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Key indicators

Single-crystal X-ray study
 T = 180 K
 Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
 R factor = 0.025
 wR factor = 0.066
 Data-to-parameter ratio = 12.9

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

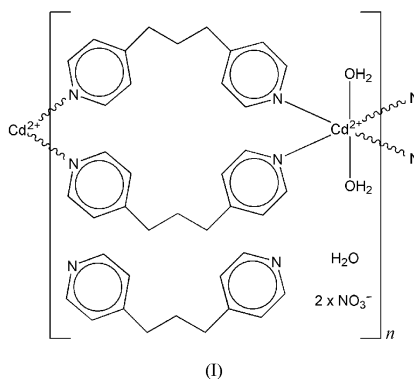
**catena-Poly[[[diaquacadmium(II)]-di- μ -4,4'-trimethylenedipyridine- $\kappa^2\text{N:N}'$]
 dinitrate 4,4'-trimethylenedipyridine
 monohydrate]**

The title compound, $\{[\text{Cd}(\text{TMD})_2(\text{H}_2\text{O})_2](\text{NO}_3)_2 \cdot \text{TMD} \cdot \text{H}_2\text{O}\}_n$, where TMD is 4,4'-trimethylenedipyridine ($\text{C}_{13}\text{H}_{14}\text{N}_2$), obtained using hydrothermal synthesis followed by slow crystallization, is formed by strong hydrogen bonds between a one-dimensional coordination polymer and uncoordinated TMD, water molecules and nitrate ions.

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Comment

We are interested in the hydrothermal synthesis of novel metal-organic frameworks which incorporate not only rigid and symmetrical spacers, but also ligands capable of inducing supramolecular isomerism into the final structure (Almeida Paz *et al.*, 2002).



The title compound, (I), contains a one-dimensional cationic coordination polymer, $[\text{Cd}(\text{TMD})_2(\text{H}_2\text{O})_2]_n^{2n+}$ (Figs. 1 and 2), where TMD is 4,4'-trimethylenedipyridine, similar to the Co and Ni analogues previously reported (Plater *et al.*, 2001). Uncoordinated TMD ligands, nitrate ions and water molecules are involved in an extensive hydrogen-bonding network with the cationic one-dimensional polymers (Table 2 and Fig. 3).

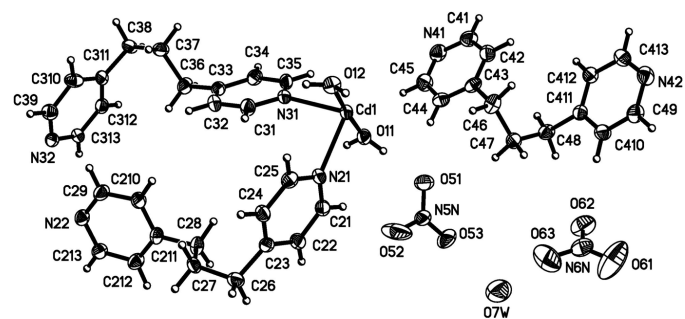


Figure 1
 The asymmetric unit of (I), represented with ellipsoids drawn at the 50% probability level for non-H atoms. H atoms on atom O7W are not shown.

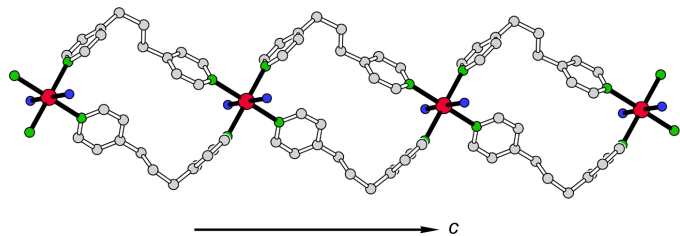


Figure 2
The one-dimensional cationic $[\text{Cd}(\text{TMD})_2(\text{H}_2\text{O})_2]_n^{2n+}$ coordination polymer in (I), running parallel to the c direction. H atoms have been omitted for clarity.

Experimental

All chemicals were obtained from commercial sources and used as received. To a solution of $\text{Cd}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.629 g, Aldrich) in distilled water (12.5 g), 4,4'-trimethylenedipyridine (TMD, 0.407 g, Aldrich) was added and the mixture was stirred thoroughly for 1 h at ambient temperature. The suspension, with the $\text{Cd}^{2+}:\text{TMD}:\text{H}_2\text{O}$ composition 1:1.01:339, was placed in a Parr stainless steel Teflon-lined vessel (21 ml, 50% filled). The reaction took place under autogeneous pressure and static conditions in a preheated oven at 418 K for 3 h. The vessel was then cooled slowly inside the oven to 298 K at a rate of 5 K h^{-1} before opening. The microcrystalline product was then cooled to 277 K. The solvent was allowed to evaporate at this temperature over a period of two months. Crystals of (I) were collected by vacuum filtration. Bulk homogeneity was confirmed using powder X-ray diffraction and CHN elemental analysis; calculated (using single-crystal data): C 52.91, H 5.47, N 12.66%; found: C 52.1, H 5.23, N 12.39%.

Selected FT-IR data (cm^{-1}): $\nu(\text{water, O-H}) = 3545 (m)$; hydrogen-bonded pyridine rings $\nu(\text{C-H}) = 3254 (m)$, $3070 (m)$ and $3026 (m)$; $\nu_{\text{antisym}}(\text{C-H in } -\text{CH}_2-) = 2925 (w)$; $\nu_{\text{sym}}(\text{C-H in } -\text{CH}_2-) = 2860 (w)$; weak overtone and combination bands observed in the range $2020\text{--}1800 \text{ cm}^{-1}$, characteristic of 4-monosubstituted pyridines (three bands); 4-monosubstituted pyridine aromatic ring $\nu(\text{C-H}) = 1607 (s)$, $1558 (m)$, $1503 (m)$, $1424 (s)$ and $1010 (m)$; 4-monosubstituted pyridine aromatic ring $\delta(\text{C-H}) = 1222 (m)$, $1068 (m)$ and $840 (m)$; characteristic vibration frequencies of the nitrate ion at $1385 (s)$, $816 (m)$ and $760 (m)$; $\delta(\text{Cd-OH}_2) = 800 (m)$; $\nu(\text{Cd-OH}_2) = 510 (m)$.

Crystal data

$[\text{Cd}(\text{C}_{13}\text{H}_{14}\text{N}_2)_2(\text{H}_2\text{O})_2] \cdot (\text{NO}_3)_2 \cdot \text{C}_{13}\text{H}_{14}\text{N}_2 \cdot \text{H}_2\text{O}$
 $M_r = 885.25$
Triclinic, $P1$
 $a = 9.8088 (3) \text{ \AA}$
 $b = 10.6080 (3) \text{ \AA}$
 $c = 12.0502 (4) \text{ \AA}$
 $\alpha = 93.500 (2)^\circ$
 $\beta = 110.936 (2)^\circ$
 $\gamma = 114.273 (2)^\circ$
 $V = 1035.21 (6) \text{ \AA}^3$

$Z = 1$
 $D_x = 1.420 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8531 reflections
 $\theta = 1.0\text{--}27.5^\circ$
 $\mu = 0.59 \text{ mm}^{-1}$
 $T = 180 (2) \text{ K}$
Block, colourless
 $0.25 \times 0.21 \times 0.16 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{\text{min}} = 0.803$, $T_{\text{max}} = 0.910$
11297 measured reflections
6863 independent reflections

6828 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\text{max}} = 26.9^\circ$
 $h = -12 \rightarrow 12$
 $k = -13 \rightarrow 13$
 $l = -15 \rightarrow 15$

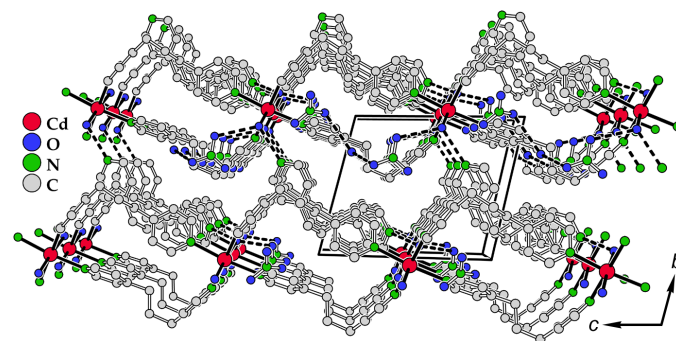


Figure 3
A perspective view of (I) along the a axis, showing the $\text{O-H} \cdots \text{O}^-$ and $\text{O-H} \cdots \text{N}$ hydrogen-bond network (black fragmented lines). H atoms have been omitted for clarity.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.066$
 $S = 1.05$
6863 reflections
533 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 0.2087P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.60 \text{ e \AA}^{-3}$
Absolute structure: (Flack, 1983), 2369 Friedel pairs
Flack parameter = 0.031 (11)

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cd1—O12	2.311 (3)	Cd1—N22 ⁱ	2.363 (3)
Cd1—O11	2.339 (3)	Cd1—N32 ⁱ	2.366 (4)
Cd1—N21	2.362 (4)	Cd1—N31	2.377 (3)
O12—Cd1—O11	176.52 (14)	N21—Cd1—N32 ⁱ	179.74 (15)
O12—Cd1—N21	91.17 (11)	N22 ⁱ —Cd1—N32 ⁱ	86.47 (12)
O11—Cd1—N21	90.38 (11)	O12—Cd1—N31	96.68 (11)
O12—Cd1—N22 ⁱ	89.69 (11)	O11—Cd1—N31	86.50 (11)
O11—Cd1—N22 ⁱ	87.10 (11)	N21—Cd1—N31	87.98 (12)
N21—Cd1—N22 ⁱ	93.77 (12)	N22 ⁱ —Cd1—N31	173.37 (13)
O12—Cd1—N32 ⁱ	88.73 (11)	N32 ⁱ —Cd1—N31	91.80 (11)
O11—Cd1—N32 ⁱ	89.74 (11)		

Symmetry code: (i) $x, y, z - 1$.

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{--H} \cdots A$	$D\text{--H}$	$\text{H} \cdots A$	$D \cdots A$	$D\text{--H} \cdots A$
O7W—H7A ⁱ ···O63	0.88 (1)	2.02 (1)	2.821 (5)	152 (3)
O7W—H7B ⁱ ···O53	0.88 (1)	2.03 (1)	2.901 (4)	178 (5)
O11—H11B ⁱ ···N42 ⁱⁱ	0.85 (1)	1.88 (1)	2.734 (4)	174 (4)
O12—H12A ⁱ ···N41 ⁱⁱⁱ	0.85 (1)	1.88 (1)	2.715 (4)	169 (5)
O12—H12B ⁱ ···O62 ^{iv}	0.85 (1)	1.99 (1)	2.834 (4)	174 (5)

Symmetry codes: (ii) $x, y, 1 + z$; (iii) $x, y - 1, z$; (iv) $1 + x, y, 1 + z$.

H atoms bound to C atoms were placed in calculated positions and allowed to ride during subsequent refinement with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Water H atoms were located in difference Fourier maps and refined with a single isotropic displacement parameter common to all H atoms. O—H distances were restrained to $0.85 (1) \text{ \AA}$ and H···H distances to $1.39 (1) \text{ \AA}$; the restraints ensure a reasonable geometry for the water molecules.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Bruker, 2001); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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