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# (2,2′-Bipyridine-$\kappa^2N,N'$)diiodidopalladium(II)

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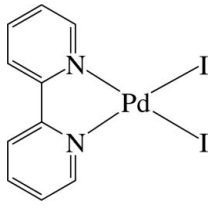
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.032; $wR$ factor = 0.071; data-to-parameter ratio = 18.0.

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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 6.60 mm$^{-1}$
$T$ = 293 K
0.25 × 0.05 × 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*.

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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.* E**65**, m1292.
Kim, N.-H., Hwang, I.-C. & Ha, K. (2009). *Acta Cryst.* E**65**, m615–m616.
Maekawa, M., Munakata, M., Kitagawa, S. & Nakamura, M. (1991). *Anal. Sci.* **7**, 521–522.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smeets, W. J. J., Spek, A. L., Hoare, J. L., Canty, A. J., Hovestad, N. & van Koten, G. (1997). *Acta Cryst.* C**53**, 1045–1047.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Vicente, J., Abad, J. A., Rink, B. & Arellano, M. C. R. (1997). Private communication (refcode PYCXMN02). CCDC, Cambridge, England.

# supporting information

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## (2,2′-Bipyridine-$\kappa^2N,N'$)diiodidopalladium(II)

**Kwang Ha**

### S1. Comment

The title complex, [$PdI_2$(bipy)] (where bipy is 2,2′-bipyridine, $C_{10}H_8N_2$), is isomorphous with [$PdBr_2$(bipy)] (Smeets *et al.*, 1997), whereas [$PdCl_2$(bipy)] crystallized in the orthorhombic space group $C222_1$ (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 $Pd^{2+}$ 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 [<N1—Pd1—$I1^a$ = 175.85 (12)°]. The complex displays intermolecular $\pi$-$\pi$ interactions between adjacent pyridine rings of the lignad (the symmetry operation for second plane *x*, -*y*, -1/2 + *z*), with a shortest centroid-centroid distance of 4.220 (4) Å (Fig. 2).

### S2. Experimental

To a solution of $Na_2PdCl_4$ (0.1991 g, 0.677 mmol) in $H_2O$ (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 $CH_3CN$ solution.

### S3. Refinement

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

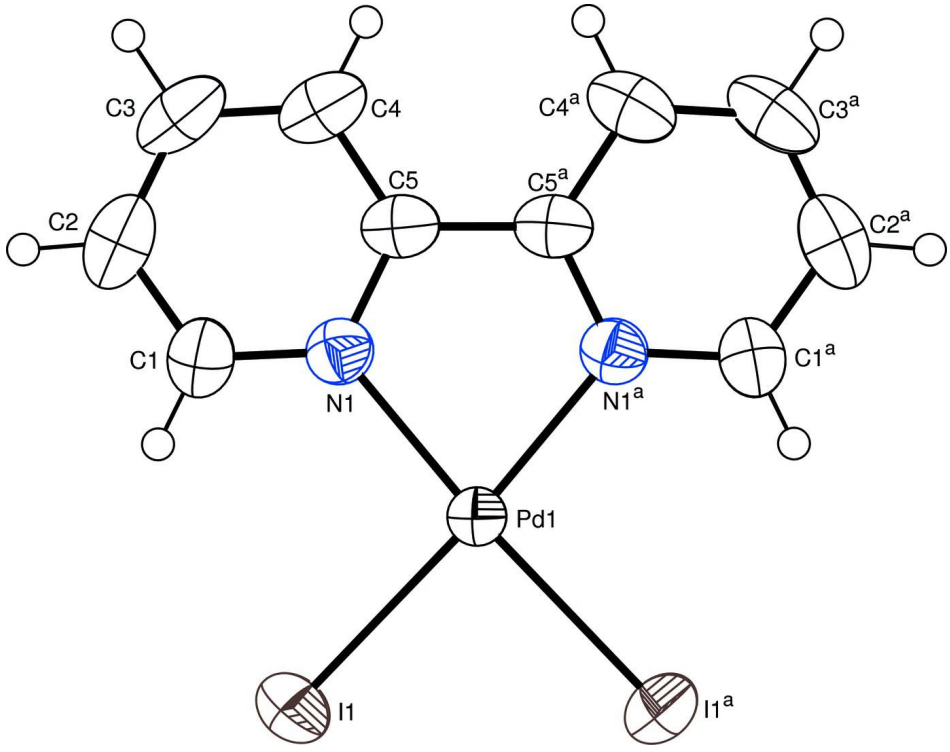


**Figure 1**

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*].

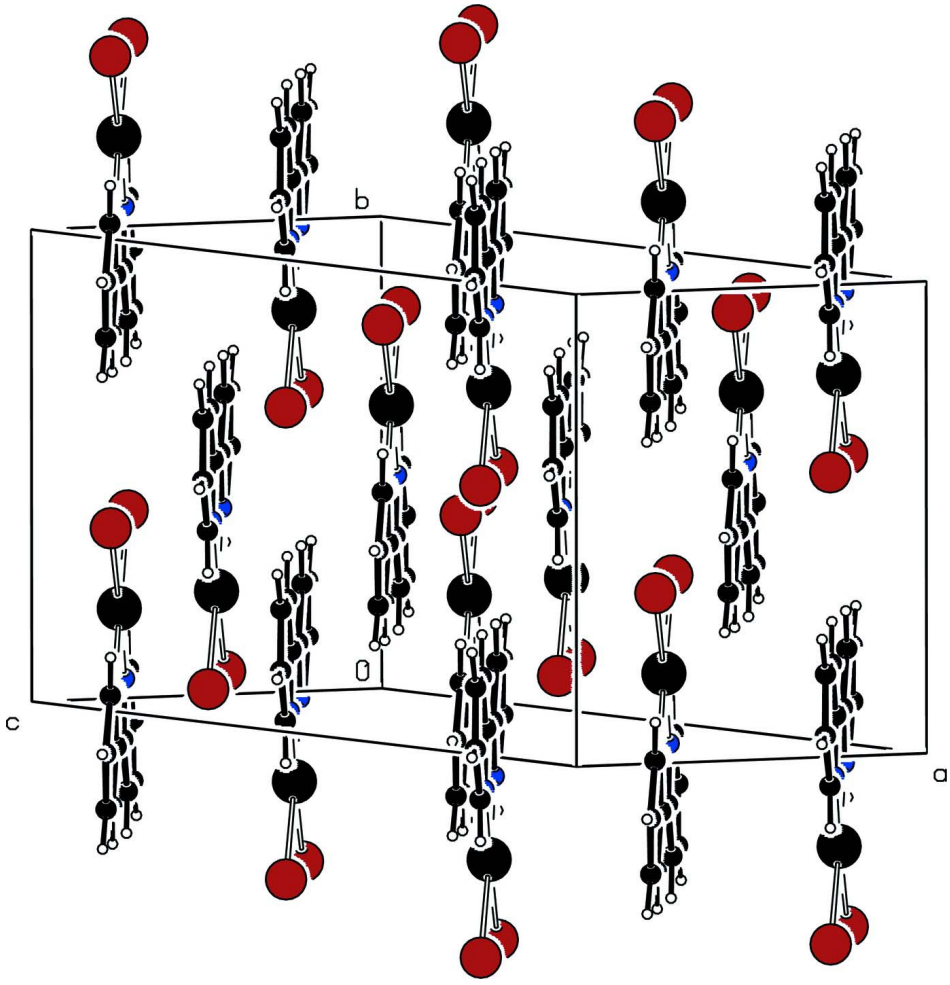


**Figure 2**

Crystal packing of the title complex.

### (2,2′-Bipyridine-$\kappa^2N,N'$)diiodidopalladium(II)

*Crystal data*

$[PdI_2(C_{10}H_8N_2)]$
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Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 17.232$ (4) Å
$b = 9.8273$ (19) Å
$c = 7.6868$ (15) Å
$\beta = 111.438$ (3)°
$V = 1211.6$ (4) Å$^3$
$Z = 4$
$F(000) = 936$
$D_x = 2.831$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 550 reflections
$\theta = 2.4$–$24.4$°
$\mu = 6.60$ mm$^{-1}$
$T = 293$ K
Needle, brown
0.25 × 0.05 × 0.05 mm

*Data collection*

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.4°$
$h = -21 \rightarrow 13$
$k = -11 \rightarrow 12$
$l = -9 \rightarrow 9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.071$
$S = 1.06$
1240 reflections
69 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
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.60$ e Å$^{-3}$
$\Delta\rho_{min} = -0.65$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

| | | | |
|---|---|---|---|
| Pd1—N1 | 2.076 (4) | C2—C3 | 1.364 (9) |
| 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 code: (i) −*x*, *y*, −*z*+1/2.