

Poly[[diaquacadmium(II)]- μ^3 -(1-carboxybenzene-3,5-dicarboxylato)]

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.028

wR factor = 0.071

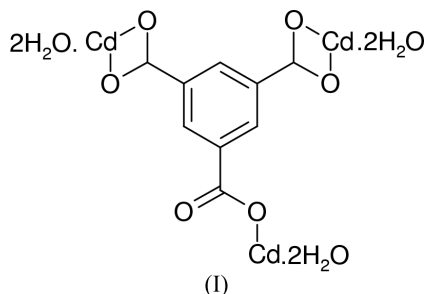
Data-to-parameter ratio = 19.7

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

The crystal structure of the title complex, poly[[diaquacadmium(II)]- μ^3 -(1-carboxybenzene-3,5-dicarboxylato- O^1 :- O^3 , O^3 ': O^5 , O^5 ')], $[\text{Cd}(\text{C}_9\text{H}_4\text{O}_6)(\text{H}_2\text{O})_2]_n$, features two-dimensional polymeric network layers which are hydrogen-bonded to one another. The Cd atom is coordinated by seven O atoms, two of which belong to water molecules arranged *trans* to each other, above and below the layers. Each polymeric layer is tiled with $(\text{Cd})_3(\text{C}_9\text{O}_6)_3$ rings formed by alternating Cd atoms and (C_9O_6) ligands. The layers are parallel to the $\bar{1}01$ crystal plane and the neighbouring layers are separated by 3.2508 (2) Å.

Comment

$[\text{Cd}(\text{C}_9\text{H}_4\text{O}_6)(\text{H}_2\text{O})_2]_n$, (I), was synthesized as part of a study into polymeric compounds containing metals and benzene-1,3,5-tricarboxylic acid (BTC) (Plater *et al.*, 1997, 1998, 1999; Yaghi *et al.*, 1996).



The title compound was shown to be polymeric, with the Cd atom coordinated to seven O atoms, the Cd–O bond lengths varying in the range 2.2479 (18)–2.5557 (16) Å (Fig. 1 and Table 1), thus showing good agreement with previously reported Cd–O values.

The O atoms coordinating each metal atom belong to three BTC molecules; two of these are bonded *via* bidentate chelating carboxylate groups (C1/O1/O2 and C8/O5/O6), whilst the third one is coordinated in a monodentate manner through its carboxylic carbonyl O3 atom. Each Cd atom is also bonded to two water molecules.

The Cd atoms and BTC ligands form a two-dimensional infinite network tiled with ‘macrocycles’, each comprising three Cd atoms and three BTC groups (Fig. 2). The layers are parallel to the $\bar{1}01$ plane, the distance between the planes of neighbouring layers being equal to 3.2508 (2) Å. Water molecules are located between the layers with the Cd–O7 and Cd–O8 vectors approximately perpendicular to the network layer planes (inclination angles are 90 and 85° for Cd–O7 and Cd–O8 respectively; Fig. 3).

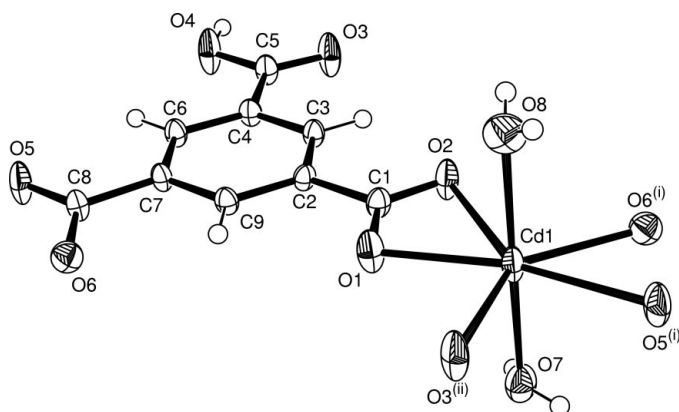


Figure 1
Asymmetric unit of the title compound. Ellipsoids are shown at 50% probability. O atoms related by symmetry are included to show the coordination sphere for Cd (symmetry transformations are as in Table 1)

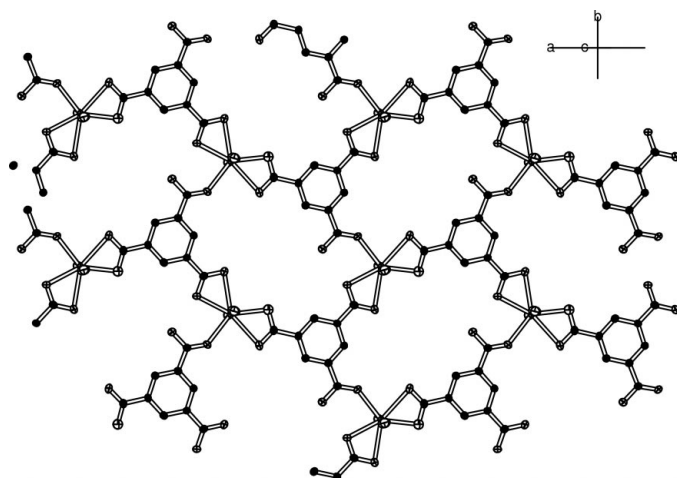


Figure 2
A fragment of polymeric network Cd-BTC layer in the crystal of the title compound.

The C5/O3/O4 carboxylic group hydrogen (H4) was objectively located from the difference Fourier map; it participates in the intra-layer hydrogen bond with one of the carboxylate O atoms (O1). The position of this carboxylic H atom is consistent with the C—O bond lengths values (see Table 1) and IR data. In contrast, the H atoms of water molecules are responsible for inter-layer hydrogen bonding (Table 2).

Experimental

Benzene-1,3,5-tricarboxylic acid (99.4 mg), cadmium acetate (128 mg) and water (10 ml) were placed inside a 23 ml Parr bomb. Once sealed, the bomb was heated to 483 K at 100 K h⁻¹. After maintaining this temperature for 2 h, the bomb was cooled to 463 K at 5 K h⁻¹, and after 6 h at 463 K was cooled to room temperature at 4 K h⁻¹. The bomb was opened and the colourless solid collected by filtration, washed with water and dried in air. IR spectra were obtained using a pressed KBr disk, using the ATI FT-IR system (Mattson Genesis series).

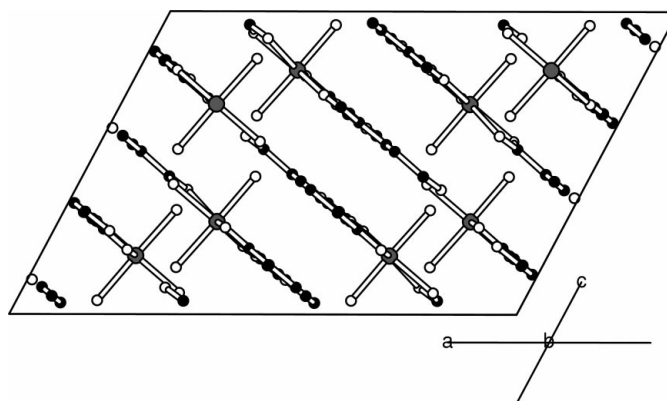


Figure 3
The 010 plane, showing orientation of Cd-BTC layers within the unit cell with Cd—H₂O bonds perpendicular to the layers.

Crystal data

[Cd(C₉H₄O₆)(H₂O)₂]
M_r = 356.55
Monoclinic, C2/c
a = 19.7328 (9) Å
b = 9.1088 (4) Å
c = 13.3636 (6) Å
β = 117.884 (1)°
V = 2123.12 (16) Å³
Z = 8

D_x = 2.225 Mg m⁻³
Mo Kα radiation
Cell parameters from 5038 reflections
θ = 2.5–31.5°
μ = 2.09 mm⁻¹
T = 293 (2) K
Block, colourless
0.3 × 0.1 × 0.1 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1999)
T_{min} = 0.679, T_{max} = 0.928
9718 measured reflections

3512 independent reflections
2892 reflections with I > 2σ(I)
R_{int} = 0.027
θ_{max} = 31.5°
h = -27 → 28
k = -13 → 11
l = -19 → 18

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.028
wR(F²) = 0.071
S = 0.97
3512 reflections
178 parameters

H atoms: see below
w = 1/[σ²(F_o²) + (0.0443P)²]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.004
Δρ_{max} = 1.50 e Å⁻³
Δρ_{min} = -1.24 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—O1	2.2851 (15)	C1—O1	1.269 (2)
Cd1—O2	2.5557 (16)	C1—O2	1.247 (3)
Cd1—O3 ⁱ	2.3011 (16)	C5—O3	1.223 (3)
Cd1—O5 ⁱⁱ	2.5225 (17)	C5—O4	1.305 (3)
Cd1—O6 ⁱⁱ	2.2479 (18)	C8—O5	1.251 (2)
Cd1—O7	2.381 (2)	C8—O6	1.273 (2)
Cd1—O8	2.309 (2)		
O6 ⁱⁱ —Cd1—O1	145.90 (6)	O1—Cd1—O5 ⁱⁱ	159.34 (6)
O6 ⁱⁱ —Cd1—O3 ⁱ	134.03 (6)	O3 ⁱ —Cd1—O5 ⁱⁱ	79.73 (6)
O1—Cd1—O3 ⁱ	80.04 (6)	O8—Cd1—O5 ⁱⁱ	88.84 (8)
O6 ⁱⁱ —Cd1—O8	89.15 (7)	O7—Cd1—O5 ⁱⁱ	93.00 (7)
O1—Cd1—O8	88.82 (8)	O6 ⁱⁱ —Cd1—O2	92.51 (6)
O3 ⁱ —Cd1—O8	95.22 (8)	O1—Cd1—O2	53.42 (5)
O6 ⁱⁱ —Cd1—O7	89.16 (7)	O3 ⁱ —Cd1—O2	133.45 (6)
O1—Cd1—O7	90.64 (7)	O8—Cd1—O2	84.56 (7)
O3 ⁱ —Cd1—O7	88.46 (8)	O7—Cd1—O2	92.04 (7)
O8—Cd1—O7	176.13 (8)	O5 ⁱⁱ —Cd1—O2	146.56 (5)
O6 ⁱⁱ —Cd1—O5 ⁱⁱ	54.57 (6)		

Symmetry codes: (i) x, y - 1, z; (ii) ½ + x, ½ - y, ½ + z.

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O7–H7A \cdots O2 ⁱ	0.73 (5)	2.11 (5)	2.836 (3)	176 (5)
O7–H7B \cdots O5 ⁱⁱ	0.63 (4)	2.24 (4)	2.867 (3)	170 (5)
O8–H8A \cdots O6 ⁱⁱⁱ	0.78 (4)	1.95 (4)	2.730 (3)	175 (4)
O8–H8B \cdots O7 ^{iv}	0.66 (4)	2.39 (4)	3.044 (3)	167 (5)
O4–H4 \cdots O1 ^v	0.92 (3)	1.71 (3)	2.604 (2)	164 (3)

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

The carboxylic acid and water H atoms (H4, H7A, H7B, H8A and H8B) were located in the difference map and were refined isotropically. Other H atoms were placed in geometrically calculated positions and included in the refinement in the riding model approximation. The highest residual density value was $1.50 \text{ e } \text{Å}^{-3}$ at 0.53 Å from O6.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* in *OSCAIL* (McArdle, 1994, 2000), *ATOMS* (Dowty, 1999)

and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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