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Three cobalt coordination polymers based on bis(1,2,4-triazol-1-ylmethyl)benzene positional isomer ligands and thiocyanate

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Three cobalt coordination polymers based on bis(1,2,4-triazol-1-ylmethyl)benzene positional isomer ligands and thiocyanate

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The reaction of three positional isomer ligands of bis(1,2,4-triazol-1-ylmethyl)benzene and $\text{Co}(\text{NCS})_2$ gives three coordination polymers $[\text{Co}(\text{obt})_2(\text{NCS})_2]_n$ (**1**), $[\text{Co}(\text{mbtz})_2(\text{NCS})_2]_n$ (**2**), and $[\text{Co}(\text{bbtz})_2(\text{NCS})_2] \cdot 2\text{DMF}$ (**3**). Polymers **1** and **2** are comprised of similar 1-D double chains. In **1**, each chain forms π - π stacking interactions with four adjacent chains (two above and two below) to extend to a 3-D supramolecular network. Polymer **3** is a neutral 2-D (4,4) network. The dangling NCS^- inserts into the window of adjacent layers in a mutual relationship and result in a 2-D \rightarrow 3-D polythreaded network in **3**. The thermal stability and the diffuse reflectance UV-Vis spectroscopy of **1**, **2**, and **3** were measured.

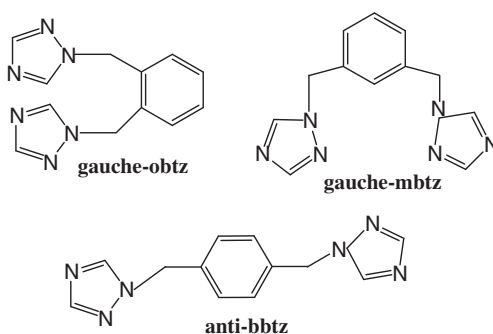
Keywords: Polythreaded; Positional isomer ligands; Coordination polymer; Co(II) complex; Bis(1,2,4-triazol-1-ylmethyl)benzene

1. Introduction

Interest in crystal engineering of coordination polymer frameworks stems from potential applications as functional materials and intriguing variety of topologies [1–5]. A strategy to construct such networks is to employ appropriate ligands that bond metal ions in different modes and provide a way to obtain intriguing architectures. When rigid bifunctional ligands are used as spacers to connect metal centers, the topology of the network is usually determined by the coordination geometry of the central metal. Contrary to rigid spacers, flexible ligands, which can adopt different conformations according to geometric needs of different metal ions, may induce coordination polymers with novel topologies [6–10]. Anions serve more than merely balancing the charges of a cationic complex and influence the structure of a supramolecular system through coordination [11–13]. Thiocyanate (SCN^-) is widely used to construct coordination polymers because of versatile coordination [14–17].

A number of mononuclear, oligonuclear, and polynuclear transition metal complexes of 1- and 4-substituted 1,2,4-triazole derivatives have been synthesized and characterized

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Scheme 1. The *gauche-obtz*, *gauche-mbtz*, and *anti-bbtz* ligands in **1**, **2**, and **3**.

with magnetic properties and novel topologies [18–24]. Our synthetic approach starts by focusing on construction of new topological frameworks and potential functional materials using flexible ligands [25–31]. Three ligands, 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (*obtz*) [32], 1,3-bis(1,2,4-triazol-1-ylmethyl)benzene (*mbtz*) [33], and 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene (*bbtz*) [34–37] (scheme 1), are positional isomers that can adopt different conformations. In order to extend our work and investigate the influence of the positions of triazole groups on structures of coordination polymers, in this work, we synthesized three cobalt(II) coordination polymers, $[\text{Co}(\text{obtz})_2(\text{NCS})_2]_n$ (**1**), $[\text{Co}(\text{mbtz})_2(\text{NCS})_2]_n$ (**2**), and $\{[\text{Co}(\text{bbtz})_2(\text{NCS})_2] \cdot 2\text{DMF}\}_n$ (**3**), by reaction of $\text{Co}(\text{NCS})_2$ and three bis(1,2,4-triazol-1-ylmethyl)benzene positional isomers. Here we report syntheses, crystal structures, thermal stabilities, and diffuse reflectance UV-Vis spectroscopies of the solids.

2. Experimental

2.1. General procedures

2.1.1. Materials and general methods. All reagents were of analytical grade and used without purification. Three bis(1,2,4-triazol-1-ylmethyl)benzene positional isomers were synthesized according to literature methods [38]. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240 C analyser. IR spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectrophotometer from 4000 to 400 cm^{-1} . TGA was carried out using a Thermal Analyst 2100 TA Instrument and SDT 2960 Simultaneous TGA-DTA Instrument in flowing dinitrogen at 10 $^\circ\text{C min}^{-1}$. Diffuse reflectance UV-Vis spectra for the solid samples were recorded on a Lambda 900 spectrometer from 200 to 1200 nm.

2.2. Synthesis of $[\text{Co}(\text{obtz})_2(\text{NCS})_2]_n$ (**1**)

A 10 mL aqueous solution of $\text{Co}(\text{SCN})_2$ (0.2 mmol) was added to a tube. Then 10 mL 1 : 1 (v/v) $\text{H}_2\text{O}/\text{CH}_3\text{OH}$ was slowly added to the tube. Finally, 10 mL CH_3OH solution

of 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obt) (0.4 mmol) was slowly added to the tube. Pink block-shaped crystals of **1** were obtained after two weeks at room temperature. Anal. Calcd for $C_{26}H_{24}CoN_{14}S_2$ (**1**) (%): C, 47.63; H, 3.69; N, 29.92. Found: C, 47.61; H, 3.66; N, 29.88. IR data (cm^{-1}): 3156w, 2069vs, 1520m, 1348w, 1288m, 1210m, 1112m, 990 m, 908w, 873w, 681 m, 650w, 468w.

2.3. Synthesis of $[Co(mbtz)_2(NCS)_2]_n$ (**2**)

The synthetic procedure of **2** was similar to the synthesis of **1**, except that 1,3-bis(1,2,4-triazol-1-ylmethyl)benzene (mbtz) (0.4 mmol) was used instead of 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obt). Anal. Calcd for $C_{26}H_{24}CoN_{14}S_2$ (**2**) (%): C, 47.63; H, 3.69; N, 29.92. Found: C, 47.56; H, 3.63; N, 29.86. IR data (cm^{-1}): 3141w, 2184vs, 1521s, 1312w, 1282m, 1204w, 1127m, 1015m, 989m, 903w, 864w, 756w, 679s, 641m.

2.4. Synthesis of $\{[Co(bbtz)_2(NCS)_2] \cdot 2DMF\}_n$ (**3**)

A 5 mL DMF solution of $Co(SCN)_2$ (0.5 mmol) was added to a DMF solution (5 mL) of 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene (bbtz) (1.0 mmol). Orange block-shaped crystals of **3** were obtained after one month at room temperature. Anal. Calcd for $C_{32}H_{38}CoN_{16}O_2S_2$ (**3**) (%): C, 47.93; H, 4.78; N, 27.96. Found: C, 47.84; H, 4.71; N, 28.04. IR data (cm^{-1}): 3114w, 2092s, 1656s, 1522m, 1387m, 1343w, 1282m, 1208w, 1130m, 1017m, 988 m, 876w, 676 m, 624 m.

2.5. X-ray structure determination

Suitable single crystals of **1**, **2**, and **3** were carefully selected under an optical microscope and glued to thin glass fibers. The diffraction data were collected on a Rigaku Mercury CCD diffractometer with graphite monochromated Mo- $K\alpha$ radiation. Intensities were collected by the ω -scan technique. The structures were solved by direct methods and refined with full-matrix least-squares (SHELXTL) [39]. The parameters of 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

3.1. Crystal structures

The crystal structures of **1** and **2** show that **1** and **2** have similar 1-D double chain structures. Due to the effect of the positional isomer ligands, **1** crystallizes in the triclinic space group $P\bar{1}$ and **2** crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit contains half Co(II), one NCS^- , one obtz in **1**, and one mbtz in **2**. The coordination geometry of Co(II) is a distorted octahedron, coordinated by four triazole nitrogen atoms from four obtz or mbtz ligands and two nitrogen atoms from two NCS^- (figures 1a and 2a).

Table 1. Crystallographic data for 1–3.

	1	2	3
Empirical formula	C ₂₆ H ₂₄ CoN ₁₄ S ₂	C ₂₆ H ₂₄ CoN ₁₄ S ₂	C ₃₂ H ₃₈ CoN ₁₆ O ₂ S ₂
Formula weight	655.64	655.64	801.83
Crystal system	Triclinic	Monoclinic	Triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> $\bar{1}$
Temperature (K)	293(2)	293(2)	193(2)
Unit cell dimensions (Å, °)			
<i>a</i>	8.459(3)	10.6273(18)	9.476(2)
<i>b</i>	8.936(4)	8.6343(14)	9.9904(15)
<i>c</i>	9.816(6)	16.059(3)	11.220(2)
α	97.416(4)	90	114.304(13)
β	86.918(5)	100.881(3)	93.630(19)
γ	95.602(6)	90	93.73(3)
Volume (Å ³), <i>Z</i>	725.5(6), 1	1447.1(4), 2	961.3(3), 1
Calculated density (g cm ⁻³)	1.501	1.505	1.385
Absorption coefficient (mm ⁻¹)	0.780	0.783	0.608
<i>F</i> (000)	337	674	417
Reflections collected	7027	15,317	9407
Unique reflections	2629 [<i>R</i> (int) = 0.0365]	3301 [<i>R</i> (int) = 0.0246]	3451 [<i>R</i> (int) = 0.0203]
Parameters	196	197	227
Goodness of fit on <i>F</i> ²	1.014	1.057	0.998
<i>R</i> ₁ [<i>I</i> > 2σ(<i>I</i>)]	0.0563	0.0326	0.0609
<i>wR</i> ₂ (all data)	0.1546	0.0839	0.1532

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

1			
Co(1)–N(3)	2.168(3)	Co(1)–N(6B)	2.177(3)
Co(1)–N(7)	2.086(3)		
N(6B)–Co(1)–N(3)	87.63(12)	N(7)–Co(1)–N(3)	91.39(12)
N(7)–Co(1)–N(6B)	88.93(12)		
N(7)–Co(1)–N(7A)	180.0(2)	N(6B)–Co(1)–N(6C)	180.00(7)
2			
Co(1)–N(3)	2.1781(14)	Co(1)–N(6B)	2.1239(13)
Co(1)–N(7)	2.1228(14)		
N(6B)–Co(1)–N(3)	87.82(5)	N(7)–Co(1)–N(3)	89.42(5)
N(7)–Co(1)–N(6B)	87.79(5)		
N(7)–Co(1)–N(7A)	180.00(2)	N(6B)–Co(1)–N(6C)	180.00(7)
3			
Co(1)–N(3)	2.148(3)	Co(1)–N(6)	2.166(3)
Co(1)–N(7)	2.110(3)		
N(3)–Co(1)–N(6)	91.89(11)	N(3)–Co(1)–N(7)	88.66(11)
N(6)–Co(1)–N(7)	90.97(11)		
N(3A)–Co(1)–N(3)	180.00(2)		

Symmetry transformations used to generate equivalent atoms: A $-x, -y+1, -z$; B $-x, -y+1, -z-1$; C $x, y, z+1$ for **1**; A $-x+2, -y+1, -z+1$; B $-x+1, -y+1, -z+1$; C $x+1, y, z$ for **2**; A $-x+1, -y, -z+1$ for **3**.

Two obtz/mbtz ligands are wrapped around each other and held together by Co(II) to form 1-D double chains. The chains extend along the *c*-axis for **1** and the *a*-axis for **2**. The Co···Co separations by obtz/mbtz ligands are equal to the *c*-axis translation for **1** (9.816(6) Å) and the *a*-axis translation for **2** (10.6273(18) Å). The obtz and mbtz ligands both exhibit *gauche* conformation. The dihedral angles between two triazole ring planes, between the N(1)–N(3)/C(9)/C(10), N(4)–N(6)/C(11)/C(12) triazole ring plane,

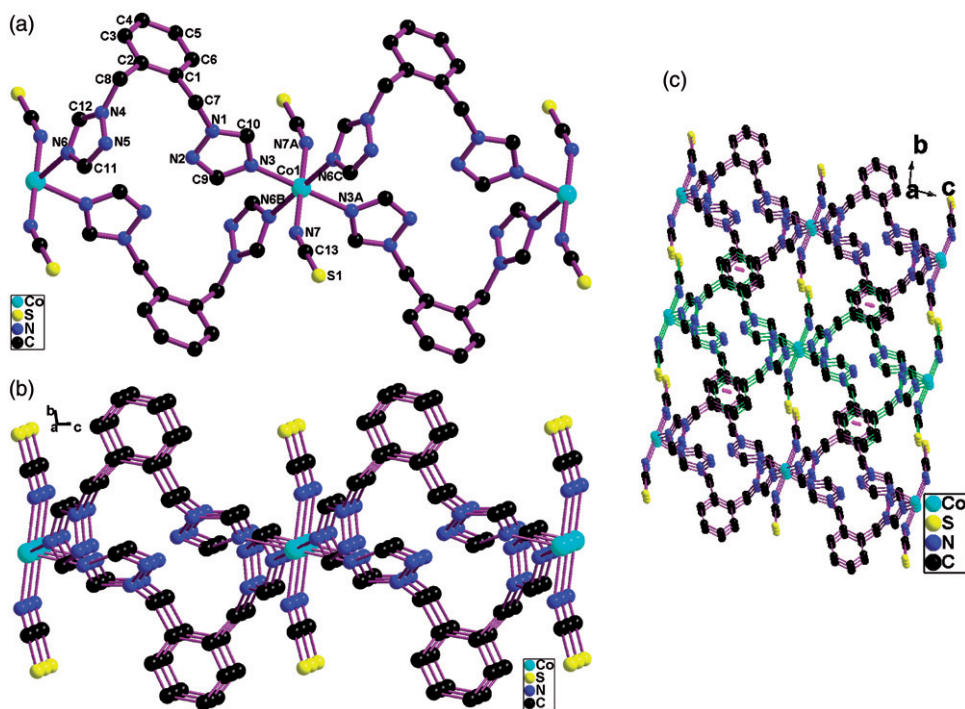


Figure 1. (a) The 1-D double chain in **1**, (b) the parallel packing of the 1-D double chains along the *a* direction in **1**, and (c) the 3-D supramolecular network in **1**. The bold dashed lines exhibit the π - π stacking interactions between the benzene rings.

and benzene ring planes are 77.2° , 79.2° , and 78.1° for **1**, and 110.1° , 83.3° , and 83.9° for **2**, respectively.

The chains of **1** are parallel stacked along the *a* direction (figure 1b) with the distances corresponding to the *a*-axis translation ($8.459(3) \text{ \AA}$), showing no $\pi \cdots \pi$ stacking interactions between obtz ligands. However, the benzene rings of adjacent chains along the *b* direction parallel overlap to each other to form a suitable space (3.61 , 3.74 \AA) for $\pi \cdots \pi$ stacking interactions. Each chain forms $\pi \cdots \pi$ stacking interactions with four adjacent chains (two above and two below) along the *b* direction. A 3-D network is constructed through these $\pi \cdots \pi$ stacking interactions (figure 1c). Janiak [40] has analyzed π - π stacking in metal complexes with aromatic nitrogen-containing ligands based on the Cambridge Structural Database. It is evident that a face-to-face π - π alignment where most of the ring-plane area overlaps is a rare phenomenon. The usual π interaction is an offset or slipped stacking, i.e. the rings are parallel displaced [40].

The chains of **2** are parallel stacked along the *b* direction (figure 2b) with the distances corresponding to the *b*-axis translation ($8.6343(14) \text{ \AA}$), showing no $\pi \cdots \pi$ stacking interactions between mbtz ligands. There are also no $\pi \cdots \pi$ stacking interactions between the mbtz between the chains along the *c* direction. Different to **1**, no $\pi \cdots \pi$ stacking interaction was observed in **2**.

Polymer **3** is a neutral 2-D (4,4) network. The asymmetry unit contains half Co(II), one NCS^- , two halves bbtz, and one disordered DMF. The coordination geometry of

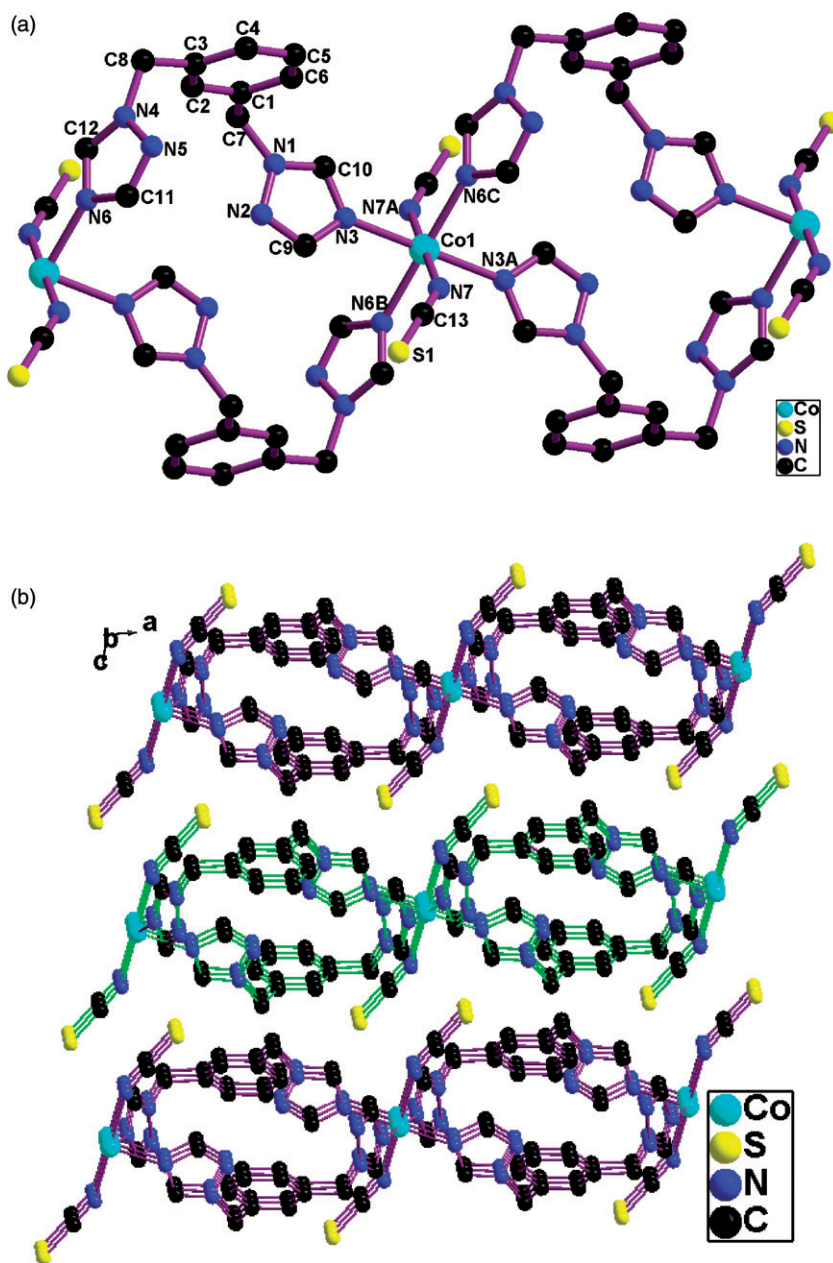


Figure 2. (a) The 1-D double chain in **2**, and (b) the packing of the 1-D double chains in **2**.

Co(II) is four nitrogen atoms from four bbtz ligands in the equatorial plane and two nitrogen atoms from two NCS⁻ (figure 3a). Each bbtz exhibits *anti* conformation. The dihedral angle between two triazole rings is 180° due to the symmetry operator. The dihedral angles between the triazole ring and the benzene ring are 88.6° and 78.7° for N1–N3 and N4–N6 bbtz ligands, respectively.

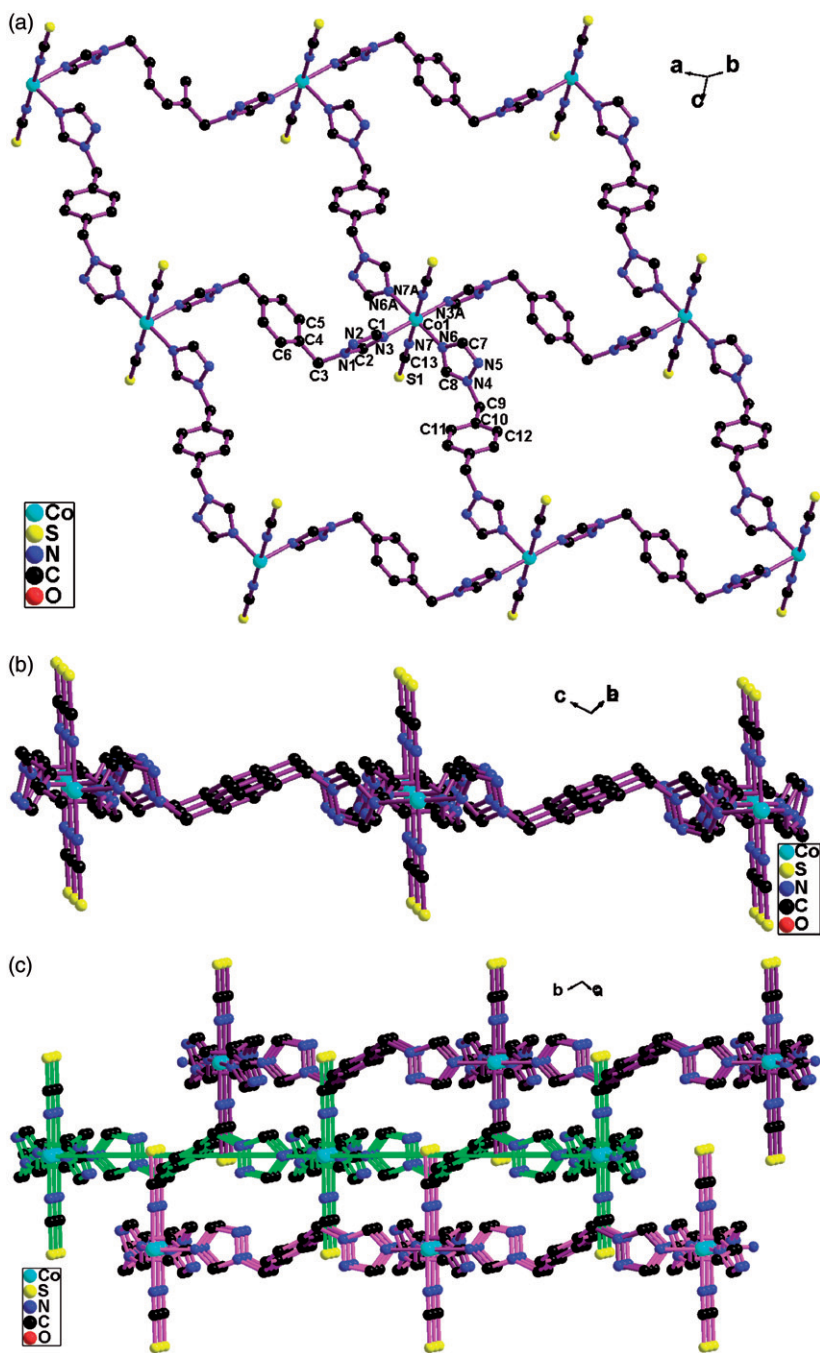


Figure 3. (a) The 2-D (4,4) network in 3, (b) side view of the 2-D layer in 3, and (c) the 2-D \rightarrow 3-D polythreaded network in 3.

Each Co(II) in **3** is bridged by four bbtz ligands to form a neutral 2-D (4,4) network with Co...Co distances of 12.210(4) and 15.138(3) Å. The network contains square grids (52-membered ring), with a Co(II) at each corner and a bbtz at each edge connecting two Co(II). The NCS⁻ ligands dangle above and below the 2-D layer (figure 3b). The 2-D layers parallel stack with an *ABAB* sequence. The dangling NCS⁻ ligands insert into the window of adjacent layers in a mutual relationship. Each window is pierced by two NCS⁻ ligands that belong to two adjacent layers (one above and one below) resulting in a 2-D → 3-D polythreaded network (figure 3c).

Polythreaded structures with finite components are unusual [41]. The few species known include polythreaded 0-D rings with side arms that give 1-D or 2-D arrays [42, 43], 1-D chains of alternating rings and rods (1-D → 1-D) [44], molecular ladders with dangling arms, resulting in (1-D → 2-D) [45] or (1-D → 3-D) [46] polythreaded arrays, 1-D chains and 2-D sheets (1-D + 2-D → 2-D) [47] or (1-D + 2-D → 3-D) [48], 2-D sheets (2-D → 2-D) [49] or (2-D → 3-D) [50–53], and (3-D + 1-D) [54]. So far, only a few 2-D → 3-D polythreaded frameworks were reported. Most of the 3-D polythreaded frameworks are constructed from simple 2-D (4,4) or (6,3) layers with dangling arms [50–53]. However, no 2-D → 3-D polythreading network based on NCS⁻ dangling arms was reported. Polymer **3** is the first coordination polymer showing 2-D → 3-D polythreading based on NCS⁻ dangling arms.

3.2. Thermal stability

All compounds are stable and retain crystallinity at room temperature for several weeks. TG experiments were carried out to explore their thermal stability (figure 4). In the TG curves, **1** and **2** were stable to 214°C and 218°C, respectively. Then **1** and **2** exhibit rapid decomposition from 230°C to 310°C and 236°C to 320°C, respectively. Weight loss did not stop at 500°C. In **3**, the lattice DMF was lost from 80°C to 175°C. The remaining substance was thermally stable upon heating to 240°C. Then **3** exhibited rapid decomposition at 250°C to 330°C and the weight loss occurred continuously and did not stop at 500°C.

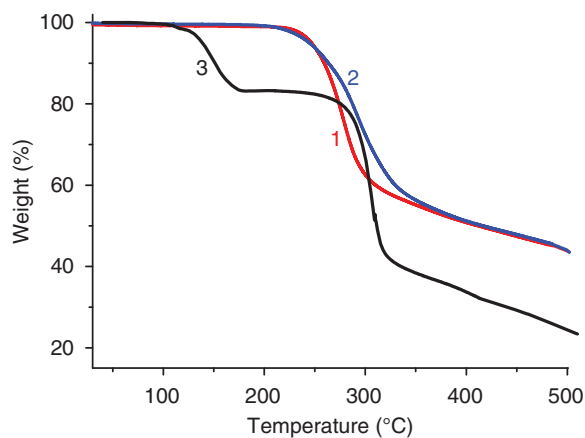


Figure 4. The TG curves of coordination polymers **1**, **2**, and **3**.

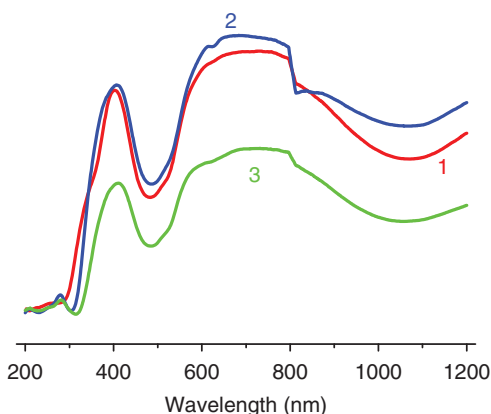


Figure 5. Diffuse reflectance UV-Vis of **1**, **2**, and **3** at solid state in BaSO₄.

3.3. Diffuse reflectance UV-Vis spectroscopy

The diffuse reflectance UV-Vis spectra of **1**, **2**, and **3** are shown in figure 5. The emission maxima at 403, 407, and 411 nm in **1**, **2**, and **3** are attributed to obtz, mbtz, and bbtz, respectively. The broad absorption bands at 600–800 nm for **1**, **2**, and **3** are assigned to electronic spectra of the d–d electronic transitions.

4. Conclusion

To investigate the effect of positional isomer ligands on the topologies, three coordination polymers, [Co(obtz)₂(NCS)₂]_n (**1**), [Co(mbtz)₂(NCS)₂]_n (**2**), and {[Co(bbtz)₂(NCS)₂]·2DMF}_n (**3**), were synthesized. Polymers **1** and **2** have similar 1-D double chains. In **1**, each chain forms π – π stacking interactions with four adjacent chains (two upper and two lower) to create a 3-D supramolecular network. Different to **1**, no π · π stacking interaction was observed in **2**. Polymer **3** is a neutral 2-D (4,4) network. The dangling NCS[–] ligands insert into the window of adjacent layers in a mutual relationship and result in a 2-D → 3-D polythreaded network. The ligands obtz, mbtz, and bbtz exhibit *gauche*, *gauche*, and *anti* conformations in **1**, **2**, and **3**, respectively. The topologies of **1**, **2**, and **3** show that position isomers play a key role in formation of different coordination polymers **1**, **2**, and **3**. Further syntheses and structural studies of coordination polymers with flexible triazole ligands are underway in our laboratory.

Supplementary material

CCDC-865976, 865977, and 865978 contains 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

Acknowledgments

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