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Cadmium Glutamate Dihydrate

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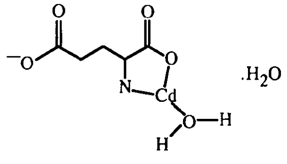
Abstract

Crystals of aqua(glutamato)cadmium hydrate, $[Cd(C_5H_7O_4)(H_2O)].H_2O$, are orthorhombic, space group $P2_12_12_1$. The compound is nearly isostructural with the corresponding zinc and copper complexes. The coordination about the cadmium is a distorted octahedron, with Cd—O1 2.282 (3), Cd—O2 2.254 (3), Cd—O3 2.303 (2), Cd—O4 2.449 (2), Cd—O5 2.255 (2) and Cd—N 2.290 (2) Å.

Comment

In our laboratories, we have been investigating the optical properties of metal–glutamate complexes. By performing the Kurtz test (Kurtz & Perry, 1968; Kurtz & Dougherty, 1978), we have found that Zn, Cu and Cd glutamate complexes have excellent second-order nonlinearity. Upon irradiation at 1064 nm, the powders show intense green light at 532 nm. Also, large single crystals can be grown from these materials, making them possible candidates for non-linear optical materials. Second-order non-linear optical materials have numerous applications as second-harmonic generators, optical parametric oscillators and electro-optic modulators.

The coordination about the Cd atom in the title compound, (I), is very similar to the square pyramid found in the corresponding Zn adduct (Gramaccioli, 1966). The additional glutamate O atom (O4) at 2.449 Å completes the distorted octahedron exhibited by both the Zn and Cu complexes (Gramaccioli & Marsh, 1966; Gramaccioli, 1966). The Cd atom lies 0.22 Å from the base of the pyramid and is approximately equidistant from all five atoms. The average distance from the Cd atom to the five nearest neighbors is 2.277 Å, which is at least 0.2 Å larger than those reported in the Cu and Zn studies (2.047 and 2.069 Å, respectively; Gramaccioli & Marsh, 1966; Gramaccioli, 1966). However, the O4 atom is at least 0.12 Å closer in the Cd complex.



(I)

There are six H atoms available for hydrogen bonding [four from the two water molecules (H8—H11) and two from the nitrogen (H6 and H7)], however, only five actively participate in hydrogen bonding. The four O—H···O distances of 2.717 (3)–2.786 (3) Å and the N—H6···O1 distance of 2.967 (3) Å are typical values indicative of moderately strong interactions. The second H atom on the nitrogen (H7) does not participate in hydrogen bonding.

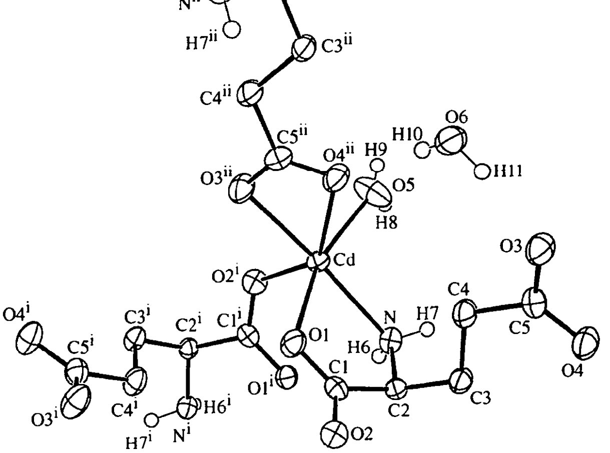


Fig. 1. The molecular structure of cadmium glutamate dihydrate along with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

A one liter solution of 0.34 *M* L-glutamic acid was heated to 373 K with gentle stirring and the pH was adjusted to 7.0 with 6 *M* NaOH. A one liter solution of 0.34 *M* cadmium acetate was added dropwise to the glutamic acid solution over a period of 1 h. The solution was removed from the heat. Cadmium glutamate was precipitated by adjusting the pH to 10.0 (10 *M* NaOH). The precipitate was then washed with ethanol and ether and dried for 48 h at 323 K. Cadmium glutamate was

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recrystallized by lowering the pH of a 0.05 *M* solution of cadmium glutamate to 2.0 (6 *M* HCl). Once the cadmium glutamate was dissolved, the pH was adjusted to 5.88 and the solvent allowed to evaporate.

Crystal data

$[Cd(C_5H_7O_4)(H_2O)].H_2O$
$M_r = 293.55$
Orthorhombic
$P2_12_12_1$
$a = 11.5747\,(13)$ Å
$b = 10.7639\,(7)$ Å
$c = 7.2556\,(7)$ Å
$V = 903.97\,(15)$ Å^3
$Z = 4$
$D_x = 2.157$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.88$–$25.88°$
$\mu = 2.41$ mm^{-1}
$T = 296$ K
Prismatic
$0.40 \times 0.30 \times 0.30$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (*SDP/PDP*; Enraf–Nonius, 1985)
$T_{\min} = 0.37$, $T_{\max} = 0.48$
1063 measured reflections
1063 independent reflections

1019 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 27.96°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 9$
3 standard reflections
frequency: 240 min
intensity decay: <3%

Refinement

Refinement on F
$R = 0.014$
$wR = 0.020$
$S = 0.91$
1019 reflections
152 parameters
Only coordinates of H atoms refined

$w = 1/\sigma^2$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cd—N	2.290 (2)	Cd—O5	2.255 (2)
Cd—O1	2.282 (3)	C1—O2	1.254 (3)
Cd—O2^{i}	2.254 (2)	C1—O1	1.259 (4)
Cd—O3^{ii}	2.303 (2)	C2—N	1.469 (4)
Cd—O4	2.449 (2)		
O2—C1—O1	124.6 (3)	C4—C3—C2	113.5 (2)
N—C2—C1	112.1 (2)	C3—C4—C5	113.4 (2)
N—C2—C3	111.7 (2)	O4—C5—O3	120.4 (3)
C1—C2—C3	109.4 (2)		

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

Table 2. *Hydrogen-bonding distances (Å)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$
O5—H8$\cdots$O6	0.61 (5)	2.12 (5)	2.726 (3)
O5—H9$\cdots$O6^{i}	0.74 (4)	1.99 (4)	2.717 (3)
O6—H10$\cdots$O4^{ii}	0.62 (4)	2.14 (5)	2.786 (3)
O6—H11$\cdots$O3	0.86 (5)	1.85 (5)	2.708 (3)
N—H6$\cdots$O1^{iii}	0.77 (4)	2.34 (4)	2.957 (3)

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

All non-H atoms were located from electron-density maps and were refined anisotropically by full-matrix least squares. H atoms were located from difference Fourier synthesis and were refined isotropically. Their bonded distances were in the ranges 0.88–1.09 (C—H), 0.77–0.86 (N—H) and 0.62–0.86 Å (O—H).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *SDP* (Frenz, 1978). Program(s) used to solve structure: *CAD*-4 *SDP*. Program(s) used to refine structure: *CAD*-4 *SDP*. Molecular graphics: *Xtal*3.4 *ORTEP* (Hall, Flack & Stewart, 1995). Software used to prepare material for publication: *Xtal*3.4 *BONDLA CIFIO*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1343). Services for accessing these data are described at the back of the journal.

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Dual Behaviour of Acetylacetonate Anions in the Hydrogen-Bonded Supramolecular Structure (Acetylacetonato-*O*,*O*′)[*trans*-(1*R*,2*R*)-diaminocyclohexane-*N*,*N*′]-platinum(II) Acetylacetonate

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Abstract

The title compound, $[Pt(C_5H_7O_2)(C_6H_{14}N_2)](C_5H_7O_2)$, involves two independent acetylacetonate anions with