–13]. Anions do more than merely balancing the charges of a cationic complex, influencing the structure of a supramolecular system through coordination [14–19]. Previously we reported the reaction of 1,4-bis(1,2,4-triazol-1-yl)butane (btb) with Cd(II) salts with different anions NO_3^-, BF_4^-, ClO_4^-, and PF_6^- gave a 1-D chain $[Cd(btb)(H_2O)_2(NO_3)_2]_n$, a threefold interpenetrated diamondoid network $\{[Cd(btb)_2(H_2O)_2](BF_4)_2\}_n$ and the threefold interpenetrated α-polonium cubic network $\{[Cd(btb)_3](ClO_4)_2\}_n$ and $\{[Cd(btb)_3](PF_6)_2\}_n$ [19].

A large number of mononuclear, oligonuclear, and polynuclear transition metal complexes of 1- and 4-substituted 1,2,4-triazole derivatives have been synthesized and characterized due to their magnetic properties and novel topologies [20–25]. Our synthetic approach starts by focusing on construction of new topological frameworks and potential functional materials using flexible bis(triazole) ligands by adjusting the

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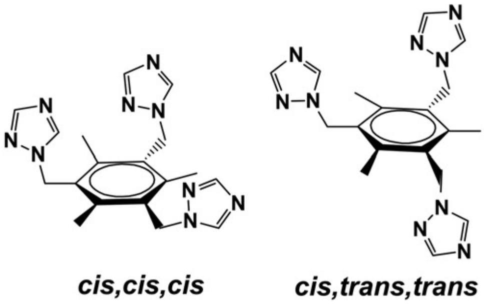

Scheme 1. Two conformations of 1,3,5-tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene.

lengths and flexibilities [26–33]. 1,3,5-Tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene (ttmb) ($C_{18}H_{21}N_9$, scheme 1) has remarkable structural features: (a) it is a flexible ligand which can adjust its conformations according to different geometric requirements of the metal ion to provide coordination polymers with different structures and properties; (b) it is a tripodal ligand with three triazole rings which may construct coordination polymers [34–36]. In addition, d^{10} metal coordination polymers exhibit interesting photoluminescence [23–25, 29, 30, 32, 36]. In the present work, we synthesized three cadmium(II) coordination polymers $\{[Cd(ttmb)_2](SO_4)(H_2O)_{16}\}_n$ (**1**), $\{[Cd(ttmb)_2(H_2O)_2](NO_3)_2(H_2O)_6\}_n$ (**2**), and $\{[Cd(ttmb)_2(H_2O)_2](ClO_4)_2(H_2O)_7\}_n$ (**3**) by the reaction of ttmb and Cd(II) salts. **1** has a (3,6)-connected $CdCl_2$-type 2-D network. The Schläfla symbol for the 2-D network of **1** is $(4^3)_2(4^6.6^6.8^3)$. However, **2** and **3** show the 2-D (4,4) network and 1-D double chain, respectively. Here, we report the syntheses, crystal structures, and luminescent properties.

2. Experimental

2.1. *Materials and general methods*

All reagents were of analytical grade and used without purification. 1,3,5-Tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene (ttmb) was synthesized similar to the synthesis of 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene [37]. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240 C analyser. Infrared (IR) spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectrophotometer from 4000 cm^{-1} to 400 cm^{-1}. Luminescence measurements were carried out in the solid state at room temperature with a Perkin-Elmer LS50B spectrofluorimeter.

2.2. *Synthesis of $\{[Cd(ttmb)_2](SO_4)(H_2O)_{16}\}_n$ (1)*

An aqueous solution (8 mL) of $CdSO_4 \cdot 2H_2O$ (0.5 mmol) was added to a tube, then a CH_3OH solution (10 mL) of ttmb (1.0 mmol) was slowly added to the tube above the aqueous solution. Colorless crystals of **1** were obtained after 2 weeks. Anal. Calcd for $C_{36}H_{74}CdN_{18}O_{20}S$ (**1**): C, 35.34; H, 6.10; N, 20.61. Found (%): C, 35.23; H, 6.01;

N, 20.52. IR data (cm^{-1}): 3437 m, 2939 m, 1550 s, 1419 s, 1284 s, 1215 m, 1134 s, 1010 m, 983 m, 790 m, 675 m.

2.3. *$\{[Cd(ttmb)_2(H_2O)_2](NO_3)_2(H_2O)_6\}_n$ (2)*

An aqueous solution (5 mL) of $Cd(NO_3)_2 \cdot 6H_2O$ (0.5 mmol) was added to a tube, then a CH_3OH (10 mL) solution of ttmb (1.0 mmol) was slowly added to the tube above the aqueous solution. Colorless crystals of **2** were obtained after 2 weeks. Anal. Calcd for $C_{36}H_{58}CdN_{20}O_{14}$ (**2**): C, 39.04; H, 5.28; N, 25.30. Found (%): C, 38.96; H, 5.23; N, 25.26. IR data (cm^{-1}): 3433 m, 3116 m, 2378 s, 1651 m, 1519 s, 1388 s, 1280 m, 1211 m, 1134 s, 1010 m, 894 m, 678 m.

2.4. *$\{[Cd(ttmb)_2(H_2O)_2](ClO_4)_2(H_2O)_7\}_n$ (3)*

An aqueous solution (5 mL) of $Cd(ClO_4)_2 \cdot 6H_2O$ (0.5 mmol) was added to a tube, then a CH_3OH solution (10 mL) of ttmb (1.0 mmol) was slowly added to the tube above the aqueous solution. Colorless crystals of **3** were obtained after 3 weeks. Anal. Calcd for $C_{36}H_{60}CdCl_2N_{18}O_{17}$ (**3**): C, 36.02; H, 5.04; N, 21.01. Found (%): C, 35.88; H, 5.01; N, 20.94. IR data (cm^{-1}): 3386 m, 1516 m, 1381w, 1280 m, 1211w, 1122vs, 1010 m, 972w, 675 m.

2.5. *Single-crystal X-ray crystallography*

Suitable single crystals of **1**, **2**, and **3** were carefully selected under an optical microscope and glued to thin glass fibers. The diffraction data were collected on a Rigaku Mercury CCD diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Intensities were collected by the ω scan technique. The structures were solved by direct methods and refined with full-matrix least-squares (SHELXTL-97) [38]. The positions of hydrogen atoms of ttmb were determined with theoretical calculation; hydrogen atoms of coordination water molecules were located from successive Fourier syntheses. No hydrogen atoms of disordered lattice water molecules were obtained. The parameters of the crystal data collection and refinement of **1**, **2**, and **3** are given in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Description of the crystal structures*

Complex **1** shows a (3,6)-connected $CdCl_2$-type 2-D network. The asymmetric unit contains one half-occupied Cd(II), which lies on an inversion center, one ttmb, one half-occupied SO_4^{2-}, and eight water molecules. Each Cd(II) displays a distorted octahedral coordination geometry, coordinated by six nitrogen atoms from six ttmb ligands (figure 1a). Each Cd(II) is 6-connected. Each ttmb shows *cis, cis, cis*-conformation in a tri-monodentate coordination, 3-connected. Each ttmb binds three Cd(II)'s forming a (3,6)-connected $CdCl_2$-type 2-D network (figure 1b and c). The Cd···Cd distances

Table 1. Crystallographic data for **1**, **2**, and **3**.

	1	**2**	**3**
Empirical formula	$C_{36}H_{74}CdN_{18}O_{20}S$	$C_{36}H_{58}CdN_{20}O_{14}$	$C_{36}H_{60}CdCl_2N_{18}O_{17}$
Formula weight	1223.59	1107.42	1200.32
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$C2/c$	$P2_1/n$	$C2/c$
Temperature (K)	223(2)	223(2)	223(2)
Unit cell dimensions (Å, °)			
a	12.050(3)	8.4563(11)	25.166(3)
b	20.441(5)	16.602(2)	9.1469(9)
c	24.029(6)	17.648(3)	22.458(2)
β	97.796(4)	100.205(2)	91.318(3)
Volume (Å^3), Z	5864(3), 4	2438.4(6), 2	5168.2(9), 4
Calculated density (g cm^{-3})	1.386	1.508	1.543
Absorption coefficient (mm^{-1})	0.489	0.531	0.611
F(000)	2560	1148	2480
Reflections collected	15,751	13,485	12,744
Independent reflections	6590 [R(int) = 0.0404]	5542 [R(int) = 0.0238]	4723 [R(int) = 0.0825]
Parameters	363	347	349
Goodness-of-fit on F^2	1.090	1.018	1.030
R_1 [$I > 2\sigma(I)$]	0.0579	0.0379	0.0892
wR_2 (all data)	0.1822	0.1081	0.2474

Table 2. Selected bond lengths (Å) and angles (°) for **1**, **2**, and **3**.

1			
Cd(1)–N(3)	2.346(3)	Cd(1)–N(6D)	2.349(3)
Cd(1)–N(9A)	2.341(3)		
N(3C)–Cd(1)–N(3)	180.00(9)	N(3)–Cd(1)–N(6D)	91.93(12)
N(3)–Cd(1)–N(6E)	88.07(12)	N(9A)–Cd(1)–N(3)	88.31(13)
N(9B)–Cd(1)–N(3)	91.69(13)	N(9A)–Cd(1)–N(6D)	87.33(13)
N(6D)–Cd(1)–N(6E)	180.0	N(9A)–Cd(1)–N(9B)	180.000(1)
2			
Cd(1)–N(3)	2.308(2)	Cd(1)–N(6B)	2.3239(18)
Cd(1)–O(1)	2.316(2)		
N(3A)–Cd(1)–N(3)	180.00(9)	N(3)–Cd(1)–N(6B)	88.46(7)
N(3)–Cd(1)–O(1)	91.94(9)	N(6B)–Cd(1)–N(6C)	180.0
O(1)–Cd(1)–N(6B)	86.26(8)	O(1A)–Cd(1)–O(1)	180.00(11)
3			
Cd(1)–N(3)	2.298(6)	Cd(1)–N(6B)	2.329(6)
Cd(1)–O(1)	2.322(9)	Cd(1)–O(2)	2.362(10)
N(3)–Cd(1)–N(3A)	174.9(3)	N(3)–Cd(1)–N(6B)	87.1(2)
N(3)–Cd(1)–N(6C)	93.1(2)	N(3)–Cd(1)–O(1)	87.43(16)
N(3)–Cd(1)–O(2)	92.57(16)	N(3A)–Cd(1)–N(6B)	93.1(2)
N(3A)–Cd(1)–O(1)	87.43(16)	N(3A)–Cd(1)–O(2)	92.57(16)
N(6B)–Cd(1)–N(6C)	175.5(3)	O(1)–Cd(1)–N(6B)	92.26(17)
N(6B)–Cd(1)–O(2)	87.74(17)	O(1)–Cd(1)–O(2)	180.000(3)

Symmetry transformations used to generate equivalent atoms: **1**: A: $-x+3/2$, $-y+1/2$, $-z+1$; B: $x-1/2$, $y+1/2$, z; C: $-x+1$, $-y+1$, $-z+1$; D: $x+1/2$, $y+1/2$, z; E: $-x+1/2$, $-y+1/2$, $-z+1$; **2**: A: $-x$, $-y+1$, $-z$; B: $-x+1/2$, $y+1/2$, $-z+1/2$; C: $x-1/2$, $-y+1/2$, $z-1/2$; **3**: A: $-x+1$, y, $-z+1/2$; B: x, $-y+2$, $z-1/2$; C: $-x+1$, $-y+2$, $-z+1$.

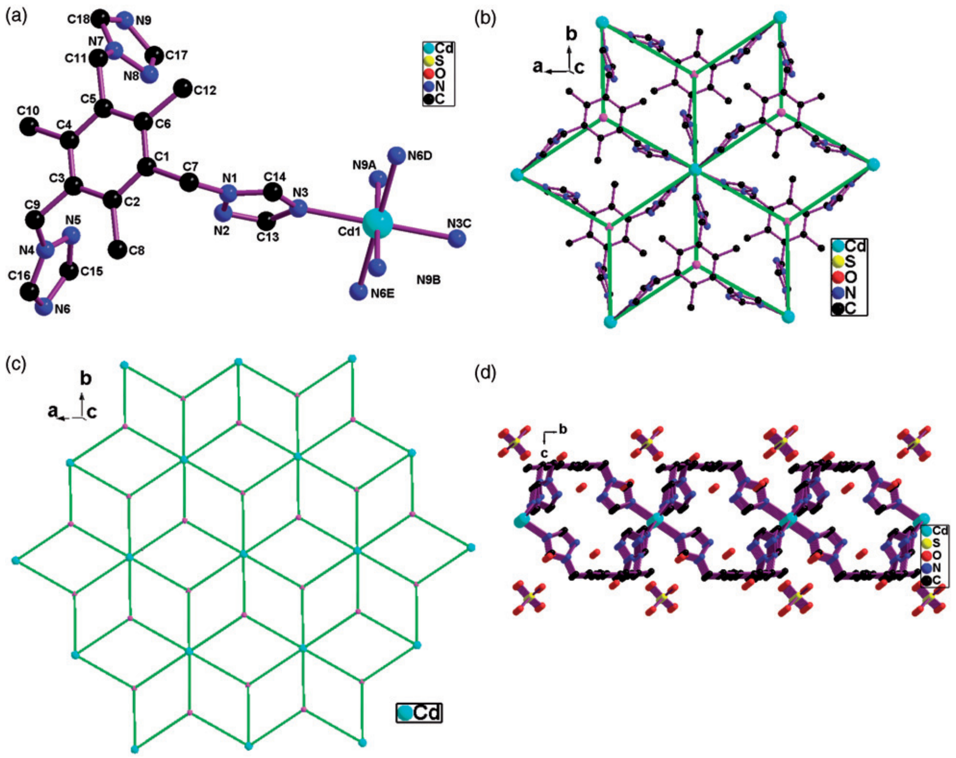

Figure 1. (a) The coordination environment of Cd(II) of **1**. (b) The 2-D network of **1**. (c) Schematic of a (3,6)-connected 2-D network of **1**. The representation of the 3-connected ttmb ligand is simplified by showing the center of benzene ring (pink dummy atom: see the online color version). (d) Viewing the 2-D network of **1** along the *a* direction.

through ttmb are 11.8642(23) and 12.050(3) Å, respectively. The Schläfla symbol for the 2-D network is $(4^3)_2(4^6.6^6.8^3)$. Among the known (3,6)-connected networks in MOFs, most structures are of the rutile network [39–41]. Examples of CdCl$_2$ topology are quite rare for metal–organic coordination polymers (MOFs), although it has been reported for some cyano-bridged MOFs [42, 43]. 1-D channels in **1** are formed along the *a* directions with SO$_4^{2-}$ and water molecules occupying the channels (figure 1d).

Complex **2** exhibits a 2-D (4,4) network. The coordination geometry of Cd(II) is distorted octahedral, formed by four triazole nitrogen atoms from four ttmb in the equatorial plane, and two water molecules in axial positions (figure 2a). All ttmb ligands show *cis, trans, trans*-conformation and two-monodentate coordination. One triazole of ttmb ligand does not coordinate to Cd(II). Each Cd(II) is square four-connected and extends to an undulated 2-D (4,4) network (figure 2b). The Cd ··· Cd separation *via* ttmb is 12.3056(12) Å. The 2-D networks stack parallel along the *a* direction with inter-layer distance equal to the *a*-axis translation (figure 2c). There are hydrogen bonding interactions between coordination water, disordered lattice water, the nitrate, and ttmb (table 3). The crystal is stable with these hydrogen bonding interactions.

Complex **3** shows a 1-D double chain. Each Cd(II) is coordinated by four triazole nitrogen atoms from four ttmb and two water molecules in a distorted octahedral geometry (figure 3a). Each ttmb has *cis, trans, trans*-conformation. The Cd ··· Cd

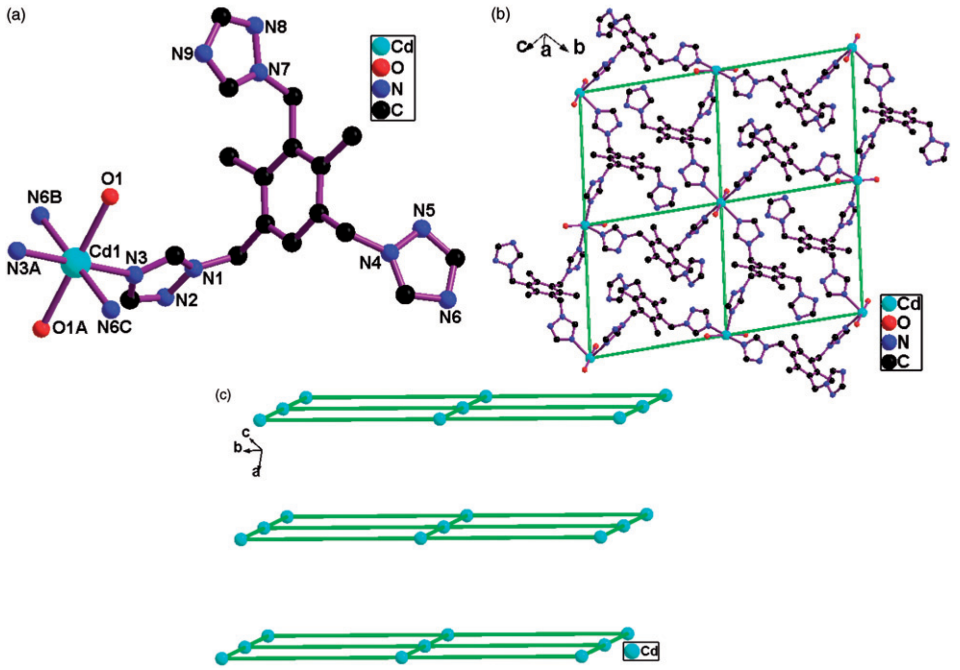

Figure 2. (a) The coordination environment for Cd(II) of **2**. (b) The 2-D (4,4) network of **2**. (c) Schematic diagram of the parallel stack of the 2-D (4,4) networks of **2**.

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

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
2				
O(1)–H(1 W)···O(2)	0.83(4)	1.94(4)	2.760(3)	172(3)
O(1)–H(2 W)···O(7)	0.82(6)	2.02(6)	2.709(4)	142(6)
O(5)–H(3 W)···O(3)^{a}	0.83(4)	2.02(4)	2.828(3)	168(4)
O(5)–H(4 W)···N(9)	0.77(4)	2.11(4)	2.877(4)	171(4)
3				
O(1)–H(1 W)···O(7)	0.85	1.99	2.767(9)	151.3(9)
O(1)–H(2 W)···O(10)^{b}	0.85	2.30	2.822(14)	120.5(9)

Symmetry transformations used to generate equivalent atoms: **2**: ^{a} $-x-1/2, y+1/2, -z+1/2$; **3**: ^{b} $-x+1, y+1, -z+1/2$.

distance *via* ttmb is 11.3019(10) Å. Two ttmb ligands are wrapped around each other and held by Cd(II) to form a 1-D double chain (figure 3b). The 1-D chains stack parallel along the *b* direction (figure 3c); hydrogen bond interactions (table 3) maintain the stability of **3**.

3.2. *Luminescence*

Solid state luminescence spectra of **1**–**3** at room temperature are compared (figure 4). Free ttmb shows weak luminescence at 400 nm (excitation at 340 nm) [36]. **1** exhibits a

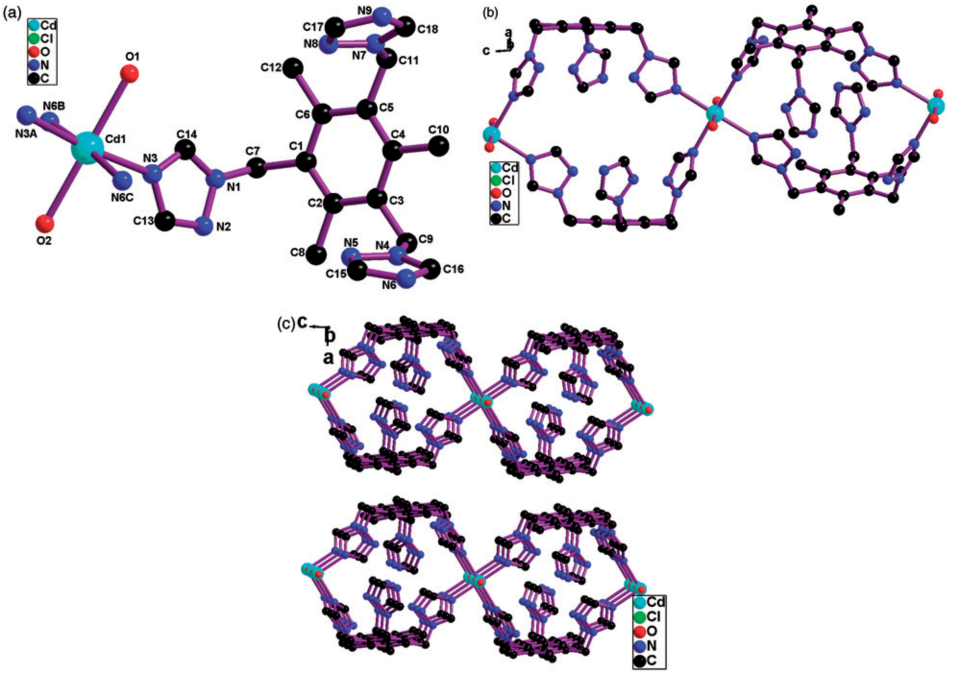

Figure 3. (a) The coordination environment of Cd(II) of **3**. (b) The 1-D chain of **3**. (c) Packing of **3** along the *b* direction.

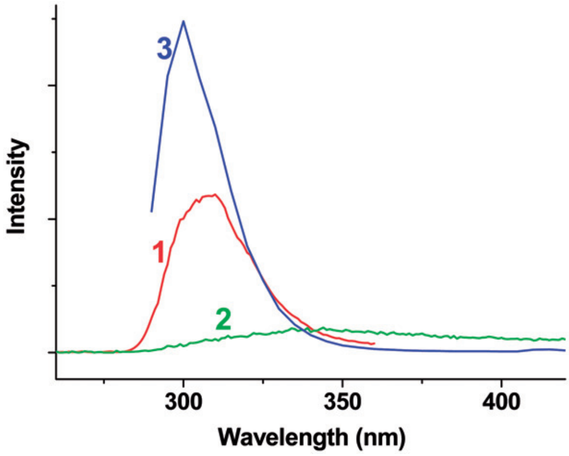

Figure 4. Solid state luminescence spectra of **1**, **2**, and **3**.

luminescence maxima at 306 nm and **2** shows a weak emission around 339 nm upon excitation at 238 nm. **3** presents a strong emission at 298 nm upon excitation at 280 nm. The weak emission of **2** can be tentatively contributed to ttmb. The emissions of **1** and **3** can be tentatively attributed to emission of metal-to-ligand charge-transfer [23–25, 29, 30, 32, 36].

4. Conclusions

Self-assembly of ttmb and Cd(II) salts gave three cadmium MOFs $\{[Cd(ttmb)_2](SO_4)(H_2O)_{16}\}_n$ (**1**), $\{[Cd(ttmb)_2(H_2O)_2](NO_3)_2(H_2O)_6\}_n$ (**2**), and $\{[Cd(ttmb)_2(H_2O)_2](ClO_4)_2(H_2O)_7\}_n$ (**3**). **1** has a (3,6)-connected 2-D network with Schläfla symbol of $(4^3)_2(4^6.6^6.8^3)$. However, **2** and **3** show 2-D (4,4) network and 1-D double chain, respectively. Each ttmb in **1** is *cis, cis, cis*-conformation, however, ttmb in **2** and **3** are *cis, trans, trans*-conformation. The nature of counteranion has influence on self-assembly of the flexible ligand with cadmium, constructing architectures with different structures. Complexes **1**, **2**, and **3** exhibit luminescence at 306, 339, and 298 nm, respectively, in the solid state at room temperature.

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre with CCDC reference numbers CCDC-837988, 837989, and 837990.

Acknowledgments

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