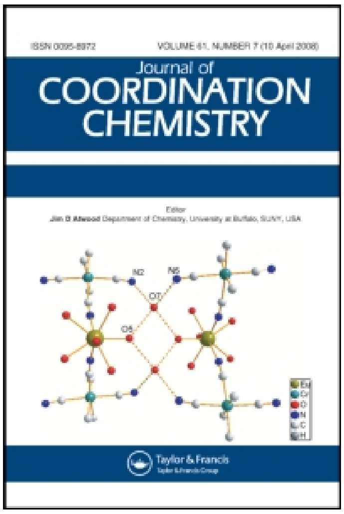

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Syntheses and structures of three new coordination polymers with the flexible 1,4-<i>bis</i>(imidazol-1-yl)butane ligand

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Syntheses and structures of three new coordination polymers with the flexible 1,4-*bis*(imidazol-1-yl)butane ligand

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Three new coordination polymers $[Co(bimb)_2(NCS)_2]_n$ (**1**), $\{[Co(bimb)_2(dca)_2] \cdot CH_3CN\}_n$ (**2**) and $[Cu(bimb)_2(NO_3)_2]_n$ (**3**) (bimb = 1,4-*bis*(imidazol-1-yl)butane, dca = dicyanamide) were synthesized and characterized. In **1**, each Co(II) links two Co(II)'s by double bimb ligands and extends to form a one-dimensional chain containing the $Co_2(bimb)_2$ 22-membered metallocycle. **2** and **3** are two-dimensional (4, 4) networks linked by bimb bridges. The conformations of the bimb ligands in **1**, **2** and **3** are analyzed.

Keywords: Cobalt complex; Copper complex; *bis*(imidazol-1-yl)butane; Crystal structure; Coordination polymer

1. Introduction

Metal-organic coordination polymers have intriguing structures and potential application as functional materials [1–7]. The flexible *bis*(imidazole) ligands 1,4-*bis*(imidazol-1-ylmethyl)benzene [8–10] and 1,2-*bis*(imidazol-1-yl)ethane [11–13] have given novel topologies in recent years. 1,4-*bis*(imidazol-1-yl)butane (bimb) is a very effective bridging ligand for construction of coordination polymers. Several bimb coordination polymers with transition metals were synthesized [14–20], but no Co-bimb complex was reported.

Pseudohalide thiocyanate (SCN^-) [21, 22] and dicyanamide (dca) ($[N(CN)_2]^-$) [23–25] are widely used to construct coordination polymers because of their versatile coordination modes and the ability to mediate strong magnetic coupling.

Previously we have synthesized a number of coordination polymers with flexible *bis*(triazole) ligands such as 1,2-*bis*(1,2,4-triazol-1-yl)ethane (bte) [12, 13], 1,4-*bis*(1,2,4-triazol-1-methyl)benzene (bbtz) [26, 27], and the *bis*(imidazole) ligand 1,2-*bis*(imidazol-1-yl)ethane (bim) [12, 13]. The ligand bimb is longer and more flexible than bim. The combination of the long flexible ligand bimb and the short anionic

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ligands (SCN^-, $[N(CN)_2]^-$ and NO_3^-) could give new topologies. Three new coordination polymers $[Co(bimb)_2(NCS)_2]_n$ (**1**), $\{[Co(bimb)_2(dca)_2]\cdot CH_3CN\}_n$ (**2**) and $[Cu(bimb)_2(NO_3)_2]_n$ (**3**) were synthesized and structurally characterized. The conformations of the bimb ligands in **1**, **2** and **3** are analyzed.

2. Experimental

2.1. *Materials and physical measurements*

All reagents were of analytical grade and used without further purification. 1,4-*bis*(imidazol-1-yl)butane (bimb) was synthesized by the literature method [16]. 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.

2.2. *Synthesis of $[Co(bimb)_2(NCS)_2]_n$ (1)*

A methanolic solution (20 mL) of bimb (0.190 g, 1.0 mmol) was added slowly to aqueous solution (20 mL) of $Co(NO_3)_2\cdot 6H_2O$ (0.146 g, 0.5 mmol) and KNCS (0.097 g, 1.0 mmol) in a tube. Red single crystals of **1** were obtained after the mixture stood at room temperature for three weeks. Yield: 0.239 g (86%). Anal. Calc. for $C_{22}H_{28}CoN_{10}S_2$: C, 47.56; H, 5.08; N, 25.22. Found: C, 47.32; H, 4.97; N, 25.06%. IR data (cm^{-1}): 3427w, 3126w, 2933w, 2076vs, 1520m, 1451w, 1382m, 1281w, 1235w, 1104m, 1034w, 942w, 841w, 764w, 664w, 633w.

2.3. *Synthesis of $\{[Co(bimb)_2(dca)_2]\cdot CH_3CN\}_n$ (2)*

A 25 mL H_2O/MeOH solution (1 : 1 v/v) of $Co(NO_3)_2\cdot 6H_2O$ (0.146 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 25 mL MeOH/CH_3CN (1 : 1 v/v) solution of bimb (0.190 g, 1.0 mmol) was added to the other leg. Orange crystals of **2** were obtained after about one month. Yield: 0.132 g (43%). Crystals of **2** lose the solvent CH_3CN, giving $[Co(bimb)_2(dca)_2]_n$ (**2a**). Anal. Calc. for $C_{24}H_{28}CoN_{14}$ (**2a**): C, 50.44; H, 4.94; N, 34.32. Found: C, 50.28; H, 4.83; N, 34.19%. IR data (cm^{-1}) for **2a**: 3126w, 2948w, 2261s, 2215m, 2161vs, 1520m, 1451w, 1328m, 1281w, 1235m, 1096m, 1034w, 942w, 872w, 834w, 749w, 664w, 625w, 517w, 440w.

2.4. *Synthesis of $[Cu(bimb)_2(NO_3)_2]_n$ (3)*

A 20 mL H_2O/MeOH solution (1 : 1 v/v) of $Cu(NO_3)_2\cdot 6H_2O$ (0.148 g, 0.5 mmol) was added to one leg of an "H-shaped" tube, and a 20 mL MeOH/CH_3CN (1 : 1 v/v) solution of bimb (0.190 g, 1.0 mmol) was added to the other leg. Green crystals of **3** were obtained after three weeks. Yield: 0.207 g (73%). Anal. Calc. for $C_{20}H_{28}CuN_{10}O_6$: C, 42.29; H, 4.97; N, 24.66. Found: C, 42.24; H, 4.87; N, 24.52%. IR data (cm^{-1}): 3124w, 2939w, 1525m, 1453w, 1383vs, 1282w, 1238w, 1108m, 1041w, 948w, 842w, 761w, 638w.

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. The 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. Structures were solved by direct methods and refined with full-matrix least-squares technique (SHELXTL-97) [28]. The positions of hydrogen atoms were determined with theoretical calculation. 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.

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

	1	**2**	**3**
Formula	$C_{22}H_{28}CoN_{10}S_2$	$C_{26}H_{31}CoN_{15}$	$C_{20}H_{28}CuN_{10}O_6$
Formula weight (g mol^{-1})	555.59	612.59	568.06
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$C2/c$	$P2_1/c$
Temperature (K)	173(2)	193(2)	223(2)
a (Å)	8.9038(8)	11.1541(16)	7.8052(15)
b (Å)	9.5907(9)	13.0599(17)	17.297(3)
c (Å)	14.3660(13)	20.679(3)	8.9172(18)
β (°)	91.068(2)	102.801(3)	108.513(4)
V (Å^3)	1226.55(19)	2937.5(7)	1141.6(4)
Z	2	4	2
$\rho_{\mathrm{Calc.}}$ (g cm^{-3})	1.504	1.385	1.653
μ(mm^{-1})	0.903	0.629	1.019
$F(000)$	578	1276	590
Reflections collected	11557	13976	10907
Unique reflections	2250 [R(int) = 0.0207]	2680 [R(int) = 0.0399]	2089 [R(int) = 0.0435]
Parameters	161	195	169
Goodness of fit	1.044	1.076	1.051
R_1 [$I > 2\sigma(I)$]	0.0260	0.0598	0.0680
wR_2 (all data)	0.0670	0.1484	0.1874

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

1			
Co(1)–N(2)	2.1434(13)	Co(1)–N(4B)	2.1691(13)
Co(1)–N(5)	2.1280(14)		
N(2)–Co(1)–N(4B)	90.21(5)	N(2)–Co(1)–N(5)	89.11(5)
N(4B)–Co(1)–N(5)	89.43(5)	N(5)–C(11)–S(1)	178.72(15)
Co(1)–N(5)–C(11)	157.08(12)		
2			
Co(1)–N(2)	2.153(3)	Co(1)–N(4)	2.149(3)
Co(1)–N(6)	2.139(3)		
N(2)–Co(1)–N(4)	93.12(10)	N(2)–Co(1)–N(6)	88.83(11)
N(4)–Co(1)–N(6)	88.84(10)		
3			
Cu(1)–N(2)	2.014(4)	Cu(1)–N(4B)	2.032(4)
Cu(1)–O(1)	2.509(4)		
N(2)–Cu(1)–N(4B)	90.60(15)	N(2)–Cu(1)–O(1)	91.01(15)
N(4B)–Cu(1)–O(1)	84.81(15)		

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

3. Results and discussion

The structure of **1** is completely different from the structures of **2** and **3** although the metal/bimb molar ratio in **1**, **2** and **3** all are 1 : 2. Complex **1** forms a one-dimensional chain (figure 1) [12, 13], while **2** and **3** have neutral two-dimensional (4, 4) networks. The key factor is that two bimb molecules link the two same metal(II) atoms in **1**, and only one bimb molecule bonds to the same metal(II) atoms in **2** and **3**.

The coordination geometry of the Co(II) atoms in **1** and **2** are similar distorted octahedrals. Each Co(II) in **1** and **2** is coordinated to four imidazole nitrogens from four different bimb ligands in the equatorial plane [Co(1)–N(2) 2.1434(13) Å, Co(1)–N(4B)) 2.1691(13) Å for **1**; Co(1)–N(2) 2.153(3) Å, Co(1)–N(4) 2.149(3) Å for **2**, and two nitrogen atoms from two thiocyanate ions for **1** [Co(1)–N(5) 2.1280(14) Å] and from two dicyanamide ions for **2** [Co(1)–N(6) 2.139(3) Å], in the axial positions. The Co–N bond lengths of **1** and **2** are similar. The dca is monodentate nitrile nitrogen coordination. The uncoordinated nitrile nitrogen of dca is disordered with occupancy factors of 0.50 assigned to N(7) and N(7B).

The coordination geometry of Cu(II) in **3** is 4 + 2 by four nitrogens from four bimb ligands in the equatorial plane [Cu(1)–N(2) 2.014(4) Å, Cu(1)–N(4B) 2.032(4) Å] and two oxygen atoms from two nitrates [Cu(1)–O(1) 2.509(4) Å] in the axial positions because of Jahn-Teller elongation. The nitrates in **3** have monodentate oxygen coordination. The coordination geometries of the Cu(II) atom in the bimb-Cu complexes $[Cu(bimb)_2(H_2O)]Cl_2 \cdot 5H_2O$, $[Cu(bimb)_2(H_2O)](NO_3)_2 \cdot H_2O$, $[Cu(bimb)_2(H_2O)]SO_4 \cdot 8H_2O$ [18] are square-pyramidal, square-pyramidal and square planar, respectively. The Cl^-, NO_3^- and SO_4^{2-}anions are uncoordinated counter-ions in the above three bimb-Cu complexes.

Each Co(II) atom connects to other Co(II) atoms by two bimb ligands, resulting in the $Co_2(bimb)_2$ 22-membered metallocycle in **1**. Two stranded bimb ligands are wrapped around each other and held together by metal atoms, forming a double chain structure along the *a* axis for **1**. The Co···Co distances of the bridging bimb are 8.904 Å. The one-dimensional chains are parallel stacked along the *b* axis for **1** and parallel extend along the *c* axis.

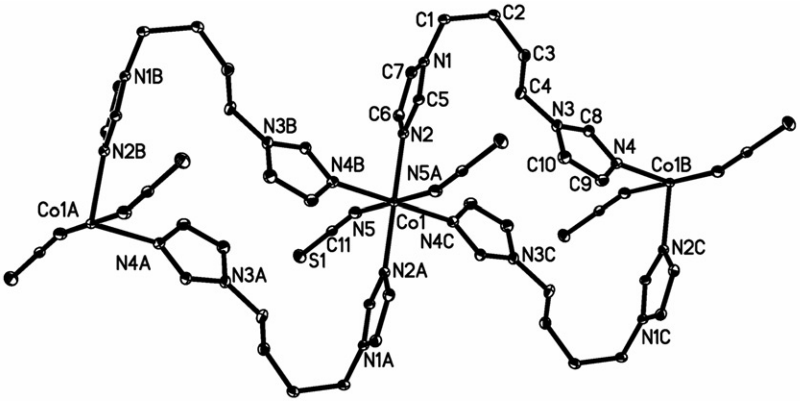

Figure 1. The one-dimensional double chain structure along the *a* direction in **1**. The hydrogen atoms are omitted for clarity.

Each metal(II) (Co(II) for **2** and Cu(II) for **3**) is bridged by four bimb ligands to form a neutral two-dimensional (4, 4) network (figures 2b and 3b). The networks contain square grids (44-membered ring), with a metal(II) atom at each corner and a bimb molecule at each edge connecting two metal(II) atoms. The edge lengths are 12.789 and

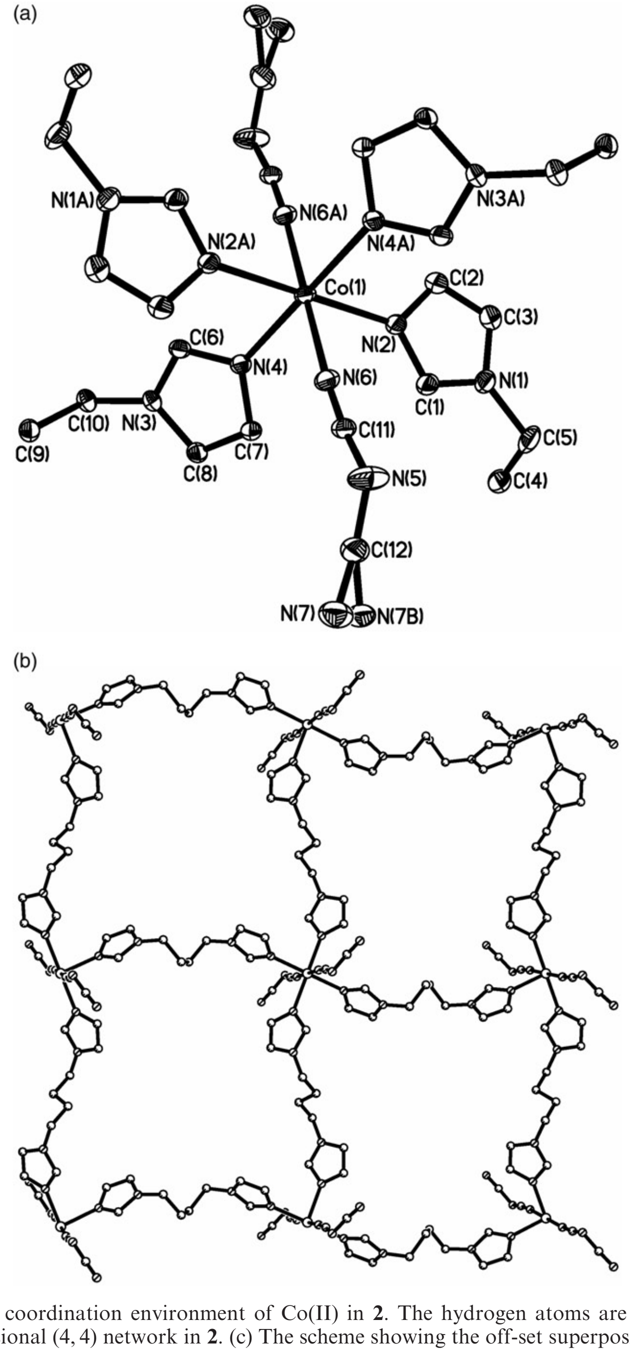

Figure 2. (a) The coordination environment of Co(II) in **2**. The hydrogen atoms are omitted for clarity. (b) The two-dimensional (4, 4) network in **2**. (c) The scheme showing the off-set superposition of the adjacent networks in **2**.

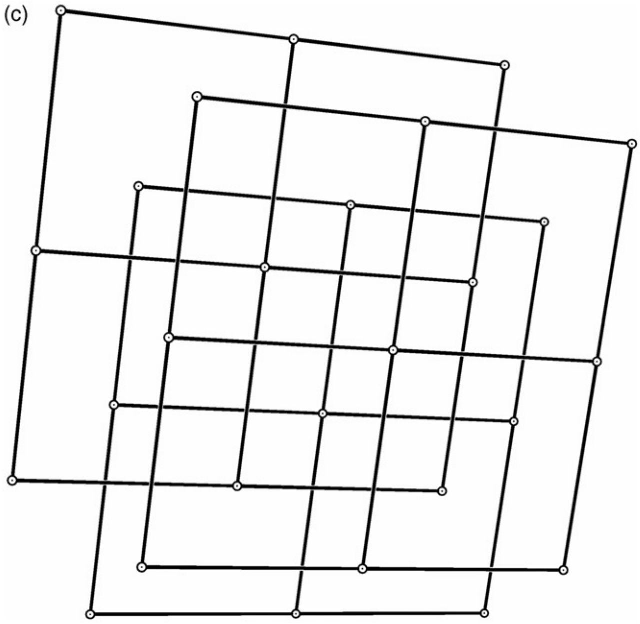

Figure 2. Continued.

13.060 Å for **2** and 13.330 Å for **3**, respectively, longer than the corresponding Co ⋯ Co separation (8.904 Å) for **1**.

The two-dimensional networks in **2** are superimposed in an interesting off-set fashion. The off-set superposition of each pair of adjacent networks by one-third of the edges divides the voids into small rectangles (figure 2c). The dicyanamide anions of one network project into the holes of the next network; distances between parallel networks are 4.397 Å. In the superposition structure, the networks are arranged in the sequence… *A*-*B*-*C*-*A*-*B*-*C*… in **2**. The two-dimensional networks in **3** are stacked in an offset fashion parallel to the *c* direction. The distance between the parallel networks is 3.253 Å. In the superposition structure, the networks are arranged in the sequence… *A*-*B*-*A*-*B*…. The nitrate anions are located in the voids in **3**. The three bimb-Cu complexes, $[Cu(bimb)_2(H_2O)]Cl_2 \cdot 5H_2O$, $[Cu(bimb)_2(H_2O)](NO_3)_2 \cdot H_2O$, $[Cu(bimb)_2(H_2O)]SO_4 \cdot 8H_2O$, all form similar two-dimensional (4, 4) networks [18].

The bimb ligands exhibit *gauche-gauche-anti, gauche-anti-gauche* and *completely anti* (*anti-anti-anti*) conformations in **1**, **2** and **3**, respectively. The dihedral angles between two imidazole planes are 77.8° for **1**, 88.8 and 0.0° for **2**, and 126.6° for **3**. The plane of $N(CH_2)_4N$ in **3** is co-planar, but poorly planar in **1** and **2**. The r.m.s. deviations of atoms from the mean planes of the $N(CH_2)_4N$ chains are 0.2447 Å in **1**, 0.4832 and 0.2086 Å in **2**, and 0.0462 Å in **3**. The plane of the $N(CH_2)_4N$ chain is steeply inclined, by 82.1 and 73.4° for **1**, 44.4° and 56.8° for **2**, 34.1 and 94.0° for **3**, to the two imidazole ring planes. The torsion angles N(1)–C(1)–C(2)–C(3), C(1)–C(2)–C(3)–C(4) and C(2)–C(3)–C(4)–N(3) are −51.4(2), 84.37(18) and 165.58(14)° for **1**, 171.1(4), 176.9(4) and −179.9(4)° for **3**.

The IR spectrum shows that imidazole ring vibrations in **1**, **2** and **3** are at 1520 and 1281, 1520 and 1281, 1525 and 1282 cm^{-1}, respectively [13]. The very strong absorption

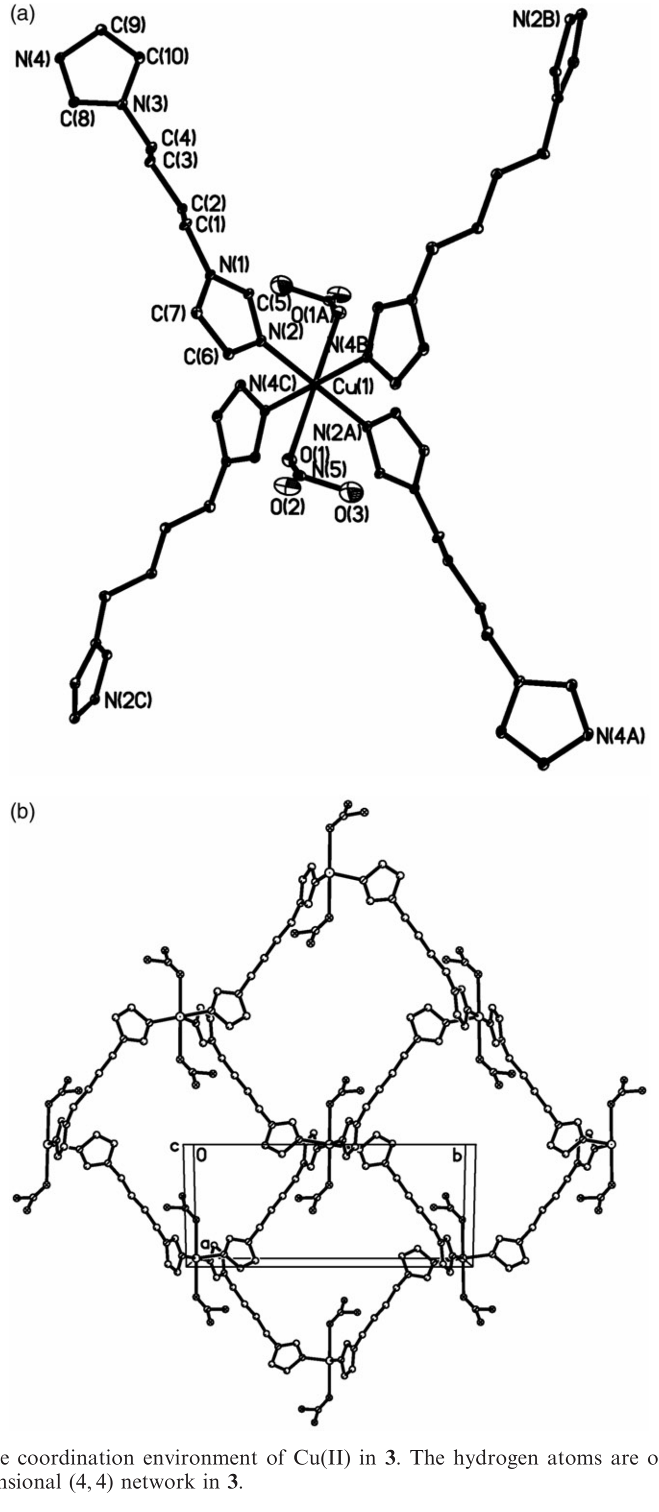

Figure 3. (a) The coordination environment of Cu(II) in **3**. The hydrogen atoms are omitted for clarity. (b) The two-dimensional (4, 4) network in **3**.

at 2076 cm^{-1} for **1** indicates the C=N stretching vibration of the thiocyanate, which is consistent with thiocyanate-N coordination [13, 21]. The strong absorptions at 2261, 2215, and 2161 cm^{-1} for **2** are assigned to C≡N symmetric stretch (ν_{sym}), the asymmetric stretch (ν_{asym}) and the combination band of ν_{sym} and ν_{asym} of dca [13, 23–25]. The smaller shift (2261 cm^{-1}) for **2** towards high frequency, compared with free dca in $Na[N(CN)_2]$ (2232 and 2179 cm^{-1}), indicates monodentate coordination of dca. The very strong absorption band at 1383 cm^{-1} for **3** is attributed to nitrate.

Reaction of the flexible *bis*(imidazole) ligand 1,4-*bis*(imidazol-1-yl)butane (bimb) with Mn(II) and non-coordinating anions BF_4^-, ClO_4^-, PF_6^- gives three-dimensional network coordination polymers $[Mn(bimb)_3](BF_4)_2$ [14], $[Mn(bimb)_3](ClO_4)_2$ and the analogous compounds containing combinations of ClO_4^-/PF_6^- or ClO_4^-, AsF_6^- [15] comprising two equivalent, mutually interpenetrating three-dimensional networks. The three bimb complexes $[Zn(bimb)_{1.5}(H_2O)(SO_4)]6H_2O$, $[Zn(bimb)_{1.5}(H_2O)_2](NO_3)_2 \cdot 2H_2O$, and $[Cd(bimb)_{1.5}(H_2O)_2(SO_4)]4H_2O$ are composed of a (6,3) network [16]. The three bimb-Cu complexes $[Cu(bimb)_2(H_2O)]Cl_2 \cdot 5H_2O$, $[Cu(bimb)_2(H_2O)](NO_3)_2 \cdot H_2O$, and $[Cu(bimb)_2(H_2O)]SO_4 \cdot 8H_2O$ form similar two-dimensional (4, 4) networks [18].

The reaction of bimb with Co(II) and anion co-ligands (NCS^- or dca), and $Cu(NO_3)_2$ yields three new coordination polymers $[Co(bimb)_2(NCS)_2]_n$ (**1**), $\{[Co(bimb)_2(dca)_2] \cdot CH_3CN\}_n$ (**2**) and $[Cu(bimb)_2(NO_3)_2]_n$ (**3**). **1** forms a one-dimensional (1D) double chain while **2** and **3** consist of neutral two-dimensional (4, 4) networks. The metal cations and anions play key roles in the structural assembly. Further synthesis of new motifs with bimb and different metal salts are under way in our lab.

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 664457, 664458 and 664459, respectively.

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