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Aquabis(hexamethylenetetramine-κN)bis(2-hydroxy-*trans*-cinnamato-κ^2O,O')cadmium trihydrate

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Key indicators

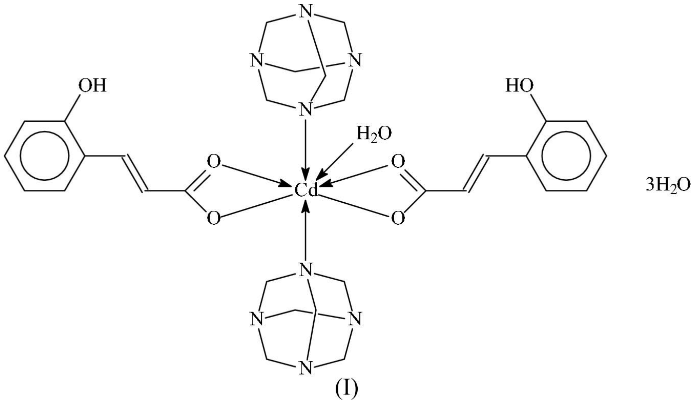
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.042
wR factor = 0.103
Data-to-parameter ratio = 15.9

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

Both carboxylate groups chelate to the Cd atom in the title compound, $[Cd(C_9H_7O_3)_2(C_6H_{12}N_4)_2(H_2O)]\cdot 3H_2O$. The four O atoms involved in chelation and one water molecule form a pentagonal plane around the Cd atom, while the N atoms of the hexamethylenetetramine ligands occupy the axial sites of the pentagonal bipyramid. Hydrogen bonds link the molecules of the complex with the solvent water molecules to form a three-dimensional network.

Comment

The present study of the hexamethylenetetramine complex of cadmium bis(2-hydroxycinnamate) originates from the report of the hexamethylenetetramine adduct of cadmium terephthalate (Banerjee *et al.*, 2003). The terephthalate adduct has one carboxyl $-CO_2$ arm binding in a monodentate manner and the other in a chelating manner, the bridging nature of the hexamethylenetetramine entity resulting in a framework motif. The six- and seven-coordinate sites of the two Cd atoms appear to be crucial to the rigidity of the framework in the context of loss of solvent water molecules. Cadmium bis(2-hydroxycinnamate) forms a 1:2 adduct with hexamethylenetetramine, crystallizing with coordinated and solvent water molecules as the title compound, (I) (Fig. 1). Chelation is relatively symmetrical in one of the carboxylate groups, but is distinctly not so in the other, and one of the two $Cd-O_{carbonyl}$ bonds is probably lengthened so as to accommodate the water molecule in the coordination sphere.



(I)

The hydroxy substituents and all four water molecules are engaged in extensive hydrogen-bonding interactions (Table 2), leading to a three-dimensional network.

Although there are a large number of structurally authenticated derivatives of 2-hydroxycinnamic acid, and the structure of the parent acid itself has been reported (Raghunathan & Pattabhi, 1979), there is only one other metal complex of this acid, namely tetraaquamagnesium bis(2-hydroxycinnamate) (Kariuki *et al.*, 1995), which has been described to date.

Experimental

A mixture of cadmium sulfate (0.50 mmol, 0.11 g), 2-hydroxycinnamic acid (1 mmol, 0.16 g) and hexamethylenetetramine (2 mmol, 0.28 g) in a 1:2 water–ethanol mixture (30 ml) was stirred for several hours until the reagents had dissolved completely. Colourless crystals of (I) were obtained from the filtered solution after a week.

Crystal data

$[Cd(C_9H_7O_3)_2(C_6H_{12}N_4)_2(H_2O)]\cdot 3H_2O$
$M_r = 791.15$
Monoclinic, $P2_1/c$
$a = 11.1249$ (6) Å
$b = 21.926$ (1) Å
$c = 14.6557$ (7) Å
$\beta = 107.096$ (1)°
$V = 3416.9$ (3) Å^3
$Z = 4$
$D_x = 1.538$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5924 reflections
$\theta = 2.2$–26.7°
$\mu = 0.71$ mm^{-1}
$T = 295$ (2) K
Block, colourless
0.34 × 0.25 × 0.15 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.585$, $T_{max} = 0.901$
21045 measured reflections
7680 independent reflections
6442 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$
$h = -14 \rightarrow 14$
$k = -24 \rightarrow 28$
$l = -17 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.103$
$S = 1.03$
7680 reflections
482 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 1.0651P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—O1	2.426 (2)	Cd1—O1*w*	2.290 (2)
Cd1—O2	2.375 (2)	Cd1—N1	2.441 (2)
Cd1—O4	2.272 (2)	Cd1—N5	2.465 (2)
Cd1—O5	2.588 (2)		
O1—Cd1—O2	54.48 (6)	O4—Cd1—O5	53.12 (7)
O1—Cd1—O4	89.43 (8)	O4—Cd1—O1*w*	133.34 (8)
O1—Cd1—O5	142.11 (7)	O4—Cd1—N1	91.09 (8)
O1—Cd1—O1*w*	136.98 (7)	O4—Cd1—N5	88.97 (8)
O1—Cd1—N1	89.36 (7)	O5—Cd1—N1	86.22 (7)
O1—Cd1—N5	86.90 (7)	O5—Cd1—N5	96.79 (7)
O2—Cd1—O4	143.87 (7)	O5—Cd1—O1*w*	80.89 (7)
O2—Cd1—O5	162.81 (7)	O1*w*—Cd1—N1	93.50 (7)
O2—Cd1—O1*w*	82.56 (7)	O1*w*—Cd1—N5	89.17 (7)
O2—Cd1—N1	90.19 (8)	N1—Cd1—N5	176.26 (7)
O2—Cd1—N5	87.56 (8)		

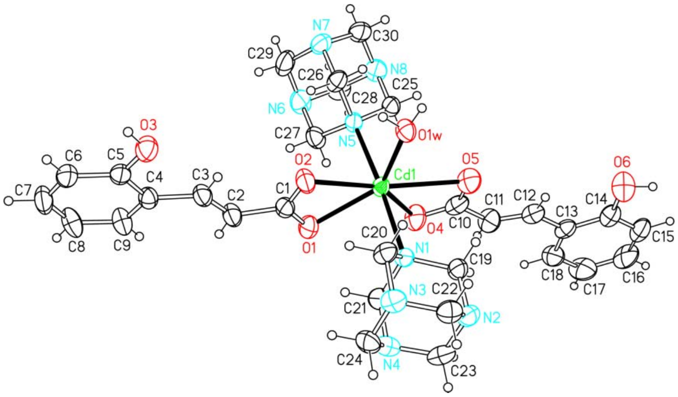


Figure 1
A plot of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The three solvent water molecules are not shown.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3o···N7^{i}	0.85 (1)	1.99 (1)	2.831 (3)	176 (3)
O6—H6o···O5^{ii}	0.85 (1)	1.87 (1)	2.701 (3)	164 (4)
O1*w*—H1*w*1···O2*w*	0.85 (1)	1.90 (1)	2.735 (3)	167 (2)
O1*w*—H1*w*2···N3^{iii}	0.85 (1)	1.98 (1)	2.817 (3)	168 (3)
O2*w*—H2*w*1···O3*w*	0.85 (1)	2.01 (2)	2.751 (5)	145 (3)
O2*w*—H2*w*2···N8^{iv}	0.85 (1)	2.06 (1)	2.892 (4)	169 (3)
O3*w*—H3*w*1···O1^{v}	0.86 (1)	2.12 (6)	2.812 (5)	138 (7)
O3*w*—H3*w*2···O4*w*^{iv}	0.85 (1)	2.12 (2)	2.96 (1)	168 (7)
O3′—H3′1···O1^{v}	0.86 (1)	2.07 (7)	2.787 (7)	141 (9)
O4*w*—H4*w*1···O4	0.86 (1)	1.86 (1)	2.722 (5)	176 (7)
O4*w*—H4*w*2···N4^{vi}	0.86 (1)	2.08 (1)	2.929 (5)	171 (6)

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

The H atoms on the sp^2-hybridized C atoms (C—H = 0.93 Å) and those on the sp^3-hybridized C atoms (C—H = 0.97 Å) were placed in calculated positions, with U_{iso}(H) set to $1.2U_{eq}$(C), and were included in the refinement in the riding-model approximation. The hydroxyl and water H atoms were located in difference Fourier maps and were refined with distance restraints of O—H = 0.85 (1) Å. As the displacement parameters of only some of these could be refined, they were all then set to $1.2U_{eq}$(O). The O3*W* water molecule is disordered over two sites, the occupancy refining to 56 (1):44 (1).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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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