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Bis(di-2-pyridylmethanediol-κ^2N,N')-palladium(II) bis(perchlorate)

Ya-Min Li,[a]* Xiao-Wei Zhao[a] and Jing-Lai Zhang[b]

[a]Institute of Chemistry and Chemical Engineering, Henan University, Kaifeng, Henan 475001, People's Republic of China, and [b]Institute of Fine Chemistry and Engineering, Henan University, Kaifeng, Henan 475001, People's Republic of China
Correspondence e-mail: liyamin2006@hotmail.com

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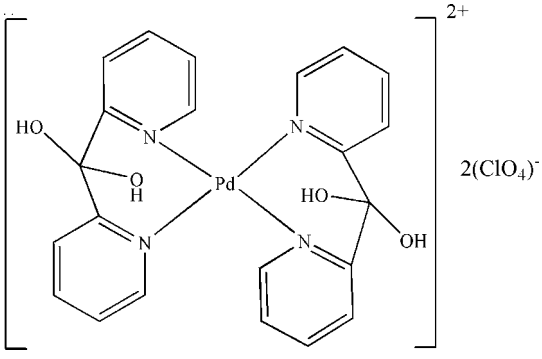
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.046; wR factor = 0.107; data-to-parameter ratio = 15.6.

In the title compound, $[Pd(C_{11}H_{10}N_2O_2)_2](ClO_4)_2$, the Pd^{II} atom, which lies on an inversion centre, adopts square-planar coordination, binding to the two N atoms of each of the two di-2-pyridylmethanediol ligands. In the crystal structure, molecules are linked by a network of O—H···O hydrogen bonds involving the hydroxyl groups and the O atoms of the perchlorate anions to form columns down a.

Related literature

A similar Pd^{II} square-planar complex of di-2-pyridylmethanediol has been reported by Sommerer *et al.* (1997).



Experimental

Crystal data

$[Pd(C_{11}H_{10}N_2O_2)_2](ClO_4)_2$
M_r = 709.72
Monoclinic, $P2_1/n$
a = 7.677 (3) Å
b = 13.929 (5) Å
c = 12.173 (5) Å
β = 99.008 (5)°
V = 1285.7 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.00 mm^{-1}
T = 293 (2) K
0.40 × 0.20 × 0.08 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.858, T_{max} = 1.000
(expected range = 0.792–0.923)
9661 measured reflections
2925 independent reflections
2518 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.107
S = 1.12
2925 reflections
187 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O5	0.82	2.03	2.842 (5)	169
O2—H2B···O6^{i}	0.82	1.84	2.636 (6)	162

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank Henan University for financial support and Professors X.-T. Wu, T.-L. Sheng and J.-J. Zhang for their help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2361).

References

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Sheldrick, G. M. (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sommerer, S. O., Jircitano, A. J., Westcott, B. L., Abboud, K. A. & Krause Bauer, J. A. (1997). *Acta Cryst.* C**53**, 707–710.

supplementary materials

Acta Cryst. (2007). E**63**, m2585 [**doi:10.1107/S1600536807045722**]

Bis(di-2-pyridylmethanediol-κ^2N,N')palladium(II) bis(perchlorate)

Y.-M. Li, X.-W. Zhao and J.-L. Zhang

Comment

The crystal structure of (I) consists of $[Pd(C_{11}H_{10}N_2O_2)_2]^{2+}$ cations and ClO_4^- anions (Fig. 1). In the cation, the metal atom lies on an inversion centre and binds to two *N,N'*-chelated di-2-pyridylmethanediol ligands in a square planar geometry. The Pd—N bond lengths are 2.025 (3) and 2.031 (3) Å. The N1—Pd—N2 angles are 86.97 (12) and 93.03 (12). A similar Pd(II) complex has already been reported by Sommerer *et al.* (1997).

In the crystal structure, there is a complex hydrogen-bonding network among the hydroxyl groups and the O atoms of the perchlorate anions, (Table 1, Fig. 2).

Experimental

The title complex was synthesized by refluxing a mixture of $Pd(OAc)_2$ (0.449 g, 2 mmol) and di-2-pyridylketone (0.185 g, 1 mmol) in ethanol/water (20 ml; 3:1 *v*/*v*) for 1 h with stirring. After cooling, solid $NaClO_4·H_2O$ (0.210 g, 1.5 mmol) was added and the solution filtered. Yellow crystals of (I) suitable for X-ray analysis were obtained as plates by slow evaporation of the filtrate over two weeks.

Refinement

H atoms were positioned geometrically and treated as riding atoms, with C—H distances of 0.93 Å and O—H distances of 0.82 Å, with $U_{iso}(H) = 1.2\ U_{eq}(C)$ and $U_{iso}(H) = 1.5\ U_{eq}(O)$.

Figures

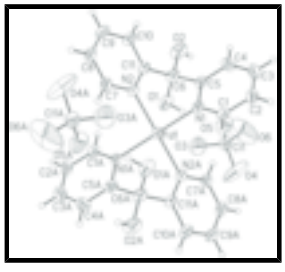


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. Atoms with the A suffix are generated by the symmetry code (−*x*, 1 − *y*, −*z*).

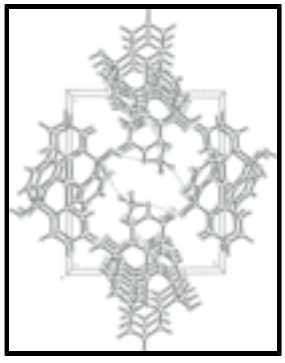


Fig. 2. A view of the crystal packing along the *a* axis. Hydrogen bonds are shown as dashed lines.

Bis(di-2-pyridylmethanediol-κ^2*N*,*N*')palladium(II) bis(perchlorate)

Crystal data

$[Pd(C_{11}H_{10}N_2O_2)_2](ClO_4)_2$	F_{000} = 712
M_r = 709.72	D_x = 1.833 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2779 reflections
a = 7.677 (3) Å	θ = 3.1–27.5º
b = 13.929 (5) Å	μ = 1.00 mm^{-1}
c = 12.173 (5) Å	T = 293 (2) K
β = 99.008 (5)º	Plate, yellow
V = 1285.7 (8) Å^3	0.40 × 0.20 × 0.08 mm
Z = 2	

Data collection

Rigaku Mercury CCD diffractometer	2925 independent reflections
Radiation source: fine-focus sealed tube	2518 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.1º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	h = −9→9
T_{min} = 0.858, T_{max} = 1.000	k = −16→17
9661 measured reflections	l = −12→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 1.6859P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2925 reflections	$\Delta\rho_{max} = 0.62$ e Å^{-3}
187 parameters	$\Delta\rho_{min} = -0.60$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
Pd1	0.0000	0.5000	0.0000	0.03376 (13)
Cl1	−0.15333 (17)	0.38024 (9)	−0.41355 (9)	0.0642 (3)
C1	0.3177 (5)	0.4007 (3)	−0.0473 (3)	0.0456 (9)
H1A	0.2952	0.3615	0.0107	0.055*
C2	0.4601 (5)	0.3814 (3)	−0.0988 (4)	0.0582 (11)
H2A	0.5330	0.3293	−0.0767	0.070*
C3	0.4931 (6)	0.4395 (4)	−0.1826 (4)	0.0671 (13)
H3A	0.5887	0.4270	−0.2189	0.081*
C4	0.3849 (6)	0.5173 (4)	−0.2141 (4)	0.0594 (12)
H4A	0.4081	0.5579	−0.2707	0.071*
C5	0.2432 (5)	0.5337 (3)	−0.1610 (3)	0.0433 (8)
C6	0.1147 (6)	0.6181 (3)	−0.1892 (3)	0.0490 (9)
C7	0.0762 (5)	0.6930 (3)	0.0970 (3)	0.0459 (9)
H7A	0.0411	0.6648	0.1592	0.055*
C8	0.1333 (6)	0.7865 (3)	0.1033 (4)	0.0560 (11)
H8A	0.1390	0.8210	0.1692	0.067*
C9	0.1817 (6)	0.8280 (3)	0.0109 (4)	0.0633 (12)
H9A	0.2187	0.8916	0.0132	0.076*
C10	0.1758 (6)	0.7757 (3)	−0.0853 (4)	0.0543 (10)
H10A	0.2097	0.8034	−0.1482	0.065*
C11	0.1187 (5)	0.6813 (3)	−0.0876 (3)	0.0410 (8)
N1	0.2095 (4)	0.4751 (2)	−0.0785 (2)	0.0378 (7)
N2	0.0696 (4)	0.6408 (2)	0.0028 (2)	0.0376 (6)
O1	−0.0599 (4)	0.5845 (2)	−0.2184 (2)	0.0600 (8)
H1B	−0.0597	0.5349	−0.2546	0.090*
O2	0.1675 (5)	0.6744 (2)	−0.2722 (3)	0.0781 (11)

H2B	0.1414	0.6475	−0.3325	0.117*
O3	−0.2926 (7)	0.4402 (4)	−0.4044 (7)	0.164 (3)
O4	−0.1980 (10)	0.2889 (3)	−0.3818 (5)	0.166 (3)
O5	−0.0060 (6)	0.4159 (3)	−0.3402 (3)	0.0981 (13)
O6	−0.1158 (11)	0.3796 (8)	−0.5176 (4)	0.236 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0400 (2)	0.0271 (2)	0.0341 (2)	0.00115 (15)	0.00552 (14)	−0.00030 (15)
Cl1	0.0799 (8)	0.0667 (7)	0.0469 (6)	−0.0032 (6)	0.0133 (5)	−0.0136 (5)
C1	0.045 (2)	0.036 (2)	0.054 (2)	0.0021 (16)	0.0036 (17)	−0.0038 (17)
C2	0.044 (2)	0.054 (3)	0.075 (3)	0.0073 (19)	0.005 (2)	−0.016 (2)
C3	0.051 (2)	0.082 (4)	0.073 (3)	0.002 (2)	0.022 (2)	−0.020 (3)
C4	0.061 (3)	0.072 (3)	0.049 (2)	−0.012 (2)	0.019 (2)	−0.003 (2)
C5	0.055 (2)	0.0387 (19)	0.0360 (18)	−0.0066 (17)	0.0056 (16)	−0.0055 (16)
C6	0.070 (3)	0.041 (2)	0.0345 (18)	−0.0004 (19)	0.0032 (17)	0.0061 (16)
C7	0.047 (2)	0.042 (2)	0.049 (2)	0.0009 (17)	0.0074 (17)	−0.0119 (17)
C8	0.055 (2)	0.045 (2)	0.068 (3)	−0.0016 (19)	0.006 (2)	−0.023 (2)
C9	0.069 (3)	0.031 (2)	0.089 (3)	−0.006 (2)	0.008 (3)	−0.010 (2)
C10	0.071 (3)	0.036 (2)	0.056 (2)	−0.0048 (19)	0.009 (2)	0.0049 (18)
C11	0.048 (2)	0.0318 (18)	0.0414 (19)	0.0000 (15)	0.0018 (16)	0.0016 (15)
N1	0.0423 (16)	0.0341 (15)	0.0372 (15)	0.0008 (12)	0.0069 (12)	−0.0038 (12)
N2	0.0438 (15)	0.0294 (15)	0.0388 (15)	−0.0012 (13)	0.0040 (12)	−0.0023 (12)
O1	0.071 (2)	0.0484 (16)	0.0521 (17)	0.0097 (14)	−0.0167 (14)	−0.0082 (13)
O2	0.135 (3)	0.0552 (19)	0.0451 (17)	−0.007 (2)	0.0183 (19)	0.0136 (15)
O3	0.109 (4)	0.096 (4)	0.297 (9)	0.020 (3)	0.066 (5)	0.006 (5)
O4	0.246 (7)	0.054 (3)	0.171 (5)	−0.029 (3)	−0.049 (5)	−0.009 (3)
O5	0.107 (3)	0.095 (3)	0.085 (3)	−0.013 (2)	−0.007 (2)	−0.034 (2)
O6	0.235 (8)	0.432 (13)	0.052 (3)	−0.126 (9)	0.055 (4)	−0.075 (5)

Geometric parameters (Å, °)

Pd1—N1	2.025 (3)	C5—N1	1.351 (5)
Pd1—N1^{i}	2.025 (3)	C5—C6	1.538 (6)
Pd1—N2^{i}	2.031 (3)	C6—O2	1.389 (5)
Pd1—N2	2.031 (3)	C6—O1	1.411 (5)
Cl1—O6	1.342 (5)	C6—C11	1.515 (5)
Cl1—O3	1.375 (5)	C7—N2	1.352 (4)
Cl1—O4	1.389 (5)	C7—C8	1.373 (6)
Cl1—O5	1.416 (4)	C7—H7A	0.9300
C1—N1	1.345 (5)	C8—C9	1.366 (7)
C1—C2	1.370 (6)	C8—H8A	0.9300
C1—H1A	0.9300	C9—C10	1.374 (6)
C2—C3	1.357 (7)	C9—H9A	0.9300
C2—H2A	0.9300	C10—C11	1.385 (5)
C3—C4	1.382 (7)	C10—H10A	0.9300
C3—H3A	0.9300	C11—N2	1.342 (5)

C4—C5	1.370 (6)	O1—H1B	0.8200
C4—H4A	0.9300	O2—H2B	0.8200
N1—Pd1—N1^{i}	180.0	O2—C6—C11	107.1 (3)
N1—Pd1—N2^{i}	93.03 (12)	O1—C6—C11	107.2 (3)
N1^{i}—Pd1—N2^{i}	86.97 (12)	O2—C6—C5	110.1 (4)
N1—Pd1—N2	86.97 (12)	O1—C6—C5	110.7 (3)
N1^{i}—Pd1—N2	93.03 (12)	C11—C6—C5	109.4 (3)
N2^{i}—Pd1—N2	180.0	N2—C7—C8	121.8 (4)
O6—Cl1—O3	111.4 (6)	N2—C7—H7A	119.1
O6—Cl1—O4	110.8 (5)	C8—C7—H7A	119.1
O3—Cl1—O4	107.8 (4)	C9—C8—C7	118.7 (4)
O6—Cl1—O5	109.2 (4)	C9—C8—H8A	120.6
O3—Cl1—O5	106.9 (4)	C7—C8—H8A	120.6
O4—Cl1—O5	110.7 (3)	C8—C9—C10	120.0 (4)
N1—C1—C2	121.6 (4)	C8—C9—H9A	120.0
N1—C1—H1A	119.2	C10—C9—H9A	120.0
C2—C1—H1A	119.2	C9—C10—C11	119.3 (4)
C3—C2—C1	118.9 (4)	C9—C10—H10A	120.4
C3—C2—H2A	120.6	C11—C10—H10A	120.4
C1—C2—H2A	120.6	N2—C11—C10	120.7 (4)
C2—C3—C4	120.1 (4)	N2—C11—C6	116.9 (3)
C2—C3—H3A	120.0	C10—C11—C6	122.3 (4)
C4—C3—H3A	120.0	C1—N1—C5	119.6 (3)
C5—C4—C3	119.2 (4)	C1—N1—Pd1	120.0 (3)
C5—C4—H4A	120.4	C5—N1—Pd1	120.4 (3)
C3—C4—H4A	120.4	C11—N2—C7	119.4 (3)
N1—C5—C4	120.6 (4)	C11—N2—Pd1	119.9 (2)
N1—C5—C6	115.9 (3)	C7—N2—Pd1	120.6 (3)
C4—C5—C6	123.5 (4)	C6—O1—H1B	109.5
O2—C6—O1	112.2 (3)	C6—O2—H2B	109.5

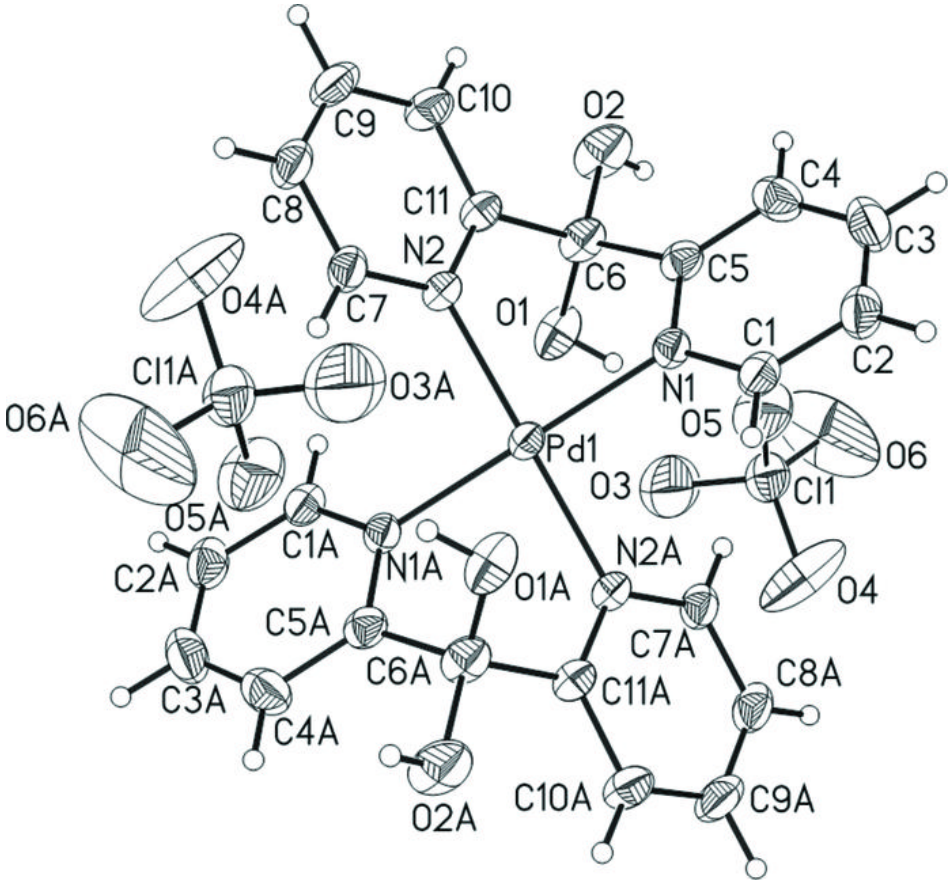
Symmetry codes: (i) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1B···O5	0.82	2.03	2.842 (5)	169
O2—H2B···O6^{ii}	0.82	1.84	2.636 (6)	162

Symmetry codes: (ii) −*x*, −*y*+1, −*z*−1.

Fig. 1


C9
C10
O2
C8
C11
C4
C3
N2
C6
C5
O4A
C7
O1
C1
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Cl1A
O3A
N1
O6A
O5
Pd1
O6
O3
Cl1
O5A
C1A
N1A
N2A
C2A
O1A
O4
C5A
C7A
C6A
C8A
C3A
C11A
C4A
O2A
C10A
C9A

Fig. 2