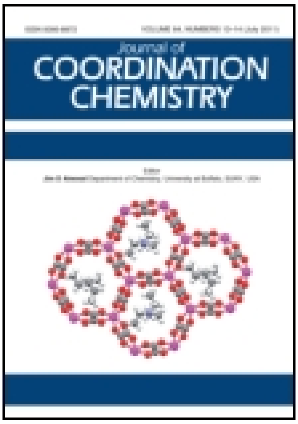

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Three 4-connected nickel coordination polymers affording a 3-D $CdSO_4$ network and two 2-D (4,4) networks

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The self-assembly reaction of 1,4-bis(1,2,4-triazol-1-yl)butane (btb) and Ni(II) salts gives three coordination polymers $\{[Ni(btb)_2(NCS)_2] \cdot H_2O\}_n$ (**1**), $[Ni(btb)_2(NCO)_2]_n$ (**2**), and $[Ni(btb)_2Cl_2]_n$ (**3**). Compound **1** is comprised of a twofold interpenetrating 4-connected $6^5 \cdot 8$-$CdSO_4$ 3-D coordination network. Compounds **2** and **3** are neutral 2-D (4,4) networks with the $ABAB \cdots$ and $ABCABC \cdots$ stacking modes, respectively. The thermal stabilities of **1**–**3** were investigated.

Keywords: 3-D $CdSO_4$ network; 4-Connected topology; Coordination polymer; Ni(II) complex; Bis(1,2,4-triazol-1-yl)butane

1. Introduction

Metal–organic frameworks (MOFs) have rapidly increased because of their intriguing topologies and potential applications as functional materials [1–8]. Ligands and metal centers are both key to the design and construction of MOFs with fascinating topology and physicochemical properties. Flexible ligands are employed in the construction of MOFs with a variety of architectures and topologies because flexible ligands can adopt different conformations according to the geometric needs of the different metal ions [9–14]. The anions serve more than merely balancing the charges of a cationic complex and influence the structure of a supramolecular system through coordination to the metal [15–23].

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 topologies [24–28]. Our synthetic approach starts by focusing on the construction of new topological frameworks and potential functional materials using flexible bis(triazole) ligands by adjusting lengths and flexibilities [29–38]. 1,4-Bis(1,2,4-triazol-1-yl)butane (btb) is a longer and flexible ligand, which can adopt different conformations with respect to the relative orientations

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of the CH_2 groups. MOFs containing btb exhibit a variety of architectures and topologies [19, 39–44].

In order to extend our work on the synthesis of topological frameworks and investigate the influence of the anions on the coordination networks of flexible ligand, in this work, we synthesize three nickel(II) coordination polymers $\{[Ni(btb)_2(NCS)_2] \cdot H_2O\}_n$ (**1**), $[Ni(btb)_2(NCO)_2]_n$ (**2**), and $[Ni(btb)_2Cl_2]_n$ (**3**) by the reaction of 1,4-bis(1,2,4-triazol-1-yl)butane (btb) and nickel salts. Compound **1** consists of a relatively rare twofold interpenetrating 4-connected $CdSO_4$ 3-D network. Compounds **2** and **3** are neutral 2-D (4,4) networks. Here we report the syntheses, crystal structures, and thermal stabilities.

2. Experimental

2.1. *General procedures*

2.1.1. Materials and general methods. All reagents were of analytical grade and used without purification. 1,4-Bis(1,2,4-triazol-1-yl)butane (btb) was synthesized according to the literature method [45]. 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 from 4000 to 400 cm^{-1}. TGA analyses were measured on a Thermal Analyst 2100 TA Instrument and SDT 2960 Simultaneous TGA-DTA Instrument in flowing dinitrogen at heating rate 10°C min^{-1}.

2.2. *Synthesis of* $\{[Ni(btb)_2(NCS)_2] \cdot H_2O\}_n$ *(1)*

A 10 mL aqueous solution of $Ni(NO_3)_2 \cdot 6H_2O$ (0.029 g, 0.1 mmol) and KSCN (0.039 g, 0.4 mmol) was added to a tube. Then 10 mL 1 : 1 (v/v) $H_2O : CH_3OH$ was slowly added to the tube. Finally, 10 mL CH_3OH solution of 1,4-bis(1,2,4-triazol-4-yl)butane (btb) (0.038 g, 0.2 mmol) was slowly added. Crystals of **1** were obtained after 1 month at room temperature. Yield: 0.047 g (47% based on btb). Anal. Calcd for $C_{18}H_{26}N_{14}NiOS_2$ (**1**) (%): C, 37.45; H, 4.54; N, 33.97. Found(%): C, 37.38; H, 4.45; N, 33.91. IR data (cm^{-1}): 3460m, 2010vs, 1616w, 1526 s, 1450w, 1348w, 1288 m, 1210w, 1135 s, 1017w, 993 m, 910w, 883w, 794w, 681 m, 650w, 468w.

2.3. *Synthesis of* $[Ni(btb)_2(NCO)_2]_n$ *(2)*

The synthetic procedure of **2** was similar to the synthesis of **1**, except that KOCN (0.033 g, 0.4 mmol) was used instead of KSCN (0.039 g, 0.4 mmol). Yield: 0.030 g (56% based on btb). Anal. Calcd for $C_{18}H_{24}N_{14}NiO_2$ (**2**) (%): C, 41.07; H, 4.59; N, 37.20. Found (%): C, 41.04; H, 4.53; N, 37.15. IR data (cm^{-1}): 3141w, 2184vs, 1644w, 1528 s, 1482w, 1374 m, 1312w, 1281 m, 1204w, 1127 s, 1050w, 10110 m, 988 m, 903w, 864w, 756w, 679 s, 641 m, 453w.

Table 1. Crystal data and structural refinement for **1**, **2**, and **3**.

	1	**2**	**3**
Formula	$C_{18}H_{26}N_{14}NiOS_2$	$C_{18}H_{24}N_{14}NiO_2$	$C_{16}H_{24}Cl_2N_{12}Ni$
Formula weight	577.36	527.22	514.08
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2/c$	$P2_1/c$	$P2_1/n$
Temperature (K)	293(2)	293(2)	223(2)
Unit cell dimensions (Å, °)			
a	8.906(2)	7.623(3)	7.444(2)
b	9.613(2	17.710(6)	17.715(5)
c	15.530(4)	8.791(3)	8.760(3)
β	90.341(6)	111.214(6)	115.490(5)
Volume (Å^3), Z	1329.6(6), 2	1106.3(7), 2	1042.7(6), 2
Calculated density (g cm^{-3})	1.442	1.583	1.637
Absorption coefficient (mm^{-1})	0.927	0.928	1.220
F(000)	600	548	532
Reflections collected	12,735	10,215	9615
Independent reflection	2437 [R(int)=0.0781]	2016 [R(int)=0.0322]	1888 [R(int)=0.0257]
Parameters	187	160	142
Goodness-of-fit on F^2	1.037	1.072	1.046
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0723$, $wR_2 = 0.2052$	$R_1 = 0.0392$, $wR_2 = 0.2052$	$R_1 = 0.0321$, $wR_2 = 0.2052$

2.4. *Synthesis of [Ni(btb)$_2$Cl$_2$]$_n$ (3)*

The synthetic procedure of **3** was similar to the synthesis of **1**, except that $NiCl_2 \cdot H_2O$ (0.015 g, 0.1 mmol) was used instead of $Ni(NO_3)_2 \cdot 6H_2O$ (0.029 g, 0.1 mmol) and KSCN (0.039 g, 0.4 mmol). Yield: 0.033 g (64% based on btb). Anal. Calcd for $C_{16}H_{24}Cl_2N_{12}Ni$ (**3**) (%): C, 37.38; H, 4.71; N, 32.70. Found (%): C, 37.32; H, 4.65; N, 32.67. IR data (cm^{-1}): 3128w, 1528 s, 1478w, 1382w, 1201 m, 1127 s, 987 m, 911w, 872w, 749w, 679 m, 648w, 471w.

2.5. *X-ray structure determination*

X-ray crystallography suitable for single crystals of **1–3** were carefully selected under an optical microscope and glued to thin glass fibers. The diffraction data were collected on a Rigaku Mercury or Saturn CCD diffractometer with graphite monochromated Mo-Kα radiation (λ = 0.71070 or 0.71075 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) [46]. The parameters of the crystal data collection and refinement of **1–3** are given in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

The crystal structure of **1** shows a 4-connected $6^5 \cdot 8$-$CdSO_4$ 3-D coordination network. Each Ni(II) is six coordinate by four triazole nitrogen atoms from four btb ligands and two nitrogen atoms from two NCS^- (figure 1). The asymmetric unit contains half

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

1			
Ni(1)–N(3)	2.104(4)	Ni(1)–N(6)	2.099(4)
Ni(1)–N(7)	2.051(5)		
N(6)–Ni(1)–N(3)	87.27(16)	N(7)–Ni(1)–N(3)	89.08(17)
N(7)–Ni(1)–N(6)	90.23(18)		
2			
Ni(1)–N(3)	2.109(2)	Ni(1)–N(6)^{ii}	2.124(2)
Ni(1)–N(7)	2.091(2)		
N(3)–Ni(1)–N(6)^{ii}	91.17(8)	N(7)–Ni(1)–N(3)	90.15(8)
N(7)–Ni(1)–N(6)^{ii}	89.00(9)		
3			
Ni(1)–N(3)	2.0150(18)	Ni(1)–N(6)^{ii}	2.0403(18)
Ni(1)–Cl(1)	2.7808(8)		
N(3)–Ni(1)–N(6)^{ii}	90.91(7)	N(3)–Ni(1)–Cl(1)	88.24(5)
N(6)^{ii}–Ni(1)–Cl(1)	90.14(6)		

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

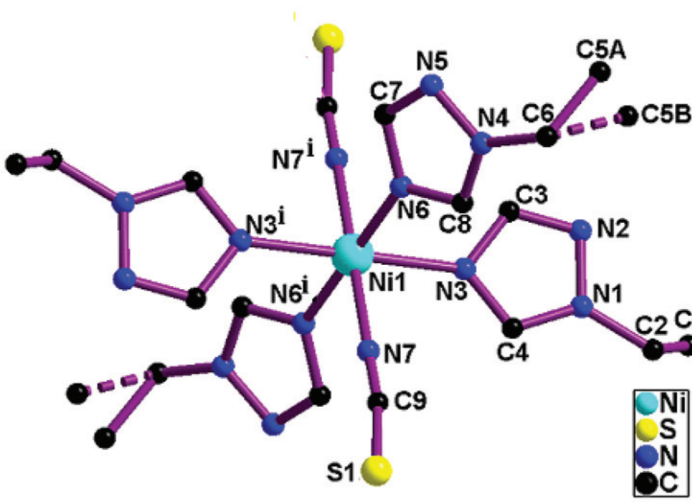

Figure 1. The coordination environment of Ni(II) in **1** (symmetry code: $^{i}-x, -y+1, -z$).

Ni(II), one NCS^-, two halves of btb, and half H_2O. The butane group (C5A and C5B) of one btb ligand, S (S1A and S1B) of NCS^- anion, and H_2O (O1) are disordered. Each btb exhibits *gauche*-*anti*-*gauche* conformation and bridges two Ni(II) atoms with the Ni⋯Ni distances of 13.105(2) and 11.851(2) Å. Each Ni(II) connects four adjacent Ni(II)'s through four btb ligands (Ni(II) is 4-connected) and extend to build a 3-D 4-connected $6^5\cdot 8$-$CdSO_4$ coordination network (figure 2). MOFs exhibiting $6^5\cdot 8$-$CdSO_4$ coordination network are relatively few [47–49]. Because single 3-D $CdSO_4$ network has large voids, it allows one identical $CdSO_4$ network to interpenetrate giving rise to a twofold interpenetrating network (figure 3), with disordered H_2O molecules occupying the gap.

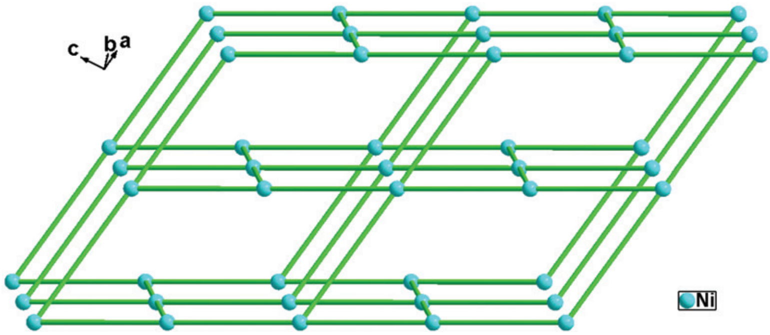

Figure 2. Schematic representation of the 3-D $CdSO_4$ topology of **1**. The sticks represent the btb ligands.

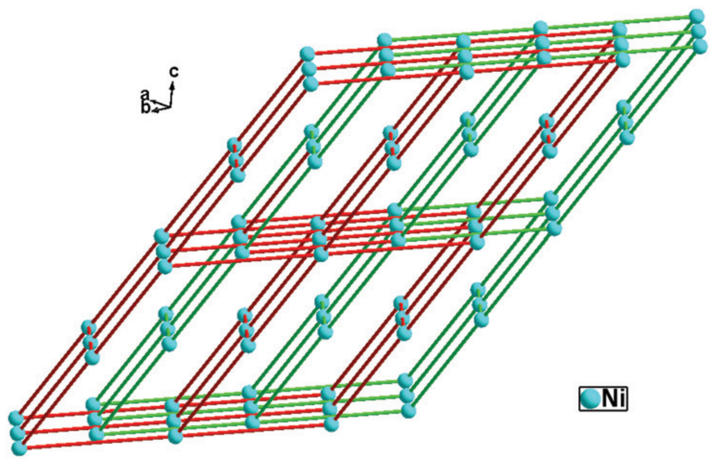

Figure 3. Schematic representation of the twofold interpenetrating 3-D $CdSO_4$ topology of **1**. The sticks represent btb.

Compounds **2** and **3** are neutral 2-D (4,4) networks. The Ni(II) is coordinated by four nitrogens from four btb ligands in the equatorial plane and two nitrogens from two NCS^- for **2** (figure 4) and two Cl^- for **3** (figure 5) in the axial positions. Each btb exhibits the completely *anti* (*anti*-*anti*-*anti*) conformation. Each Ni(II) is bridged by four btb to form a neutral 2-D (4,4) network with a $Ni \cdots Ni$ distance of 13.420(3) Å for **2** (figure 6) and 13.425(3) Å for **3** (figure 7). The network contains square grids (44-membered ring), with a Ni(II) at each corner and a btb at each edge connecting two Ni(II)'s. The 2-D (4,4) networks for **2** stack with $ABAB \cdots$ mode along the *c* direction with inter-sheet distance of half the *c*-axis translation (4.396(3) Å) (figure 8). However, the 2-D (4,4) networks for **3** parallel stack with $ABCABC \cdots$ mode along the *c* direction with the inter-sheet distance of one-third the *c*-axis translation (2.920(3) Å) (figure 9). The NCO^- and Cl^- of one sheet project into the holes of the next sheet.

MOFs containing btb exhibit a variety of architectures and topologies. The structural variety depends on the counter anions and co-ligands [19, 39–44]. For example, the

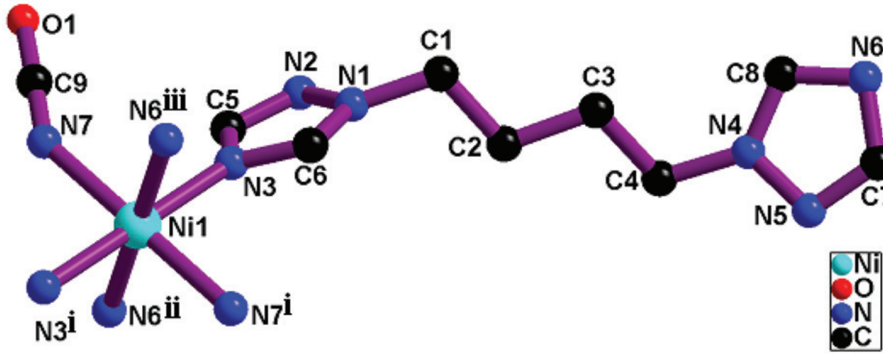

Figure 4. The coordination environment of Ni(II) in **2** (symmetry codes: $^{i}-x+1$, $-y+1$, $-z$; $^{ii}x+1$, $-y+1/2$, $z-1/2$; $^{iii}-x$, $y+1/2$, $-z+1/2$).

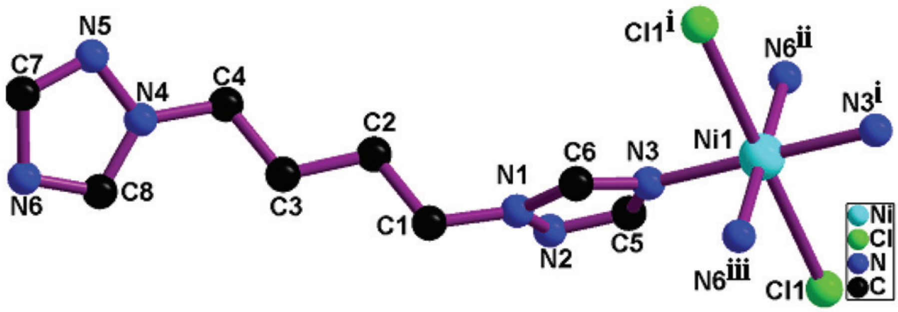

Figure 5. The coordination environment of Ni(II) in **3** (symmetry codes: $^{i}-x+1$, $-y$, $-z$; $^{ii}x-3/2$, $-y+1/2$, $z-1/2$; $^{iii}-x+5/2$, $y-1/2$, $-z+1/2$).

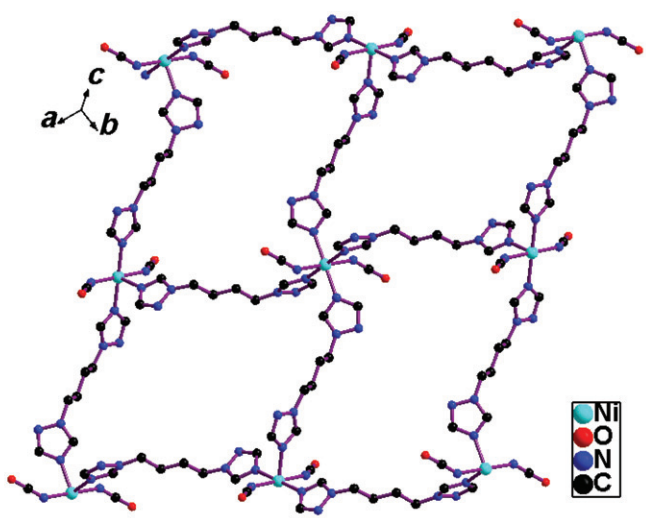

Figure 6. 2-D network of **2**.

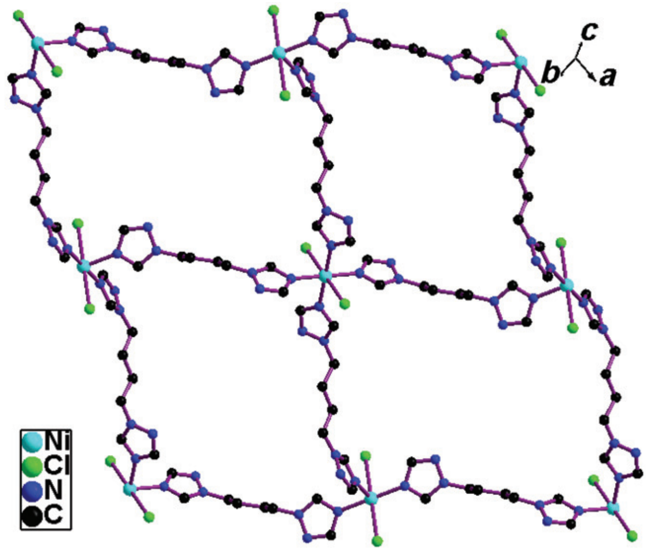

Figure 7. 2-D network of **3**.

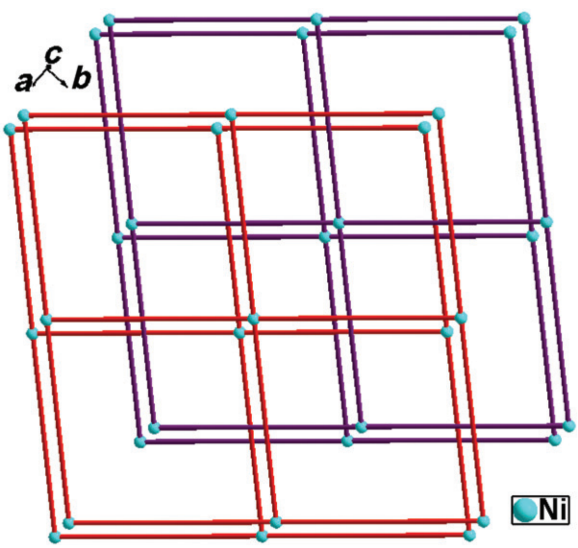

Figure 8. Schematic representation of the $ABAB\cdots$ parallel stacking mode of **2**. The long sticks represent btb.

cadmium MOFs with different anions $[Cd(btb)(H_2O)_2(NO_3)_2]_n$, $\{[Cd(btb)_2(H_2O)_2](BF_4)_2\}_n$, $\{[Cd(btb)_3](ClO_4)_2\}_n$, and $\{[Cd(btb)_3](PF_6)_2\}_n$ show 1-D chain, threefold interpenetrated diamondoid network and threefold interpenetrated α-polonium cubic network, respectively [19]. The zinc MOFs, $[Zn(btb)(H_2O)_3(SO_4)]_n$ has a 1-D chain structure and $\{[Zn(btb)_2(H_2O)_2](NO_3)_2 \cdot 2H_2O\}_n$ has an undulated 2-D (4,4) network while $\{[Zn(btb)_3](BF_4)_2\}_n$ and $\{[Zn(btb)_3](ClO_4)_2\}_n$ exhibit similar

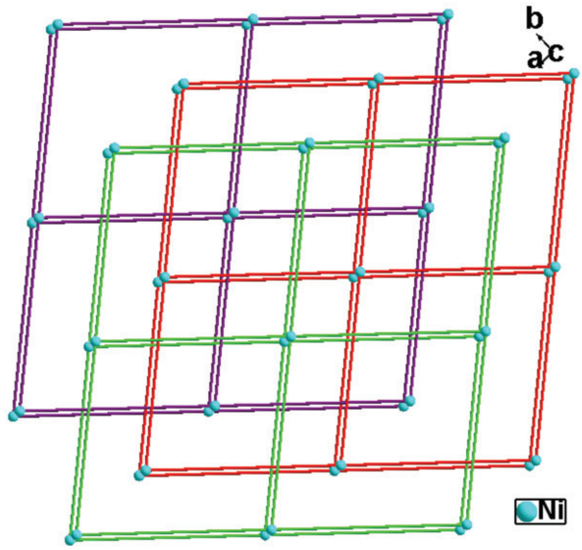

Figure 9. Schematic representation of the *ABCABC*··· parallel packing mode of **3**. The long sticks represent btb.

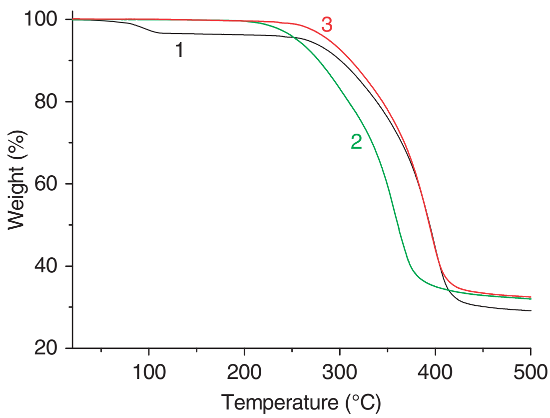

Figure 10. The TG curves of **1**–**3**.

threefold interpenetrating α-polonium cubic networks [43]. $[Mn(btb)_2(NCS)_2]_n$ has different interpenetrated network structures composed of 1-D and 2-D with the same chemical composition [44]. However, the 4-connected $6^5 \cdot 8$-$CdSO_4$ 3-D coordination network of btb complex was not observed previously.

TG experiments were carried out to explore thermal stabilities (figure 10). In the TG curve of **1**, the lattice water was lost from 50°C to 116°C. The remaining substance is thermally stable to 230°C. Compounds **2** and **3** were stable to 210°C and 232°C, respectively. Then **2** and **3** display a rapid decomposition from 250°C to 410°C.

4. Conclusion

The self-assembly reaction of 1,4-bis(1,2,4-triazol-1-yl)butane (btb) and different Ni(II) salts gives three coordination polymers $\{[Ni(btb)_2(NCS)_2]\cdot H_2O\}_n$ (**1**), $[Ni(btb)_2(NCO)_2]_n$ (**2**), and $[Ni(btb)_2Cl_2]_n$ (**3**). Ni(II) is 4-connected by four btb in **1**–**3**. However, **1** is composed of a twofold interpenetrating $6^5\cdot 8$-$CdSO_4$ 3-D coordination network. Compounds **2** and **3** are similar neutral 2-D (4,4) networks. The 2-D sheets of **2** stack with $ABAB\cdots$ stacking mode with relative long inter-sheet distance (4.396(3) Å). The 2-D sheets of **3** stack with $ABCABC\cdots$ stacking mode with relative short inter-sheet distance (2.930(3) Å). The different stacking modes of **2** and **3** may be due to relatively long linear NCO^- and short single Cl^-. The btb ligands show the *gauche–anti–gauche* conformation in **1** and the completely *anti* (*anti–anti–anti*) conformation in **2** and **3**. These results show that NCS^-, NCO^-, and Cl^- play key roles in the formation of **1**–**3**. Further syntheses and structural studies of coordination polymer with flexible triazole ligands are under way in our laboratory.

Supplementary material

CCDC-818974, 818975, 818976 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif

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