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Dichlorido(2,9-dipropoxy-1,10-phenanthroline-κ^2N,N')cadmium(II)

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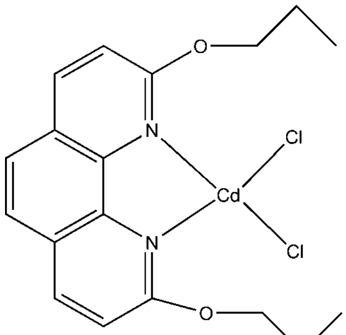
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.009 Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 16.3.

In the title complex, [$CdCl_2(C_{18}H_{20}N_2O_2)$], the Cd^{II} ion is coordinated by two N atoms from a bis-chelating 2,9-dipropoxy-1,10-phenanthroline ligand and two Cl atoms in a distorted tetrahedral environment. The two Cd—Cl bond distances are significantly different from each other and the N—Cd—N bond angle is acute. In the crystal structure, there are π–π stacking interactions between symmetry-related phenanthroline ring systems, with a centroid–centroid distance of 3.585 (3) Å.

Related literature

For details of the coordination chemistry of 1,10-phenanthroline derivatives, see: Arpi *et al.* (2006); Bie *et al.* (2006). For synthetic details, see: Pijper *et al.* (1984).



Experimental

Crystal data

[$CdCl_2(C_{18}H_{20}N_2O_2)$]
M_r = 479.66
Tetragonal, $I4_1/a$
a = 31.3159 (10) Å
c = 8.1662 (5) Å
V = 8008.5 (6) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 1.37 mm^{-1}
T = 291 K
0.18 × 0.07 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.791, T_{max} = 0.947
21000 measured reflections
3722 independent reflections
2656 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.02
3722 reflections
228 parameters
21 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—N2	2.285 (4)	Cd1—Cl2	2.3623 (14)
Cd1—N1	2.297 (4)	Cd1—Cl1	2.4182 (15)
N2—Cd1—N1	72.66 (13)	N2—Cd1—Cl1	103.88 (10)
N2—Cd1—Cl2	121.73 (11)	N1—Cd1—Cl1	109.63 (9)
N1—Cd1—Cl2	119.18 (10)	Cl2—Cd1—Cl1	120.03 (5)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to Mrs Li (Wuhan University) for her assistance with the X-ray crystallographic analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2892).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, m1245 [doi:10.1107/S1600536809038124]

Dichlorido(2,9-dipropoxy-1,10-phenanthroline-κ^2N,N')cadmium(II)

Cao-Yuan Niu, Xian-Fu Zheng and Yu-Li Dang

S1. Comment

The compound 1,10-phenanthroline and its derivatives have been used with d^{10} metals to synthesize some luminescent materials (Arpi, *et al.*, 2006; Bie, *et al.*, 2006). The compound 2,9-diethoxy-1,10-phenanthroline has been shown to possesses antimycoplasmal activity in the presence of copper (Pijper, *et al.*, 1984). Herein, we present the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig.1. The Cd^{II} ion is four-coordinated to two nitrogen atoms from one 1,10-phenanthroline ring (N1, N2) and two chlorine atoms (Cl1, Cl2), defining a distorted tetrahedral coordination geometry. The two Cd—Cl bond distances are significantly different from each other. The the N—Zn—N bond angle is acute. In the crystal structure, there are *π*–*π* stacking interactions between phenanthroline ring systems with centroid to centroid distances of 3.5847 (1) Å (Fig. 2).

S2. Experimental

2,9-Dipropoxy-1,10-phenanthroline was prepared according to the literature procedure (Pijper, *et al.*, 1984). Slow evaporation of a mixture of the ligand (0.027 g, 0.1 mmol) and cadmium dichloride (0.016 g, 0.1 mmol) in 30 ml methanol afforded suitable colourless block crystals in about 7 days (yield about 45%).

S3. Refinement

C-bound H atoms were placed in calculated positions and refined using a riding model [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms and C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms]. The final difference Fourier map had a highest peak 0.94 Å from atom C17 and a deepest hole at 0.85 Å from atom H18, but were otherwise featureless.

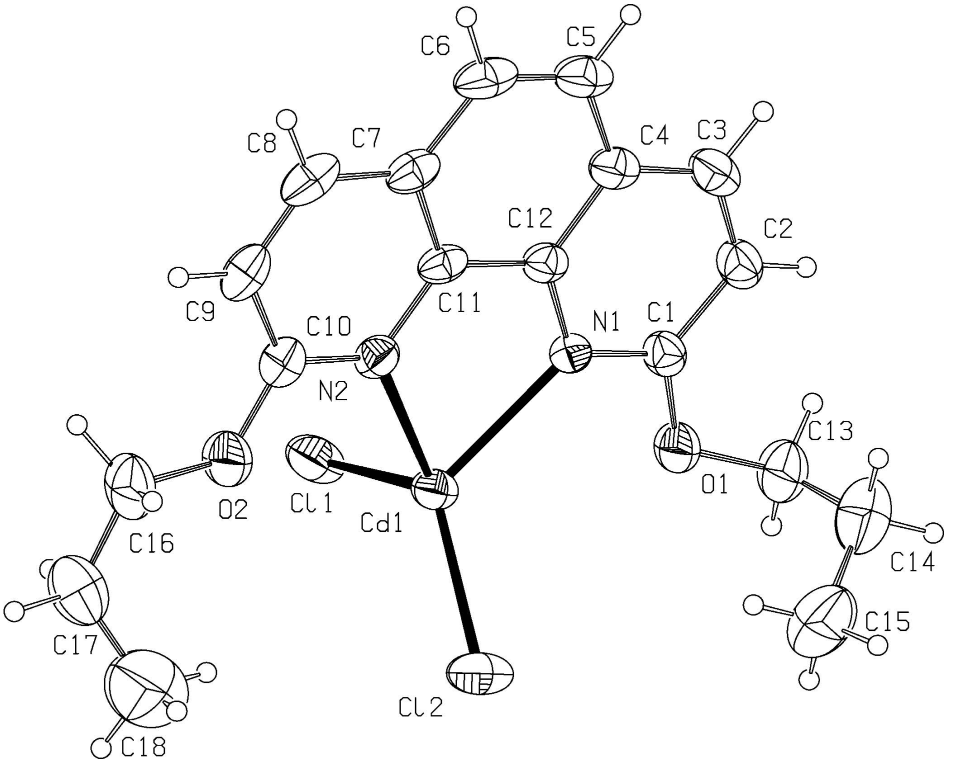

Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

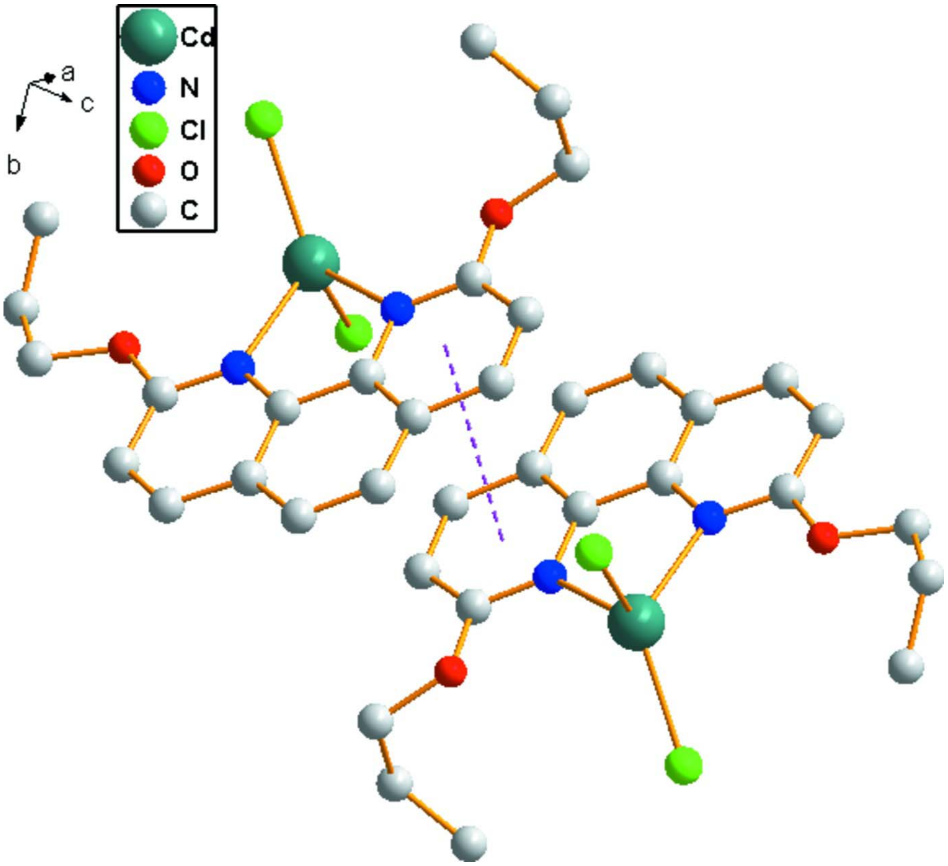

Figure 2

Packing diagram showing the π-π interaction (purple dotted line). All H atoms have been omitted for clarity.

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Crystal data

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Tetragonal, $I4_1/a$
Hall symbol: -I 4ad
$a = 31.3159$ (10) Å
$c = 8.1662$ (5) Å
$V = 8008.5$ (6) Å^3
$Z = 16$
$F(000) = 3840$
$D_x = 1.591$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3326 reflections
$\theta = 2.6$–$25.5°$
$\mu = 1.37$ mm^{-1}
$T = 291$ K
Needle, colourless
0.18 × 0.07 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.791$, $T_{max} = 0.947$
21000 measured reflections
3722 independent reflections
2656 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -36 \rightarrow 37$
$k = -37 \rightarrow 29$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.101$
$S = 1.02$
3722 reflections
228 parameters
21 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
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 20.3768P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.80$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ 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 (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
Cd1	0.329113 (11)	0.831007 (11)	0.02856 (5)	0.05269 (15)
N1	0.26377 (11)	0.80406 (11)	−0.0466 (4)	0.0434 (9)
N2	0.32414 (12)	0.76370 (11)	0.1350 (5)	0.0498 (9)
Cl1	0.37894 (5)	0.82330 (5)	−0.19572 (19)	0.0758 (4)
Cl2	0.33196 (5)	0.89205 (5)	0.19815 (19)	0.0805 (5)
O1	0.24631 (11)	0.86473 (11)	−0.1694 (4)	0.0632 (9)
O2	0.38663 (13)	0.77153 (12)	0.2600 (5)	0.0803 (12)
C1	0.23473 (14)	0.82433 (15)	−0.1329 (6)	0.0497 (11)
C2	0.19693 (16)	0.80477 (18)	−0.1842 (6)	0.0606 (13)
H2	0.1768	0.8200	−0.2440	0.073*
C3	0.19006 (16)	0.76326 (19)	−0.1456 (6)	0.0640 (14)
H3	0.1653	0.7498	−0.1815	0.077*
C4	0.22027 (16)	0.73999 (15)	−0.0509 (6)	0.0534 (12)
C5	0.21500 (19)	0.69696 (18)	−0.0030 (7)	0.0678 (16)
H5	0.1904	0.6823	−0.0328	0.081*
C6	0.2456 (2)	0.67679 (17)	0.0860 (8)	0.0715 (16)
H6	0.2417	0.6484	0.1154	0.086*
C7	0.28325 (17)	0.69825 (15)	0.1348 (6)	0.0564 (13)
C8	0.3164 (2)	0.67952 (17)	0.2272 (7)	0.0697 (16)
H8	0.3142	0.6510	0.2572	0.084*
C9	0.3512 (2)	0.70209 (17)	0.2734 (7)	0.0659 (15)
H9	0.3727	0.6895	0.3355	0.079*
C10	0.35403 (17)	0.74526 (16)	0.2245 (6)	0.0582 (13)
C11	0.28890 (15)	0.74128 (14)	0.0899 (6)	0.0474 (11)
C12	0.25695 (14)	0.76273 (14)	−0.0050 (5)	0.0464 (11)

C13	0.2158 (2)	0.8939 (2)	−0.2393 (8)	0.0847 (19)
H13A	0.1992	0.8794	−0.3228	0.102*
H13B	0.2307	0.9176	−0.2903	0.102*
C14	0.1869 (3)	0.9102 (3)	−0.1088 (12)	0.118 (3)
H14A	0.1706	0.8865	−0.0652	0.141*
H14B	0.1670	0.9302	−0.1572	0.141*
C15	0.2103 (3)	0.9323 (3)	0.0324 (12)	0.133 (3)
H15A	0.2297	0.9125	0.0826	0.199*
H15B	0.1899	0.9418	0.1121	0.199*
H15C	0.2259	0.9564	−0.0089	0.199*
C16	0.41947 (19)	0.7591 (2)	0.3744 (9)	0.094 (2)
H16A	0.4306	0.7311	0.3465	0.112*
H16B	0.4081	0.7580	0.4848	0.112*
C17	0.4545 (3)	0.7927 (3)	0.3621 (14)	0.155 (4)
H17A	0.4765	0.7853	0.4407	0.187*
H17B	0.4671	0.7903	0.2541	0.187*
C18	0.4437 (3)	0.8368 (3)	0.3878 (16)	0.182 (4)
H18A	0.4241	0.8459	0.3045	0.272*
H18B	0.4691	0.8538	0.3825	0.272*
H18C	0.4307	0.8400	0.4935	0.272*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0529 (2)	0.0449 (2)	0.0603 (3)	−0.00643 (15)	−0.00647 (17)	−0.00164 (17)
N1	0.044 (2)	0.041 (2)	0.045 (2)	0.0000 (16)	−0.0002 (17)	−0.0051 (17)
N2	0.056 (2)	0.042 (2)	0.052 (2)	0.0049 (18)	−0.0024 (19)	−0.0005 (18)
Cl1	0.0642 (8)	0.0880 (10)	0.0751 (10)	−0.0247 (7)	0.0081 (7)	−0.0212 (8)
Cl2	0.0959 (11)	0.0639 (9)	0.0818 (10)	−0.0271 (8)	0.0134 (8)	−0.0216 (8)
O1	0.059 (2)	0.060 (2)	0.071 (2)	0.0051 (17)	−0.0177 (18)	0.0077 (18)
O2	0.073 (3)	0.070 (3)	0.098 (3)	0.009 (2)	−0.033 (2)	0.009 (2)
C1	0.046 (3)	0.058 (3)	0.045 (3)	0.001 (2)	−0.003 (2)	−0.005 (2)
C2	0.051 (3)	0.074 (4)	0.057 (3)	0.001 (3)	−0.009 (2)	−0.010 (3)
C3	0.050 (3)	0.084 (4)	0.058 (3)	−0.015 (3)	0.005 (3)	−0.022 (3)
C4	0.056 (3)	0.055 (3)	0.049 (3)	−0.010 (2)	0.013 (2)	−0.011 (2)
C5	0.071 (4)	0.060 (3)	0.073 (4)	−0.022 (3)	0.023 (3)	−0.018 (3)
C6	0.094 (4)	0.043 (3)	0.078 (4)	−0.018 (3)	0.033 (4)	−0.011 (3)
C7	0.075 (3)	0.041 (3)	0.053 (3)	0.000 (2)	0.021 (3)	−0.004 (2)
C8	0.100 (5)	0.042 (3)	0.067 (4)	0.012 (3)	0.029 (3)	0.005 (3)
C9	0.082 (4)	0.056 (3)	0.060 (3)	0.024 (3)	0.008 (3)	0.010 (3)
C10	0.061 (3)	0.055 (3)	0.058 (3)	0.010 (2)	0.001 (3)	0.002 (3)
C11	0.061 (3)	0.037 (2)	0.044 (3)	−0.004 (2)	0.015 (2)	−0.006 (2)
C12	0.049 (3)	0.046 (3)	0.045 (3)	−0.005 (2)	0.009 (2)	−0.008 (2)
C13	0.080 (4)	0.080 (4)	0.094 (5)	0.018 (3)	−0.021 (4)	0.017 (4)
C14	0.101 (6)	0.116 (6)	0.136 (7)	0.036 (5)	0.000 (6)	0.013 (6)
C15	0.142 (8)	0.119 (7)	0.138 (8)	0.032 (6)	0.017 (6)	−0.025 (6)
C16	0.076 (4)	0.101 (5)	0.104 (5)	0.015 (4)	−0.034 (4)	0.017 (4)
C17	0.111 (6)	0.151 (6)	0.204 (8)	0.025 (5)	−0.095 (6)	−0.038 (6)

C18	0.170 (8)	0.168 (7)	0.207 (8)	−0.013 (6)	−0.038 (7)	−0.011 (7)

Geometric parameters (Å, º)

Cd1—N2	2.285 (4)	C7—C8	1.411 (8)
Cd1—N1	2.297 (4)	C8—C9	1.353 (8)
Cd1—Cl2	2.3623 (14)	C8—H8	0.9300
Cd1—Cl1	2.4182 (15)	C9—C10	1.412 (7)
N1—C1	1.314 (5)	C9—H9	0.9300
N1—C12	1.355 (5)	C11—C12	1.433 (6)
N2—C10	1.321 (6)	C13—C14	1.488 (10)
N2—C11	1.359 (6)	C13—H13A	0.9700
O1—C1	1.349 (6)	C13—H13B	0.9700
O1—C13	1.439 (6)	C14—C15	1.530 (11)
O2—C10	1.343 (6)	C14—H14A	0.9700
O2—C16	1.443 (6)	C14—H14B	0.9700
C1—C2	1.397 (7)	C15—H15A	0.9600
C2—C3	1.355 (7)	C15—H15B	0.9600
C2—H2	0.9300	C15—H15C	0.9600
C3—C4	1.423 (7)	C16—C17	1.524 (8)
C3—H3	0.9300	C16—H16A	0.9700
C4—C12	1.403 (6)	C16—H16B	0.9700
C4—C5	1.413 (7)	C17—C18	1.436 (8)
C5—C6	1.358 (8)	C17—H17A	0.9700
C5—H5	0.9300	C17—H17B	0.9700
C6—C7	1.415 (7)	C18—H18A	0.9600
C6—H6	0.9300	C18—H18B	0.9600
C7—C11	1.408 (6)	C18—H18C	0.9600
N2—Cd1—N1	72.66 (13)	O2—C10—C9	125.0 (5)
N2—Cd1—Cl2	121.73 (11)	N2—C11—C7	121.7 (5)
N1—Cd1—Cl2	119.18 (10)	N2—C11—C12	118.1 (4)
N2—Cd1—Cl1	103.88 (10)	C7—C11—C12	120.1 (4)
N1—Cd1—Cl1	109.63 (9)	N1—C12—C4	123.1 (4)
Cl2—Cd1—Cl1	120.03 (5)	N1—C12—C11	118.3 (4)
C1—N1—C12	119.1 (4)	C4—C12—C11	118.6 (4)
C1—N1—Cd1	125.6 (3)	O1—C13—C14	109.7 (5)
C12—N1—Cd1	115.1 (3)	O1—C13—H13A	109.7
C10—N2—C11	120.0 (4)	C14—C13—H13A	109.7
C10—N2—Cd1	124.4 (3)	O1—C13—H13B	109.7
C11—N2—Cd1	115.4 (3)	C14—C13—H13B	109.7
C1—O1—C13	120.3 (4)	H13A—C13—H13B	108.2
C10—O2—C16	121.0 (4)	C13—C14—C15	113.8 (7)
N1—C1—O1	112.7 (4)	C13—C14—H14A	108.8
N1—C1—C2	122.4 (5)	C15—C14—H14A	108.8
O1—C1—C2	124.9 (5)	C13—C14—H14B	108.8
C3—C2—C1	119.0 (5)	C15—C14—H14B	108.8
C3—C2—H2	120.5	H14A—C14—H14B	107.7

C1—C2—H2	120.5	C14—C15—H15A	109.5
C2—C3—C4	120.8 (5)	C14—C15—H15B	109.5
C2—C3—H3	119.6	H15A—C15—H15B	109.5
C4—C3—H3	119.6	C14—C15—H15C	109.5
C12—C4—C5	120.4 (5)	H15A—C15—H15C	109.5
C12—C4—C3	115.5 (4)	H15B—C15—H15C	109.5
C5—C4—C3	124.1 (5)	O2—C16—C17	106.4 (5)
C6—C5—C4	120.6 (5)	O2—C16—H16A	110.4
C6—C5—H5	119.7	C17—C16—H16A	110.4
C4—C5—H5	119.7	O2—C16—H16B	110.4
C5—C6—C7	121.2 (5)	C17—C16—H16B	110.4
C5—C6—H6	119.4	H16A—C16—H16B	108.6
C7—C6—H6	119.4	C18—C17—C16	119.1 (8)
C11—C7—C8	116.4 (5)	C18—C17—H17A	107.5
C11—C7—C6	119.1 (5)	C16—C17—H17A	107.5
C8—C7—C6	124.5 (5)	C18—C17—H17B	107.5
C9—C8—C7	121.6 (5)	C16—C17—H17B	107.5
C9—C8—H8	119.2	H17A—C17—H17B	107.0
C7—C8—H8	119.2	C17—C18—H18A	109.5
C8—C9—C10	118.2 (5)	C17—C18—H18B	109.5
C8—C9—H9	120.9	H18A—C18—H18B	109.5
C10—C9—H9	120.9	C17—C18—H18C	109.5
N2—C10—O2	113.0 (4)	H18A—C18—H18C	109.5
N2—C10—C9	122.0 (5)	H18B—C18—H18C	109.5

N2—Cd1—N1—C1	179.1 (4)	Cd1—N2—C10—O2	5.6 (6)
Cl2—Cd1—N1—C1	61.9 (4)	C11—N2—C10—C9	1.4 (7)
Cl1—Cd1—N1—C1	−82.0 (4)	Cd1—N2—C10—C9	−173.5 (4)
N2—Cd1—N1—C12	−5.3 (3)	C16—O2—C10—N2	171.6 (5)
Cl2—Cd1—N1—C12	−122.5 (3)	C16—O2—C10—C9	−9.3 (8)
Cl1—Cd1—N1—C12	93.6 (3)	C8—C9—C10—N2	−0.6 (8)
N1—Cd1—N2—C10	−179.2 (4)	C8—C9—C10—O2	−179.6 (5)
Cl2—Cd1—N2—C10	−65.1 (4)	C10—N2—C11—C7	−0.6 (7)
Cl1—Cd1—N2—C10	74.2 (4)	Cd1—N2—C11—C7	174.7 (3)
N1—Cd1—N2—C11	5.8 (3)	C10—N2—C11—C12	179.0 (4)
Cl2—Cd1—N2—C11	119.8 (3)	Cd1—N2—C11—C12	−5.7 (5)
Cl1—Cd1—N2—C11	−100.8 (3)	C8—C7—C11—N2	−0.8 (7)
C12—N1—C1—O1	−178.1 (4)	C6—C7—C11—N2	179.1 (4)
Cd1—N1—C1—O1	−2.7 (6)	C8—C7—C11—C12	179.6 (4)
C12—N1—C1—C2	−0.2 (7)	C6—C7—C11—C12	−0.5 (7)
Cd1—N1—C1—C2	175.2 (3)	C1—N1—C12—C4	0.6 (7)
C13—O1—C1—N1	−169.7 (5)	Cd1—N1—C12—C4	−175.3 (3)
C13—O1—C1—C2	12.5 (7)	C1—N1—C12—C11	−179.7 (4)
N1—C1—C2—C3	−0.8 (8)	Cd1—N1—C12—C11	4.5 (5)
O1—C1—C2—C3	176.8 (5)	C5—C4—C12—N1	−179.8 (4)
C1—C2—C3—C4	1.5 (8)	C3—C4—C12—N1	0.1 (7)
C2—C3—C4—C12	−1.1 (7)	C5—C4—C12—C11	0.5 (7)
C2—C3—C4—C5	178.7 (5)	C3—C4—C12—C11	−179.7 (4)

C12—C4—C5—C6	−1.0 (7)	N2—C11—C12—N1	0.8 (6)
C3—C4—C5—C6	179.2 (5)	C7—C11—C12—N1	−179.6 (4)
C4—C5—C6—C7	0.7 (8)	N2—C11—C12—C4	−179.4 (4)
C5—C6—C7—C11	0.0 (8)	C7—C11—C12—C4	0.2 (6)
C5—C6—C7—C8	180.0 (5)	C1—O1—C13—C14	77.9 (7)
C11—C7—C8—C9	1.5 (7)	O1—C13—C14—C15	57.3 (9)
C6—C7—C8—C9	−178.4 (5)	C10—O2—C16—C17	170.5 (6)
C7—C8—C9—C10	−0.8 (8)	O2—C16—C17—C18	55.8 (12)
C11—N2—C10—O2	−179.6 (4)		