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Syntheses and structures of three new coordination polymers with the flexible 1,2-bis(1,2,4-triazol-1-yl)ethane ligand

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Three new coordination polymers { $[Ni(bte)_2(NCO)_2](H_2O)\}_n$ (1), { $[Ni(bte)_2(N_3)_2](H_2O)\}_n$ (2) and { $[Ag(bte)_2](NO_3)\}_n$ (3) (bte = 1,2-bis(1,2,4-triazol-1-yl)ethane) were synthesized and characterized by X-ray crystallography, IR and thermogravimetric analysis. The coordination geometry of the Ni(II) atom is distorted octahedral, coordinated equatorially by four nitrogen atoms from four triazole rings of four symmetry-related bte ligands, and axially by two nitrogen atoms from two isocyanate anions in 1 and azide anions in 2, respectively. The structures of 1 and 2 are both polymeric, with 18-membered spiro-fused rings and each 18-membered ring involving two inversion-related bte molecules. The coordination geometry of the Ag(I) atom in 3 is distorted tetrahedral, formed by four nitrogen atoms from four triazole rings of four symmetry-related bte ligands. Each bte ligand links two Ag(I) atoms and forms a two-dimensional undulated (4,4) network in 3.

Keywords: Nickel complex; Silver complex; Crystal structure; Coordination polymer; Bis(triazole) ligand

1. Introduction

The design and synthesis of metal-organic framework structures have been intensely studied for their unexpected properties for potential application as functional materials as well as the intriguing architectures and topologies [1–6]. The structural motifs of coordination polymers depend on the coordination geometry of the central atom, the geometry and the flexibility of the ligand molecules, the coordinated (and/or non-coordinated) counter ions and the reaction conditions. When a linear rigid bidentate ligand is used as spacer to connect metal centers, the topology of the coordination network is usually determined by the coordination geometry of the central atom and the geometry of the ligand. Contrary to the rigid spacers, flexible ligands, which can

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adopt various conformations, may induce coordination polymers with novel topologies. Rigid rod-like N-donor ligands have been employed and a variety of topological architectures have been synthesized [7-10]. However, coordination polymers with flexible triazole ligands have not been well studied to date [11-15].

In the design and preparation of functional coordination frameworks, we found that besides the linear pyridyl-like ligands, the flexible ligand 1,2-bis(1,2,4-triazol-1-yl)ethane [16] is also an effective organic bridging ligand to construct coordination frameworks [17-21]. In this work, three novel coordination polymers $\{[Ni(bte)_2 (NCO)_2](H_2O)\}_n$ (1), $\{[Ni(bte)_2(N_3)_2](H_2O)\}_n$ (2) and $\{[Ag(bte)_2](NO_3)\}_n$ (3) were synthesized and characterized by using 1,2-bis(1,2,4-triazol-1-yl)ethane (bte).

2. Experimental

2.1. Materials and physical measurements

All reagents were of analytical grade and used without further purification. 1,2-bis(1,2,4-triazol-1-yl)ethane (bte) was prepared following the literature method [22]. Elemental analyses for C, H and N were performed on a Perkin-Elmer 240C analyser. IR spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectro-photometer in the 4000–400 cm⁻¹ region. Thermogravimetric data were collected on Thermal Analyst 2100 TA Instrument and SDT 2960 Simutaneous TGA-DTA Instrument in flowing dinitrogen at a heating rate 10° C min⁻¹.

Caution: The azide salts of the metal complexes with organic ligands are potentially explosive. Only a small amount of the material should be prepared and handled carefully.

2.2. Synthesis of $\{[Ni(bte)_2(NCO)_2](H_2O)\}_n$ (1)

A methanolic solution (15 mL) of 1,2-bis(1,2,4-triazol-1-yl)ethane (bte) (0.082 g, 0.5 mmol) was added slowly to an aqueous solution (15 mL) of Ni $(NO_3)_2 \cdot 6H_2O$ (0.145 g, 0.5 mmol) and NaNCO (0.088 g, 1.4 mmol) with stirring. The resulting solution was filtered and the filtrate was allowed to stand in air at room temperature. After several days, well-shaped blue single crystals of 1 were obtained. Yield: 0.093 g (76%). Found: C, 34.41; H, 3.72; N, 39.94. Calcd for C₁₄H₁₈N₁₄NiO₃ (1): C, 34.38; H, 3.71; N, 40.10%. IR absorption bands (KBr, cm⁻¹): 3532m, 3482m, 3427m, 3136m, 2188sh, 2169vs, 1668w, 1526s, 1440m, 1353w, 1318w, 1288m, 1209w, 1131s, 1014m, 989m, 892w, 676m, 646m, 627w and 511w cm⁻¹.

2.3. Synthesis of $\{[Ni(bte)_2(N_3)_2](H_2O)\}_n$ (2)

A 25 mL H₂O/MeOH solution (1:1, v/v) of bte (0.082 g, 0.5 mmol) was added to one leg of a H-shape tube and 25 mL of a H₂O/MeOH solution (1:1, v/v) of Ni(NO₃)₂·6H₂O (0.145 g, 0.5 mmol), NaN₃ (0.150 g, 2.5 mmol) was added to the other leg of the tube. The tube was allowed to stand in air at room temperature for about one month. The pale blue single crystals **2** suitable for X-ray analysis were obtained. Yield: 0.083 g (68%). Found: C, 29.34; H, 3.62; N, 51.43. Calcd for $C_{12}H_{18}N_{18}NiO$ (**2**): C, 29.47; H, 3.71; N, 51.55%. IR absorption bands (KBr, cm⁻¹): 3472m, 3372m, 3136m, 2051sh, 2033vs, 1667w, 1528s, 1439w, 1385w, 1354w, 1289m, 1211w, 1130s, 1015m, 992m, 903w, 675m, 644w and 513w cm⁻¹.

2.4. Synthesis of $\{[Ag(bte)_2](NO_3)\}_n$ (3)

A methanolic solution (15 mL) of bte (0.082 g, 0.5 mmol) was added slowly to 15 mL H₂O/CH₃CN (1:1, v:v) solution of Ag(NO₃) (0.043 g, 0.25 mmol) with stirring. The resulting solution was filtered and the filtrate was allowed to stand in air at room temperature. Colorless single crystals of **3** were obtained after one week. Yield: 0.058 g (47%). Found: C, 28.81; H, 3.16; N, 36.42. Calcd for C₁₂H₁₆AgN₁₃O₃ (**3**): C, 28.93; H, 3.24; N, 36.55%. IR absorption bands (KBr, cm⁻¹): 3106m, 1628w, 1524s, 1462m, 1385vs, 1281s, 1215m, 1134s, 1015m, 992w, 884m, 826w, 702w, 675m and 652w cm⁻¹.

2.5. Crystal structure determination

The single crystals 1, 2 and 3 with the approximate dimensions $0.60 \times 0.59 \times 0.31 \text{ mm}^3$, $0.30 \times 0.15 \times 0.10 \text{ mm}^3$ and $0.43 \times 0.39 \times 0.30 \text{ mm}^3$ were selected for lattice parameter determination and collection of the intensity data using a Rigaku Mercury CCD diffractometer with graphite monochromated Mo K_{α} radiation ($\lambda = 0.71073 \text{ Å}$). Intensities were collected by the ω scan technique. The structures were solved by direct methods and refined with the full-matrix least-squares technique (SHELXTL-97) [23]. The positions of the hydrogen atoms of the carbon atoms were calculated. The parameters for the crystal data collection and refinement of complexes 1, 2 and 3 are given in table 1. Bond lengths and angles are shown in table 2.

3. Results and discussion

The IR spectrum shows that the triazole ring vibrations in complexes 1, 2 and 3 are at 1526 and 1288, 1528 and 1289, 1524 and 1281 cm⁻¹, respectively [12, 13]. For comparison the triazole ring vibrations for the free bte ligand [16] are at 1516 and 1273 cm⁻¹. The strong absorption bands at 2188 and 2169 cm⁻¹ in 1 are attributed to two terminal N-bound NCO⁻ anions. The strong absorption bands at 2051 and 2033 cm⁻¹ in 2 are attributed to two terminal N-bound N₃⁻ anions. The very strong absorption band in 3 at 1385 cm⁻¹ is assigned to free NO₃⁻ anion.

Thermogravimetric analysis of **1** shows that the lattice water molecule was lost in a continuous fashion (Obsd. 3.9; Calcd 3.7%) from 40 to 120° C, then one bte molecule was lost from 206 to 302° C (Obsd. 33.8; Calcd 33.5%). When the temperature reached 486°C, the residue (14.9%) is assigned to NiO (Calcd 15.3%). Thermogravimetric analysis of **2** shows that the lattice water molecule was lost in a continuous fashion (Obsd. 3.8; Calcd 3.7%) from 40 to 120° C, then the weight was constant up to 230° C. When the temperature reached 236° C, an explosion occurred. Thermogravimetric analysis of **3** shows that **3** was stable up to 203° C, and began to decompose above this temperature.

Complexes 1 and 2 are isomorphous and thus have almost identical infinite, double-stranded chain structures. Complex 3 is composed of a two-dimensional

Complexes	1	2	3
Empirical formula	C ₁₄ H ₁₈ N ₁₄ NiO ₃	C ₁₂ H ₁₈ N ₁₈ NiO	C ₁₂ H ₁₆ AgN ₁₃ O ₃
Formula weight	489.13	489.15	498.25
Temperature [K]	193.15	193.15	193.15
Wavelength [Å]	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/m$	$P2_1/m$	$P\overline{1}$
a [Å]	7.4101(10)	7.578(2)	8.4098(19)
b [Å]	14.7889(18)	14.588(4)	10.052(2)
c [Å]	9.8821(14)	9.793(3)	11.328(3)
α [°]	90.00	90.00	97.094(4)
β [°]	109.491(3)	110.358(7)	99.516(5)
γ [°]	90.00	90.00	95.481(5)
V [Å ³]	1020.9(2)	1015.0(5)	930.6(3)
Z	2	2	2
F(000)	504	504	500
$\rho \left[\text{g cm}^{-3} \right]$	1.591	1.601	1.778
$\mu [\mathrm{mm}^{-1}]$	1.002	1.006	1.130
Crystal size (mm)	$0.60 \times 0.59 \times 0.31$	$0.30 \times 0.15 \times 0.10$	$0.43 \times 0.39 \times 0.30$
θ range for data collection (°)	3.00 to 27.48	3.27 to 27.48	3.02 to 27.48
Index ranges	$-9 \le h \le 9,$ $-16 \le k \le 19,$ $-12 \le l \le 12$	$-9 \le h \le 9,$ $-15 \le k \le 18,$ $-12 \le l \le 12$	$-10 \le h \le 10,$ $-13 \le k \le 12,$ $-14 \le l \le 11$
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Independent reflections	2429 $[R(int) = 0.0172]$	2325 [R(int) = 0.0595]	4163 [R(int) = 0.0235]
Goodness of fit S	1.079	1.094	1.084
Final R_1 and wR_2 [$I > 2\sigma(I)$]	0.0350, 0.0943	0.0739, 0.1275	0.0295, 0.0646
R_1 and wR_2 indices (all data)	0.0371, 0.0962	0.0900, 0.1341	0.0326, 0.0658
Largest diff. Peak and hole $(e \text{ Å}^{-3})$	0.729 and -0.459	0.563 and -0.379	0.348 and -0.664

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

undulated (4,4) network. 1,2,4-triazole and its derivatives are versatile ligands [11]. Non-substituted 1,2,4-triazole ligand either prefers monodentate coordination through N1 or more frequently observed, bidentate bridging mode N1, N2 [11]. The unusual tridentate bridging mode N1, N2, N4 for the deprotonated 1,2,4-triazole were also observed [24, 25]. However the flexible ligand 1,2-bis(1,2,4-triazol-1-yl)ethane (bte) prefers the N4 coordination mode to the N1 coordination mode in complexes 1, 2, 3 and all other complexes containing the bte ligand due to the steric hinderance for the N1 atoms [17–21].

Figures 1 and 2 show the local coordination of the Ni(II) atom in 1 and 2, respectively. The coordination geometry of the Ni(II) atom is distorted octahedral, formed equatorially by four nitrogen atoms at the 4-position of the triazole rings of four symmetry-related bte ligands [Ni(1)–N(3) 2.1154(13) Å, Ni(1)–N(6C) 2.1185(13) Å in 1; Ni(1)–N(3) 2.100(3) Å, Ni(1)–N(6B) 2.108(3) Å in 2], and axially by two nitrogen atoms from two isocyanato ligands [Ni(1)–N(7) 2.100(2) Å, Ni(1)–N(8) 2.089(2) Å] in 1 and azido ligands [Ni(1)–N(7) 2.133(5) Å, Ni(1)–N(10) 2.104(5) Å] in 2. The isocyanato and azido ligands are both monodentate N-bound ligands. The isocyanato ligand in 1 is quasi-linear as expected [the N–C–O bond angles are 177.7(3) and 178.6(3)°] [26]. The Ni–N–C (NCO) bond angles are 136.17(19) and 138.21(19)°. The azido ligand in 2 is also quasi-linear as expected [the N–N–N bond angles]

Complex 1				
Ni(1)–N(3)	2.1154(13)	Ni(1)-N(3A)	2.1154(13)	
Ni(1)–N(6C)	2.1185(13)	Ni(1)–N(6B)	2.1185(13)	
Ni(1)–N(7)	2.100(2)	Ni(1)–N(8)	2.089(2)	
N(3)–Ni(1)–N(3A)	93.22(7)	N(3)-Ni(1)-N(6C)	179.28(5)	
N(3)-Ni(1)-N(6B)	86.78(6)	N(6B)-Ni(1)-N(6C)	93.22(7)	
N(3)–Ni(1)–N(7)	91.00(6)	N(3)–Ni(1)–N(8)	89.14(6)	
N(6C)-Ni(1)-N(7)	89.72(5)	N(6C)-Ni(1)-N(8)	90.14(6)	
N(7)–Ni(1)–N(8)	179.79(7)	N(7)-C(7)-O(1)	177.7(3)	
N(8)-C(8)-O(2)	178.6(3)	Ni(1)-N(7)-C(7)	136.17(19)	
Ni(1)-C(8)-C(8)	138.21(19)			
	Comp	lex 2		
Ni(1)–N(3)	2.100(3)	Ni(1)–N(3A)	2.100(3)	
Ni(1)-N(6B)	2.108(3)	Ni(1)–N(6C)	2.108(3)	
Ni(1)–N(7)	2.133(5)	Ni(1)–N(10)	2.104(5)	
N(3)–Ni(1)–N(3A)	92.67(18)	N(3)-Ni(1)-N(6B)	87.58(12)	
N(3)-Ni(1)-N(6C)	178.81(15)	N(6B)-Ni(1)-N(6C)	92.14(18)	
N(3)–Ni(1)–N(7)	91.61(13)	N(3)-Ni(1)-N(10)	88.49(13)	
N(6B)-Ni(1)-N(7)	87.22(13)	N(6B)-Ni(1)-N(10)	92.68(13)	
N(7)–Ni(1)–N(10)	179.85(19)	N(7)-N(8)-N(9)	178.4(6)	
N(10)-N(11)-N(12)	177.6(7)	Ni(1)–N(7)–N(8)	122.7(4)	
Ni(1)–N(10)–N(11)	122.3(4)			
	Comp	lex 3		
Ag(1)-N(3)	2.3096(19)	Ag(1)-N(6A)	2.3360(18)	
Ag(1)–N(9)	2.2333(18)	Ag(1) - N(12B)	2.364(2)	
N(3)-Ag(1)-N(6A)	89.84(7)	N(3)-Ag(1)-N(9)	127.11(7)	
N(3)-Ag(1)-N(12B)	102.35(7)	N(6A) - Ag(1) - N(9)	123.19(7)	
N(6A) - Ag(1) - N(12B)	105.99(7)	N(9)-Ag(1)-N(12B)	105.43(7)	

Table 2. Selected bond lengths [Å] and angles [°] for 1, 2 and 3; symmetry codes: 1 A x, -y+1/2, z; B x-1, y, z; C x-1, -y+1/2, z; 2 A x, -y+1/2, z; B x+1, y, z; C x+1, -y+1/2, z; 3 A x-1, y, z; B x, y-1, z.

are 178.4(6) and 177.6(7)°] [27, 28]. The Ni–N–N (N₃) bond angles are 122.7(4) and 122.3(4)°.

The Ni–N(triazole) bond lengths in the equatorial plane of **1** are in the range 2.1154(13)–2.1185(13) Å, close to the corresponding values [2.100(3)–2.108(3) Å] of **2**. While Ni–N bond lengths of axial positions of **1** are 2.089(2) and 2.100(2) Å, similar to the corresponding values of **2** [2.104(5) and 2.133(5) Å]. The Ni–N(triazole) distances of 2.100(3)–2.1185(13) Å in **1** and **2**, correspond to the values [2.095(2)–2.106(2) Å] reported in [Ni(trzppo)₄Cl₂] · 6H₂O (trzppo = β -(1,2,4-triazol-1-yl) propiophenone) [29].

Complexes 1 (figure 3) and 2 have almost identical infinite double-stranded chain structures. The bte ligand exhibits a *gauche* conformation in 1 and 2. The Ni(II) atoms are linked by bte ligands *via* the nitrogen atoms at the 4-position of the triazole, and a rhombic network is formed. The size of rhomb $6.4645(16) \text{ Å} \times 5.5264(16) \text{ Å}$ in 1 is very close to the size $6.492(4) \text{ Å} \times 5.537(4) \text{ Å}$ in 2. The r.m.s deviations of N(1)–N(3)/C(1)/C(2) and N(4)–N(6)/C(3)/C(4) triazole ring atoms from the mean planes of the rings are 0.0032(9) and 0.0020(10) Å in 1, and 0.0034(24) and 0.0013(22) Å in 2. The dihedral angle between two triazole ring planes of one bte is $57.05(5)^{\circ}$ in 1, close to the corresponding value $59.85(12)^{\circ}$ in 2. The bte ligand is twisted during coordination; the torsion angle N(1)–C(5)–C(6)–N(4) is $48.89(20)^{\circ}$ in 1, very close to the corresponding value $49.59(43)^{\circ}$ in 2. Two strands of the bte ligands are wrapped around one another and are held together by the Ni(II) atoms, forming the infinite



Figure 1. Local coordination of the Ni(II) in complex **1** with 30% thermal ellipsoids; H-atoms were omitted for clarity.



Figure 2. Local coordination of the Ni(II) in complex **2** with 30% thermal ellipsoids; H-atoms were omitted for clarity.



Figure 3. View of the infinite double-stranded chain in complex 1, showing the water molecules.

double-stranded chain structures in **1** and **2**. Each chain consists of 18-membered spirofused rings, in which two Ni(II) atoms are joined by two bte molecules. The chains in **1** and **2** extend along the *a* axis, and all of the Ni(II) atoms in one chain are on the axial line; the Ni–Ni separations across the bte ligands are equal to the *a*-axis translation [7.4101(10) Å for **1** and 7.578(2) Å for **2**], shorter than the metal–metal distances in related double-stranded chain complexes (8.369(4) Å in [Zn(bte)₂(dca)₂]_n (dca = dicyanamide) [19], 8.515(2) Å in [Mn(bte)₂(dca)₂]_n [21], 7.923(2) Å in [Mn(*gauche*-bte)₂(NCS)₂]_n of [Mn(*gauche*-bte)₂(NCS)₂]_n · [Mn(*anti*-bte)(H₂O)₂(NCS)₂]_n [21]), and obviously shorter than the values reported in the complexes with the *anti* conformation bte (12.521(2) Å in [Mn(*anti*-bte)(H₂O)₂(NCS)₂]_n of [Mn(*gauche*bte)₂(NCS)₂]_n · [Mn(*anti*-bte)(H₂O)₂(NCS)₂]_n [21] and 12.473(2) Å in [Cu(TTA)₂]₂(bte) (TTA = 1,1,1-trifluoro-3-(2-thenoyl)acetone) [18]).

Figure 4 shows the local coordination of the Ag(I) atom in 3. Each Ag(I) atom is four-coordinate with four nitrogen atoms at the 4-position of triazole rings of four symmetry-related bte ligands [Ag(1)-N(3) 2.3096(19) Å, Ag(1)-N(6A) 2.3360(18) Å,Ag(1)-N(9) 2.2333(18) Å, Ag(1)-N(12B) 2.364(2) Å in a distorted tetrahedron. The Ag–N bond lengths are in the range 2.2333(18)–2.364(2) Å, corresponding to values in the triazole complex $[Ag(btm)(NO_2)]$ (Ag–N 2.314(4)Å) and longer than those values in $[Ag(btm)(NO_3)]$ [Ag-N 2.163(2) and 2.164(2) Å] (btm = 1,2-bis(1,2,4-triazol-1-yl)methane) [12]. Each bridging bte ligand links two Ag(I) atoms and each Ag(I) atom bonds four bte ligands to form a two-dimensional (4,4) network containing $Ag_4(bte)_4$ units (figure 5). Two independent bte ligands both exhibit gauche conformation. The r.m.s deviations of N(1)-N(3)/C(1)/C(2), N(4)-N(6)/C(3)/C(4), N(7)-N(9)/C(4)C(7)/C(8) and N(10)-N(12)/C(9)/C(10) triazole ring atoms from the mean planes of the rings are 0.0029(13), 0.0027(13), 0.0010(13) and 0.0026(13) A, respectively. The bte ligand is twisted during coordination; the torsion angles N(1)-C(5)-C(6)-N(4)(ligand molecule A) is $-60.7(2)^{\circ}$ and N(7)–C(11)–C(12)–N(10) (ligand molecule B) is $82.5(2)^{\circ}$. The dihedral angles between two triazole ring planes of the same ligand molecule A and B are $42.03(10)^{\circ}$ and $57.96(8)^{\circ}$, respectively. The Ag \cdots Ag separation across the bridging bte ligand molecules A and B are 8.4098(19) and 10.052(2) A, respectively, corresponding to the a and b axis translations, respectively. The $Ag \cdots Ag$ distances for ligand A are about 1.6 Å shorter than those for ligand B, corresponding to the change of the bte ligand molecules from angular for A to more linear



Figure 4. Local coordination of the Ag(I) in complex 3 with 30% thermal ellipsoids; H-atoms were omitted for clarity.



Figure 5. The two-dimensional (4,4) network in 3 viewed along the *c*-axis.



Figure 6. Viewing the tightly packed structure in 3.

connectors for ligand B. The most intriguing feature of the structure is that the basic two-dimensional grid is puckered (figure 6), different from previously reported grid-like structures in which the basic grids are usually coplanar or quasi-coplanar [30, 31]. The puckered shape of the basic grid in **3** is understandable because the bte ligand exhibits the *gauche* conformation in **3**. There are weak C–H···O hydrogen bond interactions between the hydrogen atoms of the bte ligand and the oxygen of the nitrate anion [C(2)–H(2A)···O(1) (-x+1, -y, -z+1) 175.4°, H(2A)···O(1) (-x+1, -y, -z+1) 2.36 Å, C(2)···O(1) (-x+1, -y, -z+1) 3.307(3) Å; C(5)–H(5A)···O(3) (-x+1, -y, -z+1) 154.6°, H(5A)···O(3) (-x+1, -y, -z+1) 2.42 Å, C(5)···O(3) (-x+1, -y, -z+1) 3.346(3) Å].

Complexes 1, 2 and 3 all have metal-to-ligand ratio 1:2 and the bte ligands all have the *gauch*e conformation. Complexes 1 and 2 have almost identical double stranded chain structures. Complex 3 exhibits a two-dimensional undulating (4,4) network. These different structures arise from different coordination geometry of the central atom and the rotational freedom of the triazole rings of the bte ligand.

The two-dimensional (4,4) network of **3** is completely different from Ag(I) complexes with similar triazole ligands 1,2-bis(1,2,4-triazol-1-yl)methane (btm) such as [Ag(btm)(NO₃)] and [Ag(btm)(NO₂)] with metal-to-ligand ratio 1:1 irrespective of the metal-to-ligand ratio employed for synthesis. The little change of the length and flexibility of the flexible triazole ligands from btm to bte by replacing methane with ethane results in completely different coordination polymers [Ag(bte)₂](NO₃) (**3**) and [Ag(btm)(NO₃)] [12]. These results show that the properties of ligands play key roles in self-assembly of coordination polymers. The rationalization of the resulting topologies is still difficult and great effort is needed to predict the supramolecular chemistry of the flexible ligands.

Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (CCDC) (E-mail: deposit@ccdc.cam.ac.uk) as supplementary materials and the CCDC reference numbers for 1, 2 and 3 are 249733, 249734 and 260881, respectively.

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