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Di-μ-nitrito-κ^3O:O,O';κ^3O,O':O-bis{[2,6-bis(pyrazol-1-yl-κN^2)pyridine-κN](nitrito-κ^2O,O')cadmium(II)}

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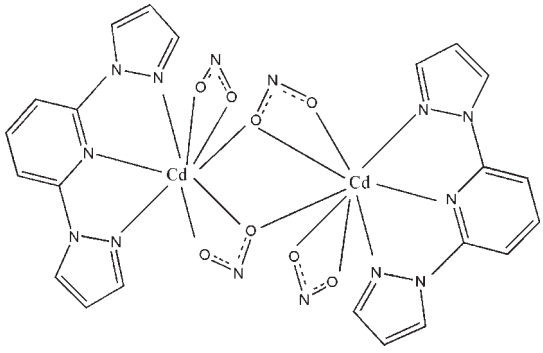
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.029; wR factor = 0.076; data-to-parameter ratio = 12.8.

In the title centrosymmetric binuclear complex, $[Cd_2(NO_2)_4(C_{11}H_9N_5)_2]$, the unique Cd^{II} ion is in a distorted dodecahedral CdN_3O_5 coordination environment. The two inversion-related Cd^{II} ions are separated by 3.9920 (6) Å and are bridged by two O atoms from two nitrite ligands. There are two types of π–π stacking interactions involving symmetry-related pyrazole rings, with centroid–centroid distances of 3.445 (2) and 3.431 (2) Å.

Related literature

For related structures, see: Yang & Sun (2008); Bessel *et al.* (1993).



Experimental

Crystal data

$[Cd_2(NO_2)_4(C_{11}H_9N_5)_2]$
M_r = 831.30
Triclinic, $P\overline{1}$
a = 7.7618 (13) Å
b = 9.5522 (16) Å
c = 10.9665 (19) Å
α = 110.285 (2)°
β = 90.616 (2)°
γ = 112.155 (2)°
V = 696.9 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.60 mm^{-1}
T = 298 K
0.32 × 0.21 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.628, T_{max} = 0.856
3815 measured reflections
2666 independent reflections
2468 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.076
S = 1.09
2666 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2917).

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supporting information

Acta Cryst. (2009). E**65**, m1318 [https://doi.org/10.1107/S1600536809039841]

Di-μ-nitrito-κ^3O:O,O';κ^3O,O':O-bis{[2,6-bis(pyrazol-1-yl-κN^2)pyridine-κN](nitrito-κ^2O,O')cadmium(II)}

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S1. Comment

2,6-Dipyrazol-1-ylpyridine is expected be a useful tridentate ligand, but complexes with it as ligand to our knowledge are somewhat rare (e.g. Yang & Sun, 2008; Bessel et al., 1993). Our interest in complexes with 2,6-dipyrazol-1-ylpyridine as a ligand has motivated us to prepare the title complex, (I), and herein we report its crystal structure.

Fig. 1 shows the molecular structure of the title complex. Each Cd^{II} ion is coordinated by five O atoms and three N atoms in a distorted dodecahedral coordination environment (see Fig. 2). It is rare for Cd^{II} to assume this coordination mode. Fig. 1 also shows that two nitrite anions function as bridging ligands, linking two inversion related Cd^{II} ions with a separation of 3.9920 (6) Å leading to a binuclear Cd^{II} complex. In the crystal there are the strong π–π stacking interactions involving the symmetry related triazole rings, with the relevant distances being Cg1···$Cg2^{i}$ = 3.445 (2) Å, Cg2···$Cg2^{ii}$ = 3.431 (2) Å, Cg1···$Cg2^{i}_{perp}$ = 3.299 Å and Cg2···$Cg2^{ii}_{perp}$ = 3.274 Å (symmetric codes: (i) -1+x, y, z; (ii) 1-x, 1-y, 2-z; Cg1 and Cg2 are the centroids of C1-C3/N1/N2; C9-C11/N4/N5 triazole rings, respectively; Cgi···Cgj_{perp} is the perpendicular distance from Cgi ring to Cgj ring).

S2. Experimental

10 ml dichloromethane solution of 2,6-Dipyrazol-1-ylpyridine (0.0692 g, 0.328 mmol) was added into 10 ml methanol solution containing $Cd(ClO_4).6H_2O$ (0.0740 g, 0.176 mmol) and sodium nitrite (0.0138 g, 0.200 mmol) and the mixed soluton was stirred for a few minutes. The colorless single crystals were obtained after the filtrate had been allowed to stand at room temperature for about one month.

S3. Refinement

All H atoms were placed in calculated positions and refined as riding with C—H = 0.93 Å, $U_{iso} = 1.2U_{eq}$(C).

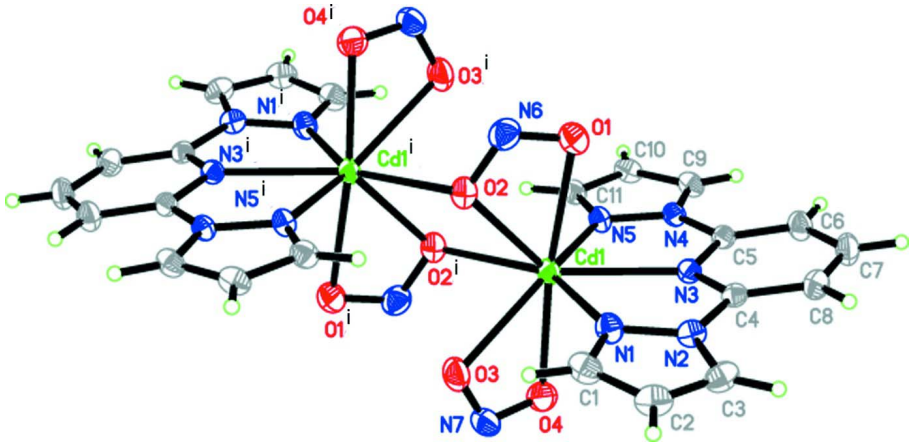

Figure 1

The molecular structure of title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (i) 1 - *x*, 2 - *y*, 2 - *z*

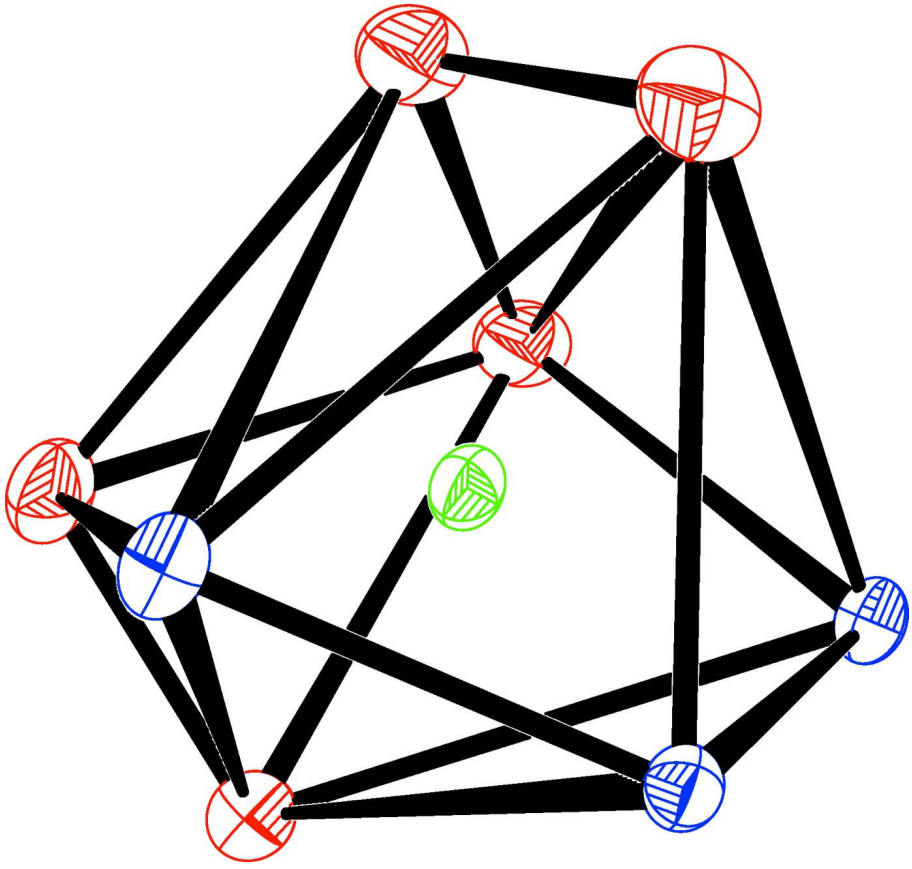

Figure 2

The coordination environment of Cd^{II}

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Crystal data

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$c = 10.9665$ (19) Å
$\alpha = 110.285$ (2)°
$\beta = 90.616$ (2)°
$\gamma = 112.155$ (2)°
$V = 696.9$ (2) Å^3
$Z = 1$
$F(000) = 408$
$D_x = 1.981$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2504 reflections
$\theta = 2.5$–27.8°
$\mu = 1.60$ mm^{-1}
$T = 298$ K
Block, colorless
0.32 × 0.21 × 0.10 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*; Sheldrick, 1996)
$T_{min} = 0.628$, $T_{max} = 0.856$
3815 measured reflections
2666 independent reflections
2468 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -7 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.076$
$S = 1.09$
2666 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.044P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.0700 (5)	0.7853 (5)	0.7004 (4)	0.0445 (8)
H1	−0.0364	0.8974	0.7345	0.053*
C2	−0.2364 (5)	0.6713 (5)	0.6144 (4)	0.0454 (8)

H2	−0.3315	0.6920	0.5817	0.055*
C3	−0.2301 (4)	0.5239 (5)	0.5885 (3)	0.0431 (8)
H3	−0.3207	0.4227	0.5338	0.052*
C4	0.0015 (4)	0.4386 (4)	0.6698 (3)	0.0296 (6)
C5	0.2520 (4)	0.4068 (3)	0.7464 (3)	0.0298 (6)
C6	0.1560 (4)	0.2398 (4)	0.7011 (4)	0.0405 (7)
H6	0.2125	0.1736	0.7117	0.049*
C7	−0.0287 (5)	0.1747 (4)	0.6390 (3)	0.0449 (8)
H7	−0.0984	0.0623	0.6074	0.054*
C8	−0.1103 (5)	0.2735 (4)	0.6234 (3)	0.0410 (8)
H8	−0.2350	0.2311	0.5835	0.049*
C9	0.5554 (4)	0.4211 (4)	0.8411 (3)	0.0357 (7)
H9	0.5291	0.3105	0.8142	0.043*
C10	0.7180 (4)	0.5479 (4)	0.9151 (3)	0.0373 (7)
H10	0.8247	0.5419	0.9481	0.045*
C11	0.6898 (4)	0.6888 (4)	0.9306 (3)	0.0364 (7)
H11	0.7781	0.7945	0.9778	0.044*
Cd1	0.35091 (3)	0.79984 (2)	0.84217 (2)	0.03281 (10)
N1	0.0340 (4)	0.7146 (3)	0.7277 (3)	0.0374 (6)
N2	−0.0654 (3)	0.5526 (3)	0.6580 (3)	0.0340 (6)
N3	0.1788 (3)	0.5057 (3)	0.7307 (2)	0.0290 (5)
N4	0.4388 (3)	0.4869 (3)	0.8142 (2)	0.0291 (5)
N5	0.5215 (3)	0.6530 (3)	0.8697 (2)	0.0323 (5)
N6	0.2056 (4)	0.8778 (4)	1.0874 (3)	0.0506 (7)
N7	0.4464 (4)	0.9665 (4)	0.6637 (3)	0.0485 (7)
O1	0.1706 (4)	0.7339 (3)	1.0265 (3)	0.0568 (7)
O2	0.3107 (4)	0.9700 (3)	1.0329 (3)	0.0542 (7)
O3	0.3877 (4)	1.0225 (3)	0.7663 (3)	0.0506 (6)
O4	0.4610 (4)	0.8348 (3)	0.6500 (3)	0.0491 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0479 (19)	0.048 (2)	0.054 (2)	0.0301 (17)	0.0146 (16)	0.0260 (18)
C2	0.0422 (18)	0.062 (2)	0.048 (2)	0.0337 (17)	0.0107 (15)	0.0256 (18)
C3	0.0323 (16)	0.056 (2)	0.0377 (18)	0.0187 (16)	0.0004 (14)	0.0135 (16)
C4	0.0314 (14)	0.0328 (15)	0.0262 (14)	0.0157 (13)	0.0086 (12)	0.0100 (12)
C5	0.0325 (14)	0.0285 (14)	0.0298 (14)	0.0139 (12)	0.0074 (12)	0.0110 (12)
C6	0.0434 (18)	0.0305 (15)	0.0486 (19)	0.0162 (14)	0.0023 (15)	0.0147 (14)
C7	0.0458 (19)	0.0280 (16)	0.051 (2)	0.0087 (14)	−0.0021 (16)	0.0111 (15)
C8	0.0353 (16)	0.0388 (18)	0.0395 (18)	0.0087 (14)	−0.0009 (14)	0.0112 (15)
C9	0.0379 (16)	0.0376 (16)	0.0449 (18)	0.0231 (14)	0.0152 (14)	0.0219 (15)
C10	0.0312 (15)	0.0472 (19)	0.0455 (18)	0.0210 (14)	0.0100 (14)	0.0256 (16)
C11	0.0314 (15)	0.0367 (16)	0.0412 (17)	0.0109 (13)	0.0041 (13)	0.0182 (14)
Cd1	0.03791 (15)	0.02645 (14)	0.03535 (15)	0.01474 (11)	0.00396 (10)	0.01137 (10)
N1	0.0360 (14)	0.0340 (14)	0.0457 (16)	0.0174 (12)	0.0059 (12)	0.0158 (12)
N2	0.0312 (13)	0.0396 (14)	0.0345 (14)	0.0175 (12)	0.0061 (11)	0.0144 (12)
N3	0.0303 (12)	0.0283 (12)	0.0305 (13)	0.0138 (10)	0.0043 (10)	0.0115 (10)

N4	0.0302 (12)	0.0271 (12)	0.0338 (13)	0.0136 (10)	0.0055 (10)	0.0137 (11)
N5	0.0324 (13)	0.0262 (12)	0.0396 (14)	0.0120 (11)	0.0054 (11)	0.0138 (11)
N6	0.0513 (18)	0.0524 (19)	0.0435 (17)	0.0200 (15)	0.0137 (14)	0.0140 (15)
N7	0.0554 (18)	0.0436 (17)	0.0508 (18)	0.0175 (15)	0.0031 (15)	0.0257 (15)
O1	0.0566 (16)	0.0422 (15)	0.0591 (17)	0.0080 (13)	0.0077 (13)	0.0181 (14)
O2	0.0708 (17)	0.0338 (13)	0.0464 (14)	0.0131 (12)	0.0090 (13)	0.0108 (11)
O3	0.0584 (15)	0.0340 (12)	0.0602 (17)	0.0202 (12)	0.0044 (13)	0.0171 (12)
O4	0.0610 (16)	0.0464 (14)	0.0462 (14)	0.0291 (13)	0.0099 (12)	0.0166 (12)

Geometric parameters (Å, º)

C1—N1	1.320 (4)	C9—C10	1.364 (4)
C1—C2	1.395 (5)	C9—H9	0.9300
C1—H1	0.9300	C10—C11	1.397 (5)
C2—C3	1.357 (5)	C10—H10	0.9300
C2—H2	0.9300	C11—N5	1.325 (4)
C3—N2	1.364 (4)	C11—H11	0.9300
C3—H3	0.9300	Cd1—O2	2.270 (3)
C4—N3	1.330 (4)	Cd1—N5	2.345 (2)
C4—C8	1.379 (4)	Cd1—O4	2.365 (3)
C4—N2	1.413 (4)	Cd1—N3	2.434 (2)
C5—N3	1.328 (4)	Cd1—N1	2.450 (3)
C5—C6	1.376 (4)	Cd1—O3	2.464 (2)
C5—N4	1.409 (4)	Cd1—O1	2.592 (3)
C6—C7	1.385 (4)	N1—N2	1.357 (4)
C6—H6	0.9300	N4—N5	1.361 (3)
C7—C8	1.371 (5)	N6—O1	1.220 (4)
C7—H7	0.9300	N6—O2	1.277 (4)
C8—H8	0.9300	N7—O3	1.240 (4)
C9—N4	1.361 (4)	N7—O4	1.264 (4)
N1—C1—C2	111.8 (3)	O4—Cd1—N3	93.58 (9)
N1—C1—H1	124.1	O2—Cd1—N1	94.34 (10)
C2—C1—H1	124.1	N5—Cd1—N1	132.58 (9)
C3—C2—C1	105.3 (3)	O4—Cd1—N1	86.53 (9)
C3—C2—H2	127.3	N3—Cd1—N1	65.49 (8)
C1—C2—H2	127.3	O2—Cd1—O3	83.63 (9)
C2—C3—N2	106.8 (3)	N5—Cd1—O3	139.36 (9)
C2—C3—H3	126.6	O4—Cd1—O3	51.23 (9)
N2—C3—H3	126.6	N3—Cd1—O3	130.34 (8)
N3—C4—C8	124.0 (3)	N1—Cd1—O3	77.14 (8)
N3—C4—N2	113.9 (3)	O2—Cd1—O1	50.02 (9)
C8—C4—N2	122.2 (3)	N5—Cd1—O1	87.76 (9)
N3—C5—C6	123.6 (3)	O4—Cd1—O1	169.63 (9)
N3—C5—N4	114.4 (2)	N3—Cd1—O1	80.15 (8)
C6—C5—N4	122.0 (3)	N1—Cd1—O1	83.36 (9)
C5—C6—C7	117.0 (3)	O3—Cd1—O1	127.86 (9)
C5—C6—H6	121.5	C1—N1—N2	104.8 (3)

C7—C6—H6	121.5	C1—N1—Cd1	137.4 (2)
C8—C7—C6	121.0 (3)	N2—N1—Cd1	117.31 (18)
C8—C7—H7	119.5	N1—N2—C3	111.2 (3)
C6—C7—H7	119.5	N1—N2—C4	120.0 (2)
C7—C8—C4	116.8 (3)	C3—N2—C4	128.7 (3)
C7—C8—H8	121.6	C5—N3—C4	117.6 (3)
C4—C8—H8	121.6	C5—N3—Cd1	119.45 (19)
N4—C9—C10	107.1 (3)	C4—N3—Cd1	122.21 (19)
N4—C9—H9	126.4	C9—N4—N5	111.0 (2)
C10—C9—H9	126.4	C9—N4—C5	128.9 (3)
C9—C10—C11	105.3 (3)	N5—N4—C5	120.0 (2)
C9—C10—H10	127.4	C11—N5—N4	105.1 (2)
C11—C10—H10	127.4	C11—N5—Cd1	136.3 (2)
N5—C11—C10	111.5 (3)	N4—N5—Cd1	118.55 (17)
N5—C11—H11	124.2	O1—N6—O2	112.5 (3)
C10—C11—H11	124.2	O3—N7—O4	113.2 (3)
O2—Cd1—N5	114.66 (9)	N6—O1—Cd1	91.5 (2)
O2—Cd1—O4	133.53 (9)	N6—O2—Cd1	105.9 (2)
N5—Cd1—O4	97.45 (9)	N7—O3—Cd1	95.73 (19)
O2—Cd1—N3	128.85 (8)	N7—O4—Cd1	99.9 (2)
N5—Cd1—N3	67.11 (8)		

N1—C1—C2—C3	−0.3 (4)	O1—Cd1—N3—C4	83.5 (2)
C1—C2—C3—N2	0.2 (4)	C10—C9—N4—N5	−0.4 (3)
N3—C5—C6—C7	−1.7 (5)	C10—C9—N4—C5	−176.8 (3)
N4—C5—C6—C7	178.2 (3)	N3—C5—N4—C9	−176.9 (3)
C5—C6—C7—C8	0.3 (5)	C6—C5—N4—C9	3.2 (5)
C6—C7—C8—C4	1.3 (5)	N3—C5—N4—N5	7.0 (4)
N3—C4—C8—C7	−1.8 (5)	C6—C5—N4—N5	−172.9 (3)
N2—C4—C8—C7	179.1 (3)	C10—C11—N5—N4	0.2 (4)
N4—C9—C10—C11	0.5 (4)	C10—C11—N5—Cd1	179.2 (2)
C9—C10—C11—N5	−0.5 (4)	C9—N4—N5—C11	0.1 (3)
C2—C1—N1—N2	0.3 (4)	C5—N4—N5—C11	176.8 (3)
C2—C1—N1—Cd1	171.8 (2)	C9—N4—N5—Cd1	−179.10 (18)
O2—Cd1—N1—C1	55.0 (4)	C5—N4—N5—Cd1	−2.4 (3)
N5—Cd1—N1—C1	−175.4 (3)	O2—Cd1—N5—C11	−56.5 (3)
O4—Cd1—N1—C1	−78.5 (3)	O4—Cd1—N5—C11	89.0 (3)
N3—Cd1—N1—C1	−174.0 (4)	N3—Cd1—N5—C11	179.8 (3)
O3—Cd1—N1—C1	−27.5 (3)	N1—Cd1—N5—C11	−178.9 (3)
O1—Cd1—N1—C1	103.8 (3)	O3—Cd1—N5—C11	53.8 (4)
O2—Cd1—N1—N2	−134.3 (2)	O1—Cd1—N5—C11	−100.0 (3)
N5—Cd1—N1—N2	−4.7 (3)	O2—Cd1—N5—N4	122.4 (2)
O4—Cd1—N1—N2	92.3 (2)	O4—Cd1—N5—N4	−92.1 (2)
N3—Cd1—N1—N2	−3.3 (2)	N3—Cd1—N5—N4	−1.34 (19)
O3—Cd1—N1—N2	143.2 (2)	N1—Cd1—N5—N4	0.1 (3)
O1—Cd1—N1—N2	−85.4 (2)	O3—Cd1—N5—N4	−127.3 (2)
C1—N1—N2—C3	−0.1 (4)	O1—Cd1—N5—N4	78.9 (2)
Cd1—N1—N2—C3	−173.6 (2)	O2—N6—O1—Cd1	−3.4 (3)

C1—N1—N2—C4	−176.7 (3)	O2—Cd1—O1—N6	2.3 (2)
Cd1—N1—N2—C4	9.8 (3)	N5—Cd1—O1—N6	127.6 (2)
C2—C3—N2—N1	−0.1 (4)	O4—Cd1—O1—N6	−112.0 (5)
C2—C3—N2—C4	176.1 (3)	N3—Cd1—O1—N6	−165.3 (2)
N3—C4—N2—N1	−12.6 (4)	N1—Cd1—O1—N6	−99.1 (2)
C8—C4—N2—N1	166.5 (3)	O3—Cd1—O1—N6	−31.1 (3)
N3—C4—N2—C3	171.5 (3)	O1—N6—O2—Cd1	4.0 (3)
C8—C4—N2—C3	−9.4 (5)	N5—Cd1—O2—N6	−66.1 (2)
C6—C5—N3—C4	1.3 (5)	O4—Cd1—O2—N6	164.6 (2)
N4—C5—N3—C4	−178.6 (2)	N3—Cd1—O2—N6	13.5 (3)
C6—C5—N3—Cd1	171.7 (3)	N1—Cd1—O2—N6	75.3 (2)
N4—C5—N3—Cd1	−8.2 (3)	O3—Cd1—O2—N6	151.8 (2)
C8—C4—N3—C5	0.6 (4)	O1—Cd1—O2—N6	−2.3 (2)
N2—C4—N3—C5	179.7 (3)	O4—N7—O3—Cd1	−1.1 (3)
C8—C4—N3—Cd1	−169.6 (2)	O2—Cd1—O3—N7	168.8 (2)
N2—C4—N3—Cd1	9.5 (3)	N5—Cd1—O3—N7	47.8 (3)
O2—Cd1—N3—C5	−98.7 (2)	O4—Cd1—O3—N7	0.71 (19)
N5—Cd1—N3—C5	5.3 (2)	N3—Cd1—O3—N7	−54.0 (2)
O4—Cd1—N3—C5	101.9 (2)	N1—Cd1—O3—N7	−95.2 (2)
N1—Cd1—N3—C5	−173.6 (2)	O1—Cd1—O3—N7	−166.11 (18)
O3—Cd1—N3—C5	141.5 (2)	O3—N7—O4—Cd1	1.2 (3)
O1—Cd1—N3—C5	−86.4 (2)	O2—Cd1—O4—N7	−17.1 (3)
O2—Cd1—N3—C4	71.3 (2)	N5—Cd1—O4—N7	−151.9 (2)
N5—Cd1—N3—C4	175.2 (2)	N3—Cd1—O4—N7	140.7 (2)
O4—Cd1—N3—C4	−88.2 (2)	N1—Cd1—O4—N7	75.6 (2)
N1—Cd1—N3—C4	−3.6 (2)	O3—Cd1—O4—N7	−0.70 (19)
O3—Cd1—N3—C4	−48.5 (3)	O1—Cd1—O4—N7	88.4 (5)