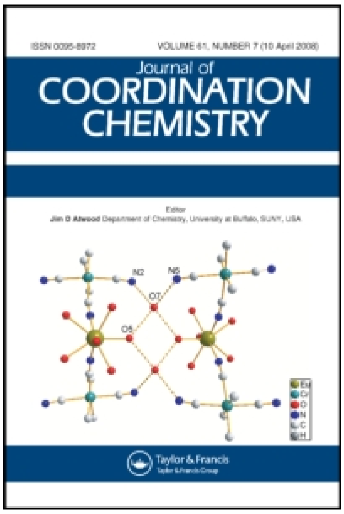

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Syntheses and structures of three zinc coordination polymers with 1-D zigzag chain, double chain, and triple chain

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Syntheses and structures of three zinc coordination polymers with 1-D zigzag chain, double chain, and triple chain

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Three new coordination polymers $[Zn(btp)(NCS)_2]_n$ (**1**), $\{[Zn(btp)_2(dca)_2]_n$ (**2**), and $\{[Zn(btp)_3](BF_4)_2\}_n$ (**3**) (btp = 1,3-*bis*(1,2,4-triazol-1-yl)propane, dca = dicyanamide) were synthesized and characterized. In **1**, **2**, and **3**, one-, double-, triple-btp ligands link two Zn(II) atoms and extend to form a 1-D zigzag chain for **1**, 1-D double chain for **2**, and 1-D triple chain for **3**. The conformations of the btp ligands in **1**, **2**, and **3** are analyzed. **1**, **2**, and **3** have emission maxima at approximately 405, 407, and 409 nm, respectively, in the solid state at room temperature.

Keywords: Zinc complex; 1,3-Bis(1,2,4-triazol-1-yl)propane; One-dimensional chain; Coordination polymer

1. Introduction

The design and construction of the coordination polymers has attracted great attention for their applications, architectures, and topologies [1–5]. The selection of ''building blocks'' is the key point in manipulating the network structures of the coordination polymers. 1,2,4-Triazole and its derivatives are very interesting ligands because they combine the coordination geometry of both pyrazole and imidazole. A large number of mononuclear, oligonuclear, and polynuclear transition metal complexes of 1,2,4-triazole derivatives have been synthesized due to their magnetic properties and novel topologies [6–12].

The pseudohalides thiocyanate (SCN^-) [12–14] and dicyanamide (dca) ($[N(CN)_2]^-$) [15–17] are widely used to construct coordination polymers because of their versatile coordination and ability to mediate strong magnetic coupling.

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Previously, we synthesized a number of coordination polymers with flexible *bis*(triazole) ligands such as 1,2-*bis*(1,2,4-triazol-1-yl)ethane (bte) [18–21], 1,3-*bis*(1,2,4-triazol-1-yl)propane [22], 1,4-*bis*(1,2,4-triazol-1-yl)butane (btb) [23, 24], 1,4-*bis*(1,2,4-triazol-1-methyl)benzene (bbtz) [25, 26], and 1,3-*bis*(1,2,4-triazol-1-methyl)benzene (mbtz) [27, 28]. Only a few btp complexes were synthesized [11, 12, 22]. The combination of the long flexible ligand btp and short anions (NCS^-, $[N(CN)_2]^-$, and BF_4^-) can give novel topologies. Three new 1-D coordination polymers $[Zn(btp)(NCS)_2]_n$ (**1**), $\{[Zn(btp)_2(dca)_2]_n$ (**2**), and $\{[Zn(btp)_3](BF_4)_2\}_n$ (**3**) (btp = 1, 3-*bis*(1,2,4-triazol-1-yl)propane, dca = dicyanamide) were synthesized, characterized, and the conformations of the btp ligands analyzed.

2. Experimental

2.1. *Materials and physical measurements*

All reagents were of analytical grade and used without purification. 1,3-*bis*(1,2, 4-triazol-1-yl)propane (btp) was synthesized according to the literature method [11]. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240C analyzer. IR spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectrophotometer in the 4000–400 cm^{-1} region. TG and DSC were measured on a Thermal Analyst 2100 TA Instrument and SDT 2960 Simultaneous TGA-DTA Instrument in flowing dinitrogen at a heating rate of 10°C min^{-1}. Luminescence measurements were carried out in the solid state at room temperature and the spectra were collected with a Perkin-Elmer LS50B spectrofluorimeter.

2.2. *Synthesis of $[Zn(btp)(NCS)_2]_n$ (1)*

A 20 mL H_2O : MeOH solution (1 : 1 v/v) of $Zn(NO_3)_2 \cdot 6H_2O$ (0.150 g, 0.5 mmol) and KSCN (0.097 g, 1.0 mmol) was added to one leg of an "H-shaped" tube, and a 20 mL H_2O : MeOH (1 : 1 v/v) solution of btp (0.178 g, 1.0 mmol) was added to the other leg of the tube. Colorless single crystals of **1** were obtained after the mixture was allowed to stand at room temperature for 3 weeks. Yield: 0.158 g (88% based on Zn). Anal. Calcd for $C_9H_{10}N_8S_2Zn$: C, 30.05; H, 2.80; N, 31.15%. Found: C, 29.87; H, 2.68; N, 31.02. IR data (cm^{-1}): 3125w, 3102w, 2094vs, 1533s, 1450w, 1382w, 1359w, 1283s, 1222w, 1135s, 996m, 973w, 877w, 799w, 670m, 650m, 474w.

2.3. *Synthesis of $[Zn(btp)_2(dca)_2]_n$ (2)*

A 20 mL H_2O : MeOH solution (1 : 1 v/v) of $Zn(NO_3)_2 \cdot 6H_2O$ (0.150 g, 0.5 mmol) and $Na[N(CN)_2]$ (0.090 g, 1.0 mmol) was added to one leg of an "H-shaped" tube, and a 20 mL H_2O : MeOH (1 : 1 v/v) solution of btp (0.178 g, 1.0 mmol) was added to the other leg of the tube. Colorless crystals of **2** were obtained after 1 month. Yield: 0.203 g (73% based on Zn). Anal. Calcd for $C_{18}H_{20}N_{14}Zn$: C, 39.03; H, 3.64; N, 45.53. Found: C, 38.87; H, 3.53; N, 45.38%. IR data (cm^{-1}): 3086w, 3029w, 2280s, 2226m, 2168vs, 1524m, 1346m, 1289m, 1227w, 1142m, 1038w, 992m, 918w, 887w, 787w, 679m, 652w, 517w.

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

	1	**2**	**3**
Formula	$C_9H_{10}N_8S_2Zn$	$C_{18}H_{20}N_{18}Zn$	$C_{21}H_{30}B_2F_8N_{18}Zn$
Formula weight ($g\,mol^{-1}$)	359.78	553.89	773.62
Crystal system	Monoclinic	Triclinic	Trigonal
Space group	C_2	$P\bar{1}$	P-3C$_1$
Temperature (K)	193(2)	193(2)	193(2)
Unit cell dimensions (Å, °)			
a	14.692(5)	7.9251(2)	10.955(2)
b	5.9948(18)	8.4584(2)	10.955(2)
c	16.724(6)	10.6767(3)	15.239(4)
α		111.635(8)	90
β	97.393(7)	93.542(8)	90
γ		114.857(9)	120
V (Å^3)	1460.7(8)	583.37(7)	1583.8(6)
Z	4	1	2
ρ_{Calcd} ($g\,cm^{-3}$)	1.636	1.577	1.622
μ (mm^{-1})	1.969	1.103	0.871
$F(000)$	728	284	788
Reflections collected	8251	5763	14391
Unique reflections	3190 [R(int) = 0.0257]	2123 [R(int) = 0.0190]	976 [R(int) = 0.0922]
Parameters	183	170	79
Goodness of fit	1.027	1.016	1.077
R_1 [$I > 2\sigma(I)$]	0.0283	0.0236	0.0506
wR_2 (all data)	0.0526	0.0624	0.1455

2.4. *Synthesis of* $\{[Zn(btp)_3](BF_4)_2\}_n$ *(3)*

A methanolic solution (15 mL) of btp (0.178 g, 1.0 mmol) was added slowly to aqueous solution (15 mL) of $Zn(BF_4)_2 \cdot 6H_2O$ (0.174 g, 0.5 mmol) in a tube. Colorless crystals of **3** were obtained after 2 weeks. Yield: 0.214 g (83% based on btp). Anal. Calcd for $C_{21}H_{30}B_2F_8N_{18}Zn$: C, 32.60; H, 3.91; N, 32.60. Found: C, 32.52; H, 3.84; N, 32.53%. IR data (cm^{-1}): 3115w, 1517s, 1449w, 1345w, 1278s, 1205m, 1130s, 1084s, 1035s, 980m, 959w, 882w, 674m, 654w, 522w.

2.5. *Crystal structure determination*

Suitable single crystals of **1**, **2**, and **3** were carefully selected under an optical microscope and glued to thin glass fibers. 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) [29]. The positions of hydrogen atoms were calculated. 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

The structure of **1** consists of zigzag chains. As depicted in figure 1(a), there are two independent zinc(II) atoms with similar distorted tetrahedral coordination

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

1			
Zn(1)–N(3)	2.0152(19)	Zn(1)–N(7)	1.938(2)
Zn(2)–N(6)	2.001(2)	Zn(2)–N(8)	1.963(2)
N(3)–Zn(1)–N(3A)	101.44(12)	N(3)–Zn(1)–N(7)	110.80(9)
N(3)–Zn(1)–N(7A)	108.91(9)	N(3A)–Zn(1)–N(7)	108.91(9)
N(3A)–Zn(1)–N(7A)	110.80(9)	N(7)–Zn(1)–N(7A)	115.12(14)
N(6)–Zn(2)–N(6B)	110.22(12)	N(6)–Zn(2)–N(8)	101.92(9)
N(6)–Zn(2)–N(8B)	117.67(9)	N(6)–Zn(2)–N(8B)	117.67(9)
N(6B)–Zn(2)–N(8B)	101.92(9)	N(8)–Zn(2)–N(8B)	108.18(14)
N(7)–C(8)–S(1)	177.6(2)	C(8)–N(7)–Zn(1)	175.5(2)
N(8)–C(9)–S(2)	178.4(2)	C(9)–N(8)–Zn(2)	166.3(2)
2			
Zn(1)–N(3)	2.1666(13)	Zn(1)–N(6^{a})	2.1502(13)
Zn(1)–N(8)	2.1528(15)	N(7)–C(8)	1.299(2)
N(7)–C(9)	1.317(2)	N(8)–C(8)	1.155(2)
N(9)–C(9)	1.153(2)		
N(3)–Zn(1)–N(6A)	87.54(5)	N(3)–Zn(1)–N(8)	92.31(5)
N(6^{a})–Zn(1)–N(8)	90.16(5)	C(8)–N(7)–C(9)	120.34(15)
N(7)–C(8)–N(8)	172.51(18)	N(7)–C(9)–N(9)	173.0(2)
3			
Zn(1)–N(3)	2.176(2)		
N(3)–Zn(1)–N(3A)	180.00(15)	N(3)–Zn(1)–N(3B)	89.83(9)
N(3)–Zn(1)–N(3C)	90.17(9)	N(3)–Zn(1)–N(3D)	89.83(9)
N(3)–Zn(1)–N(3E)	90.17(9)	N(3C)–Zn(1)–N(3D)	180.00(14)
N(3B)–Zn(1)–N(3E)	180.00(16)		

Symmetry codes: **1** A: $-x, y, -z$, B: $-x+1, y, -z+1$; **2** A: $-x+1, -y+2, -z+2$, B: $x, y-1, z-1$, C: $-x+1, -y+1, -z+1$; **3** A: $-x, -y, -z$, B: $x-y, x, -z$, C: $-y, x-y, z$, D: $y, -x+y, -z$, E: $-x+y, -x, z$.

environments in **1**. Each zinc(II) is located on the C_2 axis and coordinated with two nitrogens of two symmetry-related btp (Zn(1)–N(3): 2.015(2) Å; Zn(2)–N(6): 2.001(2) Å), and two nitrogens of two symmetry-related NCS^- (Zn(1)–N(7): 1.938(2) Å; Zn(2)–N(8): 1.963(2) Å), forming distorted tetrahedral geometry. The monodentate N-bound NCS^- ligand is quasi-linear (N(7)–C(8)–S(1): 177.6(2)°; N(8)–C(9)–S(2): 178.4(2)°). The Zn–N–C (NCS) bond angles are 175.5(2)° for Zn(1)–N(7)–C(8) and 166.3(2)° for Zn(2)–N(8)–C(9). Each btp ligand links two zinc(II)'s forming a 1-D zigzag chain [19, 22]. The Zn(1) $\cdots$ Zn(2) separation via the bridging btp is 10.403 Å in **1**, close to the metal-metal separation 10.4472 Å in $[Co(btp)_2(NCS)_2]$ [12]. The Zn(1) $\cdots$ Zn(1C) and Zn(2) $\cdots$ Zn(2A) distances are 20.792(3) Å. Adjacent parallel chains stack so that the convex NCS^- ligands bow of one chain extends into the concave of the neighboring chain (figure 1b). The shortest interchain Zn(1) $\cdots$ Zn(1D) and Zn(2) $\cdots$ Zn(2D) distances are 5.995 Å, corresponding to the b axis translation.

The coordination geometry of the Zn(II) in **2** is distorted octahedral. Each Zn(II) center is coordinated to four triazole nitrogens from four different btp ligands in the equatorial plane [Zn(1)–N(3): 2.1666(13) Å; Zn(1)–N(6A): 2.1502(13) Å], and two nitrogens from two dicyanamides [Zn(1)–N(8): 2.1528(15) Å] in the axial positions (figure 2a). The dca coordinates monodentate through nitrile nitrogen.

The nitrile C–N bond lengths of dca are 1.155(2) Å for N(8)–C(8) and 1.153(2) Å for N(9)–C(9). The bond angle related to amide nitrogen C(8)–N(7)–C(9) is 120.34(15)°, corresponding to the amide nitrogen N(7) with sp^2 hybrid. Those bond angles related to

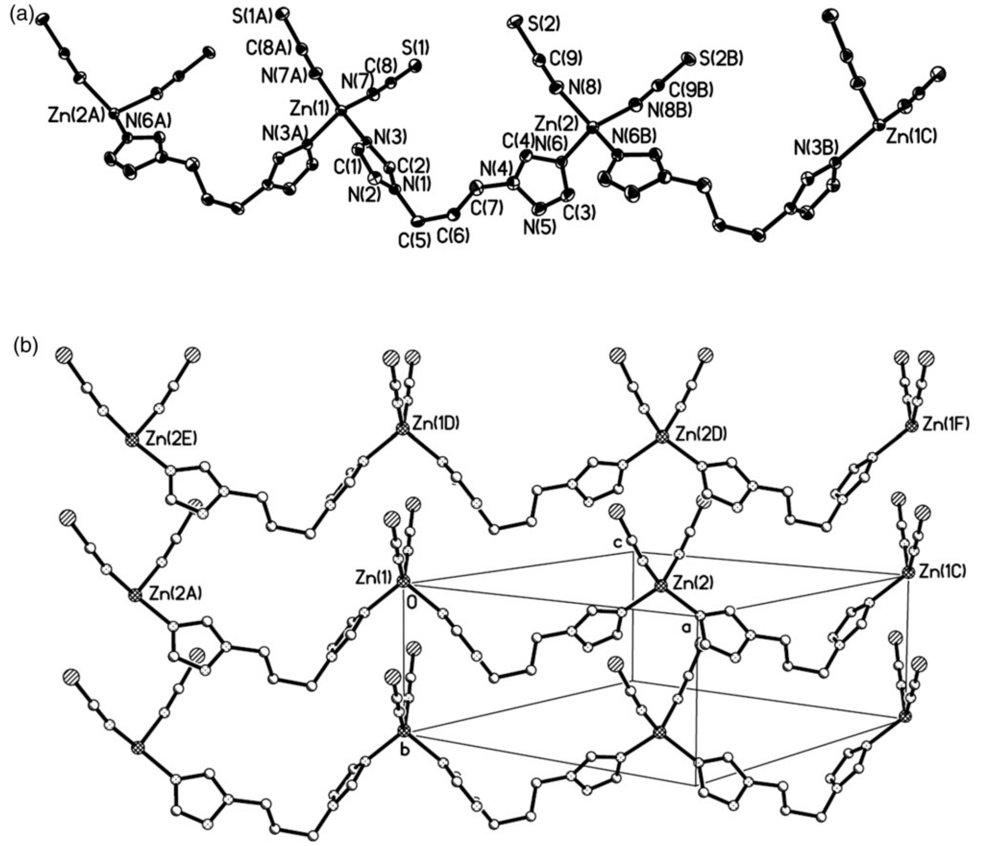

Figure 1. (a) The 1-D double chain structure along *a* in **1** (symmetry codes: A: $-x$, y, $-z$; B: $-x+1$, y, $-z+1$; C: $x+1$, y, z.). The hydrogen atoms are omitted for clarity. (b) The packing of the 1-D chains in **1** (symmetry codes: A: $-x$, y, $-z$; B: $-x+1$, y, $-z+1$; C: $x+1$, y, z; D: x, $y-1$, z; E: $x-1$, $y-1$, $z-1$; F: $x+1$, $y-1$, $z+1$).

nitrile N(7)–C(8)–N(8) and N(7)–C(9)–N(9) are 172.51(18) and 173.0(2)°, respectively, corresponding to N(8), C(8), C(9), and N(9).

Each Zn(II) connects to another Zn(II) by two btp, resulting in a $Zn_2(btp)_2$ 20-membered metallocycle in **2**. Two strands of btp are wrapped around each other and held together by Zn(II), forming a double chain structure [12, 19–21, 23, 25, 27, 28] along the *c*-axis. The Zn $\cdots$ Zn distances separated via the bridging btp are 10.906 Å, close to the Zn $\cdots$ Zn metal–metal separation 10.403 Å in **1**. The 1-D chains are stacked parallel along the *a*-axis and extend along the *b*-axis (figure 2b).

The structure of **3** consists of 1-D triple chain cations and BF_4^- anions. Each zinc(II) center is located on the C_3-axis and six-coordinate to six nitrogens of six symmetry-related btp ligands (Zn(1)–N(3): 2.176(2) Å) in the distorted octahedral coordination (figure 3a). Bond angles N–Zn–Zn are close to 90°. Neighboring Zn(II)'s locate on the same C_3-axis and connect to other Zn(II) atoms by three btp ligands. The three strands of btp are wrapped around each other, held together by zinc, forming a triple chain [30, 31] structure along the *c*-axis. The Zn $\cdots$ Zn distances separated via the bridging btp are 7.620 Å, corresponding to half of *c*-axis translation and obviously shorter than

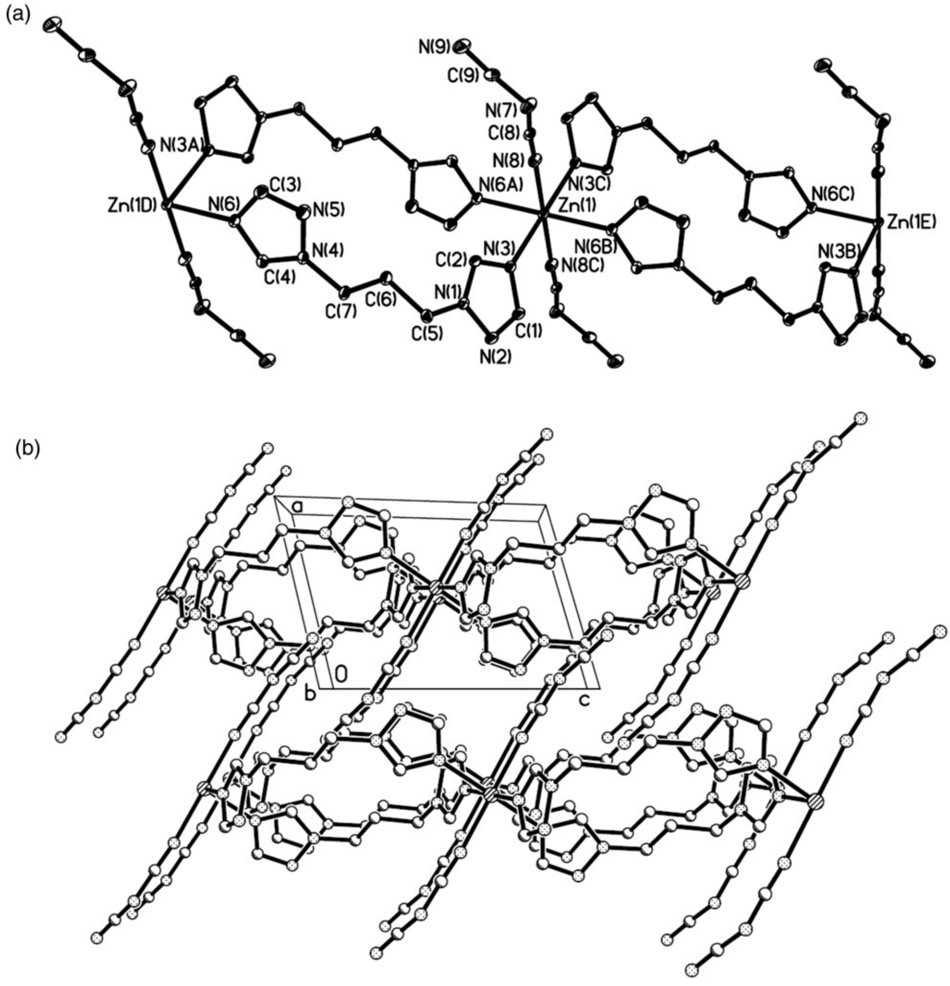

Figure 2. (a) The coordination environment of Zn(II) in **2** (symmetry codes: A: $-x+1$, $-y+2$, $-z+2$; B: x, $y-1$, $z-1$; C: $-x+1$, $-y+1$, $-z+1$; D: x, $y+1$, $z+1$; E: x, $y-1$, $z-1$). The hydrogen atoms are omitted for clarity. (b). The packing of 1-D chains in **2**.

Zn $\cdots$ Zn separations 10.403 Å in **1** and 10.906 Å in **2**. The 1-D triple chains are stacked parallel along the *b*-axis and extend along the *a*-axis (figure 3b).

The btp ligands exhibit *anti*-*gauche*, *anti*-*anti*, and *gauche*-*gauche* conformation in **1**, **2**, and **3**, respectively. The Zn $\cdots$ Zn distances separated by the btp bridges are 10.403 Å for **1**, 10.906 Å for **2**, and 7.620 Å for **3**. The dihedral angles between two triazole planes are 74.4° for **1**, 103.9° for **2**, and 145.0° for **3**. The plane of $(CH_2)_3$ chain in **3** has disorder with r.m.s. deviations of the atoms from the mean plane of C(3)/C(4)/C(3F)/C(4F) 0.1528 Å. The plane of $(CH_2)_3$ chain is steeply inclined, 75.8 and 101.8° for **1**, 157.6 and 81.2° for **2**, 72.5 and 72.5° for **3**, to the two triazole ring planes.

The IR spectrum shows triazole ring vibrations [19, 20] in **1**, **2**, and **3** at 1533 and 1283, 1524 and 1289, 1517 and 1278 cm^{-1}, respectively. The strong absorption of **1** at 2094 is attributed to C=N stretch of the cyanate group, consistent with thiocyanate–N

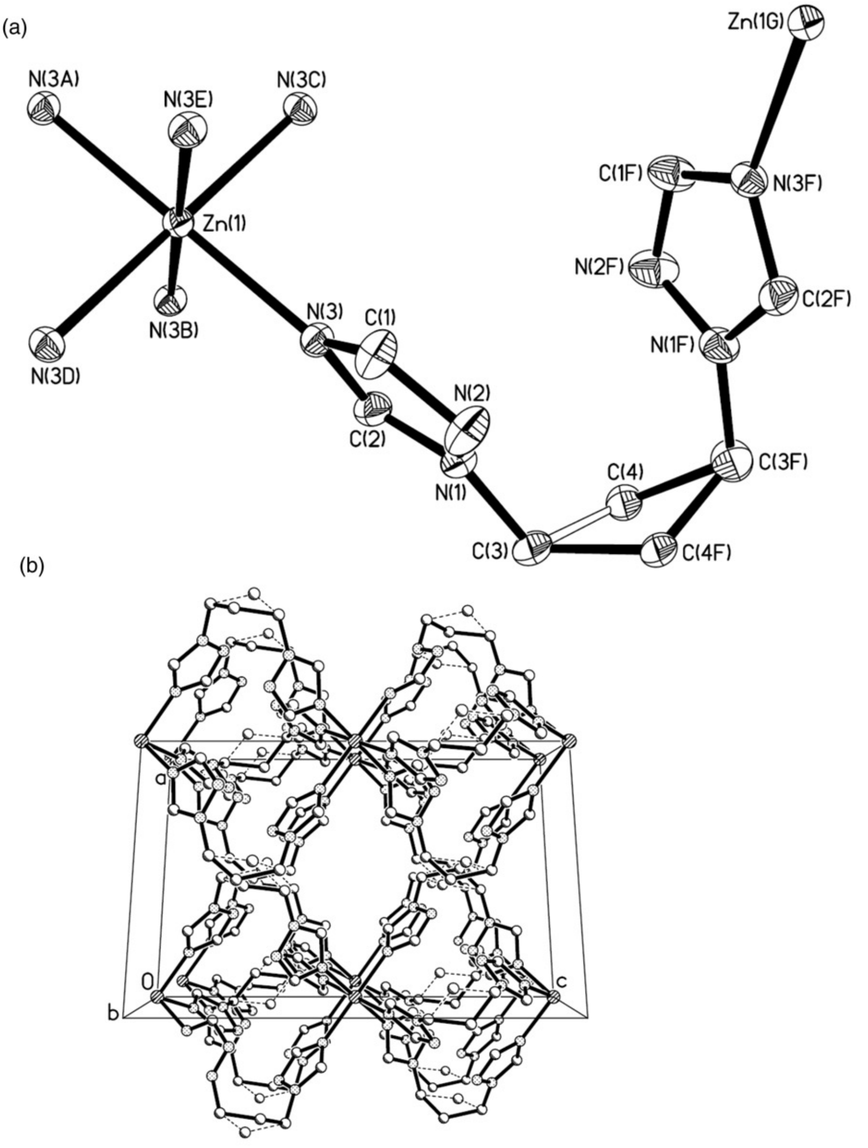

Figure 3. (a) The coordination environment of Zn(II) in **3** (symmetry codes: A: $-x$, $-y$, $-z$; B: $x-y$, x, $-z$; C: $-y$, $x-y$, z; D: y, $-x+y$, $-z$; E: $-x+y$, $-x$, z; F: y, x, $-z+1/2$; G: x, y, $z+1/2$). The hydrogen atoms are omitted for clarity. (b) The packing of 1-D chains in **3**.

coordination [12–14, 19, 20]. The strong absorption bands of **2** at 2280, 2226, and 2168 cm^{-1} are assigned to the CN symmetric stretch (ν_{sym}), the asymmetric stretch band (ν_{asym}), and the combination band of ν_{sym} and ν_{asym} of dca. The small shift towards high frequencies, compared with dca in $\mathrm{NaN(CN)_2}$ (2232 and 2179 cm^{-1}), confirmed monodentate dca [15–17]. Strong absorptions at 1130, 1084, and 1035 cm^{-1} in **3** are attributed to disordered $\mathrm{BF_4^-}$.

The TGA studies showed that **1**, **2**, and **3** are stable, beginning decomposition above 234, 158, and 253°C, respectively. The solid state luminescence spectra of **1**, **2**, and **3** at

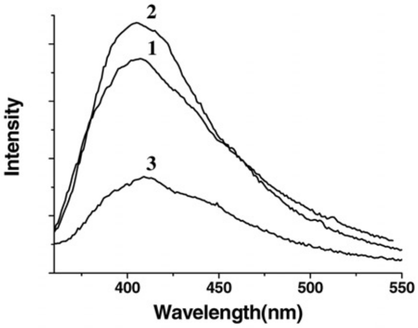

Figure 4. Solid state emission of **1**, **2**, and **3**.

room temperature (figure 4) reveal emission maxima at 405, 407, and 409 nm, respectively, upon excitation at 350 nm. In comparison, free btp molecule in the solid state displays an emission band at 397 nm when excited at 340 nm. The emissions of **1**–**3** can be attributed to the ligand-to-metal charge transfer (LMCT) [32–34].

Reaction of the flexible 1,3-*bis*(1,2,4-triazol-1-yl)propane (btp) with Cu(II) with ClO_4^-, and $CF_3SO_3^-$ gives two 2-D network coordination polymers $\{[Cu(btp)_2(CH_3CN)(H_2O)](CF_3SO_3)_2\}_n$ and $\{[Cu(btp)_2(CH_3CN)_2](ClO_4)_2\}_n$ [11]. Reaction of btp with Co(II) and NCS^- gives a 1-D double chain $[Co(btp)_2(NCS)_2]_n$ [12]. The reaction of btp with Zn(II)/Cd(II) and 4-hydroxybenzoate (phba) give $[Zn(phba)_2(btp)]_2$ containing the neutral $Zn_2(btp)_2$ metallacycle and $[Cd(phba)_2(btp)(H_2O)]_n$ forming 1-D zigzag chain [22].

Reaction of btp with Zn(II) and NCS^-, dca, or BF_4^- yields three new coordination polymers $[Zn(btp)(NCS)_2]_n$ (**1**), $\{[Zn(btp)_2(dca)_2]_n$ (**2**), and $\{[Zn(btp)_3](BF_4)_2\}_n$ (**3**). In **1**, **2**, and **3**, one-, double-, triple-btp ligands link two Zn(II) atoms and extend to form a 1-D zigzag chain for **1**, 1-D double chain for **2**, and 1-D triple chain for **3**. Further synthesis of new motifs with btp and different metal salts are underway in our lab.

Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (CCDC) (Email: deposit@ccdc.cam.ac.uk) as supplementary materials and the CCDC reference numbers for **1**, **2**, and **3** are 274707, 702049, and 702050, respectively.

Acknowledgments

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