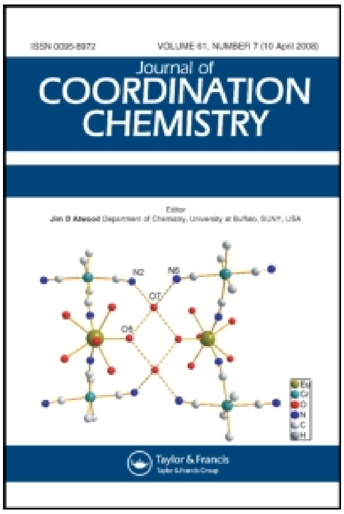

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Hydrogen-bonded assemblies of trinuclear metal string complexes

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Hydrogen-bonded assemblies of trinuclear metal string complexes

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Three kinds of trinuclear metal string complexes, $[Ni_3(dpa)_4(L^1)_2]\cdot H_2O\cdot C_2H_5OH$ ($L^1 = (E)$-3-(2-hydroxyl-phenyl)-acrylic acid) (**1**), $[Ni_3(dpa)_4(L^2)_2]\cdot 2C_2H_5OC_2H_5$ ($L^2 = (E)$-3-(3-hydroxyl-phenyl)-acrylic acid) (**2**) and $[Ni_3(dpa)_4(L^3)_2]\cdot 3CH_2Cl_2\cdot 1.5CH_3OH$ ($L^3 = (E)$-3-(4-hydroxyl-phenyl)-acrylic acid) (**3**). ($dpa^- =$ *bis*(2-pyridyl)amido), have been synthesized. The structures of **1** and **2** have been analyzed by the X-ray single-crystal diffraction showing hydrogen-bonded networks.

Keywords: Metal string complexes; *bis*(2-Pyridyl)amido; Polynuclear nickel complexes; Hydrogen-bonded network; Acrylic acid

1. Introduction

Noncovalent interactions play a special role in supramolecular chemistry used in the recognition motifs to assemble various types of supramolecular species. Particularly, these are H-bonds, stacking interactions, electrostatic interactions, hydrophobic interactions, charge-transfer interactions, and metal-coordination [1–7]. Because of its versatility and directionality, hydrogen bonds are suitable for generating supramolecular polymers [8–14].

Trinuclear nickel complexes, $[Ni_3(dpa)_4X_2]$ ($dpa^- =$ *bis*(2-pyridyl)amido, $X^- =$ a series of axial ligands) have been extensively investigated in the past decade [15, 18–34]. Such trinuclear metal string complexes can be the building blocks or "supramolecular synthons" for construction of supramolecular polymers. In addition to the special geometry of linear molecules, they have two axial positions that can be coordinated by different functional ligands. A new hydrogen bond pattern, Ni–F $\cdots$ H–O has been found in the hydrogen-bonded network of $[Ni_3(dpa)_4F_2][Ni_3(dpa)_4(H_2O)_2](BF_4)_2\cdot 2CH_3OH$ [15]. The hydrogen-bonded framework of trichromium metal string complexes have also been studied [16]. The results show that axial ligands and counter anions play an important role in solid state of metal string complexes. We have studied

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acrylic acids systematically in lanthanide complexes; these studies indicate that acrylic acids are a type of flexible carboxylato ligand with an interesting H-bond interaction [17]. In this work, (*E*)-3-(2 or 3 or 4)-hydroxyl-phenyl)-acrylic acid) has been introduced as an axial ligand in trinickel metal string complexes. The crystal structures of two complexes, $[Ni_3(dpa)_4(L^1)_2] \cdot H_2O \cdot C_2H_5OH$ ($L^1 =$ (*E*)-3-(2-hydroxyl-phenyl)-acrylic acid) (**1**) and $[Ni_3(dpa)_4(L^2)_2] \cdot 2C_2H_5OC_2H_5$ ($L^2 =$ (*E*)-3-(3-hydroxyl-phenyl)-acrylic acid) (**2**) have been studied. However, we were unable to obtain a good crystal of $[Ni_3(dpa)_4(L^3)_2] \cdot 3CH_2Cl_2 \cdot 1.5CH_3OH$ (**3**) ($L^3 =$ (*E*)-3-(4-hydroxyl-phenyl)-acrylic acid) for X-ray diffraction. CHN analysis, IR spectrum and TGA measurements of the three complexes have been done. The results of TGA indicate that H-bond networks of the three complexes are stable.

2. Experimental

2.1. *General procedures*

All chemicals purchased were of reagent grade and used without further purification. IR spectra were recorded on a Nicolet Fourier Transform IR, MAGNA-IR 500 spectrometer in the range of 500–4000 cm^{-1} using KBr disc technique. Thermogravimetric analysis is carried out in a Perkin-Elmer spectrometer from 30.0 to 344.0°C for **1** (2.3003 mg), 30.0 to 494.0°C for **2** (2.2537 mg) and 30.0 to 490.0°C for **3** (3.2522 mg) at a rate of 5.0°C.

2.2. *Preparation of complexes*

The synthesis method for the three complexes is the same and described for **1**. To a solution of $Ni_3(dpa)_4Cl_2$ (25 mg, 0.027 mmol) in 10 mL CH_2Cl_2 was added $AgBF_4$ (10.51 mg, 0.054 mmol). The reaction mixture was stirred for 2 h at room temperature. After filtering, the filtrate was added to a 10 mL ethanol solution of the sodium salt of L^1. Dark purple single crystals were obtained by ether diffusion. Yield: 20.80 mg (65.22%). Anal. Calcd for $C_{59}H_{50}Ni_3N_{12}O_8$: C, 57.55; H, 4.09; N, 13.65. Found: C, 57.93; H, 4.18; N, 13.58. Selected IR (KBr, cm^{-1}): 3403.1(m), 1642.0(s), 1607.2(s), 1590.5(s), 1563.1(m), 1465.8(s), 1459.5(s), 1425.4(s), 1363.5(s), 1312.7(s), 1158.2(m), 1061.1(m), 1012.2(m), 764.6(m), 734.3(m), 699.0(w), 637.2(w). $[Ni_3(dpa)_4(L^2)_2] \cdot 2C_2H_5OC_2H_5$ (**2**): Yield: 25.20 mg (70.17%). Anal. Calcd for $C_{66}H_{66}Ni_3N_{12}O_8$: C, 59.53; H, 5.00; N, 12.63. Found: C, 59.55; H, 4.98; N, 12.66. Selected IR (KBr, cm^{-1}): 3998.9(m), 1639.7(s), 1605.6(s), 1590.1(s), 1560.5(m), 1465.3(s), 1455.9(s), 1424.8(s), 1366.2(s), 1312.0(s), 1156.3(m), 1058.7(m), 1010.8(m), 765.3(m), 735.1(m), 694.8(w), 636.9(w). $[Ni_3(dpa)_4(L^3)_2] \cdot 3CH_2Cl_2 \cdot 1.5C_2H_5OH$ (**3**): Yield: 21.45 (58.27%). Anal. Calcd for $C_{64}H_{59}Ni_3N_{12}Cl_6O_{7.5}$: C, 51.06; H, 3.95; N, 11.17. Found: C, 51.45; H, 3.90; N, 11.13. Selected IR (KBr, cm^{-1}): 3400.8(m), 1640.5(s), 1608.8(s), 1591.6(s), 1566.2(m), 1467.1(s), 1456.6(s), 1425.8(s), 1364.5(s), 1311.1(s), 1154.3(m), 1060.5(m), 1011.3(m), 766.2(m), 733.7(m), 695.6(w), 635.9(w).

2.3. *Single-crystal X-ray diffraction*

Dark purple crystals ($0.12 \times 0.12 \times 0.05$ mm for **1** and $0.40 \times 0.40 \times 0.22$ mm for **2**) were mounted on glass fibers. Data collection was carried out on a Siemens SMART diffractometer with a CCD detector with Mo-Kα radiation ($\lambda = 0.71079'$) at 150 K. The θ range for data collection was from 1.47 to 27.50° for **1** and 1.65 to 27.50° for **2**. The completeness to 2θ was 99.9%. An empirical absorption was based on the symmetry-equivalent reflections and an absorption correction was applied with maximum and minimum transmission factors of 0.784 and 0.655 for **1** and 0.827 and 0.683 for **2** with the SORTAV program [35]. The structure was solved by direct methods with the SHELXS 97 program [36]. All non-hydrogen atomic positions were located in difference Fourier maps and refined anisotropically based on F^2 using SHELXL 97 [37]. The hydrogen atoms (H3A) in the hydroxyl group of ligand and (H4, H4′) in water were located from the difference Fourier map and refined isotropically. The remaining hydrogen atoms were placed in their geometrically generated positions.

3. Results and discussion

3.1. *Synthesis of complexes 1 to 3*

Complexes **1**, **2** and **3** were prepared from $Ni_3(dpa)_4Cl_2$ in air at room temperature. All products keep the $[Ni_3(dpa)_4]^{2+}$ core similar to the starting material. After removing two axial Cl^- by using $AgBF_4$, the sodium salts of acrylic acid ligands (L^1, L^2 and L^3) were added to the above solution giving products in high yields. Single crystals of **3** were more difficult to obtain than for **1** and **2**. This has also been observed in the lanthanide complex of L^3 [17]. At present, we cannot rationalize this result; but studies of transition metal complexes with acrylic acid ligands (L^1, L^2 and L^3) are ongoing to provide more data.

3.2. *Crystal structures of complexes 1 and 2*

Crystallographic data of **1** and **2** are summarized in table 1. Molecular structures are shown in figure 1. All the skeleton structures of the three complexes in this work are the $[Ni_3(\mu_3\text{-dpa})_4]^{2+}$ units, only different from each other in the axial ligands. Complexes **1** and **2** are the center-symmetrical. The Ni–Ni distances are 2.4162(4) and 2.4080(6) Å for **1** and **2**, respectively. The differences of Ni–Ni distances in the trinickel metal string complexes depend on the different axial ligands. After checking all the Ni–Ni distances in the trinickel metal string complexes, it has been found that the longest Ni–Ni distance (2.4470(7) Å) is in the $[Ni_3(\mu_3\text{-dpa})_4(CN)_2]$ with a strongest σ-donor axial ligand [38] and the shortest one (2.385(2) Å) is in the $[Ni_3(\mu_3\text{-dpa})_4(\mu_{1,3}\text{-}N_3)](PF_6)$ [24] with the μ-1,3 bridging azide. In this case, the axial ligands are acrylic acids that are weak σ-donor ligands. Influence by the σ-donor ability of the axial ligand on Ni–Ni distance has also been confirmed by pentanickel(II) $[Ni_5(\mu_5\text{-tpda})_4X_2]$ complexes [39]. Ni–N distances in the two complexes are divided into two ranges: 2.066(3)–2.118(3) Å for terminal Ni(II) ions and 1.881(4)–1.888(4) Å for the central Ni(II) ions (tables 2 and 3), consistent with those in the other trinickel metal string complexes [15, 18–34].

Table 1. The crystallographic data of **1** and **2**.

	1	**2**
Empirical formula	$C_{59}H_{50}Ni_3N_{12}O_8$	$C_{66}H_{66}Ni_3N_{12}O_8$
Fw	1231.24	1331.44
Space group (Å, °)	*C*2/*c*	Fddd
a	25.3400(3)	15.8523(2)
b	18.2488(2)	23.9475(4)
c	15.8873(2)	34.8684(6)
β	122.9291(5)	90
V (Å^3)	6166.39(13)	13236.8(4)
Z	4	8
D_c (g cm^{-3})	1.326	1.336
μ (mm^{-1})	0.967	0.907
No. of data/restraints/parameters	7096/3/439	3817/2/224
R indices [$I > 2\sigma(I)$]	$R_1 = 0.0506$, $wR_2 = 0.1507$	$R_1 = 0.0683$, $wR_2 = 0.1804$
R indices (all data)	$R_1 = 0.0762$, $wR_2 = 0.1707$	$R_1 = 0.0874$, $wR_2 = 0.1992$
GOF	1.057	1.077

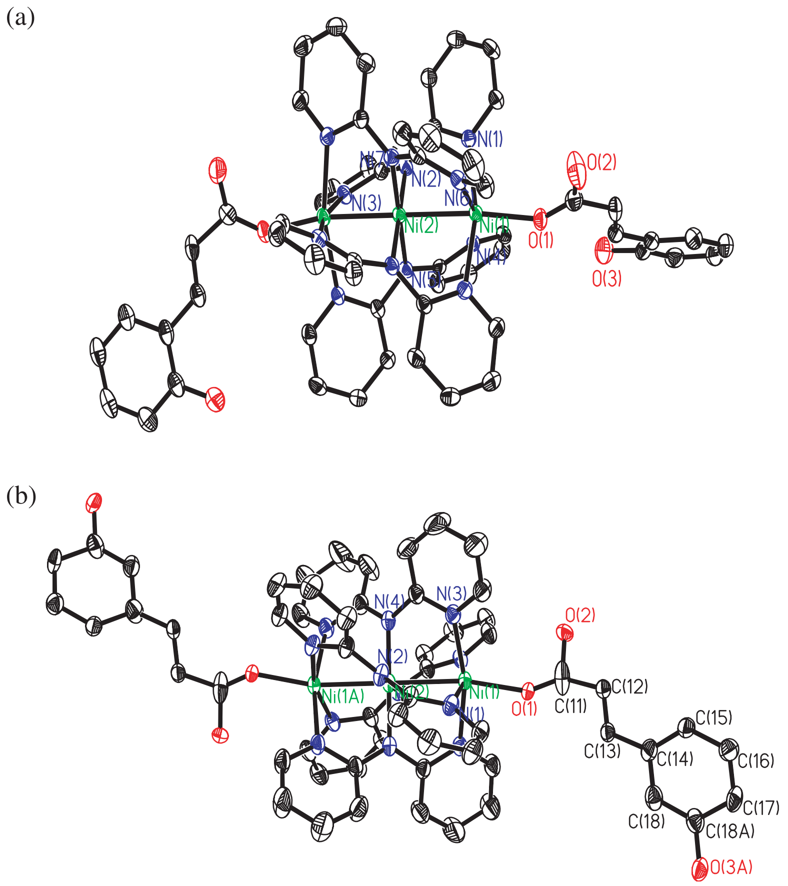

Figure 1. The molecular structures of the $Ni_3(dpa)_4(L^1)_2] \cdot 3.5H_2O \cdot 0.5C_2H_5OH$ (**1**) (a) and $[Ni_3(dpa)_4(L^2)_2] \cdot 2C_2H_5OC_2H_5$ (**2**) (b). The solvent molecules and hydrogen atoms are omitted for clarity in both pictures.

Table 2. Selected bond lengths (Å) and angles (°) of **1**.

Ni(1)–Ni(2)	2.4160(4)	Ni(1)–O(1)	2.009(2)
Ni(1)–N(1)	2.069(2)	Ni(1)–N(6)	2.118(3)
Ni(1)–N(3)#1	2.080(3)	Ni(1)–N(4)	2.117(3)
Ni(2)–N(2)	1.882(2)	Ni(2)–N(7)	1.881(3)
Ni(2)–N(5)	1.880(3)		
Ni(1)–Ni(2)–Ni(1)#1	179.65(3)	O(1)–Ni(1)–Ni(2)	169.75(7)
O(1)–Ni(1)–N(4)	88.41(9)	O(1)–Ni(1)–N(1)	99.24(9)
O(1)–Ni(1)–N(6)	108.01(10)	O(1)–Ni(1)–N(3)#1	96.39(9)
N(3)–Ni(1)–N(1)	90.39(13)	N(4)–Ni(1)–N(6)	163.57(10)
N(1)–Ni(1)–N(6)	88.17(10)	N(1)–Ni(1)–N(4)	88.71(9)
N(1)–Ni(1)–N(3)#1	164.32(10)	N(3)#1–Ni(1)–N(4)	90.60(10)
N(3)#1–Ni(1)–Ni(2)	82.10(7)	N(1)–Ni(1)–Ni(2)	82.31(7)
N(4)–Ni(1)–Ni(2)	81.48(7)	N(6)–Ni(1)–Ni(2)	82.12(7)
N(5)–Ni(2)–N(7)	180.0	N(5)–Ni(2)–N(2)	90.06(7)
N(7)–Ni(2)–N(2)	89.94(7)	N(2)–Ni(2)–N(2)#1	179.89(15)
N(5)–Ni(2)–Ni(1)	89.823(14)	N(7)–Ni(2)–Ni(1)	90.177(14)
N(2)–Ni(2)–Ni(1)	89.57(8)	N(2)–Ni(2)–Ni(1)#1	90.43(8)

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

Table 3. Selected bond lengths (Å) and angles (°) of **2**.

Ni(1)–Ni(2)	2.4080(6)	Ni(1)–O(1)	2.020(5)
Ni(1)–N(3)	2.073(3)	Ni(1)–N(1)	2.102(3)
Ni(2)–N(2)	1.886(3)	Ni(2)–N(4)	1.888(4)
Ni(1)–Ni(2)–Ni(1)#1	180.0		
O(1)–Ni(1)–N(3)	104.96(18)	O(1)–Ni(1)–N(1)	107.39(18)
O(1)–Ni(1)–Ni(2)	167.78(14)	N(3)–Ni(1)–N(1)	90.39(13)
N(3)–Ni(1)–N(3)#1	165.36(17)	N(3)–Ni(1)–N(1)	90.39(13)
N(1)–Ni–N(1)#1	164.26(17)	N(3)–Ni(1)–Ni(2)	82.68(8)
N(1)–Ni(1)–Ni(2)	82.13(8)	N(2)–Ni(2)–N(4)	90.000(1)
N(2)–Ni(2)–Ni(1)	90.0	N(4)–Ni(2)–Ni(1)	90.0
N(2)–Ni(2)–N(2)#1	180.0		

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

The O–Ni(II) bond lengths are 2.010(2) for **1** and 2.020(5)′ for **2**, comparable to the distance of O(H_2O)–Ni(II) (2.020′) [15]. The terminal Ni(II) ions in **1** and **2** are five-coordinate, as a distorted square pyramid (tables 2 and 3). The coordination number of the central Ni(II) is four with a well-defined planar square geometry in both complexes. Obviously, the bulk of the axial ligand was responsible for the distortion in the coordination geometry of the terminal Ni(II) ions. The three Ni(II) ions in **1** are almost linear (Ni(1)–Ni(2)–Ni(1)#1: 179.65(3)°) and those in **2** are exactly straight (Ni(1)–Ni(2)–Ni(1)#1: 180.0°). Compared with the other trinickel complexes, the distortion of coordination geometry of the Ni(II) ions in **1** and **2** are normal [15, 18–34].

Introducing (*E*)-3-(2 or 3 or 4)-hydroxyl-phenyl)-acrylic acid) as the axial ligand to trinickel metal string complex alters the hydrogen-bonded network in the solid state of these trinuclear metal string complexes. Hydroxide groups in axial ligand are key factors in assembly of H-bonded networks. In **1**, stereo-hindrance of hydroxide group in 2-position of benzene ring makes it impossible to form H-bond with oxygen atoms of carboxylato ligand directly. Thus, solvent water molecules act as a bridge to link molecules through H-bond. The details are O(4)–H4b ··· O(2): 1.934, 2.622′ 143.0° and O(4A) ··· O(3A)–H3AA; 1.769, 2.692′, 177.1°) (figure 2). This unit as basic structural

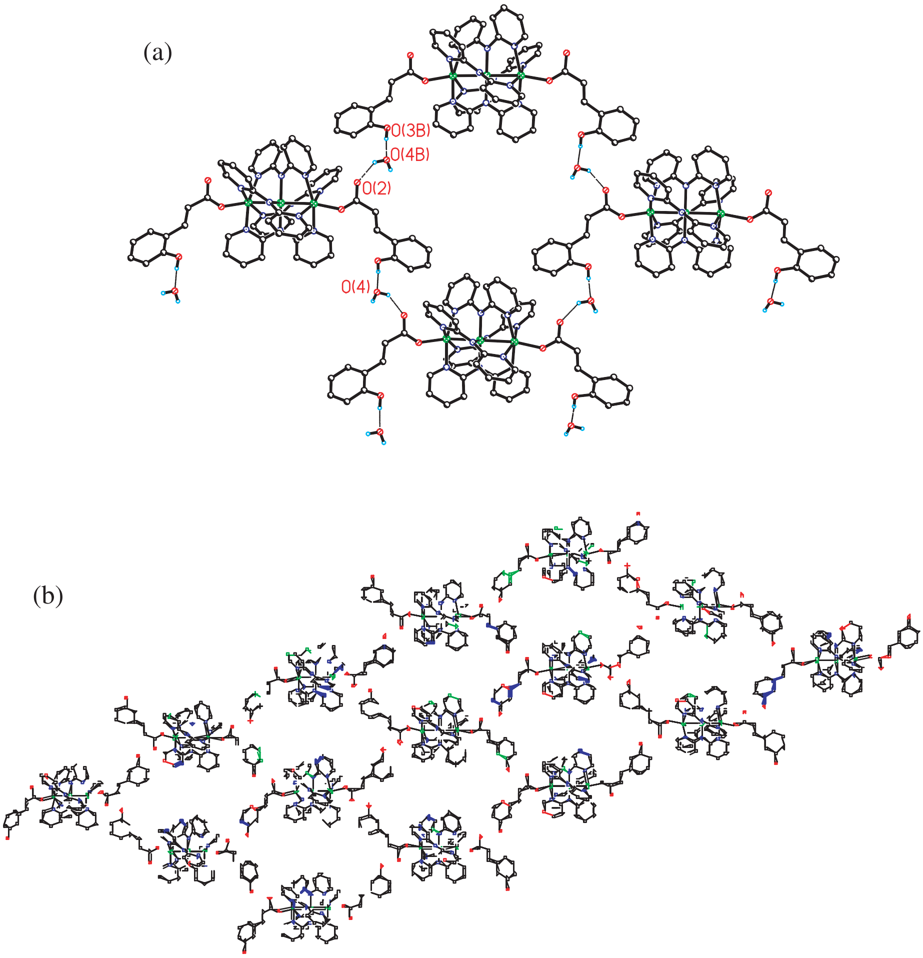

Figure 2. The 2D layer structures of **1** (a) and **2** (b).

motif extends to two-dimensional, hydrogen-bonded network in the crystal lattice. In **2**, hydrogen atoms of hydroxide group cannot be found. Therefore, the details of the hydrogen bonding are lost. However, it can be expected that hydrogen bonding interactions exist between hydroxide groups and carboxylato O atoms of adjacent molecules based on the O(3) $\cdots$ O(2B) (2.680′) distance. The two-dimensional molecular packing shows that no solvent molecules link the hydroxide groups and carboxylato O atoms of adjacent molecules as in **1**. Complex **2**, as a supramolecular synthon, forms the two-dimensional supramolecular network directly. Solvent ether molecules are sited between the layers.

3.3. *TGA analysis of complexes 1, 2 and 3*

TGA of **1** was measured from 30 to 344°C. Water and ethanol were lost between 120 to 232°C (residue 86.73%). From 232 to 344°C, both axial ligands (L^1) were lost (residue 61.01%). Based on the result of **1**, the measurement of TGA of **2** has performed between 30 to 494°C. No weight change was observed up to 280°C. Ether molecules were lost between 280 to 302°C (residue 88.91%). From 302 to 386°C, L^2 ligands were lost 24.20% (residue 64.61%). Then, from 386 to 494°C, the metal string complex cation, $[Ni_3(dpa)_4]^{2+}$, was decomposed and all dpa^- ligands were lost (residue 18.44%). Similar with **2**, solvents and L^3 axial ligands were lost between 120 to 346°C (residue 57.11%). Starting temperature of weight loss of **3** is quite similar with that of **1**. Finally, the skeleton of $[Ni_3(dpa)_4]^{2+}$ in **3** was broken down up to 484°C.

4. Conclusions

The trinuclear metal string complexes of various metal centers (Ni, Cu, Cr, Co, Ru, Rh) with *bis*(2-pyridyl)amido ligand have been synthesized and studied based on the potential applications as molecular metal wires. This work shows that trinickel metal string complexes can act as the building blocks or ‘supramolecular synthons’ because they have a well-defined linear trinuclear skeleton with two axial ligands that are easily replaced by another functional ligand. The acrylic acid ligand with hydrogen-bonding group in this work results in different solid-state packing, including different hydrogen-bonded networks.

Supplementary material

Crystallographic data (excluding structure factors) for the structure(s) reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers: CCDC-257068 **1** and CCDC-257069 **2**. Copies of the data can be obtained free of charge on application to CCDC, 12, Union Road, Cambridge CB2 1EZ, UK (Fax: (44)1223 336-033; Email: deposit@ccdc.cam.ac.uk).

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