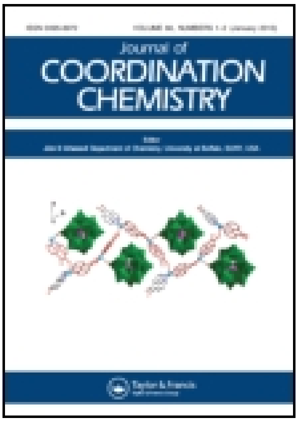

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Synthesis and characterization of two new metal-organic frameworks with interpenetrated structures and luminescent properties

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Synthesis and characterization of two new metal–organic frameworks with interpenetrated structures and luminescent properties

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Two new metal–organic compounds, $[Ag_2(HADC)_2(bimh)]$ (**1**) and $[Cd(ADC)(bpp)]_n$ (**2**) [H_2ADC = 1,3-adamantanedicarboxylic acid, bimh = 1,6-bis(2-methyl-imidazole-1-yl)hexane, bpp = 1,3-bis(4-pyridyl)propane], have been synthesized and characterized. Compound **1** exhibits a discrete symmetric unit with 0D→2D interpenetrating structure. Compound **2** crystallizes in a chiral space group *P212121* and presents a threefold interpenetrated 3D diamondoid network containing three helical chains. Thermal stability, X-ray powder diffraction, and luminescence for **1** and **2** are also measured and discussed.

Keywords: Crystal engineering; Interpenetrating structure; Luminescent properties

1. Introduction

Interpenetrating network structures are fascinating subjects in crystal engineering of coordination polymers owing to their attractive topologies, intriguing structural features [1], interesting properties, and potential applications ranging from drug delivery vehicles to sensor devices [2]. Interpenetrating networks have been the most investigated type of entanglement. They have provided a longstanding fascination for chemists, and a variety of appealing interpenetrating structures have been reported and reviewed by Batten and Robson [3]. The self-assembly of metal–organic frameworks with unique structures and functions is influenced by many factors, such as metal-to-ligand molar ratio, the nature of organic ligands, the coordination geometry of metal ions, the presence of solvent molecules, reaction temperature, and pH of the solution [4]. Therefore, it is a challenge to synthesize the predicted products.

Usually, V-shaped ligands and long flexible ligands lead to interpenetrating structures and helical modes [5]. 1,3-Adamantanedicarboxylic acid (H_2ADC) is a V-shaped ligand, and the

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*Corresponding author. Email: jjc@wxc.edu.cn angle between the coordinating sites is 110° and it can be used effectively to form interpenetrating and helical structures. Moreover, the bis(imidazole)/bis(pyridyl) ligand with flexible –CH_2– spacers is a good representative of nitrogen-donor bridging ligand, which is able to bend and rotate freely while coordinating [6]. Recently, significant work has been carried out using different metal ions with flexible bis(imidazole)/bis(pyridyl) ligands [7].

In previous work, we reported an unusual independent 1D metal–organic nanotube with 1D→2D interdigitation based on 1,3-adamantanedicarboxylic acid [8]. In this contribution, we designed and synthesized two flexible ligands, 1,6-bis(2-methyl-imidazole-1-yl)-hexane (bimh) and 1,3-bis(4-pyridyl)propane (bpp), with a long methylene skeleton to construct new frameworks. We, herein, present two new metal–organic frameworks with interpenetrated structures, $[Ag_2(HADC)_2(bimh)]$ (**1**) and $[Cd(ADC)(bpp)]_n$ (**2**). Single-crystal X-ray structural analysis shows that **1** forms a discrete symmetric unit with 0D→2D interpenetrating structure. Compound **2,** crystallized in a chiral space group *P212121* presents a threefold interpenetrated 3D diamondoid network containing three helical chains. Fluorescent properties, TG analysis, and XRPD of **1** and **2** are discussed.

2. Experimental

2.1. *Materials and measurements*

All commercially available chemicals were of reagent grade and used without purification. The ligand bimh was prepared according to literature [9]. Elemental analyzes were recorded on a Perkin-Elmer model 240C instrument. Infrared spectra on KBr pellets were recorded on a Nicolet 170SX FT-IR spectrophotometer from 4000 to 400 cm^{-1}. Thermal analyzes were performed on a NETZSCH STA449C microanalyzer with a heating rate of 10 °C min^{-1} under N_2. X-ray powder diffraction patterns were recorded with a Pigaku D/Max 3III diffractometer. Luminescence spectra for the solid samples were recorded on a Hitachi F-4500 fluorescence spectrophotometer at room temperature. The luminescent lifetimes were measured with an Edinburgh FLS920 fluorescence spectrometer at room temperature.

2.2. *Preparation of $[Ag_2(HADC)_2(bimh)]$ (1)*

A mixture of H_2ADC (0.10 mM, 22.4 mg) and bimh (0.10 mM, 24.6 mg) in 5 mL CH_3OH was layered onto a solution of $AgNO_3$ (0.20 mM, 34.0 mg) in 5 mL H_2O, and then was kept in dark at room temperature resulting in colorless block crystals after a week. Yield: 55%. Anal. Calcd for $C_{38}H_{52}Ag_2N_4O_8$: C, 50.23; H, 5.77; N, 6.17. Found: C, 50.42; H, 5.58; N, 6.35. IR (KBr, cm^{-1}): 3436 (m), 3152 (w), 2985 (m), 1798 (w), 1702 (s), 1623 (m), 1557 (s), 1486 (m), 1430 (m), 1374 (s), 1313 (m), 1228 (m), 1133 (w), 1089 (m), 1062 (w), 1003 (m), 872 (w), 824 (m), 768 (m), 654 (m), 613 (m).

2.3. *Preparation of $[Cd(ADC)(bpp)]_n$ (2)*

A mixture of H_2ADC (0.10 mM, 22.4 mg) and bpp (0.10 mM, 19.8 mg) in 5 mL CH_3OH was layered onto a solution of $Cd(NO_3)_2$ (0.20 mM, 47.2 mg) in 5 mL H_2O, and then was kept at room temperature resulting in colorless block crystals after a week. Yield: 50%.

Table 1. Selected crystallographic data for **1** and **2**.

Complexes	**1**	**2**
Empirical formula	$C_{38}H_{52}Ag_2N_4O_8$	$C_{25}H_{28}CdN_2O_4$
Formula mass	908.58	532.90
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/n$	*P212121*
a [Å]	14.017(2)	11.2710(10)
b [Å]	9.6066(14)	13.0896(11)
c [Å]	14.198(2)	15.6887(14)
α (°)	90	90
β (°)	103.978(3)	90
γ (°)	90	90
V [Å^3]	1855.2(5)	2314.6(4)
Z	2	4
μ [mm^{-1}]	1.113	0.977
F [000]	932.0	1088.0
Reflections collected	3345	4164
Final R[a] indices [I>2σ(I)]	$R_1 = 0.0437$, $wR_2 = 0.1979$	$R_1 = 0.0288$, $wR_2 = 0.0924$

[a] $R_1 = \sum||F_o|-|F_c||/\sum|F_o|$; $wR_2 = [\sum w(F_o^2-F_c^2)^2/ \sum w(F_o^2)^2]^{1/2}$.

Anal. Calcd for $C_{25}H_{28}CdN_2O_4$: C, 56.34; H, 5.30; N, 5.26. Found: C, 56.51; H, 5.09; N, 5.21. IR (KBr, cm^{-1}): 3427 (m), 3082 (w), 2985(m), 1628 (w), 1612 (s), 1557 (w), 1508 (m), 1430 (w), 1348 (s), 1300 (m), 1247 (w), 1072 (m), 1016 (w), 879 (w), 815 (m), 727 (m), 702 (w), 668 (w), 623 (w), 579 (m), 512 (m), 418 (w).

2.4. *Crystallographic data collection and structure determination*

Single-crystal X-ray diffraction analyzes of **1** and **2** were carried out on a Bruker SMART APEX II CCD diffractometer equipped with graphite monochromated Mo*Kα* radiation ($\lambda = 0.71073$ Å) using ϕ/ω scan technique at room temperature. The structures were solved using direct methods and successive Fourier difference synthesis (SHELXS-97) [10], and refined using full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97) [10, 11]. The crystallographic data and selected bond lengths and angles for **1** and **2** are listed in tables 1 and S1. Crystallographic data for the structural analyzes have been deposited at the Cambridge Crystallographic Data Center.

3. Results and discussion

3.1. *Crystal structure*

3.1.1. [Ag$_2$(HADC)$_2$(bimh)] (1). Single-crystal X-ray diffraction analysis shows that **1** crystallizes in the centrosymmetric $P2_1/n$ space group. The discrete [Ag$_2$(HADC)$_2$(bimh)] unit consists of one independent Ag(I), as shown in figure 1. The geometry around Ag1 can be described as a linear [AgNO], coordinated by one oxygen from one HADC ligand and one nitrogen from one bimh [Ag1–O1 2.0923(42) Å, Ag1–N2 2.1055(38) Å]. In **1**, *trans*-conformation bimh bridges pairs of silvers and HADC is incompletely deprotonated with one carboxylic group adopting monodentate coordination, while another is not coordinated. Compound **1**, due to partial deprotonation of H_2ADC, can act as H–bond

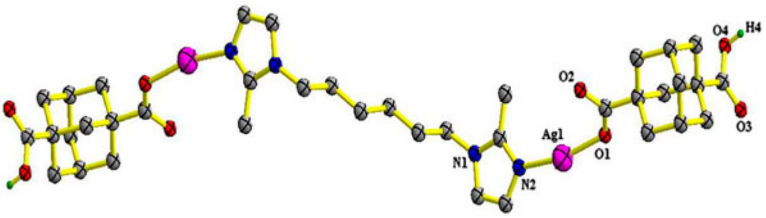


Figure 1. Local coordination environment of the Ag (I) ion in **1**.

acceptors and donors in the framework of **1**. Discrete units are connected through hydrogen–bonding interactions (O4–H4···O2 2.5524(64) Å), resulting in a 2-D network (figure 2). Systematic investigation has been reported on structures of Ag(I) polycarboxylate incorporating secondary ligands, revealing that weak interactions could play an important role in interlinking low-dimensional entities to high-dimensional supramolecular frameworks [12]. Adjacent 2D gird layers stack parallel, and arrange in an offset fashion as observed along the *a* axis (figure S1).

Though one carboxylic group is not coordinated in HADC, 1,3-adamantanedicarboxylic acid is a V-shaped ligand, and the angle between the coordinating sites is ca. 110° and can improve the interpenetrating structure. The flexible 1,6-bis(2-methyl-imidazole-1-yl)-hexane ligand with six methylene groups results in a fascinating type of OD→2D interpenetrating architecture. A representation of the beautiful topology is presented in figure 3.

3.1.2. $[Cd(ADC)(bpp)]_n$ (2).

Single-crystal X-ray diffraction analysis reveals **2** with a chiral *P212121* space group. The structure of **2** contains an octahedral Cd(II), coordinated to two ADC ligands and two bpp ligands. The Cd1–N bond lengths from 2.3012(31) to

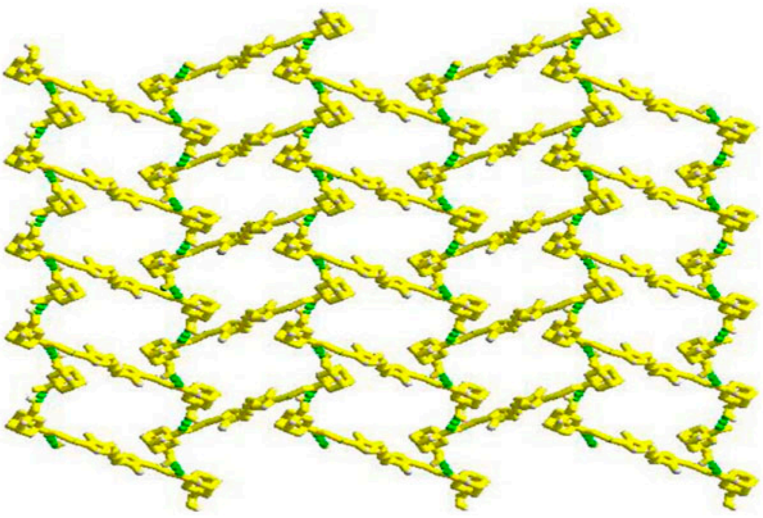


Figure 2. 2D network grid through the hydrogen–bonding interactions.

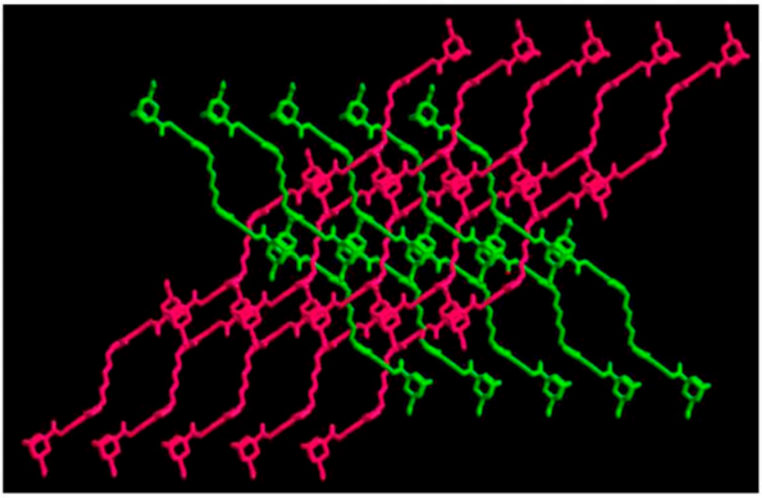


Figure 3. Fascinating 0D→2D interpenetrating architecture.

2.3512(32) Å and the Cd1–O bond lengths (2.2177(29)–2.4060(38) Å) are in agreement with those reported in other Cd(II) complexes of N, O–mixed ligands [13]. The coordination geometry around Cd(II) is shown in figure 4. Each ADC is bis-bidentate chelating to connect adjacent Cd(II) ions forming a left-handed helical chain with a pitch of 24.196 Å. Synchronously, Cd(II) ions are also bridged by bpp ligands to form a right-handed helical chain with a pitch of 13.090 Å, as shown in figure 5.

The structure of **2** contains chiral microchannels which are made of right-handed helical chains [–Cd^{2+}–bpp–Cd^{2+}–ADC–Cd^{2+}]$_n$ with dimensions of 24.61 × 23.47 Å^2 (figure 6). Further analysis indicates that it is a typical diamondoid framework containing large

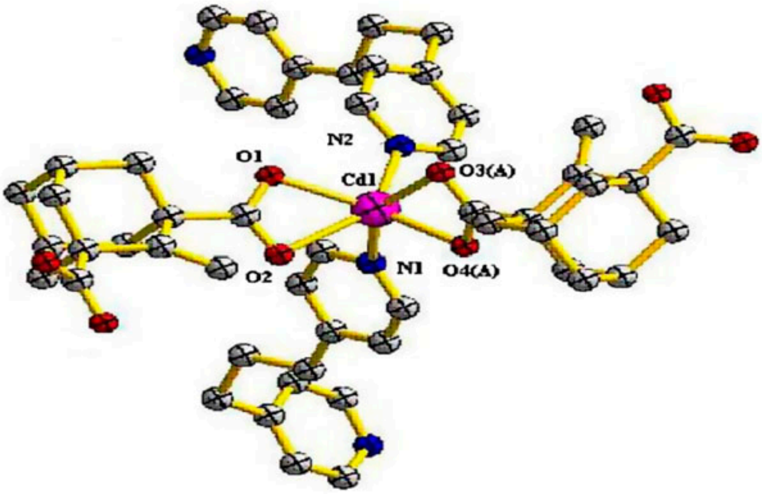


Figure 4. Coordination environment of Cd(II) atom.

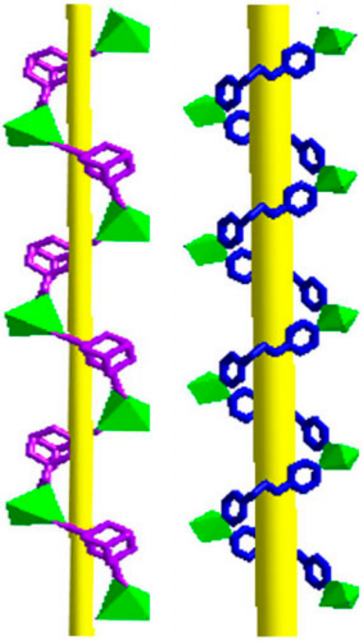


Figure 5. The two kinds of helical chains.

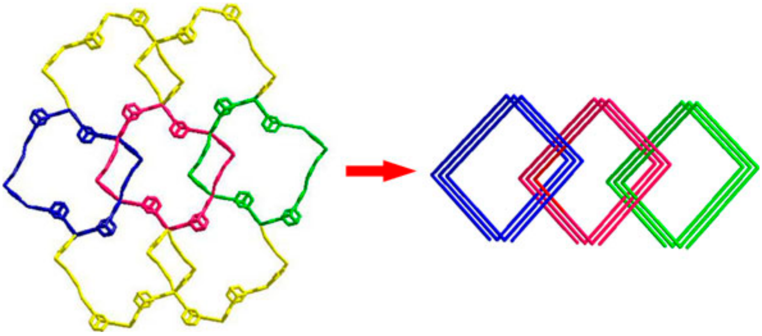


Figure 6. The chiral microchannels structure.

adamantanoid cages. Because of the spacious nature of the single network, two identical diamondoid networks interpenetrate in a normal mode giving rise to a 3D→3D interpenetrating dia array, as illustrated in figure 7.

3.2. *Fluorescent properties*

Solid-state luminescence of **1** and **2** and free ligands is depicted in figure 8. The emission spectrum for **1** shows a main peak at 395 nm upon excitation at 280 nm. Whereas, pure bimh exhibits a fluorescent emission band at 440 nm (λ_{ex} = 280 nm, solid H_2ADC does not

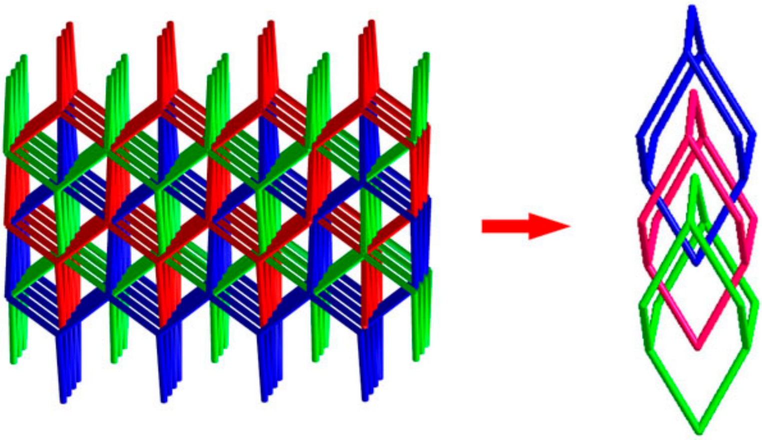


Figure 7. The 3D→3D interpenetrating dia array.

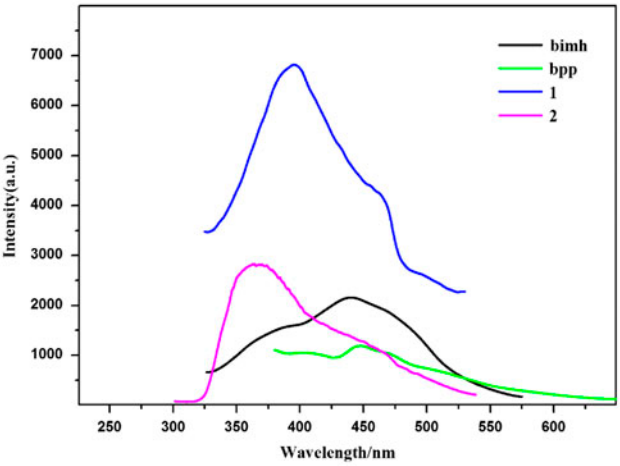
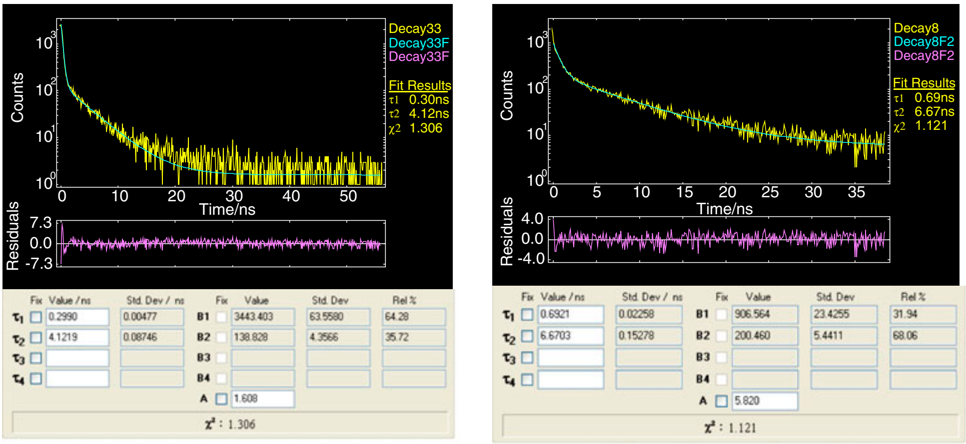


Figure 8. Photoluminescent spectra of **1** and **2** and the free ligands.

possess any luminescence at room temperature). The emission peak of **1** is blue-shifted by 45 nm compared to pure bimh. Complex **2** shows a main peak at 365 nm upon excitation at 280 nm, blue-shifted by about 81 nm relative to that of the pure bpp ligand (λ_{max} = 446 nm, λ_{ex} = 280 nm for bpp). According to literature, the emissions of **1** and **2** are probably assigned to ligand to metal charge-transfer (LMCT) transitions [14].

Lifetimes in **1** and **2** are monitored at room temperature upon excitation at 280 nm and all data are well fitted as shown in figure 9. The lifetime of 395 nm emission of **1** shows 0.2990 ns (64.28%)/4.1219 ns (35.72%), χ^2 = 1.306, and the lifetime at 365 nm emission of **2** shows 0.6921 ns (31.94%)/6.6703 ns (68.06%), χ^2 = 1.121. Thus, compounds **1** and **2** may be excellent candidates for potential photoactive materials.



	Fix	Value /ns	Std. Dev / ns		Fix	Value	Std. Dev	Rel %
τ_1	☐	0.2990	0.00477	B1	☐	3443.403	63.5580	64.28
τ_2	☐	4.1219	0.08746	B2	☐	138.828	4.3566	35.72
τ_3	☐			B3	☐			
τ_4	☐			B4	☐			
				A	☐	1.608		

χ^2 : 1.306

	Fix	Value /ns	Std. Dev / ns		Fix	Value	Std. Dev	Rel %
τ_1	☐	0.6921	0.02258	B1	☐	906.564	23.4255	31.94
τ_2	☐	6.6703	0.15278	B2	☐	200.460	5.4411	68.06
τ_3	☐			B3	☐			
τ_4	☐			B4	☐			
				A	☐	5.820		

χ^2 : 1.121

Figure 9. Lifetime at 395 nm emission of **1** and lifetime at 365 nm emission of **2**.

3.3. *XRPD pattern and thermal stability analysis*

To confirm the phase purity of **1** and **2**, the X-ray powder diffraction (XRPD) patterns were checked at room temperature. Although minor differences can be observed in the positions, intensities, and widths of some peaks, compounds **1** and **2** were obtained as a single phase (see figure S2 in the Supplementary material).

Thermal stabilities of **1** and **2** were also examined (see figure S3 in the Supplementary material). For **1**, the framework is stable up to 190 °C, and then decomposition of organic components occurs (Obsd 76.08%, Calcd 76.21%). Finally, the residual weight of 23.92% may be attributed to the percentage (expected 23.79%) of Ag. **2** that is stable up to 150 °C. The framework collapsed from 150 to 770 °C, suggesting that the organic ligands decompose slowly over a wide temperature range. The remaining weight of 24.01% is in accord with the mass of CdO residue (Calcd: 24.06%).

4. Conclusion

The research is focused on synthesis, structural characterization, and properties of two new metal–organic compounds based on 1,3-adamantanedicarboxylic acid (H_2ADC) and N-auxiliary ligands. Compound **1** exhibits a discrete symmetric unit with 0D→2D interpenetrating structure. Compound **2** presents a threefold interpenetrated 3D diamondoid network containing three helical chains. Thermal stability, X-ray powder diffraction, and luminescent properties for **1** and **2** are also measured and discussed.

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Supplementary material

Crystallographic data for **1** and **2** have been deposited at the Cambridge Crystallographic Data Center as supplementary publications (CCDC: 938827 and 941237). The data can be obtained free of cost at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internet.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk. Supplemental data for this article can be accessed http://dx.doi.org/10.1080/00958972.2013.844800.

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