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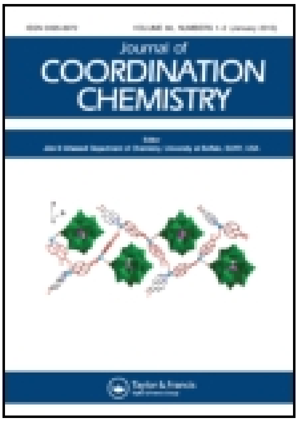

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Three double- or triple-stranded chains constructed from different cobalt(II) salts and a flexible bis-triazole ligand

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Three double- or triple-stranded chains constructed from different cobalt(II) salts and a flexible bis-triazole ligand

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Three coordination compounds, $\{[Co(btrp)_2(H_2O)_2]\cdot NO_3\cdot H_2O\}_n$ (**1**), $\{[Co(btrp)_2(H_2O)_2]\cdot H_2O\cdot 2H_2btc\}_n$ (**2**), and $\{[Co(btrp)_3]\cdot 2ClO_4\}_n$ (**3**) (btrp = 1,3-bis(1,2,4-triazol-1-yl)propane; H_3btc = benzene-1,3,5-tricarboxylic acid), have been prepared *via* solvothermal method and characterized by single-crystal X-ray diffraction and elemental analyses. Compound **1** possesses a 1-D double-stranded chain composed of ribbons of 20-membered cycles. Binuclear water clusters link adjacent nitrate anions to form a 1-D supramolecular helix in the structure. Compound **2** has a 1-D double-stranded chain wherein free H_2btc ligands constitute 1-D negative chains through classical hydrogen-bonding interactions (O–H $\cdots$ O). Compound **3** exhibits a triple-stranded 1-D chain. For **1**–**3**, 3-D supramolecular structures are consolidated by interchain weak hydrogen-bonding interactions as well as electrostatic interactions.

Keywords: 1,3-Bis(1,2,4-triazol-1-yl)propane; Double-stranded chain; Triple-stranded chain; Supramolecular structure; Hydrogen-bond interaction

1. Introduction

Coordination polymers have aroused much interest as materials owing to potential new electronic, optical, magnetic, and catalytic properties, as well as intriguing structural motifs [1–10]. A key step for construction of polymeric transition metal complexes is to select appropriate multidentate bridging ligands [11, 12]. Recently, new flexible bispolyazole-type ligands such as 1- or 4-substituted 1,2,4-triazole rings tethered by an alkyl spacer have been used to obtain a wide variety of polynuclear molecules and linear coordination polymers [13–16]. 1,3-Bis(1,2,4-triazol-1-y1)propane (btrp) is an excellent synthon for the construction of extended structures with the following distinctive characteristics: (i) btrp with large space length has flexibility and conformational freedom that allow it to bend and rotate to conform to the coordination environment of metal ions, creating structural diversity; (ii) btrp ligand can donate four nitrogen atoms in coordinating with metal ions to obtain unexpected structures.

Recently, we reported the crystal structures of a series of transition metal (Cd^{2+}, Zn^{2+}, and Cu^{2+}) coordination polymers with flexible bis(triazole) propane ligands [17–20]. To extend our work in the area, three different 1-D double- or triple-stranded chains,

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$\{[Co(btrp)_2(H_2O)_2]\cdot NO_3\cdot H_2O\}_n$ (**1**), $\{[Co(btrp)_2(H_2O)_2]\cdot H_2O\cdot 2H_2btc\}_n$ (**2**), and $\{[Co(btrp)_3]\cdot 2ClO_4\}_n$ (**3**), were fabricated by changing the anions. The role of the anions on the resulting structures of polymers is discussed. Infrared (IR) spectra and thermal stabilities of the complexes are also discussed.

2. Experimental

2.1. *General remarks*

Deionized water was used as solvent. The reagents and solvents employed were commercially available and used as received. btrp was synthesized as reported [21].

2.2. *Synthesis of 1*

A mixture of $Co(NO_3)_2\cdot 6H_2O$ (0.10 mmol, 0.029 g), btrp (0.1 mmol, 0.018 g), H_2O (2 mL), and CH_3CN (2 mL) were placed in a 15 mL Teflon-lined steel vessel and heated to 90°C for 3 days, and then cooled to room temperature at $1^\circ C\,h^{-1}$. Pink crystalline **1** were obtained (yield 38% based on Co). Elemental Anal. Calcd (%): C, 27.50; H, 4.62; N, 32.08. Found: C, 27.04; H, 4.15; N, 32.54.

2.3. *Synthesis of 2*

A mixture of $Co(NO_3)_2\cdot 6H_2O$ (0.20 mmol, 0.058 g), btrp (0.2 mmol, 0.036 g), H_3btc (0.2 mmol, 0.042 g), NEt_3 (0.2 mmol, 28 µL), and H_2O (8 mL) were placed in a 15 mL Teflon-lined steel vessel and heated to 90°C for 3 days, and then cooled to room temperature at the rate of $1^\circ C\,h^{-1}$. Pink crystalline **2** were obtained (yield 30% based on Co). Elemental Anal. Calcd (%): C, 43.30; H, 4.09; N, 18.94. Found: C, 43.72; H, 3.81; N, 19.28.

2.4. *Synthesis of 3*

A mixture of $Co(ClO_4)_2\cdot 5H_2O$ (0.10 mmol, 0.035 g), btrp (0.1 mmol, 0.018 g), H_2O (2 mL), and CH_3CN (2 mL) were placed in a 15 mL Teflon-lined steel vessel and heated to 90°C for 3 days, and then cooled to room temperature at the rate of $1^\circ C\,h^{-1}$. Pink crystalline **3** were obtained (yield 35% based on Co). Elemental Anal. Calcd (%): C, 31.82; H, 3.82; N, 31.82. Found: C, 31.44; H, 3.45; N, 31.62.

2.5. *X-ray crystallography*

Single-crystal X-ray diffraction measurements of **1**–**3** were carried out with a Bruker Smart CCD diffractometer and a graphite crystal monochromator situated in the incident beam for data collection at room temperature. Lorentz polarization and absorption corrections were applied. The structures were solved by direct methods and refined by full-matrix least-squares techniques using SHELXS-97 and SHELXL-97.

Table 1. Crystallographic data and structure refinement details for **1**–**3**.

Complex	**1**	**2**	**3**
Empirical formula	$C_{14}H_{28}CoN_{14}O_{10}$	$C_{16}H_{18}Co_{0.5}N_6O_{7.50}$	$C_{21}H_{30}Cl_2CoN_{18}O_8$
Formula weight	611.44	887.66	792.48
Temperature (K)	293(2)	293(2)	293(2)
Crystal system	$P2_1/c$	$P\bar{1}$	$P\bar{3}c1$
Space group	Monoclinic	Triclinic	Trigonal
Unit cell dimensions (Å, °)			
a	8.802(2)	9.907(7)	10.9673(4)
b	17.287(6)	10.108(8)	10.9673(4)
c	10.353(3)	10.154(8)	15.2101(9)
α	90	77.18(7)	90
β	125.36(3)	67.43(7)	120
γ	90	89.399(6)	90
Volume (Å^3), Z	1269.7(7), 2	912.36(12), 1	1584.39(12), 2
Calculated density (Mg m^{-3})	1.599	1.616	1.661
$F(000)$	634	459	786
θ range for data collection (°)	2.36–25.01	2.67–25.01	2.68–24.99
Limiting indices	$-10 \le h \le 6$; $-20 \le k \le 20$; $-12 \le l \le 12$	$-11 \le h \le 5$; $-11 \le k \le 12$; $-11 \le l \le 12$	$-12 \le h \le 12$; $-12 \le k \le 13$; $-18 \le l \le 9$
Reflections collected	2267	3401	939
Independent reflection	1995 [R(int) = 0.0271]	2815 [R(int) = 0.0161]	790 [R(int) = 0.0282]
Goodness-of-fit on F^2	1.051	1.053	1.071
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0553$, $wR_2 = 0.1556$	$R_1 = 0.0369$, $wR_2 = 0.0916$	$R_1 = 0.0498$, $wR_2 = 0.1457$
R indices (all data)	$R_1 = 0.0783$, $wR_2 = 0.1687$	$R_1 = 0.0451$, $wR_2 = 0.0945$	$R_1 = 0.0564$, $wR_2 = 0.1519$.
Largest difference peak and hole (e Å^{-3})	0.708 and −0.390	0.461 and −0.622	0.798 and −0.590

All non-hydrogen atoms were refined anisotropically and hydrogen atoms were located and refined isotropically. Crystallographic data for **1**–**3** are summarized in table 1. Selected bond distances and angles of **1**–**3** are listed in table 2. Hydrogen bond data are shown in table 3.

3. Results and discussion

3.1. *Crystal structure of 1*

Compound **1** is a double-stranded chain composed of ribbons of 20-membered rings. Each ring in **1** involves two cobalts and two btrp; Co1 is in a general position. As depicted in figure 1(a), Co1 is six coordinate in a distorted octahedral coordination sphere defined by two oxygen atoms occupying axial positions, while the equatorial positions are finished by four different nitrogen donors from four btrp ligands. The btrp ligands have *trans* conformations with N $\cdots$ N distances of 7.567 Å and a dihedral angle of the two triazole rings of 113.5°. Each btrp is a bidentate linker by using two terminal nitrogen atoms of triazole rings, bridging Co^{2+} to build a 1-D double-stranded chain (figure 1b).

O1 from coordinated water and O5 from lattice water generate strong intermolecular hydrogen bonds and construct binuclear water clusters, in which O1 is a hydrogen donor

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

1					
Co(1)–O(1)	2.1014(17)	Co(1)–N(1)	2.160(2)	Co(1)–N(4)#2	2.168(2)
Co(1)–O(1)#1	2.1014(17)	Co(1)–N(1)#1	2.160(2)	Co(1)–N(4)#3	2.169(2)
O(1)–Co(1)–O(1)#1	180.000(1)	O(1)#1–Co(1)–N(1)	89.07(8)	O(1)–Co(1)–N(4)#2	90.53(8)
O(1)–Co(1)–N(1)	90.93(8)	N(1)–Co(1)–N(1)#1	179.998(1)	N(1)–Co(1)–N(4)#2	93.41(8)
O(1)–Co(1)–N(4)#3	89.47(8)	N(1)–Co(1)–N(4)#3	86.59(8)		
2					
Co(1)–O(1)#1	2.0757(19)	Co(1)–N(1)	2.158(2)	Co(1)–N(6)#1	2.165(2)
Co(1)–O(1)	2.0757(19)	Co(1)–N(1)#1	2.158(2)	Co(1)–N(6)	2.165(2)
O(1)#1–Co(1)–O(1)	179.999(2)	O(1)–Co(1)–N(1)	88.69(8)	O(1)–Co(1)–N(6)#1	93.54(9)
O(1)–Co(1)–N(6)	86.46(9)	N(1)–Co(1)–N(6)	94.16(9)	N(6)#1–Co(1)–N(6)	179.999(1)
O(1)#1–Co(1)–N(1)	91.31(8)	N(1)–Co(1)–N(1)#1	180.0	N(1)–Co(1)–N(6)#1	85.84(9)
3					
Co(1)–N(6)	2.157(5)	Co(1)–N(6)#1	2.157(5)	Co(1)–N(6)#2	2.157(5)
Co(1)–N(6)#3	2.157(5)	Co(1)–N(6)#3	2.157(5)	Co(1)–N(6)#3	2.157(5)
N(6)#1–Co(1)–N(6)#2	90.3(2)	N(6)#1–Co(1)–N(6)#4	180.0(3)	N(6)#1–Co(1)–N(6)	90.3(2)
N(6)#1–Co(1)–N(6)#3	89.7(2)	N(6)#2–Co(1)–N(6)#4	89.7(2)	N(6)–Co(1)–N(6)#5	179.999(1)

Symmetry transformations used to generate equivalent atoms: **1**: #1: $-x+1$, $-y+2$, $-z+2$; #2: x, y, $z+1$; #3: $-x+1$, $-y+2$, $-z+1$. **2**: #1: $-x$, $-y+1$, $z+1$. **3**: #1: $-y+2$, $x-y+1$, z; #2: $-x+y+1$, $-x+2$, z; #3: $x-y+1$, x, $-z$; #4: y, $-x+y+1$, $-z$; #5: $-x+2$, $-y+2$, $-z$.

Table 3. Selected hydrogen-bond data for **1–3**.

D–H···A	H···A	D···A	D–H···A
1			
O1–H1A···O5#2	1.778	2.629	175.00
O1–H1B···O4#2	1.918	2.767	173.61
O5–H5D···O2#5	1.949	2.802	176.85
O5–H5C···N3#5	2.006	2.846	167.81
O5–H5D···N7#5	2.676	3.459	153.17
O1–H1B···N7#2	2.669	3.427	148.73
C5–H5B···O5#5	2.593	3.327	132.62
2			
O1–H1A···O7#2	1.759	2.610	175.27
O1–H1B···O5#3	1.897	2.746	172.90
O4–H4C···O4#3	1.678	2.475	155.02
O4–H4C···O5#3	2.630	3.180	123.61
O6–H6A···O6#4	1.651	2.467	161.05
O8–H8A···N5#3	2.551	3.082	121.93
O8–H8A···O6	2.652	3.187	122.45
3			
C3–H3B···O1	2.346	3.0820	133.42
C4–H4A···O3	2.221	2.950	132.64

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

and O5 a hydrogen-bond acceptor (figure 1b). The hydrogen-bond distance is 2.637(4) Å (O1–H1A···O5). Binuclear water clusters link adjacent nitrate anions to form 1-D helical supramolecular chains along the crystallographic *b*-axis (figure 1c and d), which

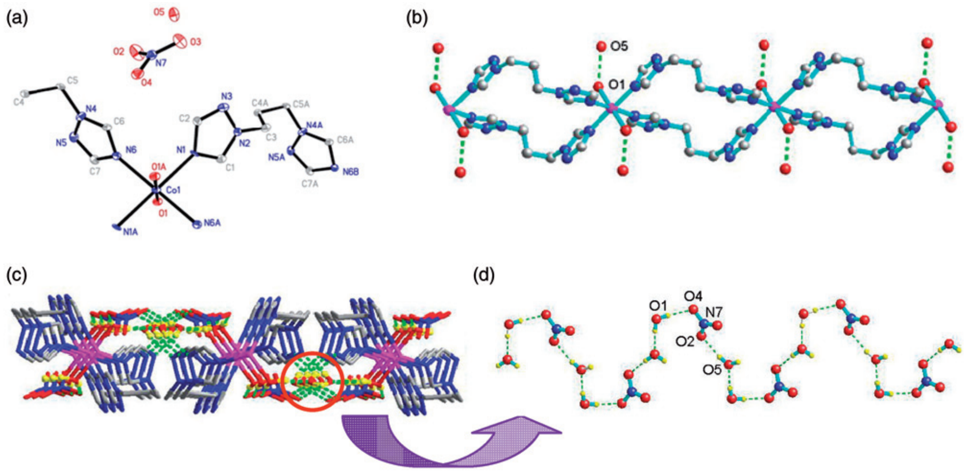

Figure 1. (a) Coordination geometry of cobalt(II) in **1**. (b) The 1-D chain structure with binuclear water clusters. (c) 2-D supramolecular architecture of **1**. (d) 1-D supramolecular helical chain constructed *via* binuclear water clusters and free nitrate anions. Dotted line indicates hydrogen bond (O–H···O).

cross-link the 1-D metal–ligand single-chains of **1** into a 2-D supramolecular architecture. O1 and O5 from binuclear clusters are hydrogen-bond donors and O2 and O4 from nitrate anions are hydrogen-bond acceptors. The corresponding hydrogen-bonding distances are 2.766(5) Å (O1–H1B···O4) and 2.811(3) Å (O5–H5D···O2) (table 3). The resulting 2-D structures are cross-linked by weak hydrogen-bond interactions (C5–H5B···O5 = 3.327(1) Å) between C–H groups in btrp ligands and oxygen in coordinated water, thus leading to formation of a 3-D supramolecular architecture (figure 2). Additionally, N7 from bidendate *trans* btrp ligands and O1 and O5 from binuclear clusters also generate intermolecular weak hydrogen bonds (O1–H1B···N7 = 3.450(1) Å, O5–H5D···N7 = 3.442(1) Å), which further stabilize the 3-D supramolecular architecture.

3.2. *Crystal structure of 2*

The X-ray structural analysis reveals that **2** is a triclinic crystal system in the space group *P*-1. The repeat unit in **2** consists of one crystallographically independent Co^{2+}, two btrp, two free H_2btc, two coordinated, and one lattice water molecule, as shown in figure 3(a). Co1 coordinates with four nitrogen atoms from four btrp, forming a quadrangle and the bond lengths of Co1–N1 and Co1–N6 are 2.160 and 2.168 Å, respectively. The Co1 and the four surrounding nitrogen atoms are coplanar. Two water molecules at the axial sites of the quadrangle coordinate to Co1 with bond length of 2.076 Å and angle of 180°.

Each cobalt(II), linked by four btrp ligands, acts as the square-planar node. The btrp exhibits *cis* conformation with the shortest N···N distance of 8.066 Å and dihedral angle of the two triazole rings of 54°, which adopt the μ_2 linking mode. As with **1**, **2** is also a 1-D double-stranded chain (figure 3b). In the framework of **2**, free H_2btc

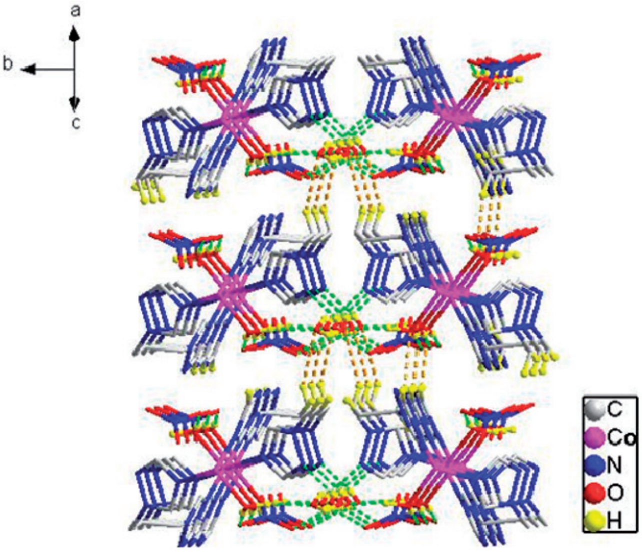

Figure 2. The 3-D supramolecular framework *via* C–H $\cdots$ O and O–H $\cdots$ O hydrogen bonds in **1**.

constitute a 1-D supramolecular chain through classical hydrogen-bonding interactions (O–H $\cdots$ O, table 3), as shown in figure 3(c). The btrp–Co chains carry positive charges, whereas the H_2btc^- supramolecular chains have negative charge. Thereupon, the 3-D supramolecular structure is consolidated by interchain weak classical hydrogen-bonding interactions as well as electrostatic interactions (figure 3d).

3.3. *Crystal structure of 3*

Compound **3** adopts very similar solid-state organization to the recently reported Fe(II) complex $[Fe(btrp)_3](ClO_4)_2$ [22]. The basic framework of **3** consists of 1-D triple-stranded cationic chains and ClO_4^-. In a distorted octahedral geometry, Co1 is coordinated with six nitrogen atoms from six btrp with Co–N distance of 2.188(7) Å (figure 4a). Each btrp is a bidentate linker, using two terminal nitrogen atoms of the triazole rings with *trans* configuration to bridge two Co(II) ions, with Co $\cdots$ Co distance of 5.254(2) Å.

Every other Co^{2+} is bridged by three btrp ligands and a triple-stranded 1-D chain structure is formed along the *c*-direction (figure 4b). These chains are further linked *via* weak hydrogen-bond interactions (C3 $\cdots$ O1 = 3.0820(1) Å, table 3) to form a 3-D supramolecular structure, in which free ClO_4^- fill the void, as shown in figure 5.

Many flexible triazole cobalt coordination polymers have been synthesized [23–26]. Zhao *et al.* [23] synthesized $[Co(btrp)_2(NCS)_2]$ (btrp = 1,3-bis(1,2,4-triazol-1-yl)propane) with a 1-D double chain structure. Li *et al.* synthesized eight cobalt coordination polymers with 1,2-bis(1,2,4-triazol-1-yl)ethane (bte) with different anions. The structures showed 1-D chains [14, 24] and 2-D layers [25]. In the compounds, though the coordinating modes of the triazole ligands are all bis-connected, the structures of the compounds are different because of the flexibility of the ligand.

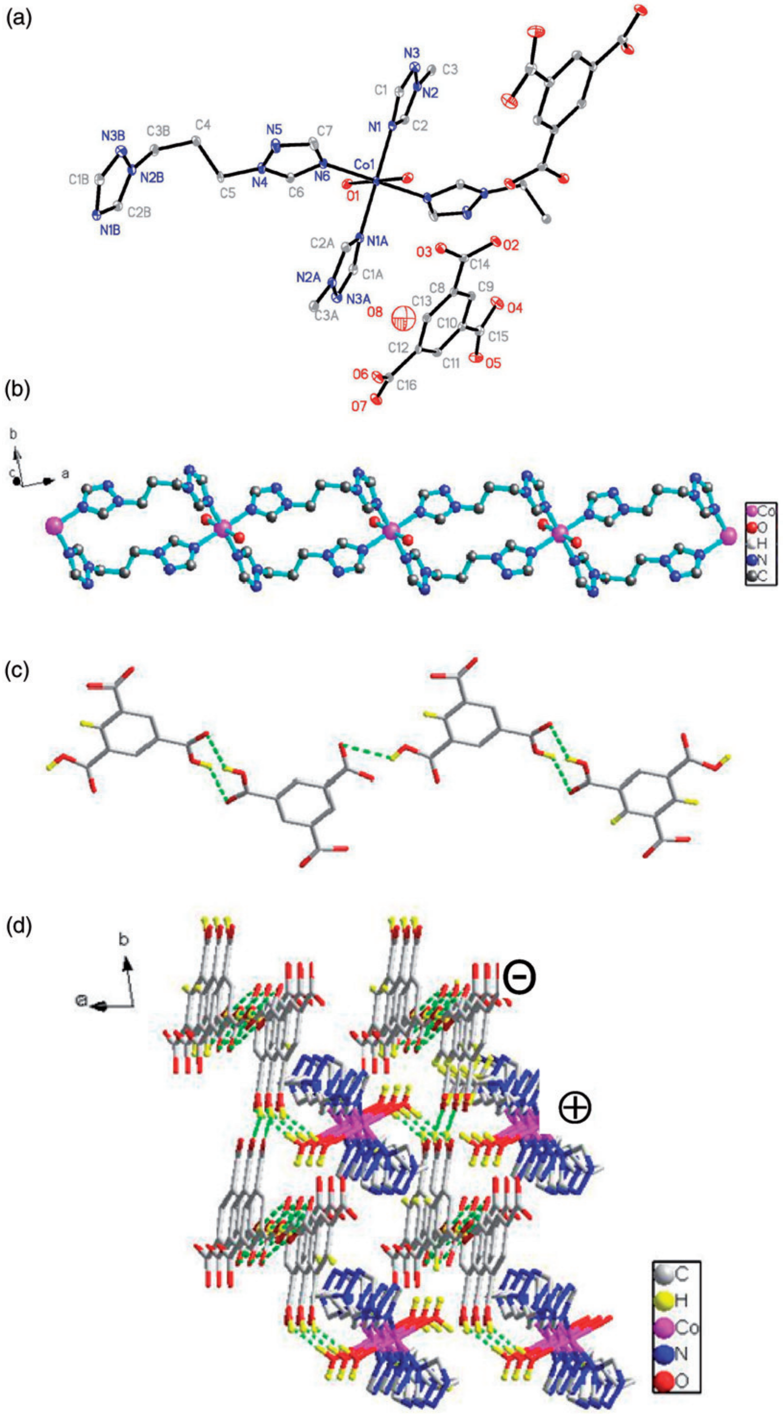

Figure 3. (a) The coordination environment of Co in **2**. (b) The extended 1-D chain linked with btrp in **2**. (c) The extended 1-D supramolecular chain linked with weak hydrogen-bonding interactions in **2**. (d) The 3-D supramolecular structure consolidated by weak interchain classical hydrogen-bonding interactions as well as electrostatic interactions in **2**.

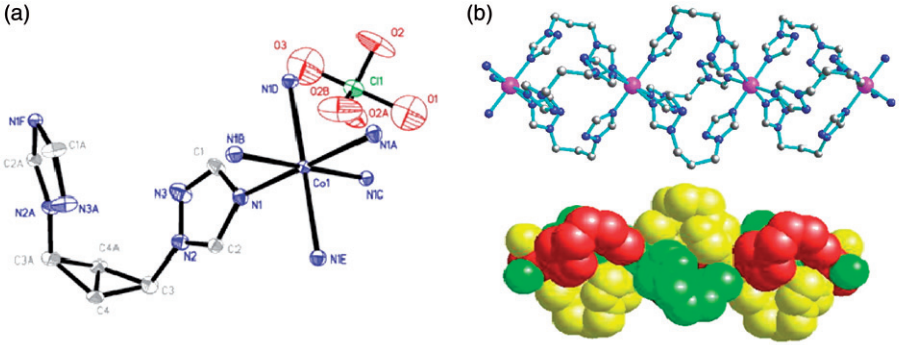

Figure 4. (a) The coordination environment of Co(II) in **3**. (b) The plait-like 1-D chain of **3** viewed along the *a*-direction (top) and space-filling diagram (bottom).

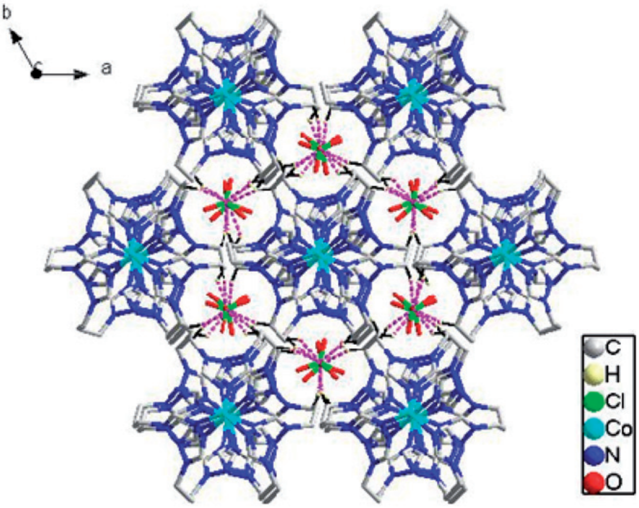

Figure 5. The 3-D structure viewed along the *c*-direction in **3**.

3.4. *IR spectra*

In IR spectra of **1**–**3** (Supplementary material), broad peaks centered at *ca* 3400 cm^{-1} indicate O–H stretching vibrations of water and/or carboxyl. IR spectra of **2** display strong absorption at 1697.9 cm^{-1}, indicating the presence of carboxyl groups coming from H_2btc^{-1}. The sharp peak appearing at 3100–3200 cm^{-1} is characteristic for the 1-triazole ligand, which should be attributed to the aromatic C–H stretching vibration (3100 cm^{-1} for **1**, 3113 cm^{-1} for **2**, and 3136 cm^{-1} for **3**).

3.5. *Thermal analyses*

Thermal behaviors of **1**–**3** were studied by thermogravimetric analysis (TGA) under nitrogen and the thermogravimetric (TG) curves are shown in

"Supplementary material." The TGA of **1** displays two mass losses. The first loss below 150°C is attributed to loss of free and coordinated water and the second weight loss is ascribed to loss of ligands around 240°C. Because of the existence of free H_2btc^{-1} in **2**, it is very unstable and decomposes gradually above 120°C. The TG curve for **3** reveals that it is stable to 240°C. With further heating, rapid mass loss occurs, assigned to decomposition of organic ligands and ClO_4^-.

4. Conclusion

Using different Co(II) salts and btrp in H_2O/CH_3CN solution, three coordination polymers (**1**–**3**) have been synthesized. Compound **3** is isomorphous to the reported $[Fe(btrp)_3](ClO_4)_2$ [22]. By using $Co(NO_3)_2 \cdot 6H_2O$, a 1-D double-stranded chain **1** containing ribbons of macrocyclic rings results, in which binuclear water clusters link adjacent nitrate anions to form a 1-D supramolecular helix. When H_3btc is added in the above reaction, a different 1-D double-stranded chain **2** is obtained. Free H_2btc^- in **2** forms a supramolecular 1-D chain through classical hydrogen-bonding interactions ($O–H \cdots O$). By using $Co(ClO_4)_2 \cdot 6H_2O$ instead of $Co(NO_3)_2 \cdot 6H_2O$, a 1-D triple-stranded structure of **3** is obtained. The btrp ligands adopt *cis* configuration in **2** and *trans* configuration in **1** and **3**. The structures of the compounds are controlled by the anions (NO_3^- in **1**, H_2btc^- in **2**, and ClO_4^- in **3**).

Supplementary material

CCDC 802915, 802916, and 802917 contain the supplementary crystallographic data for **1**, **2**, and **3**. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223-336033; or E-mail: deposit@ccdc.cam.ac.uk).

Acknowledgments

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