

A new polymorph of poly[tetraaquad- μ_3 -malonato-dicadmium(II)]Xu-Cheng Fu,^{a,b} Ming-Tian Li,^a
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Key indicators

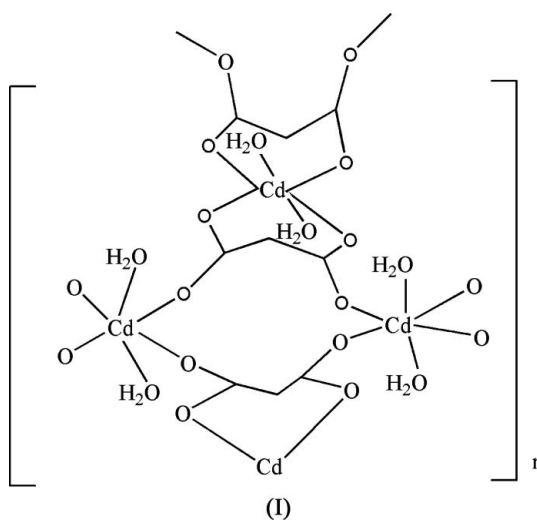
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.030
 wR factor = 0.086
Data-to-parameter ratio = 12.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $[\text{Cd}_2(\text{C}_3\text{H}_2\text{O}_4)_2(\text{H}_2\text{O})_4]_n$, was previously reported by Chung, Hong, Do & Moon [*J. Chem. Soc. Chem. Commun.* (1995), pp. 2333–2335; *J. Chem. Soc. Dalton Trans.* (1996), pp. 3363–3369]. We present here a new monoclinic polymorph which has a polymeric structure in which one Cd atom lies on an inversion centre and the other on a crystallographic twofold rotation axis.

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Comment

The crystal structure of title compound, (I), was previously reported by Chung *et al.* (1995, 1996). We present here the structure of a new monoclinic polymorph of (I) in the space group $C2/c$ (Fig. 1).



The structure of (I) can be described as a three-dimensional network of metal ions which are linked by malonate bridges and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Fig. 2).

Two of the coordinated water molecules are linked to Cd1 and the other two to Cd2. Atom Cd1 lies on an inversion centre and is octahedrally coordinated by six O atoms from two malonate ligands, O1 and O3, and two water molecules, O5. The Cd2 ion, which lies on a twofold axis, is also coordinated by six O atoms. Two of them, O6, belong to water molecules and the other four, O2 and O4, derive from four different malonate ligands, forming an irregular octahedron. The Cd2–O bonds are significantly longer than the Cd1–O bonds. Each malonate anion acts as bidentate chelate ligand to Cd1 through O1 and O3, and as a monodentate bridging ligand to Cd2 through O2 and O4, forming an infinite two-dimensional network in the (101) plane.

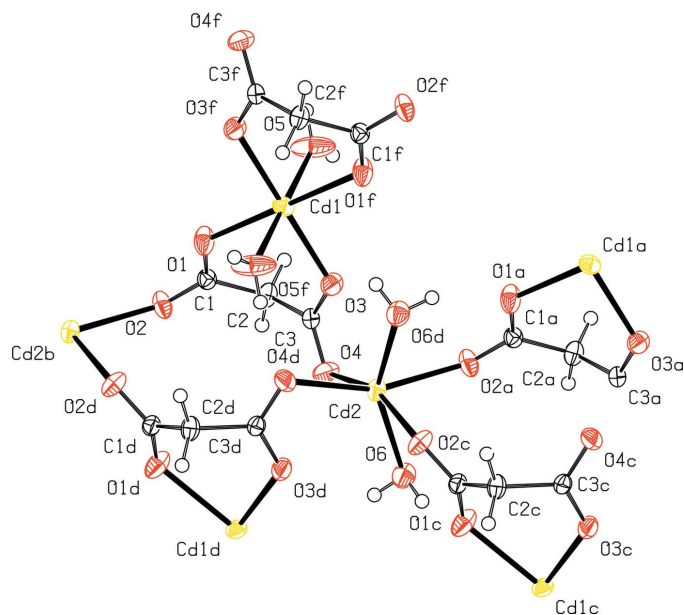


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 35% probability level. [Symmetry codes: (a) $x, -1 + y, z$; (b) $x, 1 + y, z$; (c) $1 - x, -1 + y, \frac{1}{2} - z$; (d) $1 - x, y, \frac{1}{2} - z$; (e) $1 - x, 1 + y, \frac{1}{2} - z$; (f) $\frac{3}{2} - x, \frac{3}{2} - y, 1 - z$; (g) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.]

Hydrogen bonding plays an important role in stabilizing the extended structure (Table 2). The overall network structure in the crystal is maintained and stabilized by the presence of O—H...O hydrogen bonds.

Experimental

Cd(OAc)₂·2H₂O (0.219 g, 1 mmol) and malonic acid (0.208 g, 2 mmol) were dissolved in an aqueous solution (20 ml) and the reaction mixture was adjusted to pH 5.5 by addition of NaOH solution. The resulting solution was stirred continuously at 328 K. After 2 h, the reaction mixture was cooled to room temperature and filtered. Colourless single crystals were obtained after leaving the filtrate to stand for one month.

Crystal data

[Cd₂(C₃H₂O₄)₂(H₂O)₄] Z = 8
M_r = 250.48 *D_x* = 2.626 Mg m⁻³
 Monoclinic, C2/c Mo Kα radiation
a = 14.680 (3) Å μ = 3.42 mm⁻¹
b = 7.2790 (15) Å *T* = 293 (2) K
c = 13.198 (3) Å Block, colourless
 β = 116.02 (3)° 0.56 × 0.43 × 0.20 mm
V = 1267.3 (4) Å³

Data collection

Bruker SMART CCD area-detector 3478 measured reflections
 diffractometer 1377 independent reflections
 φ and ω scans 1353 reflections with *I* > 2σ(*I*)
 Absorption correction: multi-scan *R*_{int} = 0.023
 SADABS, (Bruker 2000) θ_{max} = 27.0°
*T*_{min} = 0.185, *T*_{max} = 0.505

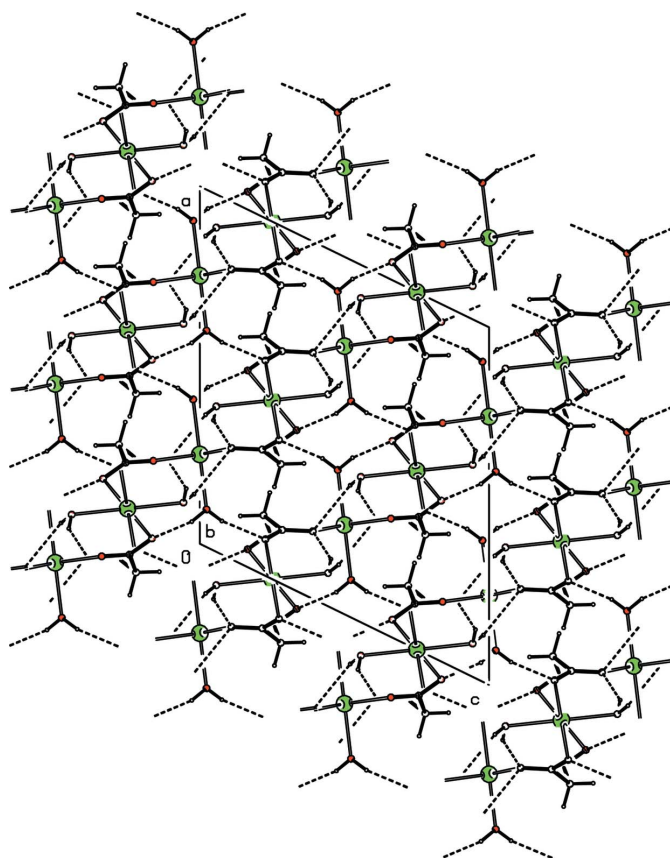


Figure 2
The crystal packing in (I). Hydrogen bonds are shown as dashed lines.

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.030
wR(*F*²) = 0.086
S = 1.24
 1377 reflections
 109 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.7017P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.75 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -2.57 \text{ e } \text{Å}^{-3}$$

Table 1

Selected geometric parameters (Å, °).

Cd1—O3	2.222 (3)	Cd2—O6	2.376 (3)
Cd1—O5	2.278 (3)	O3—C3	1.267 (4)
Cd1—O1	2.301 (2)	O4—C3	1.249 (4)
Cd2—O4	2.314 (3)	O1—C1	1.261 (4)
Cd2—O2 ⁱⁱⁱ	2.316 (2)	O2—C1	1.252 (4)
O3 ⁱ —Cd1—O3	180	O4—Cd2—O2 ⁱⁱⁱ	159.19 (9)
O5—Cd1—O5 ⁱ	180	O4—Cd2—O2 ^{iv}	94.68 (10)
O3 ⁱ —Cd1—O1 ⁱ	84.94 (10)	O6 ⁱⁱ —Cd2—O6	151.61 (14)
O1 ⁱ —Cd1—O1	180	O2—C1—O1	123.8 (3)
O4 ⁱⁱ —Cd2—O4	86.41 (14)	O4—C3—O3	121.1 (3)

Symmetry codes: (i) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $-x + 1, y, -z + \frac{1}{2}$; (iii) $-x + 1, y - 1, -z + \frac{1}{2}$; (iv) $x, y - 1, z$; (v) $x, y + 1, z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5B \cdots O2 ^{vi}	0.83 (2)	2.02 (2)	2.829 (4)	167 (6)
O5—H5B \cdots O6 ^{vii}	0.83 (2)	2.69 (6)	3.138 (4)	116 (5)
O5—H5A \cdots O4 ^{viii}	0.84 (2)	1.91 (2)	2.720 (4)	164 (5)
O6—H6B \cdots O1 ⁱⁱⁱ	0.84 (2)	1.98 (3)	2.742 (4)	150 (5)
O6—H6A \cdots O3 ⁱⁱ	0.83 (2)	2.70 (6)	3.077 (4)	109 (5)
O6—H6A \cdots O1 ^{ix}	0.83 (2)	2.58 (3)	3.325 (4)	150 (5)

Symmetry codes: (ii) $-x+1, y, -z+\frac{1}{2}$; (iii) $-x+1, y-1, -z+\frac{1}{2}$; (vi) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (vii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (viii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ix) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

All H atoms in water molecules were located in a difference Fourier map and refined freely with isotropic displacement parameters. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with $C-H = 0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The deepest hole in the final difference map was 1.71 Å from atom C3 and 1.66 Å from Cd2.

Data collection: *SMART* (Bruker 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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