

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(2,2'-Bipyridine- κ^2N,N')diiodido-palladium(II)

Kwang Ha

School of Applied Chemical Engineering, The Research Institute of Catalysis, Chonnam National University, Gwangju 500-757, Republic of Korea
Correspondence e-mail: hakwang@chonnam.ac.kr

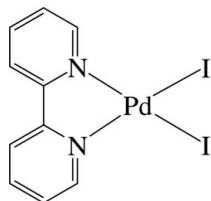
Received 10 November 2009; accepted 11 November 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.009$ Å; R factor = 0.032; wR factor = 0.071; data-to-parameter ratio = 18.0.

The asymmetric unit of the title complex, $[PdI_2(C_{10}H_8N_2)]$, contains one half of the formula unit. The Pd^{2+} ion is located on a twofold rotation axis and is four-coordinated in a slightly distorted square-planar environment by two N atoms of the chelating 2,2'-bipyridine ligand and two iodide ions. The compound displays intermolecular $\pi-\pi$ interactions between the pyridine rings of the ligand, the shortest centroid-centroid distance being 4.220 (4) Å.

Related literature

For the crystal structures of $[PdX_2(bipy)]$ ($bipy = 2,2'$ -bipyridine; $X = Cl$ or Br), see: Maekawa *et al.* (1991); Smeets *et al.* (1997). For the crystal structures of $[PdX_2(bipy)] \cdot CH_2Cl_2$ ($X = Cl$ or Br), see: Vicente *et al.* (1997); Kim *et al.* (2009); Kim & Ha (2009).



Experimental

Crystal data

$[PdI_2(C_{10}H_8N_2)]$
 $M_r = 516.38$

Monoclinic, $C2/c$
 $a = 17.232$ (4) Å

$b = 9.8273$ (19) Å
 $c = 7.6868$ (15) Å
 $\beta = 111.438$ (3)°
 $V = 1211.6$ (4) Å³
 $Z = 4$

Mo $K\alpha$ radiation $\mu = 6.60$ mm⁻¹ $T = 293$ K $0.25 \times 0.05 \times 0.05$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{min} = 0.139$, $T_{max} = 0.719$

3458 measured reflections
1240 independent reflections
1049 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.071$
 $S = 1.06$
1240 reflections

69 parameters
H-atom parameters constrained
 $\Delta\rho_{max} = 0.60$ e Å⁻³
 $\Delta\rho_{min} = -0.65$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pd1—N1	2.076 (4)	Pd1—I1	2.5704 (6)
N1—Pd1—N1 ⁱ	79.4 (2)		

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

This work was supported by a Korea Research Foundation Grant funded by the Korean Government (MOEHRD) (KRF-2007-412-J02001).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2486).

References

- Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kim, N.-H. & Ha, K. (2009). *Acta Cryst.* **E65**, m1292.
Kim, N.-H., Hwang, I.-C. & Ha, K. (2009). *Acta Cryst.* **E65**, m615–m616.
Maekawa, M., Munakata, M., Kitagawa, S. & Nakamura, M. (1991). *Anal. Sci.* **7**, 521–522.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Smeets, W. J. J., Spek, A. L., Hoare, J. L., Canty, A. J., Hovestad, N. & van Koten, G. (1997). *Acta Cryst.* **C53**, 1045–1047.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
Vicente, J., Abad, J. A., Rink, B. & Arellano, M. C. R. (1997). Private communication (refcode PYCXMN02). CCDC, Cambridge, England.

supplementary materials

Acta Cryst. (2009). E65, m1588 [doi:10.1107/S1600536809047771]

(2,2'-Bipyridine- κ^2N,N')diiodidopalladium(II)

K. Ha

Comment

The title complex, [PdI₂(bipy)] (where bipy is 2,2'-bipyridine, C₁₀H₈N₂), is isomorphous with [PdBr₂(bipy)] (Smeets *et al.*, 1997), whereas [PdCl₂(bipy)] crystallized in the orthorhombic space group C222₁ (Maekawa *et al.*, 1991).

The asymmetric unit of the title complex contains one half of the formula unit. The complex is disposed about a twofold rotation axis through Pd atom with the special position at (0, *y*, 1/4) (Wyckoff letter *e*). The Pd²⁺ ion is four-coordinated in a slightly distorted square-planar environment by two N atoms of the chelating 2,2'-bipyridine ligand and two iodide ions (Fig. 1). The main contribution to the distortion is the tight N1—Pd1—N1^{*a*} [symmetry code: (*a*) -*x*, *y*, 1/2 - *z*] chelate angle [79.4 (2)°], which results in non-linear *trans* arrangement [\angle N1—Pd1—I1^{*a*} = 175.85 (12)°]. The complex displays intermolecular π - π interactions between adjacent pyridine rings of the ligand (the symmetry operation for second plane *x*, -*y*, -1/2 + *z*), with a shortest centroid-centroid distance of 4.220 (4) Å (Fig. 2).

Experimental

To a solution of Na₂PdCl₄ (0.1991 g, 0.677 mmol) in H₂O (20 ml) were added KI (1.1230 g, 6.765 mmol) and 2,2'-bipyridine (0.1057 g, 0.677 mmol), and refluxed for 3 h. The precipitate obtained was separated by filtration, and washed with water and acetone, and dried at 70 °C, to give a red-brown powder (0.2999 g). Crystals suitable for X-ray analysis were obtained by slow evaporation from a CH₃CN solution.

Refinement

H atoms were positioned geometrically and allowed to ride on their respective parent atoms [C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

Figures

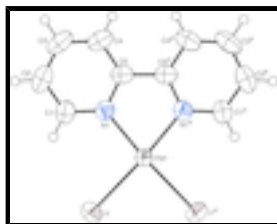


Fig. 1. The structure of the title complex, with displacement ellipsoids drawn at the 50% probability level for non-H atoms [Symmetry code: (*a*) -*x*, *y*, 1/2 - *z*].

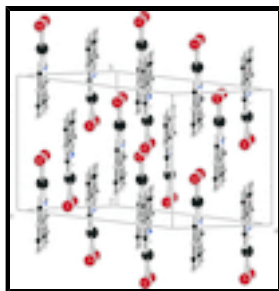


Fig. 2. Crystal packing of the title complex.

(2,2'-Bipyridine- κ^2N,N')diiodidopalladium(II)

Crystal data

[PdI₂(C₁₀H₈N₂)]

$M_r = 516.38$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 17.232\ (4)\ \text{\AA}$

$b = 9.8273\ (19)\ \text{\AA}$

$c = 7.6868\ (15)\ \text{\AA}$

$\beta = 111.438\ (3)^\circ$

$V = 1211.6\ (4)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 936$

$D_x = 2.831\ \text{Mg m}^{-3}$

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

Cell parameters from 550 reflections

$\theta = 2.4\text{--}24.4^\circ$

$\mu = 6.60\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Needle, brown

$0.25 \times 0.05 \times 0.05\ \text{mm}$

Data collection

Bruker SMART 1000 CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ \text{K}$

φ and ω scans

Absorption correction: Multi-scan
(*SADABS*; Bruker, 2000)

$T_{\min} = 0.139$, $T_{\max} = 0.719$

3458 measured reflections

1240 independent reflections

1049 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 26.4^\circ$

$\theta_{\min} = 2.4^\circ$

$h = -21 \rightarrow 13$

$k = -11 \rightarrow 12$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.071$

$S = 1.06$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

1240 reflections $\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
 69 parameters $\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$
 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.0000	-0.19021 (5)	0.2500	0.03813 (17)
I1	0.10045 (3)	-0.37899 (4)	0.43052 (5)	0.06273 (19)
N1	0.0755 (3)	-0.0277 (4)	0.3822 (6)	0.0431 (10)
C1	0.1524 (4)	-0.0339 (6)	0.5132 (7)	0.0546 (14)
H1	0.1767	-0.1188	0.5499	0.066*
C2	0.1966 (4)	0.0802 (7)	0.5952 (9)	0.0655 (17)
H2	0.2495	0.0725	0.6871	0.079*
C3	0.1617 (4)	0.2050 (6)	0.5399 (9)	0.0656 (18)
H3	0.1902	0.2837	0.5944	0.079*
C4	0.0840 (4)	0.2128 (6)	0.4029 (9)	0.0611 (17)
H4	0.0597	0.2972	0.3627	0.073*
C5	0.0418 (4)	0.0954 (5)	0.3248 (8)	0.0446 (12)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0378 (3)	0.0339 (3)	0.0385 (3)	0.000	0.0091 (2)	0.000
I1	0.0636 (3)	0.0471 (3)	0.0633 (3)	0.01213 (18)	0.0064 (2)	0.00869 (16)
N1	0.043 (2)	0.043 (2)	0.045 (2)	-0.002 (2)	0.018 (2)	-0.0005 (19)
C1	0.046 (3)	0.062 (4)	0.050 (3)	-0.003 (3)	0.012 (3)	-0.004 (3)
C2	0.050 (4)	0.087 (5)	0.056 (4)	-0.019 (4)	0.016 (3)	-0.015 (3)
C3	0.065 (4)	0.060 (4)	0.082 (4)	-0.029 (4)	0.039 (4)	-0.028 (3)
C4	0.067 (4)	0.050 (3)	0.074 (4)	-0.018 (3)	0.036 (4)	-0.015 (3)
C5	0.050 (3)	0.039 (3)	0.056 (3)	-0.001 (2)	0.033 (3)	-0.002 (2)

Geometric parameters (\AA , $^\circ$)

Pd1—N1	2.076 (4)	C2—C3	1.364 (9)
--------	-----------	-------	-----------

supplementary materials

Pd1—N1 ⁱ	2.076 (4)	C2—H2	0.9300
Pd1—I1 ⁱ	2.5704 (6)	C3—C4	1.370 (9)
Pd1—I1	2.5704 (6)	C3—H3	0.9300
N1—C1	1.341 (7)	C4—C5	1.378 (7)
N1—C5	1.345 (6)	C4—H4	0.9300
C1—C2	1.372 (8)	C5—C5 ⁱ	1.480 (12)
C1—H1	0.9300		
N1—Pd1—N1 ⁱ	79.4 (2)	C3—C2—C1	119.0 (6)
N1—Pd1—I1 ⁱ	175.85 (12)	C3—C2—H2	120.5
N1 ⁱ —Pd1—I1 ⁱ	96.48 (12)	C1—C2—H2	120.5
N1—Pd1—I1	96.48 (12)	C2—C3—C4	119.0 (6)
N1 ⁱ —Pd1—I1	175.85 (12)	C2—C3—H3	120.5
I1 ⁱ —Pd1—I1	87.61 (3)	C4—C3—H3	120.5
C1—N1—C5	118.5 (5)	C3—C4—C5	120.0 (6)
C1—N1—Pd1	127.1 (4)	C3—C4—H4	120.0
C5—N1—Pd1	114.4 (4)	C5—C4—H4	120.0
N1—C1—C2	122.6 (6)	N1—C5—C4	120.9 (6)
N1—C1—H1	118.7	N1—C5—C5 ⁱ	115.9 (3)
C2—C1—H1	118.7	C4—C5—C5 ⁱ	123.2 (4)
N1 ⁱ —Pd1—N1—C1	-178.7 (6)	C2—C3—C4—C5	0.8 (9)
I1—Pd1—N1—C1	2.1 (5)	C1—N1—C5—C4	-2.1 (8)
N1 ⁱ —Pd1—N1—C5	0.5 (3)	Pd1—N1—C5—C4	178.6 (4)
I1—Pd1—N1—C5	-178.7 (3)	C1—N1—C5—C5 ⁱ	177.9 (6)
C5—N1—C1—C2	2.3 (9)	Pd1—N1—C5—C5 ⁱ	-1.4 (7)
Pd1—N1—C1—C2	-178.5 (4)	C3—C4—C5—N1	0.6 (9)
N1—C1—C2—C3	-0.9 (10)	C3—C4—C5—C5 ⁱ	-179.4 (6)
C1—C2—C3—C4	-0.6 (10)		

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

Fig. 1

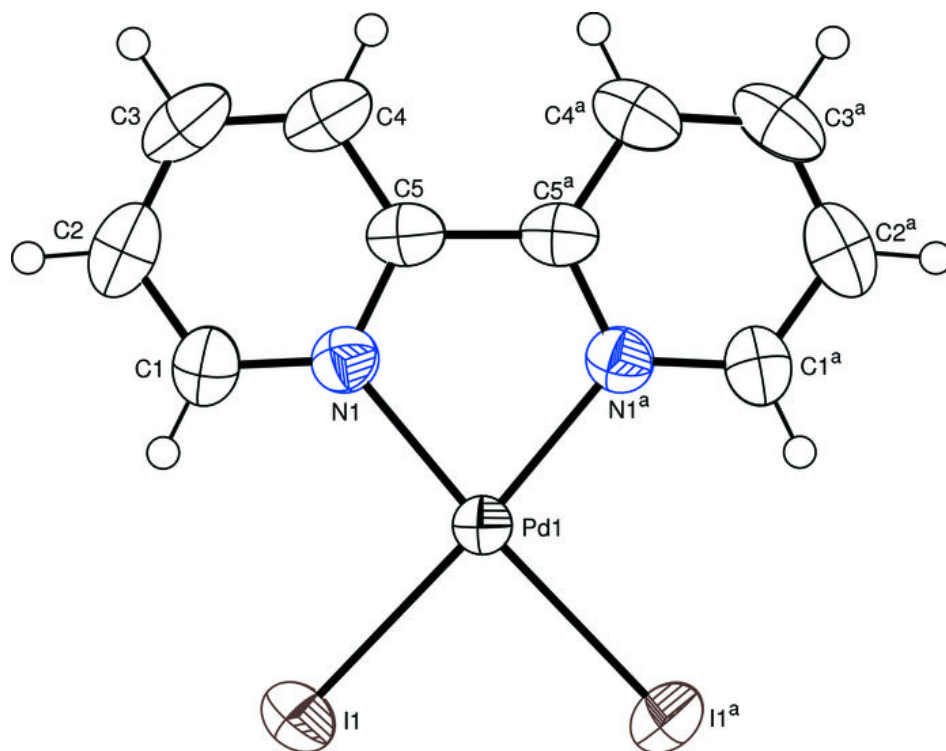


Fig. 2

