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Bis(2-hydroxybenzoato-κO)bis(1,10-phenanthroline-$\kappa^2 N,N'$)cadmium(II) monohydrate

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In the crystal structure of the title compound, $[Cd(C_7H_5O_3)_2(C_{12}H_8N_2)_2]\cdot H_2O$, the Cd^{II} ion is coordinated by two 1,10-phenanthroline (phen) ligands and two 2-hydroxybenzoate anions, resulting in a distorted CdN_4O_2 octahedral coordination geometry. A partially overlapped arrangement and short face-to-face separation of 3.47 (2) Å indicate the existence of π–π stacking between parallel phen ligands.

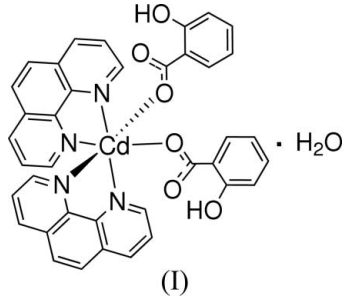
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.036
wR factor = 0.107
Data-to-parameter ratio = 18.0

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

Comment

Intermolecular π–π stacking is an important interaction and is correlated with electron-transfer processes in some biological systems (Deisenhofer & Michel, 1989). As part of investigation into the nature of π–π stacking in metal complexes (Chen *et al.*, 2003), we report here the structure of the title Cd^{II} complex, (I).



(I)

The crystal structure of (I) consists of Cd^{II} complexes and uncoordinated water molecules. The Cd^{II} ion assumes a distorted CdN_4O_2 octahedral geometry, formed by two 1,10-phenanthroline (phen) ligands and two 2-hydroxybenzoate (2-HBA) anions (Fig. 1). This geometry is similar to that found in the Cd^{II} complexes with 4-HBA anions (Pan *et al.*, 2006). The two phen ligands chelate to the Cd^{II} ion in a *cis* configuration, being roughly perpendicular to each other with a dihedral angle of 79.77 (6)°.

A partially overlapped arrangement is observed between parallel phen ligands (Fig. 2), in which the face-to-face separation of 3.47 (2) Å strongly suggests the existence of π–π stacking between them. Within the 2-HBA anions, the hydroxyl groups are intramolecularly hydrogen bonded with the carboxyl groups. The uncoordinated water molecule links to the complexes *via* O—H···O hydrogen bonding (Table 2).

Experimental

A water/ethanol solution (20 ml, 1:1) containing $Cd(NO_3)_2\cdot 4H_2O$ (0.62 g, 2 mmol), 2-hydroxybenzoic acid (0.32 g, 2 mmol), Na_2CO_3 (0.21 g, 2 mmol) and phen (0.40 g, 2 mmol) was refluxed for 3 h. After

cooling to room temperature, the solution was filtered. Single crystals of (I) were obtained from the filtrate after 2 weeks.

Crystal data

$[Cd(C_7H_5O_3)_2(C_{12}H_8N_2)_2]\cdot H2O$
M_r = 765.04
Monoclinic, $P2_1/n$
a = 10.838 (3) Å
b = 25.941 (5) Å
c = 11.698 (3) Å
β = 93.119 (11)°
V = 3284.0 (13) Å^3
Z = 4
D_x = 1.547 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.72 mm^{-1}
T = 295 (2) K
Chunk, colorless
0.32 × 0.19 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan *ABSCOR* (Higashi, 1995)
T_{min} = 0.791, T_{max} = 0.881
32337 measured reflections
7511 independent reflections
5999 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.107
S = 1.12
7511 reflections
417 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.4959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd—O1*B*	2.273 (6)	Cd—N2	2.428 (2)
Cd—O2*A*	2.351 (5)	Cd—N3	2.415 (2)
Cd—O4	2.249 (2)	Cd—N4	2.399 (2)
Cd—N1	2.363 (2)		
O1*B*—Cd—O4	87.20 (17)	O4—Cd—N1	111.27 (8)
O1*B*—Cd—N1	116.61 (15)	O4—Cd—N2	83.82 (9)
O1*B*—Cd—N2	170.59 (16)	O4—Cd—N3	149.25 (10)
O1*B*—Cd—N3	103.13 (15)	O4—Cd—N4	83.10 (9)
O1*B*—Cd—N4	87.58 (15)	N1—Cd—N2	69.60 (7)
O2*A*—Cd—O4	118.42 (16)	N1—Cd—N3	90.12 (8)
O2*A*—Cd—N1	83.65 (14)	N1—Cd—N4	151.53 (8)
O2*A*—Cd—N2	150.56 (15)	N2—Cd—N3	83.41 (8)
O2*A*—Cd—N3	84.60 (14)	N2—Cd—N4	88.57 (7)
O2*A*—Cd—N4	111.80 (14)	N3—Cd—N4	68.73 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*WA*—H1*A*···O2*A*	0.98	2.18	3.145 (9)	168
O1*WB*—H1*B*···O2*B*	0.94	2.03	2.969 (10)	172
O1*WB*—H2*B*···O5^{i}	0.98	2.05	3.036 (8)	178
O3*A*—H3*A*···O1*A*	0.82	1.80	2.533 (10)	148
O3*B*—H3*B*···O1*B*	0.82	1.88	2.563 (10)	139
O6—H6*A*···O5	0.82	1.84	2.573 (5)	148

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

One 2-HBA anion is disordered over two sites. Occupancies were refined and converged to 0.506 (4) and 0.494 (4), respectively; in the final cycles of refinement they were fixed at 0.5. The uncoordinated water molecule is also disordered, and the occupancy factors were fixed at 0.5 for each component. Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, U_{iso}(H) = 1.5U_{eq}(O). Other H atoms were placed in calculated positions, C—H = 0.93 and O—H = 0.82 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C) and U_{iso}(H) = 1.5U_{eq}(O).

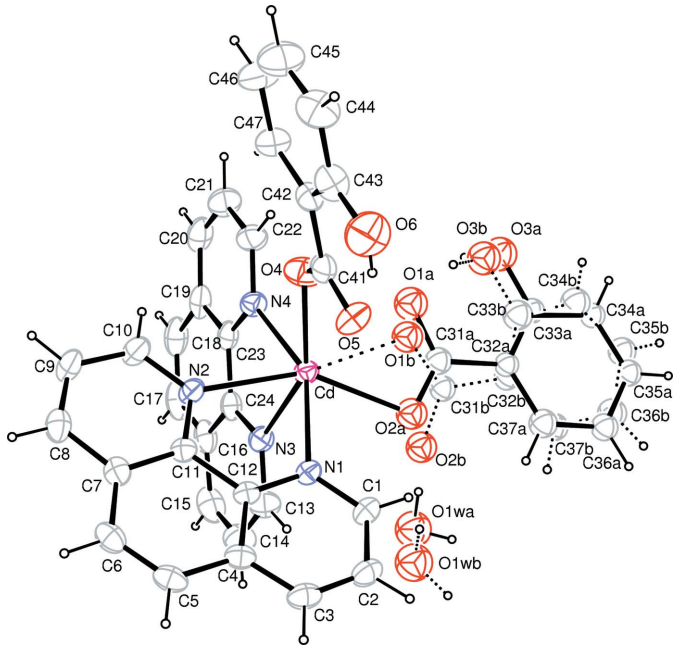


Figure 1
The molecular structure of (I) with 25% probability displacement ellipsoids (arbitrary spheres for H atoms). Suffixes a and b indicate the two disordered components.

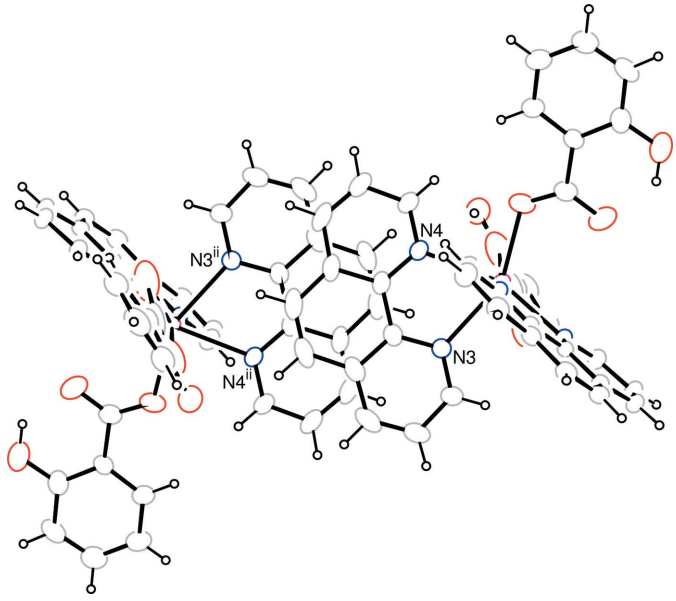


Figure 2
π–π Stacking between parallel N3-phen and N3^{ii}-phen ligands [symmetry code: (ii) $1-x, 1-y, 1-z$]. One disordered 2-HBA component has been omitted for clarity. The disordered water molecules have been omitted.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for*

Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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