Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis(9-hydroxyfluorene-9-carboxylato-κ^2O,O')-tripyridinecadmium(II)

Shan Gao,* Li-Hua Huo, Zhen-Zhong Lu and Hui Zhao

School of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China

Correspondence e-mail:
shangao67@yahoo.com

Received 1 November 2004
Accepted 2 November 2004
Online 6 November 2004

In the title complex, $[Cd(C_{14}H_9O_3)_2(C_5H_5N)_3]$, the Cd^{II} atom has a pentagonal–bipyramidal geometry, with four carboxylate O atoms from two bidentate 9-hydroxyfuorene-9-carboxylate ligands and one pyridine N atom in the equatorial plane, and two further pyridine N atoms in the axial positions. Intermolecular O—H$\cdots$O hydrogen bonds give rise to a chain structure.

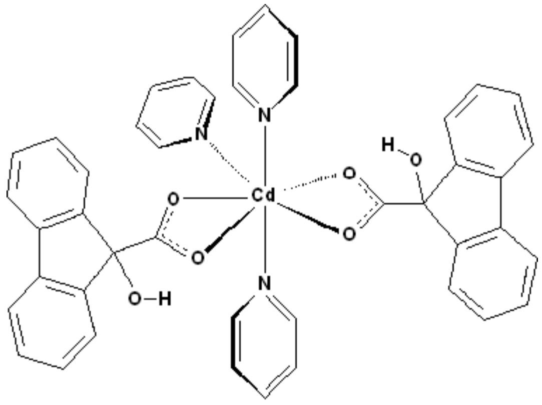
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.102
Data-to-parameter ratio = 17.2

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

Comment

There has been considerable interest in α-hydroxylate derivatives of fluorene-9-carboxylic acid in structural chemistry and biochemistry (Gao *et al.*, 2004; Feng & Liu, 2002; Liu & Wang, 1996; Bkouche, 1994). 9-Hydroxyfluorene-9-carboxylic acid, which could bind to a metal center through its carboxyl and/or hydroxyl O atoms, is a potentially multidentate ligand. To date, only some copper(II) complexes incorporating 9-hydroxyfluorene-9-carboxylate and *N*-heterocyclic amine ligands have been reported, resulting in mononuclear (Zheng *et al.*, 1999; Feng & Liu, 2002), binuclear (Liu & Yu, 1992; Yu *et al.*, 1996) and two-dimensional layer (Liu, 1992) structures. In all these complexes, the 9-hydroxyfluorene-9-carboxylate ligands display a five-membered chelate ring mode through the hydroxyl O atom and one carboxyl O atom. Recently, we obtained the title complex, (I) (Fig. 1), in which the 9-hydroxyfluorene-9-carboxylate ligands coordinate to the Cd^{II} atom *via* the two carboxyl O atoms and show a four-membered chelate ring mode. The crystal structure of (I) is reported here.



(I)

Compound (I) consists of two 9-hydroxyfluorene-9-carboxylate molecules and three pyridine molecules coordinated to one Cd^{II} atom, which has a pentagonal–bipyramidal geometry. The equatorial plane is defined by four carboxyl O atoms from two independent bidentate 9-hydroxyfluorene-9-

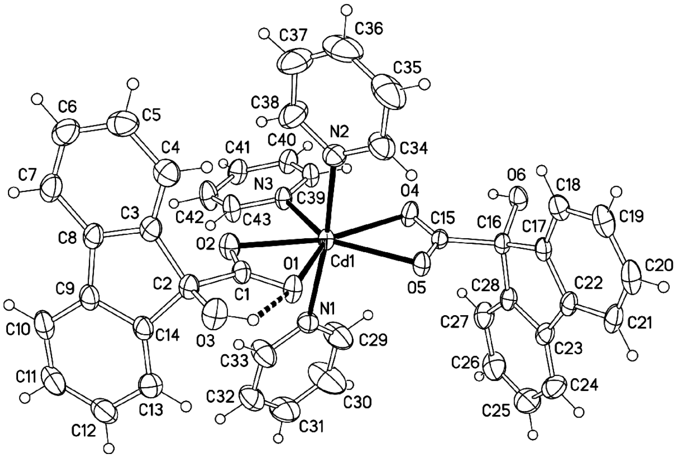


Figure 1
View of (I), with 30% probability displacement ellipsoids (arbitrary spheres for the H atoms). The O—H···O hydrogen bond is denoted by a dashed line.

carboxylate ligands and one pyridine atom N3 with an r.m.s. deviation from the mean plane of 0.07 Å. The two axial positions are occupied by atoms N1 and N2 from two pyridine molecules, with an N1—Cd1—N2 bond angle of 171.59 (7)°. The Cd—O and Cd—N bond lengths are in the ranges 2.331 (2)–2.554 (2) and 2.333 (2)–2.351 (2) Å, respectively.

There is a hydroxyl group in each of the two 9-hydroxyfluorene-9-carboxylate ligands, indicating that the ligand bears a formal charge of −1. The carboxyl groups of the anionic ligands show their normal delocalization leading to an equalization of the C—O bond lengths (Table 1). The fluorene moieties of the two independent ligands are both close to planar, with r.m.s. deviations of 0.013 (3) and 0.041 (3) Å for the C2- and C16-containing groups, respectively, and are almost perpendicular to their attached carboxyl group mean planes, with dihedral angles of 87.2 (2) and 85.3 (2)°, respectively, for the C2 and C16 groups. In the C2-containing 9-hydroxyfluorene-9-carboxylate ligand, a relatively acute intramolecular O—H···O hydrogen bond is formed between the uncoordinated hydroxyl O atom and the carboxyl O atom, which stabilizes the position of the fluorene group (Fig. 1 and Table 2). Conversely, in the C16 group, an intermolecular O—H···O hydrogen bond is formed with an adjacent mononuclear unit, giving rise to a chain propagating parallel to the *c* axis (Fig. 2).

Experimental

The ligand 9-hydroxyfluorene-9-carboxylic acid was prepared following the literature method of Zheng *et al.* (1999). The title complex was prepared by the addition of cadmium dinitrate tetrahydrate (0.303 g, 1 mmol) to an ethanol solution containing 9-hydroxyfluorene-9-carboxylic acid (0.44 g, 2 mmol) and pyridine (3 ml). The mixed solution was allowed to evaporate at room temperature and colorless single crystals were isolated from the solution at room temperature over several days. Analysis calculated for $C_{43}H_{33}CdN_3O_6$: C 64.55, H 4.16, N 5.25%; found: C 64.51, H 4.18, N 3.24%.

Crystal data

$[Cd(C_{14}H_9O_3)_2(C_5H_5N)_3]$
$M_r = 800.13$
Monoclinic, $P2_1/n$
$a = 10.266$ (2) Å
$b = 26.359$ (5) Å
$c = 13.462$ (3) Å
$\beta = 91.62$ (3)°
$V = 3641.4$ (13) Å^3
$Z = 4$
$D_x = 1.459$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 32513 reflections
$\theta = 3.1$–27.4°
$\mu = 0.65$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.39 × 0.25 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.784$, $T_{\max} = 0.891$
32752 measured reflections
8302 independent reflections
7477 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 27.5°$
$h = -13 \rightarrow 13$
$k = -34 \rightarrow 34$
$l = -14 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.102$
$S = 1.05$
8302 reflections
484 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 1.2795P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.61$ e Å^{-3}
$\Delta\rho_{\min} = -0.73$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—O1	2.554 (2)	Cd1—N3	2.351 (2)
Cd1—O2	2.394 (2)	C1—O1	1.237 (3)
Cd1—O4	2.525 (2)	C1—O2	1.237 (3)
Cd1—O5	2.331 (2)	C15—O4	1.239 (3)
Cd1—N1	2.344 (2)	C15—O5	1.238 (3)
Cd1—N2	2.333 (2)		
O2—Cd1—O4	163.86 (6)	N1—Cd1—O4	96.09 (7)
O5—Cd1—O1	90.24 (6)	N1—Cd1—O1	89.17 (7)
O2—Cd1—O1	52.39 (5)	N2—Cd1—O1	83.66 (8)
O4—Cd1—O1	143.29 (5)	N2—Cd1—O4	92.29 (7)
O5—Cd1—N2	90.63 (7)	N2—Cd1—N1	171.59 (7)
O5—Cd1—N1	93.80 (7)	N2—Cd1—N3	97.37 (8)
O5—Cd1—N3	133.21 (6)	N2—Cd1—O2	85.45 (7)
O5—Cd1—O4	53.24 (6)	N3—Cd1—O2	84.10 (6)
O5—Cd1—O2	142.62 (6)	N3—Cd1—O4	80.34 (6)
N1—Cd1—N3	84.78 (8)	N3—Cd1—O1	136.37 (6)
N1—Cd1—O2	86.69 (7)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H44···O1	0.85 (3)	1.96 (3)	2.559 (3)	126 (3)
O6—H45···O3$^{\rm i}$	0.84 (3)	2.07 (3)	2.847 (3)	153 (3)

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$.

The carbon-bound H atoms were placed in calculated positions and were allowed to ride on their parent C atoms [C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)]. The H atoms of the hydroxyl groups were located in a difference Fourier map, and refined with O—H distance restraints of 0.85 (1) Å and the constraint $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank the National Natural Science Foundation of China (No. 20101003), Heilongjiang Province Natural Science Foundation (No. B0007), the scientific fund of Remarkable Teachers of Heilongjiang Province (No. 1054G036) and Heilongjiang University for supporting this work.

References

Bkouche, W. I. (1994). *Acta Cryst.* C**50**, 62–64.
Feng, Y. L. & Liu, S. X. (2002). *Chin. J. Struct. Chem.* **2**, 142–145.
Gao, S., Liu, J.-W., Huo, L.-H. & Zhao, H. (2004). *Acta Cryst.* E**60**, m1725–m1727.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Liu, S.-X. (1992). *Acta Cryst.* C**48**, 22–24.
Liu, S. X. & Wang, X. (1996). *Chem. J. Chin. Univ*. **17**, 169–172.
Liu, S.-X. & Yu, Y.-P. (1992). *Acta Cryst.* C**48**, 652–655.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure.* Rigaku/MSC Inc., 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.

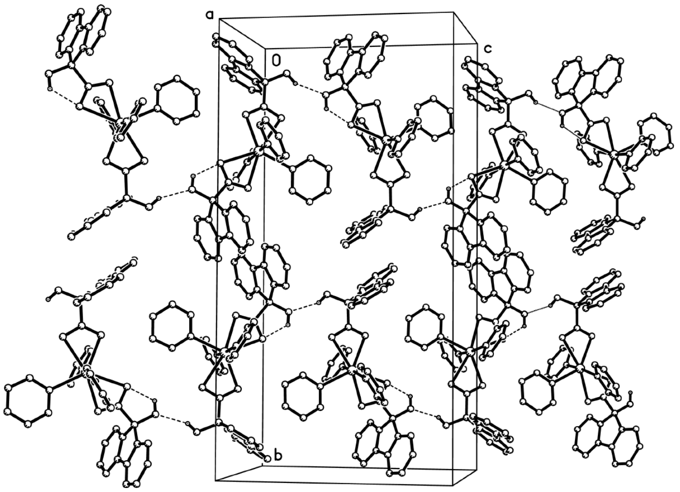


Figure 2
The crystal packing of (I), with the O—H···O hydrogen bonds denoted by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Yu, Y.-P., Zhu, D.-L. & Liu, S.-X. (1996). *Acta Cryst.* C**52**, 628–630.
Zheng, Y., Xu, D.-M. & Liu, S.-X. (1999). *Inorg. Chim. Acta*, **294**, 163–169.