

Poly[μ -aqua-aqua(μ_4 -2-oxidobenzene-1,3-dicarboxylato)(μ_2 -2-oxidobenzene-1,3-dicarboxylato)cadmium(II)dicopper(II)]

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Key indicators

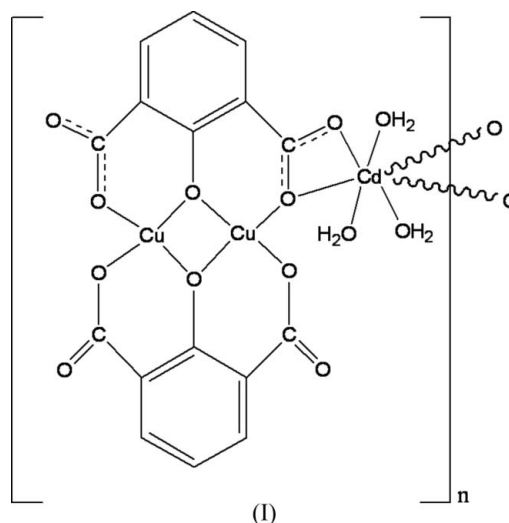
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.027
 wR factor = 0.068
Data-to-parameter ratio = 12.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the molecule of the title compound, $[\text{CdCu}_2(\text{C}_8\text{H}_3\text{O}_5)_2(\text{H}_2\text{O})_2]_n$, each 2-oxidobenzene-1,3-dicarboxylate trianion is bonded to two Cu atoms through O atoms of the carboxylates and the deprotonated hydroxy group in chelating mode, forming $[\text{Cu}_2(\text{C}_8\text{H}_3\text{O}_5)_2]^{2-}$ units. A pair of $[\text{Cu}_2(\text{C}_8\text{H}_3\text{O}_5)_2]^{2-}$ units is then coordinated to each Cd atom, forming an infinite chain. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

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Comment

Although some metal coordination polymers with benzenedicarboxylate have been reported, syntheses involving *in situ* reaction of benzenedicarboxylic acids are quite rare (Tao *et al.*, 2002; Jiang *et al.*, 2005). We report here the crystal structure of the title compound, (I). The 2-oxidobenzene-1,3-dicarboxylate trianion found in (I) was generated *in situ* by the hydroxylation of benzene-1,3-dicarboxylic acid during the synthesis.



The single-crystal X-ray structure determination showed that (I) is a heterometallic coordination polymer containing bis(2-oxidobenzene-1,3-dicarboxylato)dicopper(II) and a Cd atom. Each Cu center has a distorted square-planar geometry (Table 1, Fig. 1). Each of the Cu atoms is roughly coordinated by four O atoms, of which two belong to the deprotonated hydroxy groups and the other two belong to carboxylate groups of two 2-oxidobenzene-1,3-dicarboxylate trianion ligands.

The Cd atom is seven-coordinated by four O atoms from two chelating carboxylate groups of 2-oxidobenzene-1,3-dicarboxylate trianion ligands and three O atoms from water

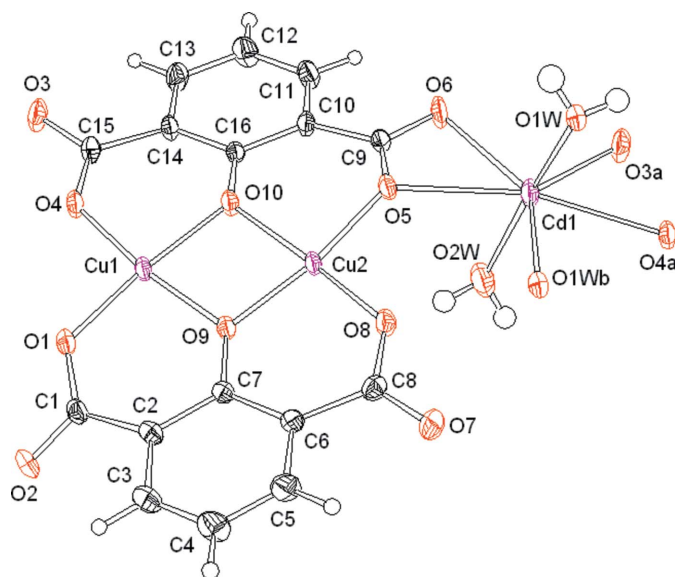


Figure 1
The structural repeat unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Two H atoms on O1Wb have been omitted for clarity [symmetry codes: (a) $-x, 1 - y, 2 - z$; (b) $x, 1 + y, z$].

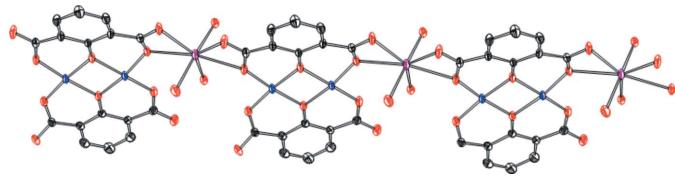


Figure 2
A view of the infinite chain. H atoms have been omitted.

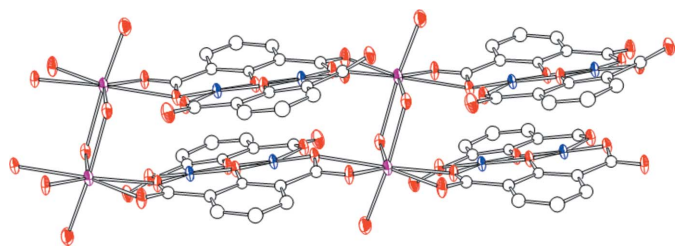


Figure 3
A view of the double chain. H atoms have been omitted.

molecules, yielding a distorted pentagonal–bipyramidal coordination (Table 1 and Fig. 1). In addition, two Cd atoms are bridged by two O atoms from water molecules, forming a centrosymmetric Cd₂O₂ parallelogram unit.

It can be seen that atoms Cu1, Cu2 and two 2-oxido-benzene-1,3-dicarboxylate trianions form a [Cu₂(C₈H₃O₅)₂]²⁻ unit (Fig. 1), and a pair of [Cu₂(C₈H₃O₅)₂]²⁻ units are then coordinated to Cd atoms, resulting in an infinite chain (Fig. 2). The most interesting feature is that Cd₂O₂ acts as a junction, connecting two infinite single chains into a double chain (Fig. 3).

The crystal structure is stabilized by intermolecular O—H...O hydrogen bonds (Table 2).

Experimental

A mixture of Cd(CH₃COO)₂·4H₂O (0.248 g), CuI (0.408 g), benzene-1,3-dicarboxylic acid (0.372 g) and Na₂CO₃ (0.292 g) in H₂O (18 ml) was kept at 418 K for 3 d in a Teflon-lined bomb (23 ml). After the reaction mixture had been slowly cooled to room temperature, blue crystals of (I) appeared (yield 0.451 g, 86.8%)

Crystal data

[CdCu₂(C₈H₃O₅)₂(H₂O)₂]
M_r = 633.72
Triclinic, P1̄
a = 8.272 (3) Å
b = 10.362 (3) Å
c = 11.373 (4) Å
α = 102.026 (2)°
β = 109.877 (3)°
γ = 103.958 (1)°

V = 843.5 (5) Å³
Z = 2
D_x = 2.495 Mg m⁻³
Mo Kα radiation
μ = 3.82 mm⁻¹
T = 294 (2) K
Prism, blue
0.15 × 0.12 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.579, T_{max} = 0.830

6580 measured reflections
3811 independent reflections
3324 reflections with I > 2σ(I)
R_{int} = 0.019
θ_{max} = 27.5°

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.027
wR(F²) = 0.068
S = 1.05
3811 reflections
296 parameters

H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(F_o²) + (0.039P)²]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.49 e Å⁻³
Δρ_{min} = -0.91 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—O2W	2.230 (3)	Cu2—O10	1.934 (2)
Cd1—O6	2.310 (2)	Cu2—Cu1	2.9532 (10)
Cd1—O1W	2.379 (2)	O10—Cu1	1.932 (2)
Cd1—O5	2.549 (2)	O9—Cu1	1.907 (2)
Cu2—O8	1.884 (2)	O4—Cu1	1.888 (2)
Cu2—O5	1.909 (2)	O1—Cu1	1.899 (2)
Cu2—O9	1.914 (2)		
O2W—Cd1—O6	86.44 (10)	O1W—Cd1—O5	96.10 (7)
O2W—Cd1—O1W	167.46 (9)	O8—Cu2—O5	93.89 (9)
O6—Cd1—O1W	100.53 (9)	O8—Cu2—O9	94.27 (9)
O2W—Cd1—O5	79.68 (8)	Cu1—O10—Cu2	99.61 (9)
O6—Cd1—O5	53.05 (7)	Cu1—O9—Cu2	101.25 (9)

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1W—H1A...O8 ⁱ	0.79 (4)	1.93 (2)	2.701 (3)	164 (5)
O1W—H1B...O1 ⁱⁱ	0.80 (4)	2.04 (3)	2.748 (3)	149 (5)
O2W—H2A...O2 ⁱⁱⁱ	0.82 (4)	1.92 (2)	2.724 (3)	170 (5)
O2W—H2B...O7 ^{iv}	0.82 (4)	1.83 (2)	2.635 (4)	169 (5)

Symmetry codes: (i) $-x, -y + 1, -z + 2$; (ii) $-x, -y, -z + 2$; (iii) $-x + 1, -y, -z + 2$; (iv) $-x + 1, -y + 1, -z + 2$.

Water H atoms were located in difference syntheses and refined isotropically [O—H = 0.79 (4)–0.82 (4) Å and U_{iso}(H) = 0.063 (16)–

0.09 (2) Å²]. The remaining H atoms were positioned geometrically, with C–H = 0.93 Å, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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