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catena-Poly[[diaquacalcium(II)]bis[μ-2-oxo-1,2-dihydropyridine-1-acetato]]

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Key indicators

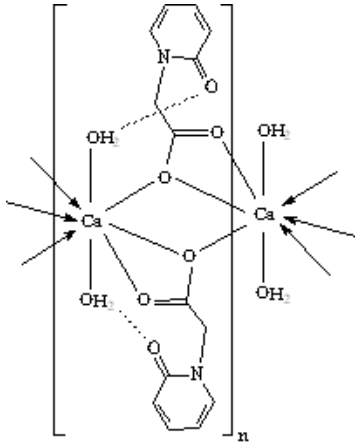
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.030
wR factor = 0.073
Data-to-parameter ratio = 15.0

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

In the title coordination polymer, $[Ca(2\text{-}OPA)_2(H_2O)_2]_n$ [2-OPA$^-$ is 2-oxo-1,2-dihydropyridine-1-acetate, $C_7H_6NO_3$], the Ca atom is eight-coordinated by six O atoms from four 2-OPA$^-$ ligands and two water molecules, and displays a dodecahedral coordination geometry. Each 2-OPA$^-$ ligand bridges two adjacent Ca atoms, forming a chain along the a-axis direction. The Ca···Ca separation within the polymer is 4.1022 (8) Å. A two-dimensional supramolecular framework is further constructed by hydrogen bonds and weak π–π stacking interactions.

Comment

2-Oxo-1,2-dihydropyridine-1-acetic acid (2-OPAH), known as an important medical intermediate (Klopman & Buyukbingol, 1988), is a potential multidentate ligand with versatile binding ability. However, there is little information on the structure of metal complexes formed by the 2-OPAH ligand. Recently, we have reported the structure of the mononuclear complexes, $[M(2\text{-}OPA)_2(H_2O)_4]$ (M = Mg, Co; Gao, Huo *et al.*, 2004; Gao, Zhang *et al.*, 2004), which are isostructural. We report here the synthesis and crystal structure of a new one-dimensional calcium-based coordination polymer, $[Ca(2\text{-}OPA)_2(H_2O)_2]_n$, (I).



(I)

As shown in Fig. 1, the asymmetric unit of (I) consists of one Ca^{II} atom, two 2-OPA$^-$ ligands and two coordinated water molecules. The Ca^{II} atom is coordinated by six O atoms from four 2-OPA$^-$ ligands and two water molecules in a dodecahedral coordination geometry (Fig. 2).

Each 2-OPA$^-$ ligand bridges two adjacent Ca^{II} atoms, forming a one-dimensional chain along the a-axis direction. The Ca···Ca distance between adjacent atoms is 4.1022 (8) Å.

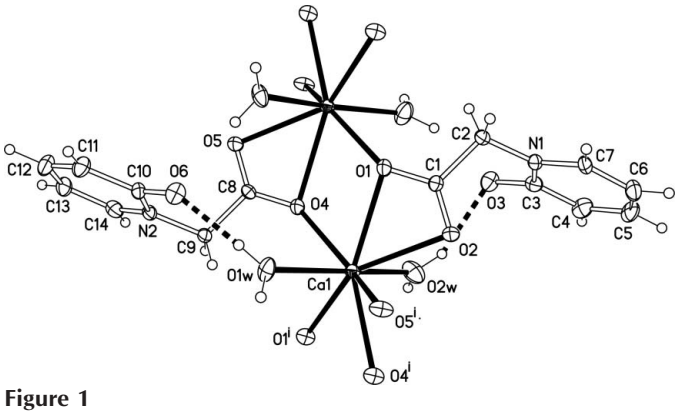


Figure 1
View of the title complex, showing 30% probability ellipsoids for the non-H atoms. Hydrogen-bond interactions are shown as dashed lines.

Moreover, there exist weak π–π stacking interactions between adjacent pyridine rings [centroid–centroid distance = 3.9 (2) Å] and hydrogen bonds, which are formed between the coordinated water molecules and carboxyl O atoms, resulting in an extended layer structure parallel to the *ab* plane (Table 2).

Experimental

The title complex was prepared by the addition of $Ca(NO_3)_2{\cdot}4H_2O$ (4.72 g, 20 mmol) to an aqueous solution of 2-oxo-1,2-dihydropyridine-1-acetic acid (5.84 g, 40 mmol). The resulting solution was stirred and the pH was adjusted to 7 with 0.2 *M* NaOH solution. After evaporation at room temperature for a week, colourless single crystals were obtained from the filtered solution. Analysis calculated for $C_{14}H_{20}CaN_2O_{10}$: C 44.21, H 4.24, N 7.36%; found: C 44.08, H 4.35, N 7.50%.

Crystal data

$[Ca(C_7H_6NO_3)_2(H_2O)_2]$
M_r = 380.37
Orthorhombic, $Pna2_1$
a = 7.9996 (16) Å
b = 8.2377 (16) Å
c = 24.153 (5) Å
V = 1591.6 (6) Å^3
Z = 4
D_x = 1.587 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 561 reflections
θ = 3.5–27.5°
μ = 0.44 mm^{-1}
T = 296 (2) K
Block, colourless
0.39 × 0.24 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.846, T_{max} = 0.928
13 703 measured reflections
3590 independent reflections
3187 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°
$h = -10 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.073
S = 1.00
3590 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.2343P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1739 Friedel pairs.
Flack parameter = 0.11 (3)

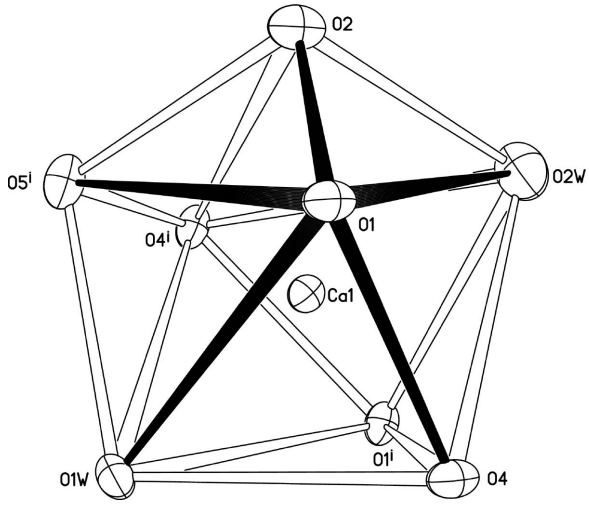


Figure 2
The coordination polyhedron of the Ca atom [symmetry code: (i) $x - \frac{1}{2}$, $\frac{1}{2} - y$, z].

Table 1
Selected geometric parameters (Å, °).

Ca1—O1	2.5766 (15)	Ca1—O5^i	2.5342 (16)
Ca1—O1^i	2.4011 (14)	Ca1—O1*W*	2.424 (3)
Ca1—O2	2.5344 (16)	Ca1—O2*W*	2.392 (4)
Ca1—O4	2.3995 (14)	O3—C3	1.274 (4)
Ca1—O4^i	2.5749 (15)	O6—C10	1.241 (4)
O1^i—Ca1—O1	150.72 (4)	O5^i—Ca1—O1	93.50 (5)
O1^i—Ca1—O2	144.56 (6)	O5^i—Ca1—O4^i	51.02 (5)
O1^i—Ca1—O4^i	69.00 (4)	O1*W*—Ca1—O1	95.83 (8)
O1^i—Ca1—O5^i	115.01 (5)	O1*W*—Ca1—O2	123.29 (8)
O1^i—Ca1—O1*W*	88.23 (8)	O1*W*—Ca1—O4^i	89.20 (7)
O2—Ca1—O1	50.97 (5)	O1*W*—Ca1—O5^i	70.18 (7)
O2—Ca1—O4^i	93.53 (5)	O2*W*—Ca1—O1	88.60 (8)
O2—Ca1—O5^i	68.45 (4)	O2*W*—Ca1—O1^i	81.53 (8)
O4—Ca1—O1	69.00 (4)	O2*W*—Ca1—O2	69.53 (9)
O4—Ca1—O1^i	83.17 (4)	O2*W*—Ca1—O4	88.44 (10)
O4^i—Ca1—O1	139.82 (4)	O2*W*—Ca1—O4^i	96.02 (9)
O4—Ca1—O2	115.00 (5)	O2*W*—Ca1—O5^i	122.97 (10)
O4—Ca1—O4^i	150.72 (4)	O2*W*—Ca1—O1*W*	165.95 (5)
O4—Ca1—O5^i	144.45 (6)	N1—C2—C1	110.81 (18)
O4—Ca1—O1*W*	80.76 (8)	N2—C9—C8	111.38 (18)

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O6	0.84 (3)	1.92 (3)	2.753 (3)	168 (4)
O1*W*—H1*W*2···O6^i	0.84 (3)	2.14 (3)	2.970 (3)	172 (3)
O2*W*—H2*W*1···O3^i	0.86 (3)	1.93 (4)	2.763 (3)	163 (4)
O2*W*—H2*W*2···O3	0.84 (3)	2.16 (4)	2.958 (4)	157 (4)

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z$.

H atoms on carbon were placed in calculated positions, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C), and were included in the refinement in the riding-model approximation. The H atoms of water molecules were located in Fourier difference maps and refined using a riding model, with O—H and H···H distance restraints of 0.85 (1) and 1.39 (1) Å, respectively, and U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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