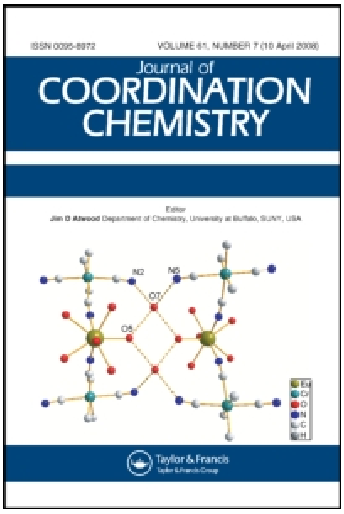

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Two non-interpenetrating 3D coordination networks with (3, 4)-connected topologies

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Two non-interpenetrating 3D coordination networks with (3, 4)-connected topologies

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Two new coordination polymers, namely $[Zn_3(1,3,5\text{-}BTC)_2(L^1)_2(H_2O)_2]\cdot 2H_2O$ (**1**) and $[Cd_3(1,2,3\text{-}BTC)_2(L^2)_3]\cdot H_2O$ (**2**) (where $L^1 =$ 1,2-*bis*(imidazol-1-ylmethyl)benzene, $L^2 =$ 1,1′-(1,4-butanediyl)*bis*(imidazole), 1,3,5-H_3BTC = 1,3,5-benzenetricarboxylic acid and 1,2,3-H_3BTC = 1,2,3-benzenetricarboxylic acid), were synthesized in hydrothermal conditions. In **1**, each 1,3,5-BTC anion coordinates to three Zn cations, and the framework of **1** can be simplified as $(6\cdot 8\cdot 10)_2(6^2\cdot 8\cdot 10^3)(8^2\cdot 10)(6^2\cdot 10)$ topology. In **2**, 1,2,3-BTC anions coordinate to three cadmiums, and the whole structure displays a $(6^2\cdot 8^4)_2(6^4\cdot 8\cdot 10)(6^2\cdot 8)_2$ network containing three different types of nodes. The luminescent properties for **1** and **2** are discussed.

Keywords: Coordination polymers; Carboxylate; *Bis*(imidazole); Topology

1. Introduction

Extended framework solids are of current importance because they may offer new materials with a range of potentially useful properties, as well as intriguing molecular topological nets and entanglements [1]. Much effort has been made towards the connections of suitable, predetermined building blocks into networks in order to obtain the desired materials. As an important family of multidentate N-donor ligands, *bis*(imidazole) ligands have been extensively employed in the preparation of metal-organic compounds in recent years [2]. In this study, We use benzenetricarboxylic acids (1,3,5-H_3BTC and 1,2,3-H_3BTC) as mixed ligands with *bis*(imidazole) ligands (as shown in chart 1), and isolated two (3, 4)-connected networks, namely $[Zn_3(1,3,5\text{-}BTC)_2(L^1)_2(H_2O)_2]\cdot 2H_2O$ (**1**) and $[Cd_3(1,2,3\text{-}BTC)_2(L^2)_3]\cdot H_2O$ (**2**). The crystal structures of these compounds and topological analyses will be represented and discussed.

2. Experimental

2.1. *Materials*

All reagents and solvents for syntheses were purchased from commercial sources and used as received. The ligands L^1 and L^2 were synthesized according to the literature [3].

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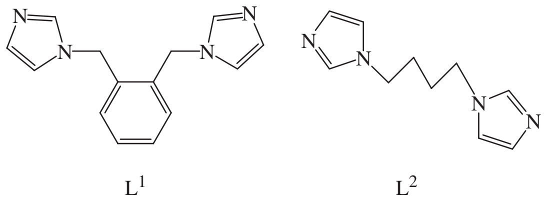

Chart 1. Structures of L^1 and L^2.

2.2. *Synthesis of the compounds*

2.2.1. Synthesis of $[Zn_3(1,3,5\text{-BTC})_2(L^1)_2(H_2O)_2]\cdot 2H_2O$ (1). A mixture of $Zn(OH)_2$ (0.045 g, 0.45 mmol), 1,3,5-H_3BTC (0.095 g, 0.45 mmol), L^1 (0.107 g, 0.45 mmol), and water (7 mL) was stirred for 10 min in air. The mixture was heated at 160°C for 4 days and then gradually cooled to room temperature at a rate of 10 °C h^{-1}. Colorless crystals of **1** were obtained. Yield: 28% based on $Zn(OH)_2$. Elemental analyses calcd for $C_{46}H_{42}Zn_3N_8O_{16}$ (1158.99): C, 47.67; H, 3.65; N, 9.67. Found: C, 47.81; H, 3.70; N, 9.60. IR (cm^{-1}): 3455 cm^{-1} (s), 3130 (s), 2527 (w), 2363 (w), 1701 (s), 1633 (s), 1575 (s), 1447 (s), 1354 (s), 1238 (m), 1098 (s), 1029 (w), 947 (m), 854 (w), 738 (s), 657 (m).

2.2.2. Synthesis of $[Cd_3(1,2,3\text{-BTC})_2(L^2)_3]\cdot H_2O$ (2). A mixture of $Cd(OH)_2$ (0.066 g, 0.45 mmol), 1,2,3-H_3BTC (0.095 g, 0.45 mmol), and L^2 (0.086 g, 0.45 mmol) in water (7 mL) was heated at 160°C for 3 days and then slowly cooled to room temperature at 10 °C h^{-1}. Colorless crystals of **2** were obtained in 51% yield based on $Cd(OH)_2$. Anal. Calcd for $C_{48}H_{50}Cd_3N_{12}O_{13}$ (1340.20): C, 43.02; H, 3.76; N, 12.54. Found: C, 43.12; H, 3.61; N, 12.44. IR (cm^{-2}): 3446 (s), 3117 (s), 2936 (m), 1554 (s), 1451 (s), 1372 (s), 1236 (m), 1100 (s), 942 (s), 862 (s), 772 (s), 715 (s), 659 (s).

2.3. *Physical measurements and X-ray crystallography*

The C, H, and N elemental analyses were conducted on a Perkin-Elmer 240C elemental analyzer. FT-IR spectra were recorded from KBr pellets in the range 4000–400 cm^{-2} on a Mattson Alpha-Centauri spectrometer. TGA was performed on a Perkin–Elmer TG-7 analyzer heated from 25 to 800°C under nitrogen. The powder XRD data were collected on a Siemens D5005 diffractometer with Cu-Kα radiation ($\lambda = 1.5418$ Å) over the 2θ range of 5–35° at room temperature.

Single-crystal X-ray diffraction data for **1** and **2** were recorded on a Bruker Apex CCD diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at 293 K. Absorption corrections were applied using multiscan technique. The structures were solved by Direct Methods of SHELXS-97 [4] and refined by full-matrix least-squares techniques using SHELXL-97 [5]. Non-hydrogen atoms were refined with anisotropic temperature parameters. The hydrogen atoms of ligands were refined as rigid groups. The disordered 1,3,5-BTC anion of **1** and L^2 in **2** were refined using isotropic O, C and N atoms split over two sites, with a total occupancy of 1. The lattice water H atoms in **2** could not be positioned reliably. Other H atoms of water molecules in idealized positions were located from difference Fourier maps.

Table 1. Crystal data and structure refinements for **1** and **2**.

	1	**2**
Formula	$C_{46}H_{42}Zn_3N_8O_{16}$	$C_{48}H_{50}Cd_3N_{12}O_{13}$
Formula weight	1158.99	1340.20
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
Unit cell dimensions (Å,°)		
a	10.812(5)	30.531(9)
b	14.844(5)	10.799(3)
c	16.829(5)	16.267(5)
α	67.13(1)	90
β	75.24(2)	107.312(10)
γ	76.91(2)	90
V(Å^3)	2381.9(15)	5120(2)
Z	2	4
D_{Calcd} (Mg m^{-3})	1.616	1.739
Absorption coefficient (mm^{-1})	1.580	1.311
F(000)	1184	2680
θ range (°)	3.01–27.47	3.14–27.48
Reflns collected/unique	23377/10731	23842/5770
Goodness-of-fit on F^3	1.042	1.015
R_1[a], wR_2[b] [$I > 2\sigma(I)$]	0.0349/0.1042	0.0733/0.1142
Max, min peaks (eÅ^{-3})	0.582/−0.373	0.773/−0.939

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $wR_2 = |\Sigma w(|F_o|^3 - |F_c|^3)|/\Sigma|w(F_o^3)^3|^{2/3}$.

The detailed crystallographic data and structure refinement parameters for **1** and **2** are summarized in table 1.

3. Results and discussion

3.1. *Crystal structure*

Selected geometric parameters for **1** and **2** are given in table 2 and hydrogen bond data are listed in table 3.

3.1.1. The structure of [Zn$_3$(1,3,5-BTC)$_2$(L^1)$_2$(H$_2$O)$_2$]·2H$_2$O (1). Part of the structure for **1** shown in figure 1(a), contains three kinds of unique Zn ions, two unique 1,3,5-BTC anions and two kinds of L^1 ligands. Zn1 is four-coordinate by two oxygens (Zn–O 2.043(2)–2.099(2) Å) from two 1,3,5-BTC anions, one water (Zn–O 2.056(2) Å) and one nitrogen (Zn–N 2.021(2) Å) from L^1; another two oxygen atoms of 1,3,5-BTC have weak interactions with Zn1 (Zn–O 2.301(2) and 2.344(2) Å) [6]. Zn2 is four-coordinate by two oxygens (Zn–O 1.980(2)–2.013(2) Å) from two 1,3,5-BTC anions, one water (Zn–O 2.066(2) Å) and one nitrogen (Zn–N 1.989(2) Å) from L^1; one oxygen of a 1,3,5-BTC has a weak interaction with Zn2 (Zn–O 2.436(2) Å). Zn3 is four-coordinate by two oxygens (Zn–O 1.933(2)–1.966(2) Å) from different 1,3,5-BTC anions and two nitrogens (Zn–N 1.999(2)–2.007(2) Å) from two L^1 ligands.

Two unique 1,3,5-BTC anions display different coordination modes [figure 2(b)], and each coordinates to three zincs. Zn$^{\mathrm{II}}$ centers are bridged by 1,3,5-BTC anions to form a 1D loop-like chain [figure 1(b)]. L^1 has two kinds of conformations, one L^1 shows

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

Compound **1**			
Zn(1)–N(4)	2.021(2)	Zn(1)–O(5)^{i}	2.043(2)
Zn(1)–O(2W)	2.056(2)	Zn(1)–O(12)^{ii}	2.099(2)
Zn(1)$\cdots$O(11)^{ii}	2.301(2)	Zn(1)$\cdots$O(6)^{i}	2.344(2)
Zn(2)–O(9)^{iii}	1.980(2)	Zn(2)–N(5)	1.989(2)
Zn(2)–O(2)	2.013(2)	Zn(2)–O(1W)	2.066(2)
Zn(2)$\cdots$O(1)	2.436(2)	Zn(3)–O(4)	1.933(2)
Zn(3)–O(7)	1.966(2)	Zn(3)–N(8)^{iv}	1.999(2)
Zn(3)–N(1)	2.007(2)		
N(4)–Zn(1)–O(5)^{i}	104.28(9)	N(4)–Zn(1)–O(2W)	101.33(9)
O(5)^{i}–Zn(1)–O(2W)	93.42(8)	N(4)–Zn(1)–O(12)^{ii}	147.17(9)
O(5)^{i}–Zn(1)–O(12)^{ii}	102.34(8)	O(2W)–Zn(1)–O(12)^{ii}	95.89(9)
N(4)–Zn(1)–O(11)^{ii}	92.69(8)	O(5)^{i}–Zn(1)–O(11)^{ii}	161.98(8)
O(2W)–Zn(1)–O(11)^{ii}	89.05(8)	O(12)^{ii}–Zn(1)–O(11)^{ii}	59.64(7)
N(4)–Zn(1)–O(6)^{i}	88.67(9)	O(5)^{i}–Zn(1)–O(6)^{i}	59.34(7)
O(2W)–Zn(1)–O(6)^{i}	152.67(8)	O(12)^{ii}–Zn(1)–O(6)^{i}	88.62(8)
O(11)^{ii}–Zn(1)–O(6)^{i}	116.09(8)	O(9)^{iii}–Zn(2)–N(5)	109.86(9)
O(9)^{iii}–Zn(2)–O(2)	102.24(8)	N(5)–Zn(2)–O(2)	137.46(9)
O(9)^{iii}–Zn(2)–O(1W)	95.91(8)	N(5)–Zn(2)–O(1W)	101.43(9)
O(2)–Zn(2)–O(1W)	102.25(9)	O(9)^{iii}–Zn(2)–O(1)	159.46(8)
N(5)–Zn(2)–O(1)	89.91(8)	O(2)–Zn(2)–O(1)	57.73(7)
O(1W)–Zn(2)–O(1)	85.16(8)	O(4)–Zn(3)–O(7)	109.60(10)
O(4)–Zn(3)–N(8)^{iv}	98.88(10)	O(7)–Zn(3)–N(8)^{iv}	120.50(10)
O(4)–Zn(3)–N(1)	122.00(9)	O(7)–Zn(3)–N(1)	97.57(10)
N(8)^{iv}–Zn(3)–N(1)	109.91(1)		
Compound **2**			
Cd(1)–N(4)^{i}	2.231(7)	Cd(1)–N(1)	2.243(7)
Cd(1)–O(6)	2.304(6)	Cd(1)–O(3)^{ii}	2.363(6)
Cd(1)–O(4)^{ii}	2.400(6)	Cd(1)–O(5)	2.467(6)
Cd(2)–N(5)	2.245(7)	Cd(2)–O(1)	2.282(7)
Cd(2)–O(2)	2.579(7)		
N(4)^{i}–Cd(1)–N(1)	102.9(3)	N(4)^{i}–Cd(1)–O(6)	130.2(3)
N(1)–Cd(1)–O(6)	103.9(3)	N(4)^{i}–Cd(1)–O(3)^{ii}	117.1(2)
N(1)–Cd(1)–O(3)^{ii}	86.3(2)	O(6)–Cd(1)–O(3)^{ii}	105.8(2)
N(4)^{i}–Cd(1)–O(4)^{ii}	89.4(2)	N(1)–Cd(1)–O(4)^{ii}	140.6(2)
O(6)–Cd(1)–O(4)^{ii}	95.6(2)	O(3)^{ii}–Cd(1)–O(4)^{ii}	55.2(2)
N(4)^{i}–Cd(1)–O(5)	84.4(2)	N(1)–Cd(1)–O(5)	90.0(2)
O(6)–Cd(1)–O(5)	54.6(2)	O(3)^{ii}–Cd(1)–O(5)	158.4(2)
O(4)^{ii}–Cd(1)–O(5)	128.8(2)	N(5)–Cd(2)–N(5)^{iii}	100.9(4)
N(5)–Cd(2)–O(1)	96.0(3)	N(5)^{iii}–Cd(2)–O(1)	145.8(3)
O(1)^{iii}–Cd(2)–O(1)	85.9(4)	N(5)–Cd(2)–O(2)	120.7(3)
O(1)^{iii}–Cd(2)–O(2)	87.4(3)	O(1)–Cd(2)–O(2)	52.7(2)
N(5)–Cd(2)–O(2)^{iii}	93.2(3)	O(2)–Cd(2)–O(2)^{iii}	127.1(3)

Symmetry code for **1**: $^{i}-x+1,\ -y,\ -z$; $^{ii}x+1,\ y, z+1$; $^{iii}-x,\ -y$ - $1,\ -z$; $^{iv}-x,\ -y,\ -z$.
Symmetry codes for **2**: $^{i}x,\ -y+1,\ z+1/2$; $^{ii}-x+1/2,\ y-1/2,\ -z+1/2$; $^{iii}-x+1,\ y,\ -z+1/2$.

a *trans* conformation [7], with Zn$\cdots$Zn distance of 13.14 Å. The other L^1 adopts a *cis* conformation [7b], with Zn$\cdots$Zn distance of 6.57 Å. If *trans* or *cis* L^1 ligands are neglected, two kinds of 2D networks result [figures 1(c), 1(d)]. These two kinds of 2D networks cross-link, resulting in a 3D network. If 1,3,5-BTC anions are considered as three-connected nodes, the structure of **1** can be symbolized as a $(6\cdot8\cdot10)_2(6^2\cdot8\cdot10^3)(8^2\cdot10)(6^2\cdot10)$ topology [figure 1(e)] [8].

$[Zn_3(1,3,5\text{-BTC})_2(L^1)_2(H_2O)_4]\cdot3H_2O$ (**3**) shows a 2D polymeric structure of 2-fold interpenetrating (6, 3) networks [3]. Although it is hard to propose definitive reasons as to why **1** and **3** adopt different configurations, pH values may exert an important influence on the architecture [9].

Table 3. Hydrogen-bonding geometry for **1** (in Å and °).

D–HA	d(D–H)	d(H···A)	d(D···A)	∠(D–H···A)
1				
O(1W)–H(1C)···O(1)[i]	0.83(2)	1.94(2)	2.763(3)	176(4)
O(4W)–H(4C)···O(10)[ii]	0.87(2)	1.93(3)	2.791(3)	170(4)
O(2W)–H(2C)···O(11)[iii]	0.87(2)	1.87(3)	2.717(3)	163(4)
O(3W)–H(3D)···O(6)[iv]	0.86(3)	1.90(3)	2.749(3)	171(5)
O(4W)–H(4D)···O(8)	0.86(2)	1.94(2)	2.796(7)	173(4)
O(3W)–H(3C)···O(3)	0.87(3)	1.89(3)	2.723(3)	158(4)

Symmetry code for **1**: [i]$-x-1, -y, -z$; [ii]$-x+1, -y-1, -z-1$; [iii]$-x+1, -y-1, -z$; [iv]$-x, -y, -z$.

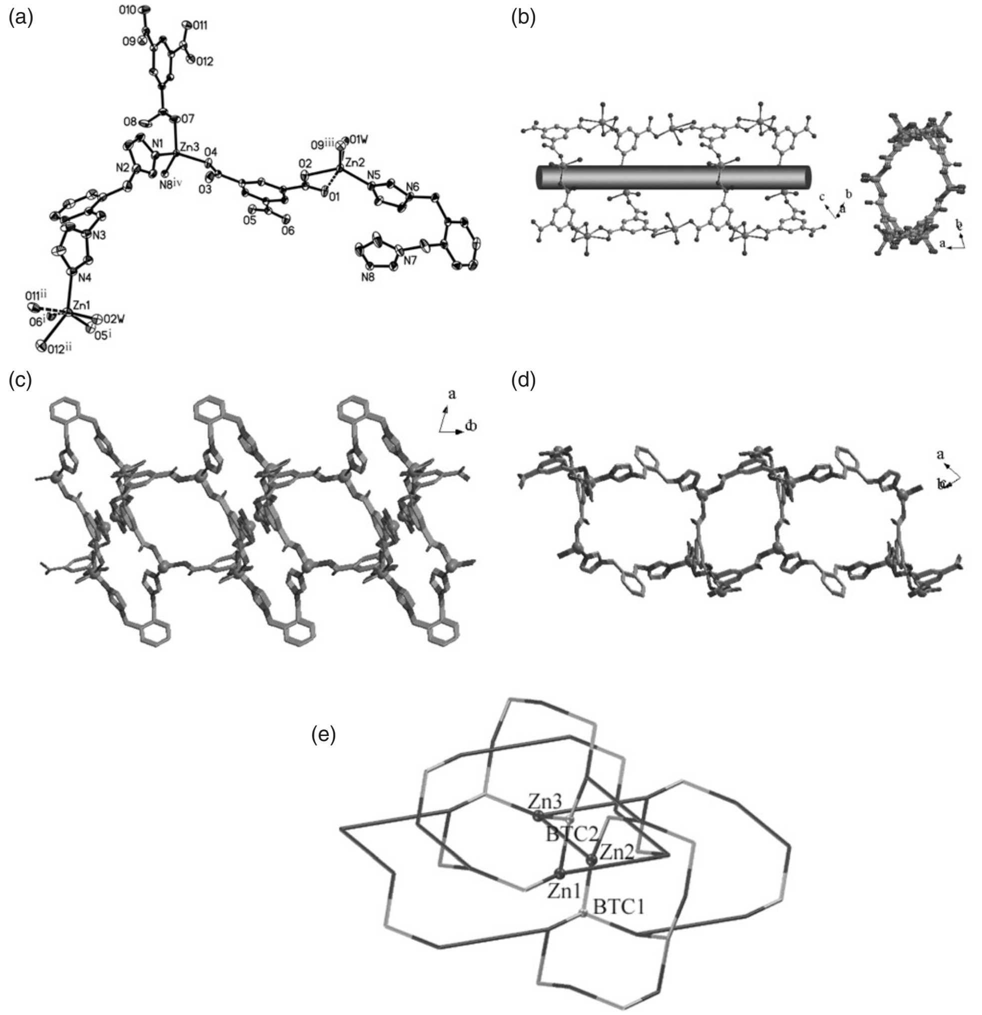

Figure 1. (a) ORTEP diagram showing the coordination environments for Zn atoms in **1**; (b) Side and front views of the loop-like chain constructed with Zn^{II} and 1,3,5-BTC anions; (c) The extended 2D structure of **1** when *trans* L^1 ligands are neglected; (d) The extended 2D structure of **1** when *cis* L^1 ligands are neglected; (e) Schematic diagram (OLEX) showing the $(6\cdot8\cdot10)_2(6^2\cdot8\cdot10^3)(8^2\cdot10)(6^2\cdot10)$ network. Symmetry code: (i) $-x+1, -y, -z$; (ii) $x+1, y, z+1$; (iii) $-x, -y-1, -z$; (iv) $-x, -y, -z$.

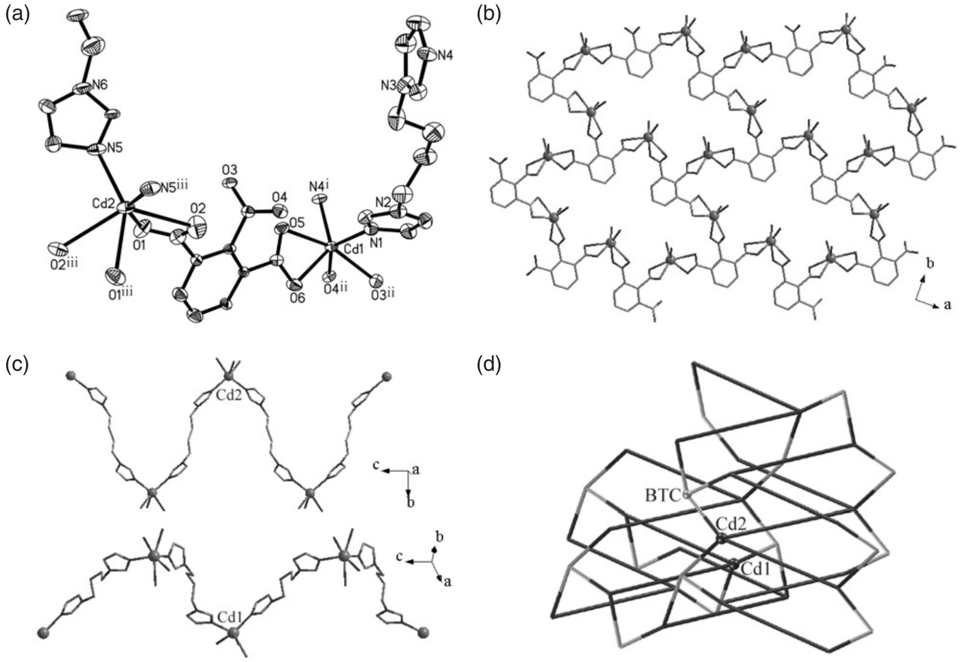

Figure 2. (a) ORTEP diagram showing the coordination environment for Cd atoms in **2**; (b) View of the 2D layer made of Cd^{II} atoms and 1,2,3-BTC anions; (c) The zig-zag chains in **2** linked by the two formed L^2 ligands, respectively; (d) Schematic diagram (OLEX) showing the $(6^4 \cdot 8^2)_2(6^4 \cdot 8 \cdot 10)(6^2 \cdot 8)_2$ network. Symmetry code: (i) $x, -y+1, z+1/2$; (ii) $-x+1/2, y-1/2, -z+1/2$; (iii) $-x+1, y, -z+1/2$.

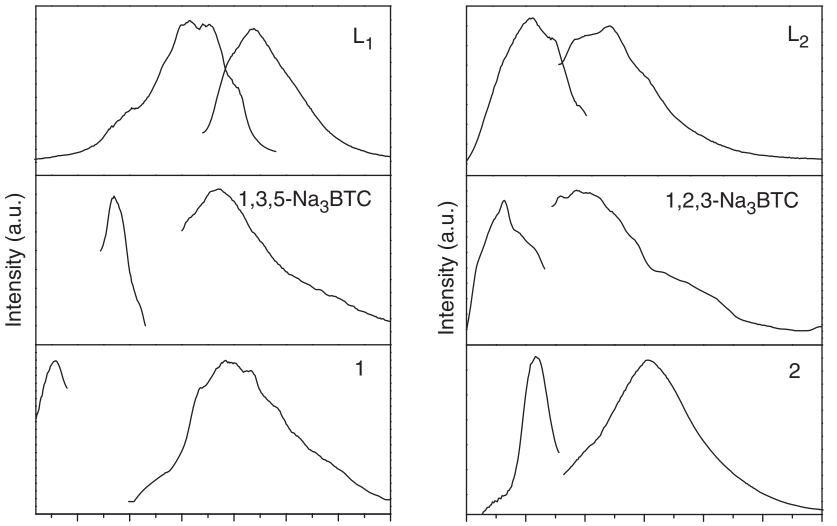

Figure 3. Solid-state photoluminescent spectra of **1** and **2** and free ligands L^1, L^2, 1,3,5-Na_3BTC and 1,2,3-Na_3BTC at room temperature.

Both **1** and **3** contain coordinated and guest water molecules. The question therefore arises as to whether they give the same product after removal of water. TGA and X-ray powder diffraction (XRPD) suggest that the compounds give different products after removal of water.

3.1.2. The structure of $[Cd_3(1,2,3\text{-}BTC)_2(L^2)_3]\cdot H_2O$ (2).

As shown in figure 2(a), the structure of **2** contains two unique Cd atoms. Cd1 is six-coordinate by four carboxylate oxygens (Cd–O 2.304(6)–2.467(6) Å) from two 1,2,3-BTC anions and two nitrogen atoms (Cd–N 2.231(7)–2.243(7) Å) from two L^2 ligands. Cd2 lies at an inversion center and is six-coordinate by four oxygens (Cd–O 2.282(7)–2.579(7) Å) from two 1,2,3-BTC anions and two nitrogens (Co–N 2.245(7) Å) from two L^2 ligands. Each 1,2,3-BTC coordinates to three Cd atoms. The cadmium atoms are linked by 1,2,3-BTC anions to form a 2D layer [figure 2(b)].

Two unique L^2 ligands display different conformations [figure 2(c)]. One coordinates to two Cd2 cations, with methylene groups showing a zig-zag conformation [3]. The other coordinates to two Cd1 cations, with methylene groups adopting an Ω conformation [10]. The $[Cd_3(1,2,3\text{-}BTC)_2]$ layers [figure 2(b)] are connected by these two L^2 ligands to form a complicated 3D framework [figure 2(d)]. The structure of **2** can be symbolized as a (3, 4)-connected network with $(6^4\cdot 8^2)_2(6^4\cdot 8\cdot 10)(6^2\cdot 8)_2$ topology with 1,2,3-BTC anions considered as three-connected nodes [8].

The analysis of network topology is important in discussion of crystal structures. Wells has listed a large number of topologies in his classic monographs on networks, in which no fewer than 30 topologically nonuniform examples were listed [8]. Although a number of experiments have provided some possible (3, 4)-connected 3D networks [11], the topologies of **1** and **2** gave two new examples to these theoretical topologies.

3.2. *Luminescent properties*

The solid-state photoluminescent spectra of **1** and **2** and free L^1, L^2, 1,3,5-Na_3BTC and 1,2,3-Na_3BTC are depicted in figure 3. The emission and excitation peaks of 1,3,5-Na_3BTC, L^1, 1,2,3-Na_3BTC and L^2 are at 435, 468, 414 and 438 nm, respectively. The emission bands for these free ligands are attributable to $\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ transitions [12]. On complexation with Zn^{II} and Cd^{II}, strong luminescences were observable in the solid state at ambient temperature. The emission peaks of **1** and **2** are at 442 nm ($\lambda_{ex} = 277$ nm) and 454 nm ($\lambda_{ex} = 356$ nm), respectively. The photoluminescence of **1** and **2** may be assigned to intra-ligand fluorescent emission of BTC anions or $\pi^* \rightarrow \pi$ transitions of neutral ligands [12].

4. Conclusion

We have provided two intriguing polymeric architectures by combining *bis*(imidazole) and benzenetricarboxylic ligands with Zn^{2+} and Cd^{2+}. This study highlights an intriguing feature of coordinative network chemistry in producing rich architectures and topologies.

Supplementary data

X-ray crystallographic files in CIF format for **1** and **2** have been deposited at the Cambridge Crystallographic Data Centre with deposition number CCDC 669074 and 660611. Copies of the data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 IEZ, UK (Fax: +44-1223-336033; Email: deposit@ccdc.cam.ac.uk).

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