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Di-μ-iodido-bis{[hydroxy(methoxy)-bis(2-pyridyl)methane-κ^3N,O,N']iodidocadmium(II)}

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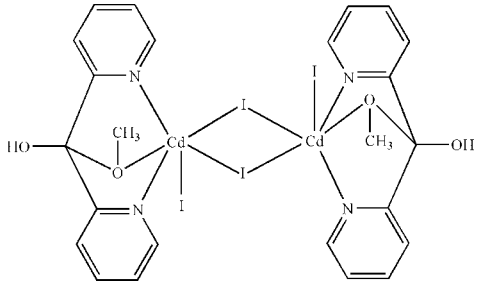
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.027; wR factor = 0.056; data-to-parameter ratio = 24.1.

In the centrosymmetric dinuclear title compound, $[Cd_2I_4(C_{12}H_{12}N_2O_2)_2]$, two μ-I atoms bridge two Cd^{II} atoms and each Cd^{II} atom is also bonded to a terminal I atom and a hydroxy-methoxy-bis(2-pyridyl)methane ligand, which functions in an N,O,N'-tridentate mode, resulting in a distorted octahedral coordination environment. Intermolecular O—H···I hydrogen bonds and π–π stacking interactions between the pyridine rings [centroid–centroid distance = 3.790 (2) Å] are present in the crystal structure.

Related literature

For general background to metal complexes with bis(2-pyridyl)ketone or derivative ligands, see: Bandoli *et al.* (1994); Breeze *et al.* (1996); Crowder *et al.* (2004); Hemmert *et al.* (1999); Katsoulakou *et al.* (2002); Kavounis *et al.* (1996); Padhi & Sahu (2008); Papadopoulos *et al.* (1996); Rattanaphani & McWhinnie (1974); Serna *et al.* (2001); Sommerer *et al.* (1993); Tangoulis *et al.* (1997).



Experimental

Crystal data

$[Cd_2I_4(C_{12}H_{12}N_2O_2)_2]$
M_r = 1164.89
Monoclinic, $P2_1/c$
a = 9.6684 (6) Å
b = 10.1083 (7) Å
c = 16.4970 (13) Å
β = 105.365 (5)°
V = 1554.64 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.38 mm^{-1}
T = 298 K
0.33 × 0.09 × 0.05 mm

Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.560, T_{max} = 0.760
12218 measured reflections
4186 independent reflections
3589 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.056
S = 1.08
4186 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cd1—N1	2.381 (3)	Cd1—I1	2.8868 (4)
Cd1—N2	2.371 (3)	Cd1—I1^{i}	2.9951 (4)
Cd1—O1	2.633 (2)	Cd1—I2	2.8082 (4)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···I2^{ii}	0.82	2.73	3.509 (3)	160

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We are grateful to the University of Urmiyeh and Shahid Beheshti University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2362).

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supporting information

Acta Cryst. (2010). E**66**, m1434–m1435 [https://doi.org/10.1107/S1600536810041681]

Di-μ-iodido-bis{[hydroxy(methoxy)bis(2-pyridyl)methane-κ^3N,O,N']iodidocadmium(II)}

Majid Esmhosseini, Nasser Safari and Vahid Amani

S1. Comment

Bis(2-pyridyl)ketone (bpk) is a molecule that can exhibit different modes of coordination. As a bidentate ligand, it may present an N,O-coordination giving a five-membered chelate ring or an N,N-coordination forming a three or six-membered ring (Crowder *et al.*, 2004; Rattanaphani & McWhinnie, 1974; Sommerer *et al.*, 1993). Frequently the coordinated bpk undergoes nucleophilic addition of water or an alcohol at the carbonylic carbon atom to form the diol or the corresponding hemiacetal, (pyridyl)$_2$C(OR)(OH), which, deprotonated, acts as a mononegative, tridentate N,O,N-donor ligand. The three donor atoms may be coordinated to one metal atom (Bandoli *et al.*, 1994; Crowder *et al.*, 2004; Kavounis *et al.*, 1996; Padhi & Sahu, 2008) or to two metal atoms in a bridging coordination (Breeze *et al.*, 1996; Hemmert *et al.*, 1999; Katsoulakou *et al.*, 2002; Papadopoulos *et al.*, 1996; Serna *et al.*, 2001; Tangoulis *et al.*, 1997). Here, we report the synthesis and structure of the title compound.

The asymmetric unit of the title compound contains a half of the molecule (Fig. 1). Two μ-I atoms bridge two Cd^{II} atoms, and each Cd^{II} atom is also bonded to a terminal I atom and an organic ligand which functions in an N,O,N′-tridentate mode, resulting in a distorted octahedral coordination environment. The Cd—I, Cd—O and Cd—N bond lengths are collected in Table 1.

In the crystal structure, intermolecular O—H···I hydrogen bonds (Table 2) and π–π interactions between the pyridine rings (Fig. 2), *Cg*···*Cg*^{i} [symmetry code: (i) -*x*, 2-y,-*z*; *Cg* is the centroid of the N1, C1–C5 ring], stabilize the structure, with a centroid-centroid distance of 3.790 (2) Å.

S2. Experimental

For the preparation of the title compound, a solution of bis(2-pyridyl)ketone (0.15 g, 0.80 mmol) in methanol (10 ml) was added to a solution of CdI_2 (0.29 g, 0.80 mmol) in methanol (5 ml) at room temperature. Crystals suitable for X-ray diffraction experiment were obtained by methanol diffusion into a colorless solution in DMSO. The crystals were isolated after one week (yield: 0.34 g, 72.9%).

S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic) and 0.96 (methyl), O—H = 0.82 Å, and with U_{iso}(H) = 1.2(1.5 for methyl and hydroxyl)U_{eq}(C,O).

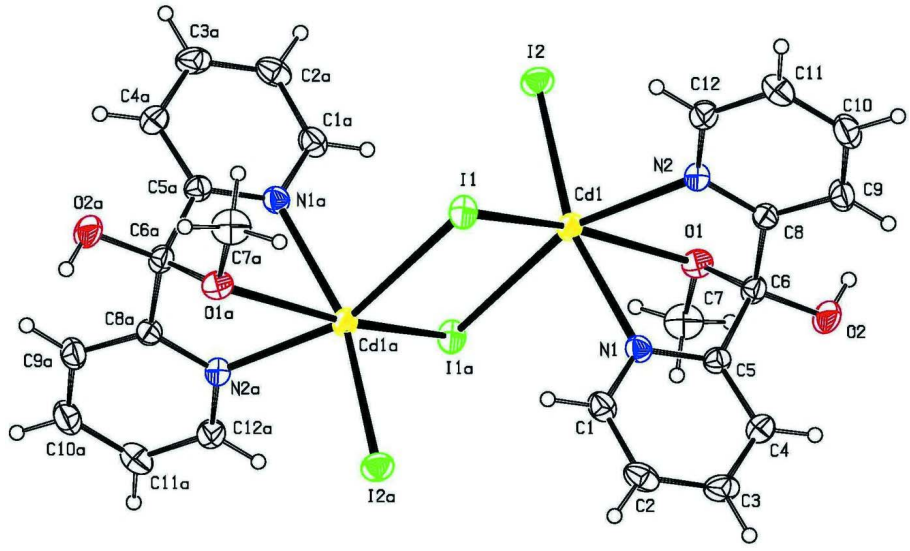

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (a) 1-x, 2-y, -z.]

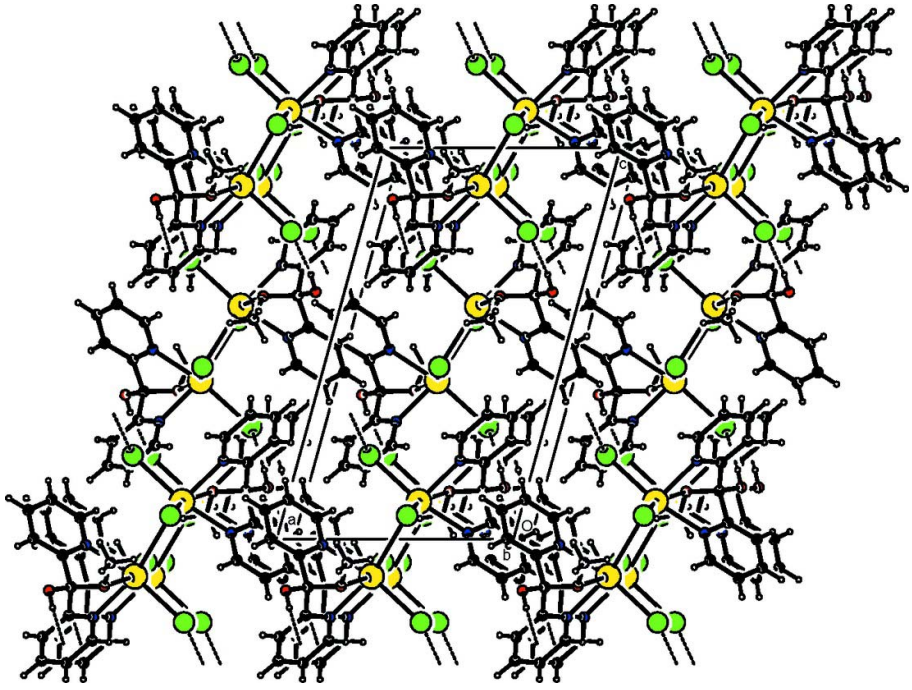

Figure 2

Packing diagram of the title compound. Dashed lines denote hydrogen bonds.

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Crystal data

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a = 9.6684 (6) Å
b = 10.1083 (7) Å

c = 16.4970 (13) Å
β = 105.365 (5)°
V = 1554.64 (19) Å^3
Z = 2
F(000) = 1072
D_x = 2.489 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 998 reflections
θ = 2.2–29.3°
μ = 5.38 mm^{-1}
T = 298 K
Needle, colorless
0.33 × 0.09 × 0.05 mm

Data collection

Bruker APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.560, T_{max} = 0.760

12218 measured reflections
4186 independent reflections
3589 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 29.3°, θ_{min} = 2.2°
h = −13→13
k = −13→13
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.056
S = 1.08
4186 reflections
174 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0193P)^2 + 1.4317P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1500 (4)	0.9278 (4)	−0.0544 (2)	0.0400 (8)
H1	0.2045	0.9914	−0.0726	0.048*
C2	0.0103 (4)	0.9080 (4)	−0.1006 (2)	0.0449 (9)
H2	−0.0289	0.9575	−0.1488	0.054*
C3	−0.0696 (4)	0.8134 (5)	−0.0738 (2)	0.0487 (10)
H3	−0.1637	0.7970	−0.1042	0.058*
C4	−0.0093 (4)	0.7425 (4)	−0.0012 (2)	0.0429 (8)
H4	−0.0622	0.6790	0.0182	0.051*
C5	0.1307 (3)	0.7683 (3)	0.0414 (2)	0.0321 (6)
C6	0.2084 (3)	0.6988 (3)	0.1231 (2)	0.0333 (7)
C7	0.3574 (5)	0.5630 (4)	0.0613 (3)	0.0546 (10)
H7A	0.3376	0.4787	0.0825	0.082*
H7B	0.4517	0.5619	0.0525	0.082*
H7C	0.2878	0.5808	0.0090	0.082*
C8	0.2258 (3)	0.7955 (3)	0.1957 (2)	0.0328 (7)
C9	0.1399 (4)	0.7896 (4)	0.2505 (2)	0.0425 (8)
H9	0.0728	0.7222	0.2462	0.051*
C10	0.1556 (4)	0.8853 (5)	0.3119 (2)	0.0499 (10)
H10	0.0983	0.8836	0.3492	0.060*

C11	0.2557 (5)	0.9825 (4)	0.3176 (2)	0.0469 (9)
H11	0.2684	1.0473	0.3590	0.056*
C12	0.3383 (4)	0.9825 (4)	0.2602 (2)	0.0409 (8)
H12	0.4066	1.0487	0.2640	0.049*
N1	0.2106 (3)	0.8594 (3)	0.01554 (17)	0.0330 (6)
N2	0.3235 (3)	0.8920 (3)	0.20032 (18)	0.0340 (6)
O1	0.3501 (2)	0.6642 (2)	0.12090 (16)	0.0373 (5)
O2	0.1277 (3)	0.5886 (3)	0.13179 (18)	0.0478 (6)
H2A	0.1654	0.5513	0.1763	0.072*
Cd1	0.45074 (2)	0.89825 (2)	0.095665 (15)	0.03330 (6)
I1	0.46360 (3)	1.17684 (2)	0.059856 (16)	0.03945 (6)
I2	0.71540 (3)	0.85526 (3)	0.213660 (16)	0.04384 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0416 (18)	0.044 (2)	0.0351 (17)	0.0046 (16)	0.0115 (14)	0.0072 (15)
C2	0.0449 (19)	0.057 (2)	0.0317 (17)	0.0187 (18)	0.0077 (15)	0.0028 (16)
C3	0.0281 (16)	0.075 (3)	0.0395 (19)	0.0080 (18)	0.0027 (14)	−0.0097 (19)
C4	0.0319 (16)	0.054 (2)	0.0418 (18)	−0.0047 (16)	0.0080 (14)	−0.0011 (17)
C5	0.0293 (15)	0.0334 (16)	0.0330 (15)	0.0016 (13)	0.0071 (12)	−0.0014 (13)
C6	0.0292 (14)	0.0314 (16)	0.0383 (17)	−0.0022 (13)	0.0073 (13)	0.0057 (14)
C7	0.061 (2)	0.040 (2)	0.061 (3)	0.0116 (19)	0.013 (2)	−0.0058 (19)
C8	0.0308 (15)	0.0351 (17)	0.0314 (15)	−0.0013 (13)	0.0062 (12)	0.0049 (13)
C9	0.0420 (18)	0.048 (2)	0.0395 (18)	−0.0023 (17)	0.0146 (15)	0.0121 (17)
C10	0.051 (2)	0.063 (3)	0.041 (2)	0.002 (2)	0.0227 (17)	0.0076 (19)
C11	0.061 (2)	0.048 (2)	0.0345 (18)	0.0048 (19)	0.0166 (17)	0.0008 (16)
C12	0.0429 (19)	0.0398 (19)	0.0408 (18)	−0.0067 (16)	0.0127 (15)	−0.0038 (16)
N1	0.0308 (13)	0.0369 (14)	0.0311 (13)	0.0025 (12)	0.0080 (11)	0.0040 (12)
N2	0.0334 (13)	0.0354 (15)	0.0344 (14)	−0.0026 (11)	0.0109 (11)	0.0015 (12)
O1	0.0343 (12)	0.0350 (12)	0.0400 (13)	0.0055 (10)	0.0054 (10)	0.0005 (10)
O2	0.0493 (15)	0.0385 (14)	0.0524 (16)	−0.0161 (12)	0.0080 (12)	0.0098 (12)
Cd1	0.02758 (11)	0.03613 (13)	0.03617 (12)	−0.00235 (9)	0.00842 (9)	0.00239 (10)
I1	0.04378 (12)	0.03060 (11)	0.04923 (13)	−0.00043 (9)	0.02155 (10)	−0.00408 (9)
I2	0.03782 (12)	0.04460 (14)	0.04293 (13)	0.00002 (10)	−0.00012 (9)	0.00529 (10)

Geometric parameters (Å, º)

C1—N1	1.340 (4)	C8—N2	1.346 (4)
C1—C2	1.378 (5)	C8—C9	1.383 (5)
C1—H1	0.9300	C9—C10	1.380 (6)
C2—C3	1.375 (6)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.365 (6)
C3—C4	1.384 (6)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.391 (5)
C4—C5	1.375 (5)	C11—H11	0.9300
C4—H4	0.9300	C12—N2	1.327 (4)
C5—N1	1.342 (4)	C12—H12	0.9300

C5—C6	1.529 (5)	Cd1—N1	2.381 (3)
C6—O2	1.389 (4)	Cd1—N2	2.371 (3)
C6—O1	1.424 (4)	Cd1—O1	2.633 (2)
C6—C8	1.520 (5)	O2—H2A	0.8200
C7—O1	1.433 (5)	Cd1—I1	2.8868 (4)
C7—H7A	0.9600	Cd1—I1^{i}	2.9951 (4)
C7—H7B	0.9600	Cd1—I2	2.8082 (4)
C7—H7C	0.9600		

N1—C1—C2	122.8 (4)	C11—C10—H10	120.2
N1—C1—H1	118.6	C9—C10—H10	120.2
C2—C1—H1	118.6	C10—C11—C12	118.5 (4)
C3—C2—C1	118.4 (3)	C10—C11—H11	120.7
C3—C2—H2	120.8	C12—C11—H11	120.7
C1—C2—H2	120.8	N2—C12—C11	122.6 (3)
C2—C3—C4	119.7 (3)	N2—C12—H12	118.7
C2—C3—H3	120.2	C11—C12—H12	118.7
C4—C3—H3	120.2	C1—N1—C5	118.1 (3)
C5—C4—C3	118.5 (4)	C1—N1—Cd1	122.2 (2)
C5—C4—H4	120.8	C5—N1—Cd1	119.7 (2)
C3—C4—H4	120.8	C12—N2—C8	118.6 (3)
N1—C5—C4	122.6 (3)	C12—N2—Cd1	123.3 (2)
N1—C5—C6	114.0 (3)	C8—N2—Cd1	118.0 (2)
C4—C5—C6	123.5 (3)	C6—O1—C7	114.5 (3)
O2—C6—O1	112.1 (3)	C6—O1—Cd1	100.48 (18)
O2—C6—C8	112.5 (3)	C7—O1—Cd1	116.5 (2)
O1—C6—C8	105.7 (3)	C6—O2—H2A	109.5
O2—C6—C5	107.5 (3)	N2—Cd1—N1	77.41 (9)
O1—C6—C5	110.0 (3)	N2—Cd1—O1	64.93 (9)
C8—C6—C5	108.9 (3)	N1—Cd1—O1	65.95 (8)
O1—C7—H7A	109.5	N2—Cd1—I2	92.54 (7)
O1—C7—H7B	109.5	N1—Cd1—I2	159.22 (7)
H7A—C7—H7B	109.5	O1—Cd1—I2	93.35 (5)
O1—C7—H7C	109.5	N2—Cd1—I1	103.52 (7)
H7A—C7—H7C	109.5	N1—Cd1—I1	97.81 (7)
H7B—C7—H7C	109.5	O1—Cd1—I1	161.21 (5)
N2—C8—C9	121.9 (3)	I2—Cd1—I1	102.242 (11)
N2—C8—C6	116.2 (3)	N2—Cd1—I1^{i}	158.66 (7)
C9—C8—C6	121.8 (3)	N1—Cd1—I1^{i}	86.16 (7)
C10—C9—C8	118.8 (4)	O1—Cd1—I1^{i}	96.05 (5)
C10—C9—H9	120.6	I2—Cd1—I1^{i}	98.429 (11)
C8—C9—H9	120.6	I1—Cd1—I1^{i}	92.019 (9)
C11—C10—C9	119.6 (3)	Cd1—I1—Cd1^{i}	87.981 (9)

N1—C1—C2—C3	−0.4 (6)	C8—C6—O1—Cd1	−60.1 (2)
C1—C2—C3—C4	1.0 (6)	C5—C6—O1—Cd1	57.4 (3)
C2—C3—C4—C5	−0.9 (6)	C12—N2—Cd1—N1	−131.9 (3)
C3—C4—C5—N1	0.2 (5)	C8—N2—Cd1—N1	44.4 (2)

C3—C4—C5—C6	178.8 (3)	C12—N2—Cd1—O1	159.0 (3)
N1—C5—C6—O2	−166.4 (3)	C8—N2—Cd1—O1	−24.7 (2)
C4—C5—C6—O2	14.9 (5)	C12—N2—Cd1—I2	66.5 (3)
N1—C5—C6—O1	−44.1 (4)	C8—N2—Cd1—I2	−117.2 (2)
C4—C5—C6—O1	137.2 (3)	C12—N2—Cd1—I1	−36.7 (3)
N1—C5—C6—C8	71.4 (3)	C8—N2—Cd1—I1	139.5 (2)
C4—C5—C6—C8	−107.3 (4)	C12—N2—Cd1—I1^{i}	−172.4 (2)
O2—C6—C8—N2	168.1 (3)	C8—N2—Cd1—I1^{i}	3.9 (4)
O1—C6—C8—N2	45.4 (4)	C1—N1—Cd1—N2	133.1 (3)
C5—C6—C8—N2	−72.7 (4)	C5—N1—Cd1—N2	−45.4 (2)
O2—C6—C8—C9	−15.8 (4)	C1—N1—Cd1—O1	−159.0 (3)
O1—C6—C8—C9	−138.5 (3)	C5—N1—Cd1—O1	22.5 (2)
C5—C6—C8—C9	103.4 (4)	C1—N1—Cd1—I2	−164.33 (19)
N2—C8—C9—C10	−0.2 (5)	C5—N1—Cd1—I2	17.2 (4)
C6—C8—C9—C10	−176.1 (3)	C1—N1—Cd1—I1	30.9 (3)
C8—C9—C10—C11	−0.6 (6)	C5—N1—Cd1—I1	−147.6 (2)
C9—C10—C11—C12	0.7 (6)	C1—N1—Cd1—I1^{i}	−60.6 (3)
C10—C11—C12—N2	0.0 (6)	C5—N1—Cd1—I1^{i}	120.9 (2)
C2—C1—N1—C5	−0.3 (5)	C6—O1—Cd1—N2	45.57 (19)
C2—C1—N1—Cd1	−178.8 (3)	C7—O1—Cd1—N2	169.9 (3)
C4—C5—N1—C1	0.4 (5)	C6—O1—Cd1—N1	−41.29 (19)
C6—C5—N1—C1	−178.3 (3)	C7—O1—Cd1—N1	83.0 (2)
C4—C5—N1—Cd1	179.0 (3)	C6—O1—Cd1—I2	136.82 (18)
C6—C5—N1—Cd1	0.3 (4)	C7—O1—Cd1—I2	−98.9 (2)
C11—C12—N2—C8	−0.8 (5)	C6—O1—Cd1—I1	−9.4 (3)
C11—C12—N2—Cd1	175.4 (3)	C7—O1—Cd1—I1	114.9 (3)
C9—C8—N2—C12	0.9 (5)	C6—O1—Cd1—I1^{i}	−124.33 (18)
C6—C8—N2—C12	177.0 (3)	C7—O1—Cd1—I1^{i}	0.0 (2)
C9—C8—N2—Cd1	−175.5 (3)	N2—Cd1—I1—Cd1^{i}	−165.25 (7)
C6—C8—N2—Cd1	0.5 (4)	N1—Cd1—I1—Cd1^{i}	−86.40 (7)
O2—C6—O1—C7	51.3 (4)	O1—Cd1—I1—Cd1^{i}	−115.52 (17)
C8—C6—O1—C7	174.3 (3)	I2—Cd1—I1—Cd1^{i}	99.076 (12)
C5—C6—O1—C7	−68.3 (4)	I1^{i}—Cd1—I1—Cd1^{i}	0.0
O2—C6—O1—Cd1	176.9 (2)		

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···I2^{ii}	0.82	2.73	3.509 (3)	160

Symmetry code: (ii) −*x*+1, *y*−1/2, −*z*+1/2.