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(2,3-Di-2-pyridylpyrazine-κ^2N^2,N^3)-diiodidopalladium(II)

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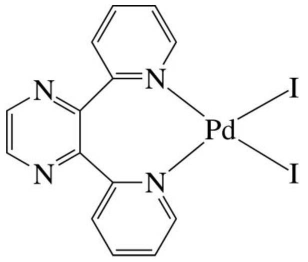
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.009 Å; R factor = 0.035; wR factor = 0.103; data-to-parameter ratio = 18.5.

The Pd^{II} ion in the title complex, [PdI$_2$(C$_{14}$H$_{10}$N$_4$)], is four-coordinated in a slightly distorted square-planar environment by the two pyridine N atoms of the chelating 2,3-di-2-pyridylpyrazine (dpp) ligand and two iodide anions. In the crystal, the pyridine rings are considerably inclined to the least-squares plane of the PdI$_2$N$_2$ unit [maximum deviation = 0.090 (2) Å], making dihedral angles of 65.0 (2) and 66.6 (2)°. The pyrazine ring is perpendicular to the unit plane, with a dihedral angle of 89.9 (2)°. The complex molecules are connected by C—H···I hydrogen bonds, forming a helical chain along the b axis.

Related literature

For the crystal structure of the yellow form of [PtBr$_2$(dpp)] which is isotypic to the title complex, see: Ha (2011).



Experimental

Crystal data

[PdI$_2$(C$_{14}$H$_{10}$N$_4$)]
M_r = 594.46
Monoclinic, $P2_1/n$
a = 8.7936 (12) Å
b = 15.528 (2) Å
c = 12.3351 (17) Å
β = 102.118 (3)°
V = 1646.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.87 mm^{-1}
T = 200 K
0.42 × 0.31 × 0.29 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.183, T_{max} = 0.243
10659 measured reflections
3513 independent reflections
2968 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.103
S = 1.05
3513 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −1.42 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pd1—N3	2.050 (5)	Pd1—I1	2.5761 (7)
Pd1—N4	2.056 (5)	Pd1—I2	2.5898 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···I1^{i}	0.95	2.99	3.776 (7)	141

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: IS2795).

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Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2011). E**67**, m1626 [doi:10.1107/S1600536811044023]

(2,3-Di-2-pyridylpyrazine-κ^2N^2,N^3)diiodidopalladium(II)

Kwang Ha

S1. Comment

The title complex, [PdI_2(dpp)] (dpp is 2,3-di-2-pyridylpyrazine, $C_{14}H_{10}N_4$), is isomorphous with the yellow form of [$PtBr_2$(dpp)] (Ha, 2011). The Pd^{II} ion is four-coordinated in a slightly distorted square-planar environment by the two pyridyl N atoms of the chelating dpp ligand and two iodide anions (Fig. 1). The contributions to the distortion are the N3—Pd1—N4 chelate angle of 87.17 (19)° and I—I repelling, and therefore the *trans* axes are slightly bent [<I1—Pd1—N4 = 173.87 (13)° and <I2—Pd1—N3 = 176.70 (13)°]. The Pd—N and Pd—I bond lengths are nearly equivalent, respectively (Table 1). In the crystal, the two pyridyl rings are considerably inclined to the least-squares plane of the PdI_2N_2 unit [maximum deviation = 0.090 (2) Å] with dihedral angles of 65.0 (2) and 66.6 (2)°, respectively. The nearly planar pyrazine ring [maximum deviation = 0.014 (4) Å] is perpendicular to the unit plane with a dihedral angle of 89.9 (2)°. The dihedral angle between the two pyridyl rings is 78.6 (2)°. The complexes are connected by C—H···I hydrogen bonds, forming a helical chain running along the *b* axis (Fig. 2 and Table 2), and stacked in columns along the *a* axis. When viewed down the *b* axis, the successive complexes stack in the opposite direction. In the columns, numerous inter- and intramolecular *π–π* interactions between the six-membered rings are present, the shortest ring centroid-centroid distance being 3.969 (4) Å.

S2. Experimental

To a solution of Na_2PdCl_4 (0.1464 g, 0.498 mmol) and KI (0.7790 g, 4.693 mmol) in MeOH (30 ml) was added 2,3-di-2-pyridylpyrazine (0.1183 g, 0.505 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with MeOH and acetone, and dried at 50 °C, to give a redbrown powder (0.2537 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 = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C)]. The highest peak (1.27 e Å^{-3}) and the deepest hole (-1.41 e Å^{-3}) in the difference Fourier map are located 0.72 Å and 0.59 Å from the atoms I2 and I1, respectively.

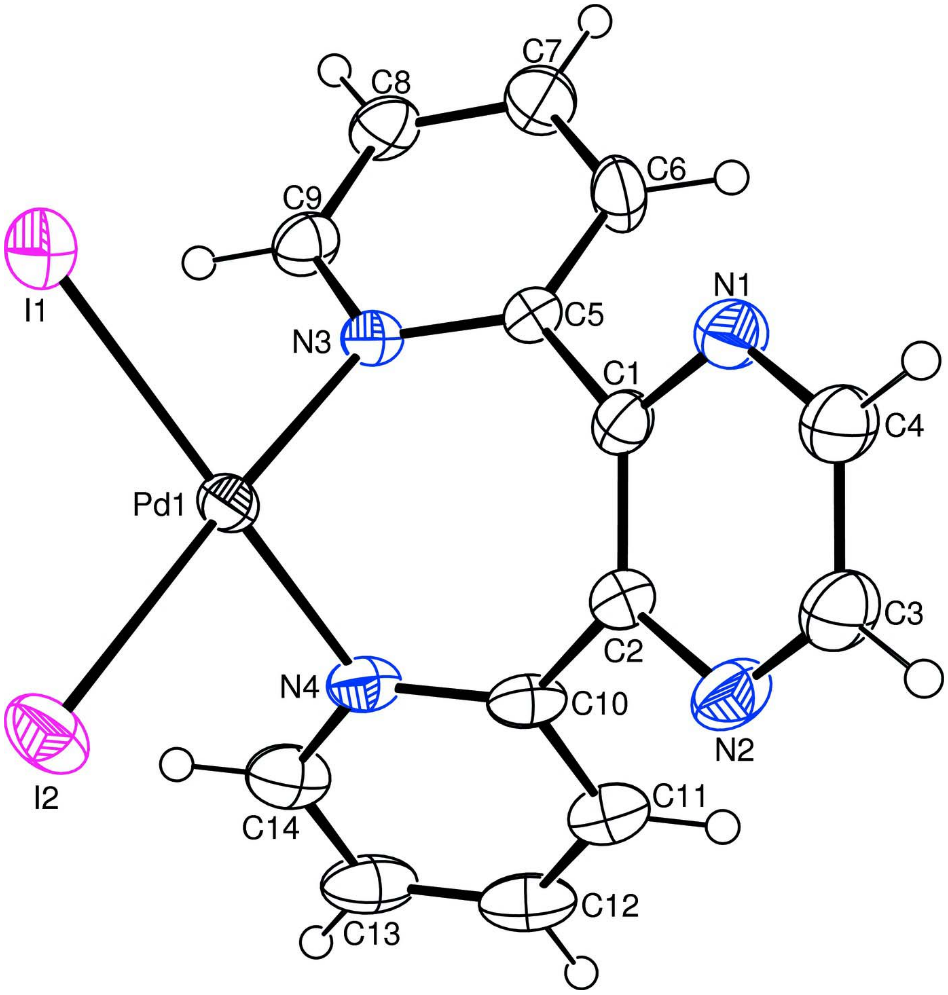

Figure 1

The structure of the title complex, with displacement ellipsoids drawn at the 40% probability level; H atoms are shown as small circles of arbitrary radius.

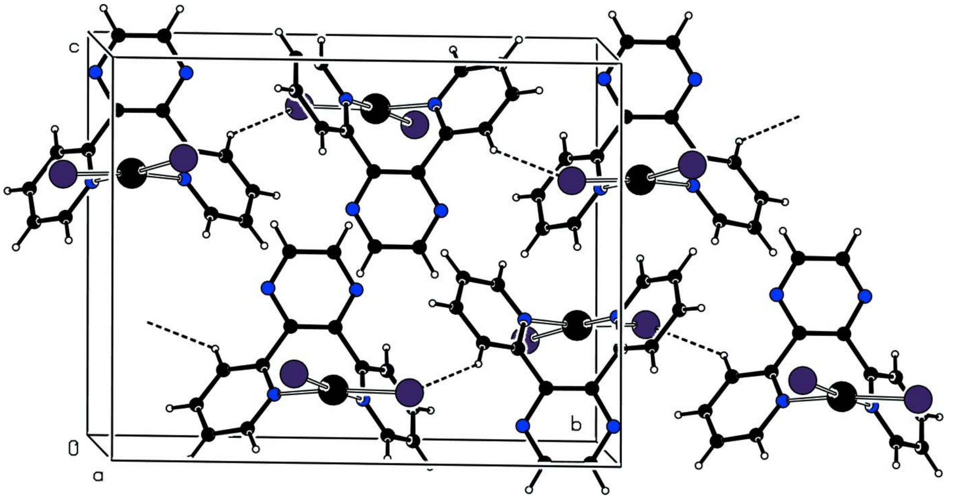

Figure 2

View of the hydrogen-bond interactions of the title complex. Hydrogen-bonds are drawn with dashed lines.

(2,3-Di-2-pyridylpyrazine-κ^2N^2,N^3)diiodidopalladium(II)

Crystal data

$[PdI_2(C_{14}H_{10}N_4)]$
$M_r = 594.46$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.7936\ (12)$ Å
$b = 15.528\ (2)$ Å
$c = 12.3351\ (17)$ Å
$\beta = 102.118\ (3)°$
$V = 1646.8\ (4)$ Å^3
$Z = 4$

$F(000) = 1096$
$D_x = 2.398$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7148 reflections
$\theta = 2.4–27.0°$
$\mu = 4.87$ mm^{-1}
$T = 200$ K
Block, red-brown
0.42 × 0.31 × 0.29 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.183$, $T_{max} = 0.243$

10659 measured reflections
3513 independent reflections
2968 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 10$
$k = -19 \rightarrow 19$
$l = -11 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.103$
$S = 1.05$
3513 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 7.4036P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.28$ e Å^{-3}
$\Delta\rho_{min} = -1.42$ 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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Pd1	0.46673 (5)	0.06358 (3)	0.67952 (3)	0.03147 (13)
I1	0.61860 (5)	−0.07875 (3)	0.68473 (4)	0.04827 (15)
I2	0.71972 (6)	0.15314 (3)	0.73389 (4)	0.05207 (16)
N1	0.1852 (6)	0.0054 (3)	0.9114 (4)	0.0419 (12)
N2	0.2351 (7)	0.1813 (4)	0.9159 (5)	0.0473 (13)
N3	0.2628 (5)	−0.0048 (3)	0.6446 (4)	0.0314 (10)
N4	0.3330 (6)	0.1733 (3)	0.6587 (4)	0.0375 (11)
C1	0.2007 (6)	0.0473 (4)	0.8187 (5)	0.0332 (12)
C2	0.2265 (7)	0.1360 (4)	0.8211 (5)	0.0347 (12)
C3	0.2229 (9)	0.1385 (5)	1.0064 (6)	0.0516 (17)
H3	0.2304	0.1690	1.0740	0.062*
C4	0.1993 (9)	0.0498 (4)	1.0047 (6)	0.0495 (16)
H4	0.1932	0.0207	1.0714	0.059*
C5	0.1683 (6)	−0.0075 (3)	0.7174 (5)	0.0309 (11)
C6	0.0425 (7)	−0.0623 (4)	0.7030 (6)	0.0430 (15)
H6	−0.0216	−0.0642	0.7561	0.052*
C7	0.0103 (7)	−0.1154 (4)	0.6092 (6)	0.0485 (16)
H7	−0.0753	−0.1541	0.5981	0.058*
C8	0.1045 (7)	−0.1106 (4)	0.5334 (5)	0.0418 (14)
H8	0.0844	−0.1452	0.4684	0.050*
C9	0.2272 (7)	−0.0552 (4)	0.5534 (5)	0.0385 (13)
H9	0.2913	−0.0517	0.5004	0.046*
C10	0.2329 (7)	0.1913 (4)	0.7239 (5)	0.0374 (13)
C11	0.1398 (8)	0.2639 (4)	0.7055 (6)	0.0470 (16)
H11	0.0711	0.2768	0.7533	0.056*
C12	0.1467 (10)	0.3171 (4)	0.6187 (6)	0.0558 (19)
H12	0.0820	0.3665	0.6050	0.067*
C13	0.2478 (10)	0.2984 (4)	0.5515 (6)	0.0554 (19)
H13	0.2543	0.3348	0.4907	0.067*
C14	0.3406 (9)	0.2259 (4)	0.5733 (5)	0.0497 (16)
H14	0.4113	0.2130	0.5270	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0364 (2)	0.0300 (2)	0.0295 (2)	−0.00616 (17)	0.01056 (17)	−0.00251 (16)

I1	0.0419 (3)	0.0503 (3)	0.0548 (3)	0.00162 (18)	0.01512 (19)	0.00031 (19)
I2	0.0562 (3)	0.0592 (3)	0.0431 (3)	−0.0268 (2)	0.0154 (2)	−0.0105 (2)
N1	0.053 (3)	0.037 (3)	0.036 (3)	−0.003 (2)	0.013 (2)	−0.002 (2)
N2	0.062 (4)	0.039 (3)	0.041 (3)	0.002 (3)	0.012 (3)	−0.011 (2)
N3	0.034 (2)	0.026 (2)	0.034 (2)	−0.0009 (18)	0.0070 (19)	0.0001 (19)
N4	0.054 (3)	0.027 (2)	0.031 (2)	−0.004 (2)	0.007 (2)	−0.0016 (19)
C1	0.032 (3)	0.034 (3)	0.034 (3)	0.001 (2)	0.007 (2)	−0.006 (2)
C2	0.038 (3)	0.030 (3)	0.037 (3)	−0.001 (2)	0.010 (2)	−0.004 (2)
C3	0.072 (5)	0.048 (4)	0.038 (4)	−0.002 (3)	0.018 (3)	−0.009 (3)
C4	0.068 (5)	0.044 (4)	0.040 (4)	−0.001 (3)	0.019 (3)	−0.004 (3)
C5	0.034 (3)	0.026 (3)	0.034 (3)	0.001 (2)	0.010 (2)	−0.003 (2)
C6	0.034 (3)	0.055 (4)	0.044 (4)	−0.009 (3)	0.017 (3)	−0.009 (3)
C7	0.037 (3)	0.046 (4)	0.062 (4)	−0.012 (3)	0.008 (3)	−0.014 (3)
C8	0.047 (4)	0.038 (3)	0.039 (3)	−0.005 (3)	0.004 (3)	−0.014 (3)
C9	0.048 (3)	0.037 (3)	0.031 (3)	0.000 (3)	0.008 (2)	−0.007 (2)
C10	0.050 (3)	0.026 (3)	0.033 (3)	−0.004 (2)	−0.001 (3)	−0.003 (2)
C11	0.053 (4)	0.033 (3)	0.052 (4)	0.003 (3)	0.003 (3)	−0.004 (3)
C12	0.069 (5)	0.029 (3)	0.060 (4)	0.000 (3)	−0.008 (4)	0.004 (3)
C13	0.092 (6)	0.032 (3)	0.036 (3)	−0.007 (3)	0.001 (4)	0.004 (3)
C14	0.071 (5)	0.041 (3)	0.036 (3)	−0.011 (3)	0.008 (3)	−0.001 (3)

Geometric parameters (Å, º)

Pd1—N3	2.050 (5)	C4—H4	0.9500
Pd1—N4	2.056 (5)	C5—C6	1.378 (8)
Pd1—I1	2.5761 (7)	C6—C7	1.400 (9)
Pd1—I2	2.5898 (6)	C6—H6	0.9500
N1—C4	1.326 (8)	C7—C8	1.375 (10)
N1—C1	1.347 (8)	C7—H7	0.9500
N2—C3	1.321 (9)	C8—C9	1.362 (9)
N2—C2	1.353 (8)	C8—H8	0.9500
N3—C5	1.346 (7)	C9—H9	0.9500
N3—C9	1.352 (7)	C10—C11	1.384 (9)
N4—C10	1.340 (8)	C11—C12	1.363 (10)
N4—C14	1.346 (8)	C11—H11	0.9500
C1—C2	1.395 (8)	C12—C13	1.367 (12)
C1—C5	1.489 (7)	C12—H12	0.9500
C2—C10	1.486 (8)	C13—C14	1.383 (11)
C3—C4	1.393 (10)	C13—H13	0.9500
C3—H3	0.9500	C14—H14	0.9500

N3—Pd1—N4	87.17 (19)	C6—C5—C1	117.9 (5)
N3—Pd1—I1	89.26 (13)	C5—C6—C7	119.2 (6)
N4—Pd1—I1	173.87 (13)	C5—C6—H6	120.4
N3—Pd1—I2	176.70 (13)	C7—C6—H6	120.4
N4—Pd1—I2	91.43 (14)	C8—C7—C6	118.9 (6)
I1—Pd1—I2	92.38 (2)	C8—C7—H7	120.5
C4—N1—C1	118.5 (5)	C6—C7—H7	120.5

C3—N2—C2	118.0 (6)	C9—C8—C7	118.6 (6)
C5—N3—C9	118.0 (5)	C9—C8—H8	120.7
C5—N3—Pd1	120.7 (4)	C7—C8—H8	120.7
C9—N3—Pd1	120.9 (4)	N3—C9—C8	123.6 (6)
C10—N4—C14	119.3 (6)	N3—C9—H9	118.2
C10—N4—Pd1	121.9 (4)	C8—C9—H9	118.2
C14—N4—Pd1	118.7 (5)	N4—C10—C11	120.8 (6)
N1—C1—C2	120.3 (5)	N4—C10—C2	120.3 (5)
N1—C1—C5	113.6 (5)	C11—C10—C2	118.9 (6)
C2—C1—C5	125.7 (5)	C12—C11—C10	120.0 (7)
N2—C2—C1	120.7 (6)	C12—C11—H11	120.0
N2—C2—C10	113.1 (5)	C10—C11—H11	120.0
C1—C2—C10	126.0 (5)	C11—C12—C13	119.3 (7)
N2—C3—C4	121.5 (6)	C11—C12—H12	120.4
N2—C3—H3	119.2	C13—C12—H12	120.4
C4—C3—H3	119.2	C12—C13—C14	119.2 (7)
N1—C4—C3	120.9 (6)	C12—C13—H13	120.4
N1—C4—H4	119.5	C14—C13—H13	120.4
C3—C4—H4	119.5	N4—C14—C13	121.4 (7)
N3—C5—C6	121.6 (5)	N4—C14—H14	119.3
N3—C5—C1	120.4 (5)	C13—C14—H14	119.3

N4—Pd1—N3—C5	−70.8 (4)	N1—C1—C5—C6	42.3 (7)
I1—Pd1—N3—C5	114.2 (4)	C2—C1—C5—C6	−130.1 (7)
N4—Pd1—N3—C9	116.0 (4)	N3—C5—C6—C7	−1.3 (10)
I1—Pd1—N3—C9	−59.1 (4)	C1—C5—C6—C7	−179.2 (6)
N3—Pd1—N4—C10	62.2 (5)	C5—C6—C7—C8	−0.6 (11)
I2—Pd1—N4—C10	−114.8 (4)	C6—C7—C8—C9	1.0 (10)
N3—Pd1—N4—C14	−114.1 (5)	C5—N3—C9—C8	−2.4 (9)
I2—Pd1—N4—C14	68.9 (5)	Pd1—N3—C9—C8	171.0 (5)
C4—N1—C1—C2	−1.8 (9)	C7—C8—C9—N3	0.6 (10)
C4—N1—C1—C5	−174.7 (6)	C14—N4—C10—C11	−1.2 (9)
C3—N2—C2—C1	2.0 (10)	Pd1—N4—C10—C11	−177.5 (4)
C3—N2—C2—C10	176.8 (6)	C14—N4—C10—C2	−177.7 (5)
N1—C1—C2—N2	−0.6 (9)	Pd1—N4—C10—C2	6.0 (7)
C5—C1—C2—N2	171.4 (6)	N2—C2—C10—N4	129.4 (6)
N1—C1—C2—C10	−174.7 (6)	C1—C2—C10—N4	−56.1 (8)
C5—C1—C2—C10	−2.7 (10)	N2—C2—C10—C11	−47.2 (8)
C2—N2—C3—C4	−1.1 (11)	C1—C2—C10—C11	127.3 (7)
C1—N1—C4—C3	2.7 (10)	N4—C10—C11—C12	1.6 (10)
N2—C3—C4—N1	−1.3 (12)	C2—C10—C11—C12	178.1 (6)
C9—N3—C5—C6	2.8 (8)	C10—C11—C12—C13	−1.0 (10)
Pd1—N3—C5—C6	−170.7 (5)	C11—C12—C13—C14	0.1 (11)
C9—N3—C5—C1	−179.4 (5)	C10—N4—C14—C13	0.3 (9)
Pd1—N3—C5—C1	7.2 (7)	Pd1—N4—C14—C13	176.7 (5)
N1—C1—C5—N3	−135.7 (6)	C12—C13—C14—N4	0.3 (11)
C2—C1—C5—N3	51.9 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···I1^{i}	0.95	2.99	3.776 (7)	141

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+3/2.