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Dichloridobis(2-dimethylamino-1,10-phenanthroline)cadmium(II)

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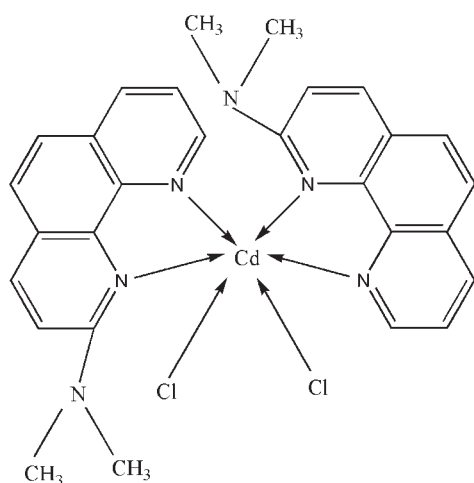
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.032; wR factor = 0.078; data-to-parameter ratio = 16.1.

In the title complex, $[\text{CdCl}_2(\text{C}_{14}\text{H}_{13}\text{N}_3)_2]$, the Cd^{II} ion lies on a twofold rotation axis and assumes a distorted octahedral CdN_4Cl_2 coordination geometry. There is a π - π stacking interaction between the symmetry-related 1,10-phenanthroline ligands with a centroid-centroid distance of 3.5578 (16) Å and a perpendicular distance of 3.445 (su?) Å between the relevant rings.

Related literature

For background to the use of 1,10-phenanthroline derivatives in coordination chemistry, see: Liu *et al.* (2008). For a related structure, see: Zhang *et al.* (2008).



Experimental

Crystal data

$[\text{CdCl}_2(\text{C}_{14}\text{H}_{13}\text{N}_3)_2]$
 $M_r = 629.85$
 Monoclinic, $C2/c$
 $a = 17.161$ (2) Å
 $b = 9.3572$ (12) Å
 $c = 16.805$ (2) Å
 $\beta = 110.343$ (2)°

$V = 2530.2$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.11$ mm⁻¹
 $T = 298$ K
 $0.36 \times 0.19 \times 0.17$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\text{min}} = 0.692$, $T_{\text{max}} = 0.834$

7147 measured reflections
 2741 independent reflections
 2512 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.078$
 $S = 1.04$
 2741 reflections

170 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.67$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5029).

References

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
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supporting information

Acta Cryst. (2009). E65, m1150 [doi:10.1107/S1600536809032358]

Dichloridobis(2-dimethylamino-1,10-phenanthroline)cadmium(II)**Hong Liang Li****S1. Comment**

Derivatives of 1,10-phenanthroline play an important role in modern coordination chemistry (Liu *et al.*, 2008), and only one complex with 2-(dimethyl)amine-1,10-phenanthroline as ligand has been published (Zhang *et al.*, 2008) so far. Now the crystal structure of title complex, which is the second complex dealing with 2-(dimethyl)amine-1,10-phenanthroline as ligand, is reported.

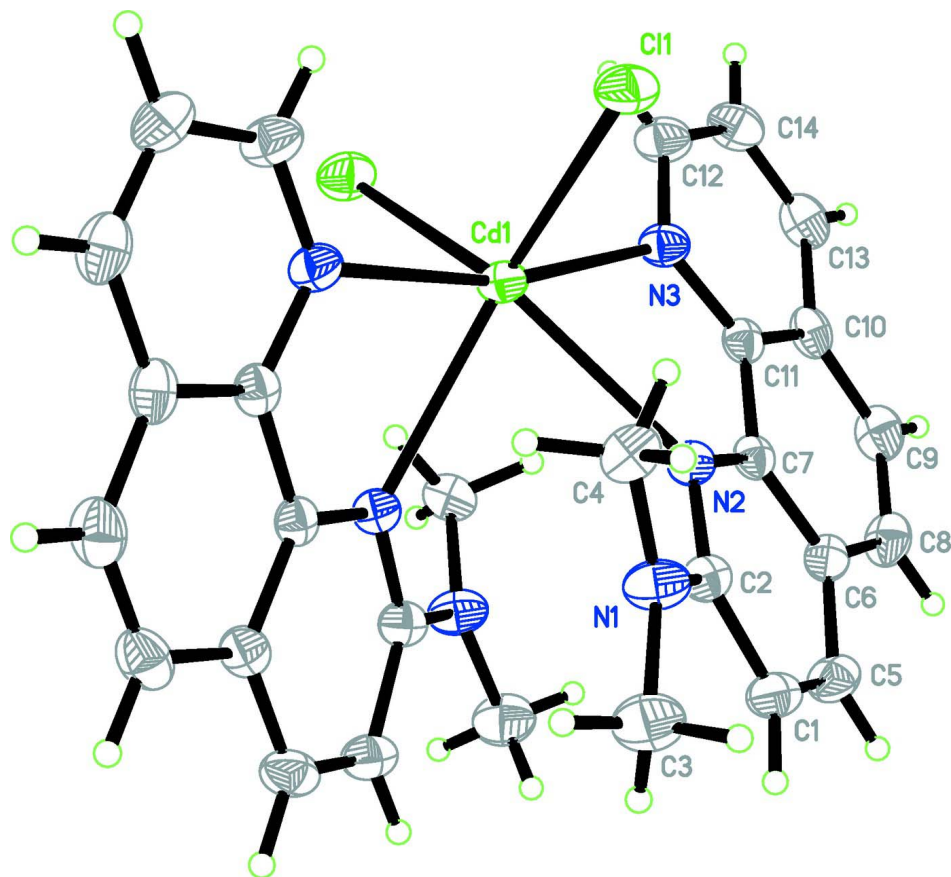
Fig. 1 and Table 1 show the coordination structure, with the Cd centre located on a crystallographic twofold axis. It is in a distorted octahedral geometry. There is a π - π stacking interaction involving symmetry related 1,10-phenanthroline ligands, with the relevant distances being $Cg1 \cdots Cg2^i = 3.5578(16)$ Å and $Cg1 \cdots Cg2^i_{\text{perp}} = 3.445$ Å and $\alpha = 3.82^\circ$ [symmetry code: (i) 1-*X*, -*Y*, -*Z*; *Cg1* and *Cg2* are the centroids of C1C2C5-C7/N2 ring and C6—C11 ring, respectively; $Cg1 \cdots Cg2^i_{\text{perp}}$ is the perpendicular distance from ring *Cg1* to ring *Cg2*ⁱ; α is the dihedral between ring plane *Cg1* and ring plane *Cg2*ⁱ].

S2. Experimental

10 ml methanol solution of 2-(dimethyl)amine-1,10-phenanthroline (0.1438 g, 0.644 mmol) was added into 5 ml H₂O solution of cadmium chloride (0.1485 g, 0.650 mmol), and the mixed solution was stirred for a few minutes. The yellow single crystals were obtained after the filtrate had been allowed to stand at room temperature for two weeks.

S3. Refinement

All H atoms were placed in calculated positions and refined as riding with C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl groups, and C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for other H atoms.

**Figure 1**

Coordination diagram of title complex with atom-numbering scheme for only asymmetric unit, and displacement ellipsoids being at the 30% probability level

Dichloridobis(2-dimethylamino-1,10-phenanthroline)cadmium(II)

Crystal data

$[\text{CdCl}_2(\text{C}_{14}\text{H}_{13}\text{N}_3)_2]$

$M_r = 629.85$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 17.161(2) \text{ \AA}$

$b = 9.3572(12) \text{ \AA}$

$c = 16.805(2) \text{ \AA}$

$\beta = 110.343(2)^\circ$

$V = 2530.2(6) \text{ \AA}^3$

$Z = 4$

$F(000) = 1272$

$D_x = 1.653 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3730 reflections

$\theta = 2.5\text{--}27.9^\circ$

$\mu = 1.11 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, yellow

$0.36 \times 0.19 \times 0.17 \text{ mm}$

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 1997)

$T_{\min} = 0.692$, $T_{\max} = 0.834$

7147 measured reflections

2741 independent reflections

2512 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -21 \rightarrow 21$

$k = -6 \rightarrow 11$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.078$
 $S = 1.04$
 2741 reflections
 170 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
 $w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.8626P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.67 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.53071 (18)	-0.2662 (3)	0.14483 (19)	0.0453 (6)
H1	0.5561	-0.3519	0.1680	0.054*
C2	0.55652 (14)	-0.1339 (3)	0.18970 (16)	0.0366 (5)
C3	0.6672 (2)	-0.2607 (3)	0.3007 (2)	0.0597 (8)
H3A	0.6275	-0.3371	0.2899	0.090*
H3B	0.6916	-0.2451	0.3608	0.090*
H3C	0.7099	-0.2855	0.2785	0.090*
C4	0.67199 (15)	-0.0021 (3)	0.29048 (17)	0.0442 (6)
H4A	0.6495	0.0734	0.2503	0.066*
H4B	0.7293	-0.0169	0.2972	0.066*
H4C	0.6679	0.0237	0.3442	0.066*
C5	0.46892 (18)	-0.2641 (3)	0.0683 (2)	0.0468 (6)
H5	0.4529	-0.3484	0.0377	0.056*
C6	0.42857 (14)	-0.1347 (3)	0.03479 (15)	0.0378 (5)
C7	0.45301 (13)	-0.0134 (2)	0.08600 (14)	0.0324 (5)
C8	0.36608 (16)	-0.1257 (3)	-0.04727 (16)	0.0483 (7)
H8	0.3509	-0.2077	-0.0804	0.058*
C9	0.32884 (17)	-0.0021 (3)	-0.07762 (17)	0.0507 (7)
H9	0.2884	0.0010	-0.1315	0.061*
C10	0.35021 (14)	0.1243 (3)	-0.02876 (15)	0.0410 (6)
C11	0.40983 (14)	0.1189 (3)	0.05419 (14)	0.0346 (5)
C12	0.39202 (17)	0.3585 (3)	0.07410 (18)	0.0488 (6)

H12	0.4049	0.4381	0.1094	0.059*
C13	0.31509 (18)	0.2587 (4)	-0.05904 (18)	0.0499 (7)
H13	0.2766	0.2671	-0.1137	0.060*
C14	0.33721 (18)	0.3755 (3)	-0.00872 (18)	0.0531 (7)
H14	0.3163	0.4652	-0.0290	0.064*
Cd1	0.5000	0.20663 (2)	0.2500	0.03387 (10)
Cl1	0.61434 (4)	0.36432 (7)	0.22647 (5)	0.05136 (18)
N1	0.62589 (13)	-0.1317 (2)	0.26011 (15)	0.0465 (5)
N2	0.51504 (11)	-0.0126 (2)	0.16313 (12)	0.0339 (4)
N3	0.42685 (13)	0.2347 (2)	0.10524 (14)	0.0396 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0450 (15)	0.0335 (13)	0.0594 (17)	0.0015 (11)	0.0208 (13)	0.0007 (12)
C2	0.0310 (11)	0.0353 (12)	0.0446 (13)	-0.0017 (9)	0.0144 (10)	-0.0014 (10)
C3	0.0533 (18)	0.0496 (16)	0.068 (2)	0.0181 (14)	0.0110 (16)	0.0104 (15)
C4	0.0343 (12)	0.0454 (14)	0.0472 (14)	0.0030 (11)	0.0069 (11)	-0.0062 (12)
C5	0.0462 (15)	0.0371 (13)	0.0596 (17)	-0.0105 (11)	0.0216 (14)	-0.0121 (12)
C6	0.0340 (12)	0.0406 (13)	0.0390 (12)	-0.0075 (10)	0.0130 (10)	-0.0056 (11)
C7	0.0285 (11)	0.0375 (12)	0.0322 (11)	-0.0063 (9)	0.0117 (9)	-0.0011 (9)
C8	0.0455 (14)	0.0565 (17)	0.0404 (14)	-0.0104 (13)	0.0118 (12)	-0.0146 (13)
C9	0.0451 (15)	0.0664 (19)	0.0334 (13)	-0.0081 (13)	0.0045 (11)	-0.0037 (13)
C10	0.0317 (12)	0.0576 (16)	0.0340 (12)	-0.0023 (11)	0.0117 (10)	0.0045 (12)
C11	0.0303 (11)	0.0406 (13)	0.0340 (11)	-0.0070 (10)	0.0124 (9)	-0.0019 (10)
C12	0.0505 (15)	0.0382 (14)	0.0493 (15)	0.0040 (12)	0.0066 (12)	0.0062 (12)
C13	0.0396 (14)	0.0670 (18)	0.0368 (14)	0.0022 (13)	0.0053 (11)	0.0113 (13)
C14	0.0520 (16)	0.0485 (16)	0.0535 (16)	0.0080 (13)	0.0115 (13)	0.0168 (14)
Cd1	0.03261 (15)	0.02812 (14)	0.03351 (15)	0.000	0.00217 (10)	0.000
Cl1	0.0486 (4)	0.0394 (4)	0.0652 (4)	-0.0099 (3)	0.0188 (3)	-0.0034 (3)
N1	0.0360 (11)	0.0370 (12)	0.0562 (13)	0.0051 (9)	0.0029 (10)	0.0034 (10)
N2	0.0299 (9)	0.0336 (10)	0.0356 (10)	-0.0019 (8)	0.0080 (8)	-0.0006 (8)
N3	0.0395 (11)	0.0342 (10)	0.0406 (12)	0.0013 (9)	0.0083 (9)	0.0032 (9)

Geometric parameters (Å, °)

C1—C5	1.353 (4)	C8—C9	1.334 (4)
C1—C2	1.437 (4)	C8—H8	0.9300
C1—H1	0.9300	C9—C10	1.413 (4)
C2—N2	1.332 (3)	C9—H9	0.9300
C2—N1	1.356 (3)	C10—C13	1.412 (4)
C3—N1	1.445 (4)	C10—C11	1.415 (3)
C3—H3A	0.9600	C11—N3	1.349 (3)
C3—H3B	0.9600	C12—N3	1.325 (3)
C3—H3C	0.9600	C12—C14	1.392 (4)
C4—N1	1.441 (3)	C12—H12	0.9300
C4—H4A	0.9600	C13—C14	1.353 (4)
C4—H4B	0.9600	C13—H13	0.9300

C4—H4C	0.9600	C14—H14	0.9300
C5—C6	1.411 (4)	Cd1—N3 ⁱ	2.332 (2)
C5—H5	0.9300	Cd1—N3	2.332 (2)
C6—C7	1.398 (3)	Cd1—N2 ⁱ	2.582 (2)
C6—C8	1.425 (3)	Cd1—N2	2.582 (2)
C7—N2	1.362 (3)	Cd1—Cl1	2.5928 (7)
C7—C11	1.446 (3)	Cd1—Cl1 ⁱ	2.5928 (7)
C5—C1—C2	118.9 (3)	N3—C11—C10	121.4 (2)
C5—C1—H1	120.6	N3—C11—C7	118.8 (2)
C2—C1—H1	120.6	C10—C11—C7	119.8 (2)
N2—C2—N1	118.9 (2)	N3—C12—C14	123.4 (3)
N2—C2—C1	121.8 (2)	N3—C12—H12	118.3
N1—C2—C1	119.2 (2)	C14—C12—H12	118.3
N1—C3—H3A	109.5	C14—C13—C10	120.2 (2)
N1—C3—H3B	109.5	C14—C13—H13	119.9
H3A—C3—H3B	109.5	C10—C13—H13	119.9
N1—C3—H3C	109.5	C13—C14—C12	118.6 (3)
H3A—C3—H3C	109.5	C13—C14—H14	120.7
H3B—C3—H3C	109.5	C12—C14—H14	120.7
N1—C4—H4A	109.5	N3 ⁱ —Cd1—N3	167.08 (10)
N1—C4—H4B	109.5	N3 ⁱ —Cd1—N2 ⁱ	67.85 (7)
H4A—C4—H4B	109.5	N3—Cd1—N2 ⁱ	123.76 (7)
N1—C4—H4C	109.5	N3 ⁱ —Cd1—N2	123.76 (7)
H4A—C4—H4C	109.5	N3—Cd1—N2	67.85 (7)
H4B—C4—H4C	109.5	N2 ⁱ —Cd1—N2	74.81 (9)
C1—C5—C6	120.4 (3)	N3 ⁱ —Cd1—Cl1	86.19 (6)
C1—C5—H5	119.8	N3—Cd1—Cl1	86.47 (6)
C6—C5—H5	119.8	N2 ⁱ —Cd1—Cl1	140.17 (4)
C7—C6—C5	116.8 (2)	N2—Cd1—Cl1	97.82 (5)
C7—C6—C8	120.8 (2)	N3 ⁱ —Cd1—Cl1 ⁱ	86.47 (6)
C5—C6—C8	122.5 (2)	N3—Cd1—Cl1 ⁱ	86.19 (6)
N2—C7—C6	123.9 (2)	N2 ⁱ —Cd1—Cl1 ⁱ	97.82 (5)
N2—C7—C11	118.5 (2)	N2—Cd1—Cl1 ⁱ	140.17 (4)
C6—C7—C11	117.5 (2)	Cl1—Cd1—Cl1 ⁱ	110.63 (3)
C9—C8—C6	121.4 (3)	C2—N1—C4	122.0 (2)
C9—C8—H8	119.3	C2—N1—C3	122.5 (2)
C6—C8—H8	119.3	C4—N1—C3	114.6 (2)
C8—C9—C10	120.7 (2)	C2—N2—C7	117.7 (2)
C8—C9—H9	119.7	C2—N2—Cd1	129.38 (16)
C10—C9—H9	119.7	C7—N2—Cd1	110.11 (14)
C13—C10—C11	117.3 (3)	C12—N3—C11	118.9 (2)
C13—C10—C9	123.0 (2)	C12—N3—Cd1	121.04 (18)
C11—C10—C9	119.7 (2)	C11—N3—Cd1	119.46 (16)
C5—C1—C2—N2	8.2 (4)	N1—C2—N2—Cd1	-30.5 (3)
C5—C1—C2—N1	-170.5 (3)	C1—C2—N2—Cd1	150.79 (19)
C2—C1—C5—C6	-2.3 (4)	C6—C7—N2—C2	2.4 (3)

C1—C5—C6—C7	-2.9 (4)	C11—C7—N2—C2	-177.1 (2)
C1—C5—C6—C8	176.8 (3)	C6—C7—N2—Cd1	-160.33 (18)
C5—C6—C7—N2	3.1 (3)	C11—C7—N2—Cd1	20.2 (2)
C8—C6—C7—N2	-176.6 (2)	N3 ⁱ —Cd1—N2—C2	7.3 (2)
C5—C6—C7—C11	-177.4 (2)	N3—Cd1—N2—C2	-179.2 (2)
C8—C6—C7—C11	2.9 (3)	N2 ⁱ —Cd1—N2—C2	-42.08 (16)
C7—C6—C8—C9	-0.3 (4)	C11—Cd1—N2—C2	97.91 (18)
C5—C6—C8—C9	-180.0 (3)	C11 ⁱ —Cd1—N2—C2	-126.03 (17)
C6—C8—C9—C10	-0.3 (4)	N3 ⁱ —Cd1—N2—C7	167.33 (14)
C8—C9—C10—C13	177.5 (3)	N3—Cd1—N2—C7	-19.10 (14)
C8—C9—C10—C11	-2.0 (4)	N2 ⁱ —Cd1—N2—C7	117.98 (17)
C13—C10—C11—N3	6.0 (3)	C11—Cd1—N2—C7	-102.02 (14)
C9—C10—C11—N3	-174.5 (2)	C11 ⁱ —Cd1—N2—C7	34.03 (18)
C13—C10—C11—C7	-174.8 (2)	C14—C12—N3—C11	1.1 (4)
C9—C10—C11—C7	4.7 (4)	C14—C12—N3—Cd1	-169.7 (2)
N2—C7—C11—N3	-6.3 (3)	C10—C11—N3—C12	-5.8 (4)
C6—C7—C11—N3	174.1 (2)	C7—C11—N3—C12	175.0 (2)
N2—C7—C11—C10	174.5 (2)	C10—C11—N3—Cd1	165.14 (17)
C6—C7—C11—C10	-5.1 (3)	C7—C11—N3—Cd1	-14.1 (3)
C11—C10—C13—C14	-1.5 (4)	N3 ⁱ —Cd1—N3—C12	-16.5 (2)
C9—C10—C13—C14	179.0 (3)	N2 ⁱ —Cd1—N3—C12	135.9 (2)
C10—C13—C14—C12	-2.9 (4)	N2—Cd1—N3—C12	-171.9 (2)
N3—C12—C14—C13	3.2 (5)	C11—Cd1—N3—C12	-72.0 (2)
N2—C2—N1—C4	-21.3 (4)	C11 ⁱ —Cd1—N3—C12	39.0 (2)
C1—C2—N1—C4	157.4 (2)	N3 ⁱ —Cd1—N3—C11	172.75 (18)
N2—C2—N1—C3	170.2 (3)	N2 ⁱ —Cd1—N3—C11	-34.9 (2)
C1—C2—N1—C3	-11.0 (4)	N2—Cd1—N3—C11	17.35 (17)
N1—C2—N2—C7	170.7 (2)	C11—Cd1—N3—C11	117.28 (18)
C1—C2—N2—C7	-8.0 (3)	C11 ⁱ —Cd1—N3—C11	-131.75 (18)

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