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Dichlorido{2,2′-[oxydi(methylene)]dipyridine-κ^3N,N,N'}cadmium(II)

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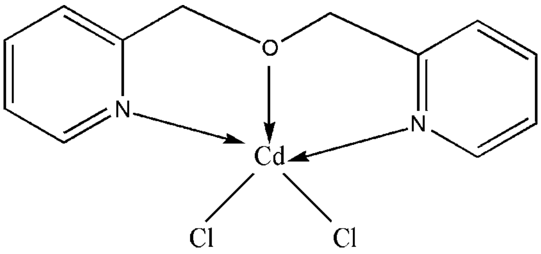
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.028; wR factor = 0.074; data-to-parameter ratio = 15.3.

In the title complex, $[CdCl_2(C_{12}H_{12}N_2O)]$, the Cd^{II} ion is coordinated by two Cl atoms [Cd—Cl = 2.4402 (9) and 2.4422 (8) Å], and by one O [Cd—O = 2.396 (2) Å] and two N atoms [Cd—N = 2.303 (2) and 2.305 (2) Å] from a tridentate 2,2′-[oxydi(methylene)]dipyridine ligand in a distorted trigonal–bipyramidal geometry. In the ligand, the two pyridyl rings make a dihedral angle of 16.85 (15)°. Weak intermolecular C—H⋯Cl hydrogen bonds link the molecules into two-dimensional layers parallel to the plane formed by the **a**+**c** and **b** vectors.

Related literature

For crystal structures of related five-coordinated Cd^{II} complexes, see: Sawitzki & von Schnering (1974); Reger *et al.* (2002); Zhang & Shi (2007).



Experimental

Crystal data

$[CdCl_2(C_{12}H_{12}N_2O)]$
M_r = 383.54
Monoclinic, $P2_1/n$
a = 8.1397 (15) Å
b = 12.597 (2) Å
c = 13.823 (3) Å
β = 90.00 (1)°
V = 1417.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.91 mm^{-1}
T = 298 (2) K
0.35 × 0.30 × 0.26 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.555, T_{max} = 0.637
6908 measured reflections
2504 independent reflections
2259 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.04
2504 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2B⋯Cl2^{i}	0.97	2.77	3.735 (3)	175
C6—H6⋯Cl1^{ii}	0.93	2.82	3.701 (4)	158

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2283).

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supplementary materials

Acta Cryst. (2007). E**63**, m2241 [**doi:10.1107/S1600536807036574**]

Dichlorido{2,2'-[oxydi(methylene)]dipyridine-κ^3N,N,N'}cadmium(II)

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Comment

Complexes containing Cd(II) are very common and useful, and play a pivotal role in the area of optics. In most known structures, Cd^{II} ions assume octahedral coordination geometry, whereas five-coordinated Cd(II) complexes are very limited (Sawitzki *et al.* 1974; Reger *et al.*, 2002; Zhang *et al.* 2007). Herewith we report the crystal structure of of the title compound, (I)− a new five-coordinated Cd(II) complex.

In (I) (Fig. 1), Cd1 has a distorted trigonal bipyramidal CdN_2OCl_2 coordination geometry. In a tridentate 2,2'-[oxydi(methylene)]dipyridine ligand, two pyridyl rings make a dihedral angle of 16.85 (15)°. In two neighbouring complexes related by inversion centre, there is a weak π-π interaction proved by short distance C3···C9^{i} of 3.453 (5) Å [symmetry code: −*x*, −*y*, −*z*]. The weak intermolecular C—H···Cl hydrogen bonds (Table 1, Fig. 2) link the molecules into two-dimensional layers parallel to the plane formed by a+c and b vectors.

Experimental

5 ml me thanol solution of 2,2'-[oxydi(methylene)]dipyridine (0.0327 g, 0.163 mmol) was added into 8 ml H_2O solution containing $CdCl_2·2.5H_2O$ (0.0402 g, 0.176 mmol), and the mixture was stirred for a few minutes. The red single crystals were obtained after the solution had been allowed to stand at room temperature for two weeks.

Refinement

All H atom were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

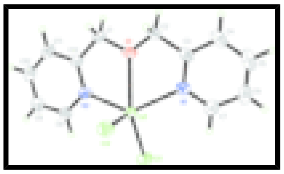

Fig. 1. View of (I), showing the atom numbering scheme and displacement ellipsoids drawn at the 30% probability level

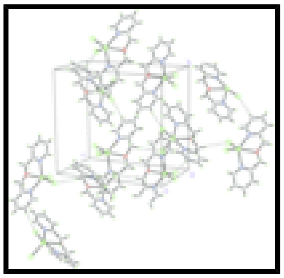

Fig. 2. A portion of the crystal packing of (I) showing the intermolecular hydrogen bonds as dashed lines.

Dichlorido{2,2′-[oxydi(methylene)]dipyridine-κ^3*N*,*N*,*N*′}cadmium(II)

Crystal data

$[CdCl_2(C_{12}H_{12}N_2O)]$

$M_r = 383.54$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.1397$ (15) Å

$b = 12.597$ (2) Å

$c = 13.823$ (3) Å

$\alpha = 90$°

$\beta = 90.00$ (1)°

$\gamma = 90$°

$V = 1417.3$ (4) Å^3

$Z = 4$

$F_{000} = 752$

$D_x = 1.797$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 5221 reflections

$\theta = 2.2$–28.2°

$\mu = 1.91$ mm^{-1}

$T = 298$ (2) K

Block, red

0.35 × 0.30 × 0.26 mm

Data collection

Bruker SMART APEX CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.555$, $T_{max} = 0.637$

6908 measured reflections

2504 independent reflections

2259 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$

$\theta_{max} = 25.0$°

$\theta_{min} = 2.2$°

$h = -8 \rightarrow 9$

$k = -12 \rightarrow 14$

$l = -13 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.074$

$S = 1.04$

2504 reflections

164 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.0413P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.51$ e Å^{-3}

$\Delta\rho_{min} = -0.50$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0071 (6)

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}
Cd1	0.11080 (2)	0.310375 (16)	0.121183 (14)	0.04107 (13)
O1	0.1024 (3)	0.32605 (17)	−0.05150 (16)	0.0573 (6)
N1	−0.0571 (3)	0.18529 (17)	0.0506 (2)	0.0463 (6)
N2	0.2095 (3)	0.47121 (19)	0.06695 (17)	0.0412 (5)
C3	−0.0493 (3)	0.1670 (2)	−0.0436 (2)	0.0454 (7)
C9	0.3299 (4)	0.6388 (3)	0.1051 (3)	0.0549 (8)
H9	0.3558	0.6898	0.1514	0.066*
C7	−0.1646 (4)	0.1293 (3)	0.1042 (3)	0.0597 (8)
H7	−0.1710	0.1429	0.1702	0.072*
C10	0.3719 (4)	0.6525 (3)	0.0100 (3)	0.0566 (8)
H10	0.4281	0.7132	−0.0092	0.068*
C8	0.2480 (4)	0.5471 (2)	0.1301 (2)	0.0497 (7)
H8	0.2181	0.5377	0.1944	0.060*
C2	0.0691 (4)	0.2318 (3)	−0.1024 (2)	0.0535 (7)
H2A	0.1699	0.1922	−0.1126	0.064*
H2B	0.0217	0.2482	−0.1650	0.064*
C4	−0.1469 (4)	0.0920 (3)	−0.0877 (3)	0.0662 (9)
H4	−0.1410	0.0808	−0.1541	0.079*
C5	−0.2547 (4)	0.0334 (3)	−0.0304 (4)	0.0787 (12)
H5	−0.3196	−0.0193	−0.0581	0.094*
C1	0.2060 (4)	0.3997 (2)	−0.0970 (2)	0.0487 (7)
H1A	0.1509	0.4302	−0.1527	0.058*
H1B	0.3052	0.3647	−0.1191	0.058*
C6	−0.2647 (4)	0.0532 (3)	0.0654 (3)	0.0705 (10)
H6	−0.3381	0.0159	0.1041	0.085*
C11	0.3312 (3)	0.5770 (3)	−0.0566 (3)	0.0530 (8)
H11	0.3581	0.5863	−0.1214	0.064*
Cl2	0.36235 (10)	0.21466 (7)	0.16135 (6)	0.0580 (2)
Cl1	−0.05175 (10)	0.36258 (7)	0.26186 (6)	0.0595 (2)
C12	0.2489 (3)	0.4856 (2)	−0.0264 (2)	0.0419 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.05013 (17)	0.04633 (18)	0.02674 (16)	−0.00318 (8)	0.00138 (10)	0.00116 (8)
O1	0.0820 (15)	0.0613 (13)	0.0286 (12)	−0.0183 (11)	0.0046 (11)	−0.0049 (10)
N1	0.0484 (13)	0.0449 (14)	0.0457 (17)	0.0009 (10)	−0.0018 (12)	−0.0020 (11)
N2	0.0461 (12)	0.0429 (13)	0.0347 (13)	0.0004 (10)	0.0025 (10)	0.0026 (11)
C3	0.0420 (14)	0.0469 (16)	0.0473 (19)	0.0082 (12)	−0.0065 (13)	−0.0114 (14)
C9	0.0514 (16)	0.0481 (18)	0.065 (2)	0.0002 (14)	−0.0015 (16)	−0.0024 (16)
C7	0.0607 (18)	0.060 (2)	0.058 (2)	−0.0040 (16)	0.0061 (16)	0.0084 (17)
C10	0.0455 (16)	0.0470 (17)	0.077 (3)	−0.0017 (13)	0.0035 (16)	0.0100 (18)
C8	0.0565 (16)	0.0504 (17)	0.0423 (17)	−0.0007 (14)	0.0017 (14)	−0.0038 (14)
C2	0.0589 (17)	0.0634 (19)	0.0381 (17)	0.0016 (15)	−0.0001 (14)	−0.0158 (16)
C4	0.0671 (19)	0.061 (2)	0.070 (2)	0.0027 (17)	−0.0153 (18)	−0.0223 (19)
C5	0.062 (2)	0.056 (2)	0.118 (4)	−0.0077 (17)	−0.017 (2)	−0.018 (2)
C1	0.0489 (15)	0.0631 (19)	0.0341 (16)	−0.0001 (13)	0.0057 (13)	0.0041 (14)
C6	0.063 (2)	0.052 (2)	0.097 (3)	−0.0113 (16)	0.001 (2)	0.009 (2)
C11	0.0474 (15)	0.0584 (19)	0.0532 (19)	0.0019 (14)	0.0077 (14)	0.0204 (16)
Cl2	0.0549 (4)	0.0674 (5)	0.0516 (5)	0.0075 (4)	−0.0007 (4)	0.0050 (4)
Cl1	0.0673 (5)	0.0726 (6)	0.0387 (4)	0.0084 (4)	0.0136 (4)	−0.0028 (4)
C12	0.0354 (13)	0.0514 (16)	0.0388 (16)	0.0056 (11)	0.0015 (11)	0.0072 (13)

Geometric parameters (Å, °)

Cd1—N1	2.303 (2)	C7—H7	0.9300
Cd1—N2	2.305 (2)	C10—C11	1.365 (5)
Cd1—O1	2.396 (2)	C10—H10	0.9300
Cd1—Cl2	2.4402 (9)	C8—H8	0.9300
Cd1—Cl1	2.4422 (8)	C2—H2A	0.9700
O1—C1	1.403 (3)	C2—H2B	0.9700
O1—C2	1.407 (4)	C4—C5	1.394 (6)
N1—C3	1.325 (4)	C4—H4	0.9300
N1—C7	1.345 (4)	C5—C6	1.349 (6)
N2—C8	1.332 (4)	C5—H5	0.9300
N2—C12	1.342 (4)	C1—C12	1.498 (4)
C3—C4	1.376 (4)	C1—H1A	0.9700
C3—C2	1.502 (5)	C1—H1B	0.9700
C9—C10	1.370 (5)	C6—H6	0.9300
C9—C8	1.378 (4)	C11—C12	1.396 (4)
C9—H9	0.9300	C11—H11	0.9300
C7—C6	1.368 (5)		
N1—Cd1—N2	132.11 (9)	N2—C8—C9	123.5 (3)
N1—Cd1—O1	67.52 (8)	N2—C8—H8	118.3
N2—Cd1—O1	67.26 (8)	C9—C8—H8	118.3
N1—Cd1—Cl2	104.85 (6)	O1—C2—C3	108.1 (2)
N2—Cd1—Cl2	102.47 (6)	O1—C2—H2A	110.1
O1—Cd1—Cl2	106.94 (6)	C3—C2—H2A	110.1

N1—Cd1—Cl1	101.53 (7)	O1—C2—H2B	110.1
N2—Cd1—Cl1	102.19 (6)	C3—C2—H2B	110.1
O1—Cd1—Cl1	139.08 (6)	H2A—C2—H2B	108.4
Cl2—Cd1—Cl1	113.98 (3)	C3—C4—C5	118.4 (4)
C1—O1—C2	116.7 (2)	C3—C4—H4	120.8
C1—O1—Cd1	118.96 (17)	C5—C4—H4	120.8
C2—O1—Cd1	115.74 (18)	C6—C5—C4	119.8 (3)
C3—N1—C7	118.8 (3)	C6—C5—H5	120.1
C3—N1—Cd1	120.5 (2)	C4—C5—H5	120.1
C7—N1—Cd1	120.8 (2)	O1—C1—C12	109.0 (2)
C8—N2—C12	118.5 (2)	O1—C1—H1A	109.9
C8—N2—Cd1	120.00 (19)	C12—C1—H1A	109.9
C12—N2—Cd1	120.97 (19)	O1—C1—H1B	109.9
N1—C3—C4	121.8 (3)	C12—C1—H1B	109.9
N1—C3—C2	118.0 (3)	H1A—C1—H1B	108.3
C4—C3—C2	120.2 (3)	C5—C6—C7	118.6 (4)
C10—C9—C8	117.8 (3)	C5—C6—H6	120.7
C10—C9—H9	121.1	C7—C6—H6	120.7
C8—C9—H9	121.1	C10—C11—C12	119.4 (3)
N1—C7—C6	122.6 (4)	C10—C11—H11	120.3
N1—C7—H7	118.7	C12—C11—H11	120.3
C6—C7—H7	118.7	N2—C12—C11	120.9 (3)
C11—C10—C9	119.9 (3)	N2—C12—C1	118.3 (2)
C11—C10—H10	120.0	C11—C12—C1	120.8 (3)
C9—C10—H10	120.0		

N1—Cd1—O1—C1	−174.5 (2)	Cd1—N1—C3—C2	−1.2 (3)
N2—Cd1—O1—C1	21.70 (19)	C3—N1—C7—C6	0.9 (5)
Cl2—Cd1—O1—C1	−75.0 (2)	Cd1—N1—C7—C6	−179.0 (3)
Cl1—Cd1—O1—C1	104.0 (2)	C8—C9—C10—C11	0.6 (4)
N1—Cd1—O1—C2	−27.6 (2)	C12—N2—C8—C9	−1.6 (4)
N2—Cd1—O1—C2	168.6 (2)	Cd1—N2—C8—C9	170.0 (2)
Cl2—Cd1—O1—C2	71.9 (2)	C10—C9—C8—N2	0.7 (5)
Cl1—Cd1—O1—C2	−109.1 (2)	C1—O1—C2—C3	−177.3 (2)
N2—Cd1—N1—C3	35.0 (3)	Cd1—O1—C2—C3	35.0 (3)
O1—Cd1—N1—C3	14.7 (2)	N1—C3—C2—O1	−22.5 (4)
Cl2—Cd1—N1—C3	−87.8 (2)	C4—C3—C2—O1	156.9 (3)
Cl1—Cd1—N1—C3	153.3 (2)	N1—C3—C4—C5	−0.8 (5)
N2—Cd1—N1—C7	−145.2 (2)	C2—C3—C4—C5	179.7 (3)
O1—Cd1—N1—C7	−165.4 (2)	C3—C4—C5—C6	1.9 (5)
Cl2—Cd1—N1—C7	92.1 (2)	C2—O1—C1—C12	−170.9 (3)
Cl1—Cd1—N1—C7	−26.8 (2)	Cd1—O1—C1—C12	−24.3 (3)
N1—Cd1—N2—C8	153.2 (2)	C4—C5—C6—C7	−1.6 (6)
O1—Cd1—N2—C8	173.5 (2)	N1—C7—C6—C5	0.2 (5)
Cl2—Cd1—N2—C8	−83.2 (2)	C9—C10—C11—C12	−0.9 (4)
Cl1—Cd1—N2—C8	35.1 (2)	C8—N2—C12—C11	1.2 (4)
N1—Cd1—N2—C12	−35.5 (2)	Cd1—N2—C12—C11	−170.33 (19)
O1—Cd1—N2—C12	−15.17 (18)	C8—N2—C12—C1	179.7 (3)
Cl2—Cd1—N2—C12	88.18 (19)	Cd1—N2—C12—C1	8.2 (3)
Cl1—Cd1—N2—C12	−153.55 (18)	C10—C11—C12—N2	0.0 (4)

C7—N1—C3—C4	−0.6 (4)	C10—C11—C12—C1	−178.5 (3)
Cd1—N1—C3—C4	179.3 (2)	O1—C1—C12—N2	10.7 (3)
C7—N1—C3—C2	178.9 (3)	O1—C1—C12—C11	−170.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2B···Cl2^{i}	0.97	2.77	3.735 (3)	175
C6—H6···Cl1^{ii}	0.93	2.82	3.701 (4)	158

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

Fig. 1

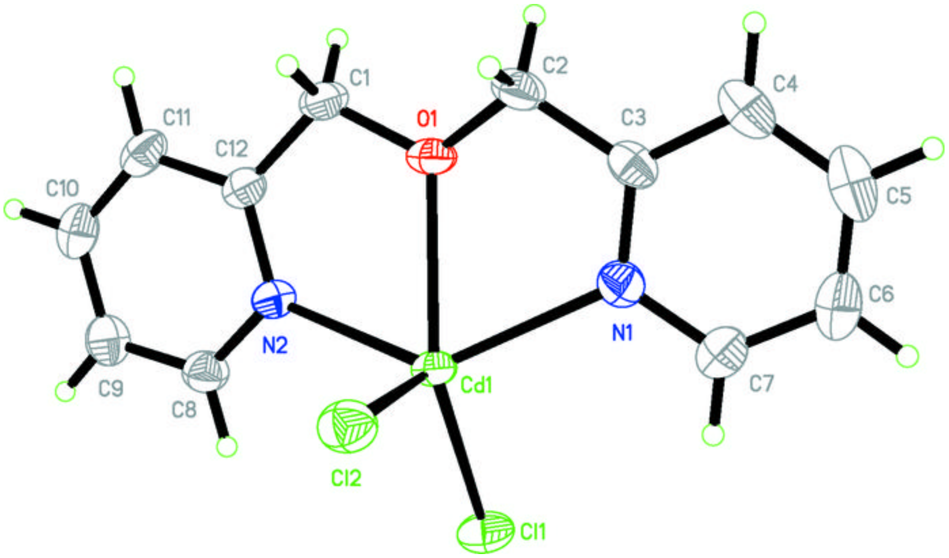

Fig. 2