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A new Zn^{II} two-dimensional coordination polymer, {[Zn(μ -4,4'-bipy)(1,4-ndc)(H₂O)₂] · (H₂O)}_n (4,4'-bipy= 4,4'-bipyridine and 1,4-ndc=1,4-naphthalenedicarboxylate)

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**A new Zn^{II} two-dimensional coordination polymer,
{[Zn(μ -4,4'-bipy)(1,4-ndc)(H₂O)₂]·(H₂O)}_n (4,4'-bipy =
4,4'-bipyridine and 1,4-ndc = 1,4-naphthalenedicarboxylate)**

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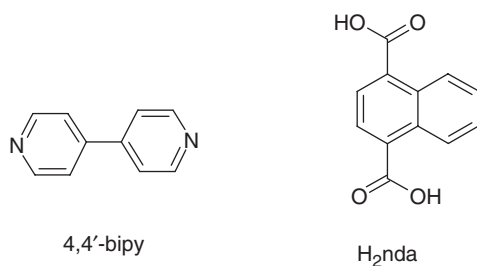
A 2D Zn^{II}(μ -4,4'-bipy) coordination polymer with 1,4-naphthalenedicarboxylate, {[Zn(μ -4,4'-bipy)(1,4-ndc)(H₂O)₂]·(H₂O)}_n, has been synthesized, characterized and studied by X-ray crystallography. The structural studies show the Zn atoms have six-coordinate geometry with a distorted octahedral environment. The 2D structure is grown by hydrogen bonds into a hybrid three-dimensional network.

Keywords: Zinc(II); 4,4'-Bipyridine; 1,4-Naphthalenedicarboxylate; Coordination polymer

1. Introduction

Metal-organic frameworks (MOFs) have received attention in recent years because of potential applications in gas storage, adsorption (separation), porosity (molecular selection), ion exchange, conduction, catalysis and nanowires [1]. Coordination of the metal with 1,4-naphthalenedicarboxylate (nda) is less investigated [2]. In constructing MOFs, dicarboxylates are selected because they may act as both counter-anions and bridging ligands to extend the architecture to 1D chains, 2D layers and 3D networks [3]. In this article, nda is utilized to build a coordination network for two reasons. It provides directional conformation via coordination interactions with versatile bonding modes as well as via non-covalent bonding interactions such as hydrogen bonding and π - π stacking interactions. It can construct MOFs with channels because of its linear structure. The 4,4'-bipyridine is used as an excellent spacer in the construction of supramolecular architectures [4]. Both ligands are of 7.0 Å, favorable for formation of a square grid or cubic framework. The reported structures of MOFs constructed

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Scheme 1. The organic ligands used in the construction of zinc(II) coordination polymer.

by carboxylates and 4,4'-bipy are 1D chains [5], 2D grids (square grids [6] or others [7]) and 2D sheets pillared by 4,4'-bipy to form a 3D network [8]. Each edge of the square grid is mostly built by linkage of two ligands.

Zn(II) complexes attract interest for appealing structures, photoluminescent and non-linear optical properties [9]. The spherical d^{10} configuration has flexible coordination environments so that geometries can vary from tetrahedral to octahedral and severe distortions in the ideal polyhedron easily occur. Due to lability of Zn complexes, formation of coordination bonds is reversible enabling rearrangement during polymerization to give highly-ordered network structures. Consequently, Zn readily accommodates various architectures giving selection of topological types of 1D, 2D and 3D polymers. The present determination of the structure 1,4-naphthalenedicarboxylate (nda) Zn(II) complex of 4,4'-bipyridine (4,4'-bipy) provides a new two-dimensional coordination polymer (Scheme 1).

2. Experimental

2.1. Physical property measurements

All reagents and solvents for synthesis and analysis were commercially available and used as received. IR spectra were recorded using Perkin-Elmer 597 with pressed potassium bromide (KBr) disks. Microanalyses were carried out using a Heraeus CHN-O-Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected.

2.2. Syntheses of $\{[Zn(\mu-4,4'-bipy)(1,4-nda)(H_2O)_2] \cdot (H_2O)\}_n$

A methanol solution (10 mL) containing 4,4'-bipyridine (0.2 mmol, 0.031 g) was added slowly into a *N,N*-dimethylformamide (10 mL) solution containing $ZnCl_2 \cdot 2H_2O$ (0.2 mmol, 0.034 g) and 1,4-naphthalenedicarboxylic acid (0.2 mmol, 0.044 g). The mixture solution was left to stand at room temperature in a covered vessel and in about three weeks colorless crystals were obtained, M.P. $>320^\circ C$, Yield: 0.049 g, 50%. Anal. Calcd for $C_{22}H_{20}N_2O_7Zn$: C, 53.95; H, 4.12; N, 5.72%. Found: C, 53.91; H, 4.06; N, 5.65%. IR (selected signals, cm^{-1}): 3563s, 3425w, 1607s, 1558s, 1510m, 1468m, 1413m, 1363s, 815m, 631m.

Table 1. Crystal data and structure refinement for $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$.

Identification code	1
Empirical formula	C ₂₂ H ₂₀ N ₂ O ₇ Zn
Formula weight	489.77
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system, space group	Monoclinic, <i>P2/c</i>
Unit cell dimensions (Å, °)	
<i>a</i>	9.8808(7)
<i>b</i>	11.5275(8)
<i>c</i>	9.8189(7)
β	109.8960(10)
Volume (Å ³)	1051.63(13)
Z, Calculated density (Mg m ⁻³)	1, 1.547
Absorption coefficient (mm ⁻¹)	1.216
<i>F</i> (000)	504
Crystal size (mm ³)	0.36 × 0.32 × 0.21
θ range for data collection	1.77 to 26.00°
Limiting indices	-12 ≤ <i>h</i> ≤ 5, -13 ≤ <i>k</i> ≤ 14, -12 ≤ <i>l</i> ≤ 11
Reflections collected/unique	5824/2059 [<i>R</i> (int) = 0.0224]
Completeness to $\theta = 26.00$	99.7%
Absorption correction	'Multi-scan'
Max. and min. transmission	0.7844 and 0.6687
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	2059/0/152
Goodness-of-fit on <i>F</i> ²	1.095
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0341, <i>wR</i> ₂ = 0.0809
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0358, <i>wR</i> ₂ = 0.0818
Largest diff. peak and hole (e Å ⁻³)	0.374 and -0.365

2.3. Crystallography

X-ray measurements were made at 298(2)K using a Bruker SMART APEX CCD diffractometer. The intensity data were collected within the theta range 1.77 to 26.00° using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and an orientation matrix for data collection were obtained from least-squares refinement. The structure was solved by direct methods and refined by full-matrix least-squares on *F*². Crystal data and refinement parameters are given in table 1. Selected bond lengths and angles are given in table 2. An ORTEP diagram and a perspective view of the packing are shown in figures 1–3.

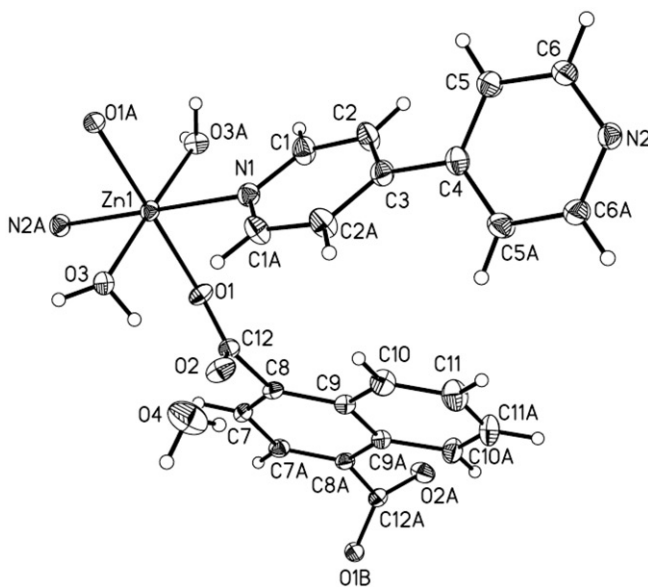
3. Results and discussion

Reaction between 4,4'-bipyridine and mixtures of zinc(II) chloride dihydrate with 1,4-naphthalenedicarboxylic acid provided crystalline materials analyzing as $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$. The IR spectrum of $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$ shows characteristic bands of 4,4'-bipy, 1,4-ndc and H₂O. The absorption band at 1607 cm⁻¹ is assigned to the C–N of 4,4'-bipy. Bands in the region 621–1067 cm⁻¹ are due to bending of C–H in or out of the aromatic plane with peaks at 631 and 815 cm⁻¹ attributed to the aromatic ring of 4,4'-bipy and 1,4-ndc [10]. The absorptions of the C–C bond of the aromatic rings occur at 1413 and 1468 cm⁻¹.

Table 2. Bond lengths (Å) and angles (°) for $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$.

Zn(1)–O(1)	2.0904(15)
Zn(1)–O(1)#1	2.0904(15)
Zn(1)–O(3)	2.0946(16)
Zn(1)–O(3)#1	2.0946(16)
Zn(1)–N(2)#2	2.184(3)
Zn(1)–N(1)	2.249(3)
O(1)–Zn(1)–O(1)#1	179.06(9)
O(1)–Zn(1)–O(3)	90.99(6)
O(1)#1–Zn(1)–O(3)	89.03(6)
O(1)–Zn(1)–O(3)#1	89.03(6)
O(1)#1–Zn(1)–O(3)#1	90.99(6)
O(3)–Zn(1)–O(3)#1	177.64(8)
O(1)–Zn(1)–N(2)#2	89.53(4)
O(1)#1–Zn(1)–N(2)#2	89.53(4)
O(3)–Zn(1)–N(2)#2	91.18(4)
O(3)#1–Zn(1)–N(2)#2	91.18(4)
O(1)–Zn(1)–N(1)	90.47(4)
O(1)#1–Zn(1)–N(1)	90.47(4)
O(3)–Zn(1)–N(1)	88.82(4)
O(3)#1–Zn(1)–N(1)	88.82(4)
N(2)#2–Zn(1)–N(1)	180.000(1)

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

Figure 1. Molecular structure of $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$.

Absorptions at 1558 and 1363 cm^{-1} are due to the asymmetric and symmetric stretching of COO^- of the 1,4-ndc [11], respectively. The $\Delta\nu$ value ($\nu_{\text{as}}-\nu_{\text{sym}}$) of 195 cm^{-1} indicates that the carboxylate group coordinates monodentate [12–16], confirmed by the crystal structure. The peaks at 3563 and 1510 cm^{-1} are due to stretching and bending of H_2O [10].

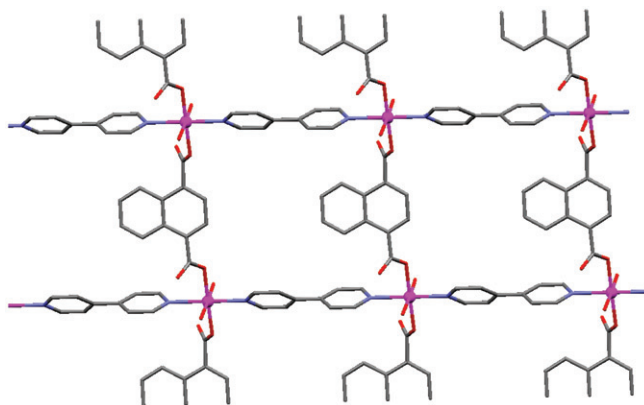


Figure 2. A perspective view of the two-dimensional layers in $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$.

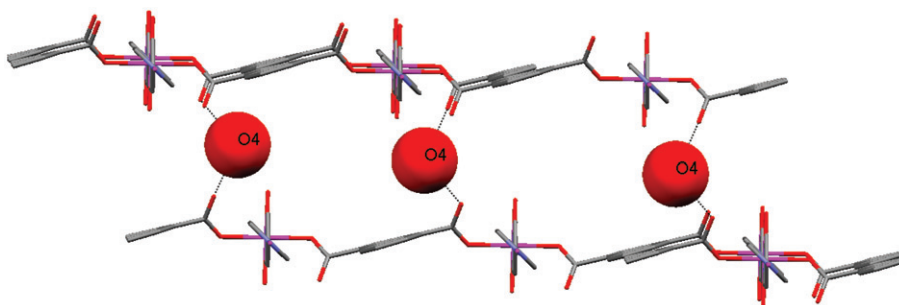


Figure 3. A perspective view of the packing down the *a* axis in $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$, showing the free water molecules lying in the interlayer region.

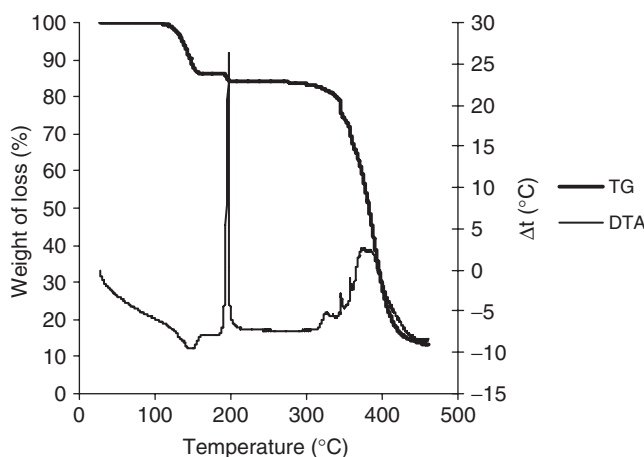
Determination of the structure of the $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$ by X-ray crystallography (tables 1 and 2) showed the complex in the solid state (figures 1 and 2) consists of two-dimensional polymeric units consisting of layers formed by bridging 4,4'-bipy and 1,4-ndc ligands. The carboxylate groups of the 1,4-ndc ligand are unidentate bridging with a $\text{Zn} \cdots \text{Zn}$ separation of 11.314 Å. These one-dimensional chains are connected through “4,4'-bipy” bridges to other nodes, resulting in two-dimensional squares with dimensions $11.314 \times 11.528 \text{ \AA}^2$. The intra-chain $\text{Zn} \cdots \text{Zn}$ distances connected by “4,4'-bipy” ligands are 11.528 Å; each Zn^{II} resides along a two-fold axis ($-x, y, 1/2 - z$) that is co-linear with the 4,4'-bipyridine ligand and has a distorted octahedral coordination sphere with $(\text{O}_2)_{\text{ndc}}(\text{O}_2)_{\text{water}}(\text{N}_2)_{\text{py}}$ donor atoms. Two nitrogens from two “4,4'-bipy” ligands are *trans* [the angle $\text{N1-Zn1-N1}^{\text{i}}$ is 180.00°].

The structures of $\text{M}(\text{H}_2\text{O})_2(4,4'\text{-bipy})[\text{C}_6\text{H}_4(\text{COO})_2] \cdot 2\text{H}_2\text{O}$ ($\text{M} = \text{Mn}^{2+}, \text{Co}^{2+}$) have recently been reported [17], where Mn^{2+} and Co^{2+} ions are moderately distorted octahedra with two waters, two oxygens of phthalate anions and two nitrogens of 4,4'-bipy. The environment of metal ions and coordination mode of ligands are very similar with our compound here. Two isostructural coordination polymers, $\text{M}(\text{H}_2\text{O})_2(4,4'\text{-bipy})[\text{C}_6\text{H}_4(\text{COO})_2] \cdot 2\text{H}_2\text{O}$ ($\text{M} = \text{Mn}^{2+}, \text{Co}^{2+}$) have a layered structure

Table 3. Hydrogen bonds for $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$ (Å) and ($^\circ$).

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
O(3)-H(3A)...O(2)	0.85	1.93	2.725(2)	155.4
O(3)-H(3B)...O(1)#5	0.85	1.90	2.744(2)	171.9
O(4)-H(4A)...O(4)#6	0.85	2.08	2.785(10)	139.5
O(4)-H(4A)...O(2)	0.85	2.19	2.800(5)	128.9
O(4)-H(4B)...O(2)#6	0.85	2.08	2.830(5)	147.4

Symmetry transformations used to generate equivalent atoms: #5x, -y+2, z-1/2; #6 -x+2, y, -z+3/2.

Figure 4. Thermal behavior of $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$.

similar to $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$. Both lattice and coordinated water molecules are involved in hydrogen bonding networks (figure 3). The water molecules are hydrogen bond donors to uncoordinated O atoms of 1,4-ndc (table 3). The coordinated water only participates in intramolecular hydrogen bonding with oxygens of carboxylate, whereas the interstitial water forms intermolecular hydrogen bonds bridging the two layers. Consequently, the 2D structure may be considered as a hybrid three-dimensional network formed from coordinative and hydrogen bonds.

The thermal decomposition of $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})\}_n$ was investigated in air from ambient to 700°C (figure 4). It does not melt and is stable up to 120°C at which temperature the water is removed with an endothermic effect. Second, strongly exothermic mass loss at 200°C may be due to elimination of coordinated water. The experimental mass loss of 12.1% is consistent with the calculated value 11.5% for elimination of three H₂O. The solid residue is suggested to be $\{[\text{Zn}(\mu\text{-}4,4'\text{-bipy})(1,4\text{-ndc})]\}$. At higher temperatures, decomposition of the residue occurs with two exothermic effects at 200 and 400°C. This final process is accompanied by the decomposition of 4,4'-bipy and "1,4-ndc", ultimately giving a brown, amorphous solid that appears to be ZnO. The XRD pattern is in agreement with the typical wurtzite ZnO diffraction (figure 5) (hexagonal phase, space group P6₃mc, with lattice constants $a = 3.24982(9)\text{Å}$, $c = 1.6021\text{Å}$, $Z = 2$, JCPDS No. 36-1451).

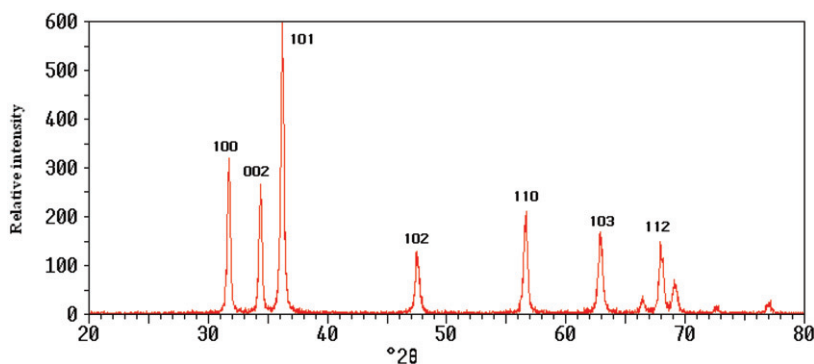


Figure 5. X-ray powder diffraction pattern of ZnO.

Supplementary material

Crystallographic data for the structure reported in the article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-659593. Copies of the data can be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: +44-1223/336033; Email: deposit@ccdc.cam.ac.uk].

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