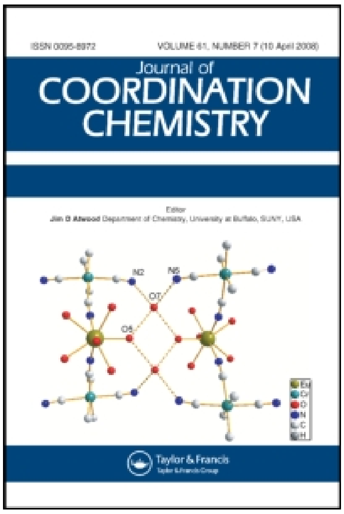

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Syntheses and crystal structures of two Cd(II) coordination polymers: one-dimensional chain $[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_{\text{<b><i>n</i>}}$ and two-dimensional sheet $[Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)]_{\text{<b><i>n</i>}}$ · <i>n</i>H_2O

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Syntheses and crystal structures of two Cd(II) coordination polymers: one-dimensional chain $[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$ and two-dimensional sheet $[Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)]_n \cdot nH_2O$

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Two new Cd(II) coordination polymers, $[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$ (1) (where $C_4H_6N_2 =$ 2-methylimidazole, $C_4H_2O_4 =$ fumarate), and $[Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)]_n \cdot nH_2O$ (2), (where $C_4H_4O_4 =$ succinates), have been prepared and structurally characterized by single crystal X-ray diffraction. Complex **1** crystallizes in the triclinic space group $P\bar{1}$ in a one-dimensional chain structure, in which carboxy is monodentate; a three-dimensional supermolecular network structure was formed through hydrogen bonding. In complex **2**, the coordination geometry of the Cd atoms is a pentagonal bipyramid, and a two-dimensional sheet is formed though carboxyl group bridging. In **1** and **2**, IR spectra indicate the presence of bridging carboxyl groups, confirmed by structure analyses.

Keywords: Coordination polymer; Cadmium; Carboxyl group; Network structure

1. Introduction

The past decade has witnessed an enormous expansion of research on coordination polymers based on metal-organic ligand coordination. Coordination polymers with specific topologies display promising properties for potential technological and industrial application as catalysts, hosts in intercalation compounds, and electronic, electro-optical, photo-optical, and magnetic materials [1–10]. Rational design and syntheses of coordination polymers using rigid di- and polycarboxylates as bridging spacers are of interest [11, 12]. Based on flexibility and conformational freedom, aliphatic dicarboxylate ligands have given rise to a variety of interesting structural motifs; π–π stacking interactions between chelating heteroaromatic *N*-donor ligands play very important roles in the construction of coordination polymers with interesting

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supramolecular architectures [13–43]. Cd(II) coordination polymers with fumarate [44] and succinates [10, 45] have been reported, $[Cd(C_3H_4N_2)_3(C_4H_2O_4)]_n \cdot 2nH_2O$ (where $C_3H_4N_2$ = imidazole, $C_4H_2O_4$ = fumarate) [44], in which one carboxyl of fumarate is monodentate and the other bidentate. For $[Cd_3(OH)_2\ (C_4H_4O_4)_2]$ (where $C_4H_4O_4$ = succinates) [10, 45], containing CdO_6 octahedra and edge-shared Cd_2O_{10} bi-octahedra, the formed 2D layers are pillared by succinate groups to constitute a 3D coordination polymer [10]; Cd_4O_{18} tetramers are joined together forming infinite Cd–O sheets [45]. Herein, we report two new Cd(II) coordination polymers $[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$ (**1**), in which each carboxyl group of fumarate is monodentate and the polymer has a one-dimensional network structure and $[Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)]_n \cdot nH_2O$ (**2**), in which the Cd atom is a seven-coordinate pentagonal bipyramidal with carboxyl group of succinates chelating bis-bidentate and chelating/bridging *bis*-bidentate coordination, and the polymer exhibiting a two-dimensional sheet structure.

2. Experimental methods

2.1. *Physical measurements*

All reagents for syntheses were purchased from commercial sources and used as received. Elemental analyses (C, H, N) were carried on an Elementar Vario EL instrument. Infrared spectra (KBr pellets) were measured on a MAGNA-IR 560 FTIR spectrometer in the 400–4000 cm^{-1} region.

2.2. *Syntheses*

$[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$ (1). $Cd(NO_3)_2 \cdot 4H_2O$ (0.31 g; 1 mmol), fumaric acid (0.12 g, 1 mmol) and 2-methylimidazole (0.16 g, 2 mmol) in the molar ratio of 1 : 1 : 2 were dissolved in distilled water (25 mL) and the pH value was adjusted to 7 with 2M NaOH. The resulting solution was heated at 60°C for 1 h with stirring. The solvent was allowed to evaporate slowly at room temperature and small colorless single crystals appeared in the bottom of the cup in yields of 25%. EA for $C_{12}H_{18}CdN_4O_6$ (**1**) (%): C 33.78, H 4.25, N 13.13; found C 33.75, H 4.25, N 13.15. The IR spectra show higher frequencies 3129 and 3192 cm^{-1}, characteristic of the 2-MeIm groups and two strong peaks corresponding to the $\nu(COO^-)$ vibration at 1553 and 1577 cm^{-1}.

$[Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)]_n \cdot 0.5nH_2O$ (2). $Cd(NO_3)_2 \cdot 4H_2O$ (0.31 g; 1 mmol), succinic acid (0.12 g, 1 mmol) and 2-methylimidazole (0.16 g, 2 mmol) in the molar ratio of 1 : 1 : 2 were dissolved in a mixture of distilled water and ethanol (25 mL) and the pH value was adjusted to 6 with NaOH. The resulting solution was heated at 80°C for 2 h with stirring. The solvent was allowed to evaporate slowly at room temperature and light colorless single crystals appeared in the bottom of the cup in yields of 40%. EA for $C_{16}H_{28}Cd_2N_4O_{12}$ (**2**) (%): C 27.72, H 4.04, N 8.08; found C 27.70, H 4.05, N 8.12. The IR spectra showed at higher frequencies 3128 and 3254 cm^{-1}, characteristic of the 2-MeIm groups. One strong peak corresponding to $\nu(COO^-)$ was observed at 1543 cm^{-1}.

2.3. *X-ray crystallography*

Diffraction data for **1** and **2** were collected at 293 K, with a R-AXIS RAPID IP diffractometer, using the ω-scan technique with Mo-Kα radiation ($\lambda = 0.71073$ Å). The unit cell parameters were obtained by a least-squares refinement of setting angles of 11882 reflections with $3.13^\circ < \theta < 27.48^\circ$ for **1**, and of 11,749 reflections with $3.06^\circ < \theta < 27.50^\circ$ for **2**. The structures were solved by direct methods and refined by full-matrix least-squares based on F^2 with the SHELXTL program package [46]. The non-hydrogen atoms were subjected to anisotropic refinement. The hydrogen atoms bound to the carbon atoms were calculated theoretically. The weighting schemes are $\omega = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.0000P]$ **1** and $\omega = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 1.4137P]$ **2** where $P = (F_o^2 + F_c^2)/3$. The crystallographic data are summarized in Table 1. Selected bonds and angles for **1** are listed in table 2, those for **2** are listed in table 3.

3. Results and discussion

3.1. *Crystal structures*

$[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$ (1). Single crystal structure analysis reveals that **1** consists of a polymer $[Cd(C_4H_6N_2)_2(C_4H_2O_4)(H_2O)_2]_n$, with Cd(II) a distorted CdN_2O_4 octahedral geometry (figure 1). Two nitrogen atoms [N(1) and N(3)] from two monodentate 2-methylimidazole groups, two carboxylate oxygen atoms [O(1) and O(4A)] from the monodentate fumarate ligand and the other two oxygen atoms [O(7) and O(8)] from two water molecules build the octahedron, the mean value of the Cd–N bonds[2.266(2) Å] being somewhat shorter than that of Cd–O$_{\text{carboxylate}}$ [2.344(2) Å] and that of the Cd–N ones in the literature [2.29(2) Å] [44]. Each carboxyl group of fumarate

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

	1	**2**
Formula	$C_{12}H_{18}Cd\,N_4O_6$	$C_{16}H_{28}Cd_2N_4O_{12}$
Formula weight	426.70	693.22
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	8.5897(17)	8.6966(17)
b (Å)	9.913(2)	10.730(2)
c (Å)	15.550(3)	12.996(3)
α (°)	83.89(3)	84.57(3)
β (°)	84.11(3)	79.93(3)
γ (°)	66.59(3)	83.90(3)
V(Å^3)	1205.5(4)	1183.7(4)
Z	3	2
D_c(g cm^{-3})	1.763	1.945
μ(mm^{-1})	1.394	1.864
Crystal size (mm^3)	0.20 × 0.15 × 0.11	0.48 × 0.37 × 0.31
θ Range (°)	$3.13 < \theta < 27.48$	$3.06 < \theta < 27.50$
Reflections measured	11882	11749
Independent reflections (R_{int})	5439(0.0419)	5368 (0.0237)
R_1, wR_2	0.0277, 0.0911	0.0278, 0.0936
Goodness-of-fit	1.059	1.211

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

Cd(1)–N(1)	2.261(2)	Cd(1)–O(8)	2.361(2)
Cd(1)–O(4)#2	2.3302(18)	Cd(1)–O(7)	2.328(2)
Cd(1)–O(1)	2.3584(18)		
N(1)–C(2)–N(2)	110.5(2)	N(1)–C(2)–C(1)	126.4(2)
N(2)–C(2)–C(1)	123.2(2)	C(4)–C(3)–N(1)	110.1(2)
C(3)–C(4)–N(2)	105.9(2)	N(3)–C(6)–N(4)	110.1(2)
N(3)–C(6)–C(5)	126.1(2)	N(4)–C(6)–C(5)	123.8(2)
C(8)–C(7)–N(4)	106.1(2)	C(7)–C(8)–N(3)	110.1(2)
O(2)–C(15)–O(1)	125.2(2)	O(2)–C(15)–C(16)	118.2(2)
O(1)–C(15)–C(16)	116.6(2)	C(17)–C(16)–C(15)	121.8(2)
C(16)–C(17)–C(18)	121.7(2)	O(3)–C(18)–O(4)	125.2(2)
O(3)–C(18)–C(17)	118.4(2)	O(4)–C(18)–C(17)	116.4(2)
N(1)–Cd(1)–N(3)	173.69(6)	N(1)–Cd(1)–O(7)	94.25(9)
N(1)–Cd(1)–O(4)#2	88.63(7)	O(4)#2–Cd(1)–O(8)	88.72(7)

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

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

Cd(1)–N(1)	2.288(4)	Cd(1)–O(9)	2.323(3)
Cd(1)–O(3)#1	2.336(3)	Cd(1)–O(7)#2	2.337(3)
Cd(1)–O(1)	2.342(2)	Cd(1)–O(8)#2	2.512(3)
Cd(1)–O(2)	2.514(2)	Cd(2)–N(3)	2.276(4)
Cd(2)–O(10)	2.288(4)	Cd(2)–O(3)#1	2.348(3)
Cd(2)–O(1)	2.373(3)	Cd(2)–O(5)	2.397(3)
Cd(2)–O(6)	2.434(3)	Cd(2)–O(4)#1	2.495(3)
O(1)–C(1)–O(2)	121.4(3)	O(1)–C(1)–C(2)	117.4(3)
C(2)–C(3)–C(4)	115.1(3)	O(6)–C(5)–O(5)	121.9(3)
O(6)–C(5)–C(6)	120.6(3)	O(5)–C(5)–C(6)	117.5(3)
C(7)–C(6)–C(5)	116.0(3)	C(6)–C(7)–C(8)	115.9(3)
Cd(1)–O(1)–Cd(2)	108.15(9)	Cd(1)#3–O(3)–Cd(2)#3	109.20(10)
C(8)–O(7)–Cd(1)#4	96.6(2)		

Symmetry transformations used to generate equivalent atoms: #1 $x-1, y, z$; #2 $x, y, z-1$; #3 $x+1, y, z$; #4 $x, y, z+1$.

is monodentate [though O(1) towards Cd(1)], and carboxyl groups are not conjugated [C–O (1) = 1.271(3) Å, C–O (2) = 1.252(3) Å]. Neighboring Cd(II) ions were connected through monodentate carboxyl bridges to form a regular infinite 1D chain with the Cd···Cd distance of 10.217 Å (figure 2). Simultaneously, the chains are interconnected though two types of hydrogen bonds O–H···O and N–H···O to form a three-dimensional network involving coordinated carboxyl oxygen O(5) of fumarate and water O(8) with O8–H8A···O5 = 2.784 Å, coordinated carboxyl oxygen O(4) and water molecule O(7) with O7–H7B···O4 = 2.741 Å $[-x+2, -y, -z]$; and the uncoordinated nitrogen atom N(2) from a 2-methylimidazole group and the uncoordinated carboxyl oxygen O(3) with N2–H2···O3 = 2.871 Å $[x, y+1, z]$, the uncoordinated nitrogen atom N(4) from a 2-methylimidazole group and the uncoordinated carboxyl oxygen O(2) with N4–H4···O2 = 2.799 Å $[-x+1, -y, -z]$, the hydrogen bond distances are comparable with the reported 2.743 to 2.844 Å [44].

$[\mathrm{Cd(C_4H_6N_2)(H_2O)(C_4H_4O_4)}]_n \cdot n\mathrm{H_2O}$ (2). The asymmetric unit contains two Cd atoms, two 2-methylimidazole, two succinate, two bound water molecules and two free

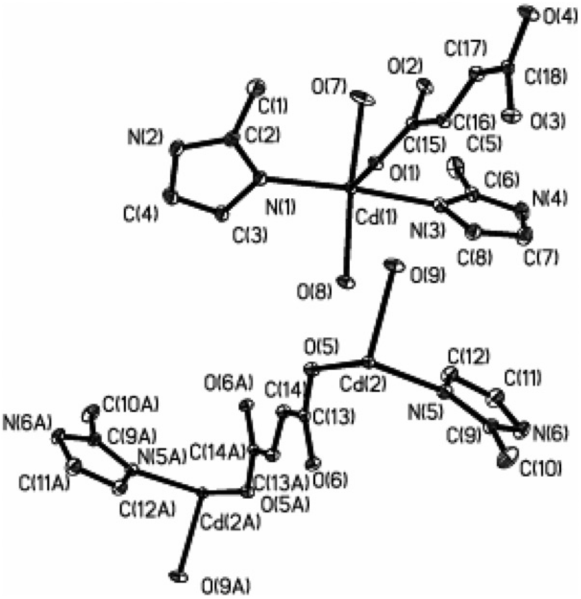

Figure 1. An ORTEP drawing of complex **1** showing the atom numbering scheme. The hydrogen atoms are omitted for clarity.

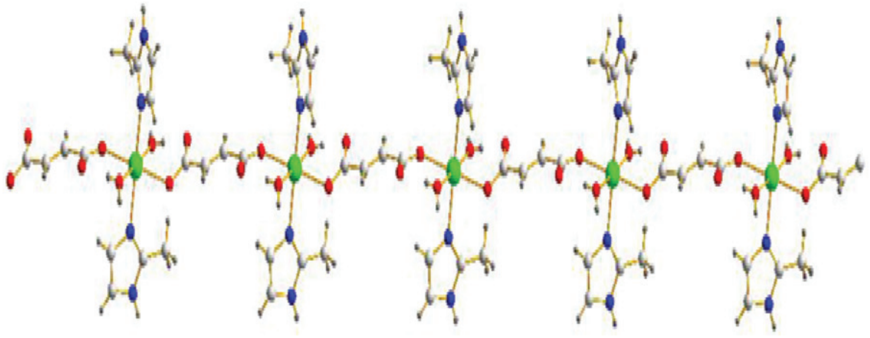

Figure 2. The one-dimensional chain structure of complex **1**.

water molecules. The cadmium atom is seven-coordinate with one nitrogen atom from 2-methylimidazole and six oxygen atoms, five from three succinates carboxyl groups and the other from water. Thus, the coordination geometry of the Cd atoms is a pentagonal bipyramid (figure 3). Five succinate oxygen atoms occupy the equatorial positions with an average Cd–O distance and O–Cd–O angle of 2.408 Å and 107.7°, respectively, comparable with the reported 2.393 Å and 107.6° [45], while the bonded water molecule and nitrogen atom of 2-methylimidazole occupy the apical positions with Cd–O distance of 2.306 Å and Cd–N distance of 2.282 Å. Succinate carboxyl group is chelating *bis*-bidentate and chelating/bridging *bis*-bidentate. Chelating *bis*-bidentate carboxyl groups connect Cd(1) and Cd(2) [through O(5) and O(6) towards Cd(2), O(7) and O(8) towards Cd(1)] with the adjacent Cd $\cdots$ Cd distance of 9.305 Å; chelating/bridging *bis*-bidentate carboxyl groups connect Cd(1) and Cd(2)

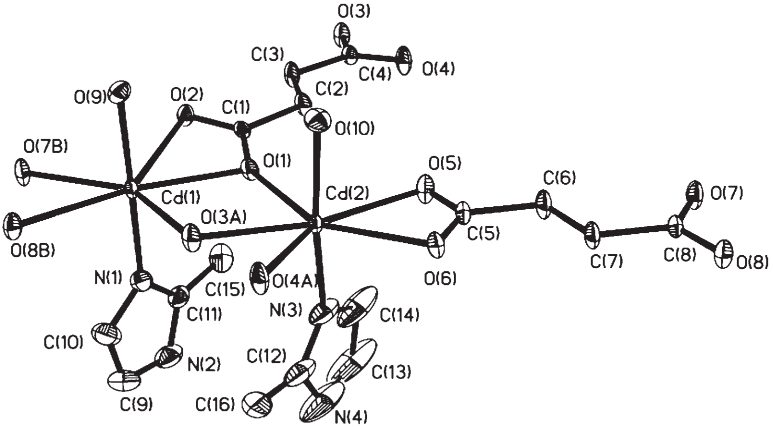

Figure 3. An ORTEP drawing of complex **2** showing the atom numbering scheme. The free H_2O molecule and hydrogen atoms are omitted for clarity.

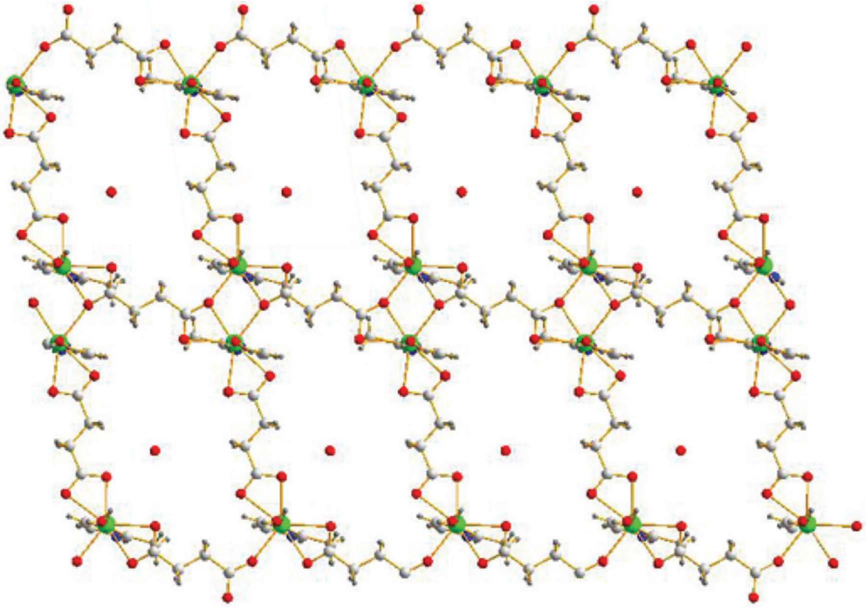

Figure 4. The two-dimensional layer structure of complex **2**.

[through O(2) towards Cd(1), O(4) towards Cd(2), O(1) and O(3) as bridges towards both Cd(1) and Cd(2)] with adjacent Cd···Cd distance of 3.818 Å. The connectivity between the Cd centers and succinate units results in perforated two-dimensional layers in the *ac*-plane, and free water molecules in the hole (figure 4). Such layers are stacked along the *b*-axis and are stabilized by hydrogen bond interactions. The three carboxyl group oxygen atoms [O(2), O(6), O(8)], two coordinated water molecules [O(9) and O(10)], two free water molecules [O(11) and O(12)], and two uncoordinated nitrogen

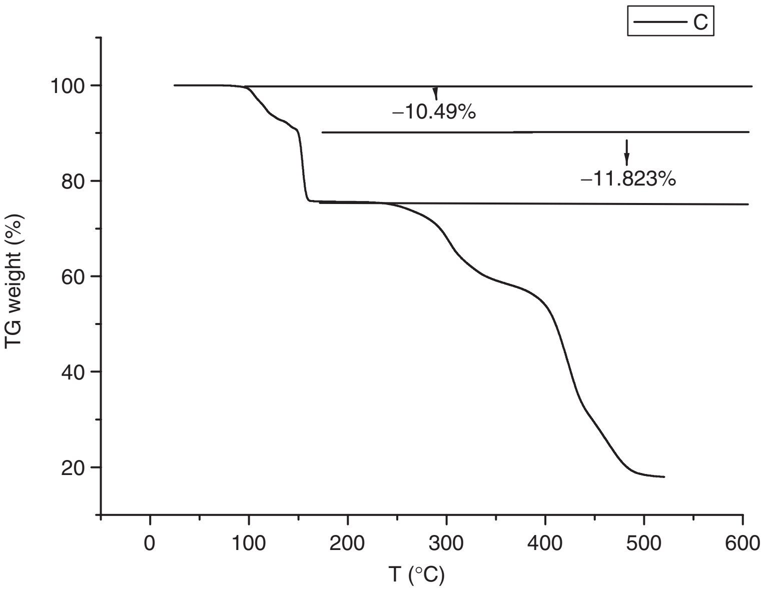

Figure 5. TGA for **2**.

atoms of 2-methylimidazole [N(2) and N(4)] participate in stabilizing cohesion of the structure with hydrogen bonds between the layers.

3.2. *TGA*

Thermal stability by thermogravimetric analysis (TGA) for **2** has been studied (see figure 5). TGA was performed at a heating rate of 10°C min^{-1} in the single crystal samples of **2**. Onset of water loss starts at 88.9°C and is completed by 150.5°C, a 10.49% weight loss was observed, which is almost equivalent to loss of four water molecules, consistent with the calculated weight content (Calcd 10.39%) (two bonded water molecules and two free water molecules). Further weight loss of 11.823% for the compound upon heating from 150.5 to 157.40°C is consistent with the calculated weight content (Calcd 11.914%) of one 2-methylimidazole molecule.

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