Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(Di-2-pyridylamine-$\kappa^2N^2,N^{2'}$)diiodidoplatinum(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

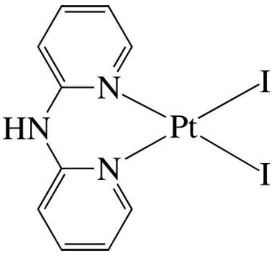
Received 10 March 2012; accepted 19 March 2012

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.013 Å; R factor = 0.031; wR factor = 0.077; data-to-parameter ratio = 17.4.

The Pt^{II} ion in the title complex, $[\mathrm{PtI_2(C_{10}H_9N_3)}]$, is four-coordinated in a distorted square-planar environment defined by the two pyridine N atoms of the chelating di-2-pyridylamine (dpa) ligand and by two I$^-$ anions. The dpa ligand is not planar, the dihedral angle between the pyridine rings being 52.8 (3)°. Pairs of complex molecules are assembled through intermolecular N—H$\cdots$I hydrogen bonds, forming a dimer-type species. The complexes are stacked in columns along the b axis and display several intermolecular π–π interactions between the pyridine rings, with a shortest ring centroid–centroid distance of 3.997 (5) Å.

Related literature

For the crystal structure of the related chlorido Pt^{II} complex $[\mathrm{PtCl_2(dpa)}]$, see: Li & Liu (2004); Tu *et al.* (2004); Zhang *et al.* (2006).



Experimental

Crystal data

$[\mathrm{PtI_2(C_{10}H_9N_3)}]$
M_r = 620.09
Monoclinic, $P2_1/n$
a = 8.2354 (6) Å
b = 9.7940 (7) Å
c = 16.4702 (12) Å
β = 102.148 (1)°
V = 1298.70 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 15.54 mm^{-1}
T = 200 K
0.16 × 0.12 × 0.08 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min}$ = 0.753, $T_{\max}$ = 1.000
7763 measured reflections
2527 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
S = 1.07
2527 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 1.47 e Å^{-3}
$\Delta\rho_{\min}$ = −1.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—N1	2.033 (7)	Pt1—I2	2.5675 (7)
Pt1—N3	2.055 (6)	Pt1—I1	2.5934 (7)
N1—Pt1—N3	85.9 (3)	I2—Pt1—I1	90.85 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$N\cdots$I1$^{\mathrm{i}}$	0.92	2.82	3.607 (7)	144

Symmetry code: (i) $-x, -y+2, -z$.

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 the Priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (2011–0030747).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2603).

References

Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Li, D. & Liu, D. (2004). *Cryst. Res. Technol.* **39**, 359–362.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tu, C., Wu, X., Liu, Q., Wang, X., Xu, Q. & Guo, Z. (2004). *Inorg. Chim. Acta*, **357**, 95–102.
Zhang, F., Prokopchuk, E. M., Broczkowski, M. E., Jennings, M. C. & Puddephatt, R. J. (2006). *Organometallics*, **25**, 1583–1591.

supplementary materials

Acta Cryst. (2012). E**68**, m479 [doi:10.1107/S1600536812011907]

(Di-2-pyridylamine-$\kappa^2N^2,N^{2\prime}$)diiodidoplatinum(II)

Kwang Ha

Comment

The title complex, [PtI_2(dpa)] (dpa = di-2-pyridylamine, $C_{10}H_9N_3$), is closely related with the previously reported analogous chlorido Pt^{II} complex [$PtCl_2$(dpa)] (Li & Liu, 2004; Tu *et al.*, 2004; Zhang *et al.*, 2006). The Pt^{II} ion is four-coordinated in a distorted square-planar environment by the two pyridine N atoms of the chelating dpa ligand and two I^- anions (Fig. 1). In the crystal, the dpa ligand is not planar. The dihedral angle between the least-squares planes of the pyridine rings is 52.8 (3)°. The two Pt—N and the two Pt—I bond lengths, respectively, are nearly equivalent (Table 1). Two complex molecules are assembled through intermolecular N—H···I hydrogen bonds, forming a dimer-type species (Fig. 2 and Table 2). The complexes are stacked in columns along the *b* axis and display several intermolecular π—π interactions between the pyridine rings, with a shortest ring centroid to centroid distance of 3.997 (5) Å.

Experimental

To a solution of K_2PtCl_4 (0.2082 g, 0.502 mmol) in H_2O (20 ml) and MeOH (10 ml) were added KI (0.7022 g, 4.230 mmol) and di-2-pyridylamine (0.0896 g, 0.523 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration and washed with H_2O and MeOH, and dried at 373 K, to give a yellow powder (0.2614 g). Crystals suitable for X-ray analysis were obtained by slow evaporation from a CH_3CN/acetone solution.

Refinement

Carbon-bound 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 nitrogen-bound H atom was located from Fourier difference maps and then allowed to ride on its parent atom in the final cycles of refinement with N—H = 0.92 Å and $U_{iso}(H) = 1.5\ U_{eq}(N)$. The highest peak (1.47 e Å^{-3}) and the deepest hole (-1.31 e Å^{-3}) in the difference Fourier map are located 0.56 Å and 0.67 Å from the atoms Pt1 and I1, respectively.

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); 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* (Sheldrick, 2008).

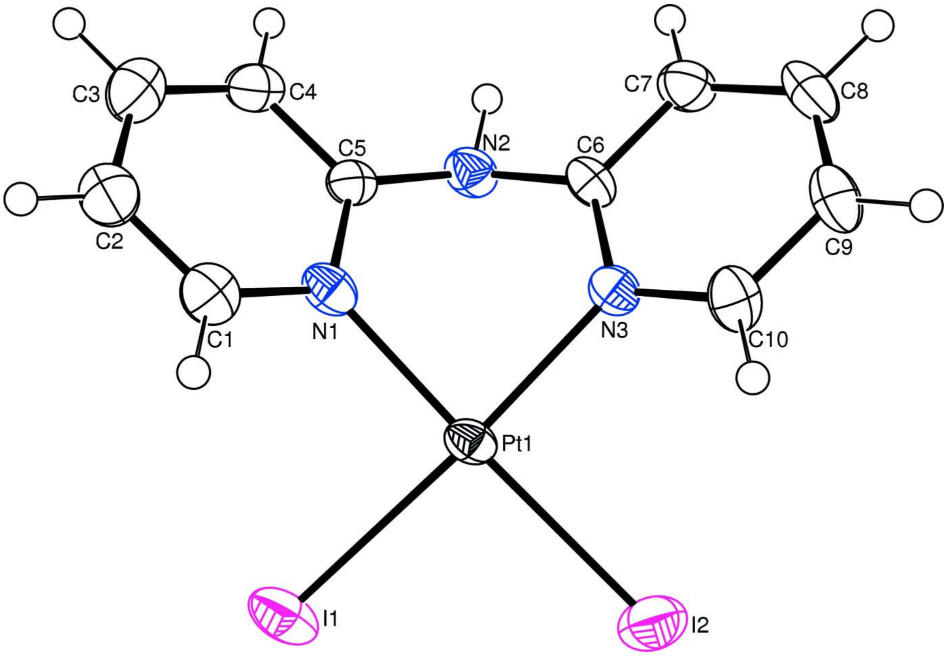


Figure 1

The molecular structure of the title complex, with displacement ellipsoids drawn at the 50% probability level for all non-H atoms.

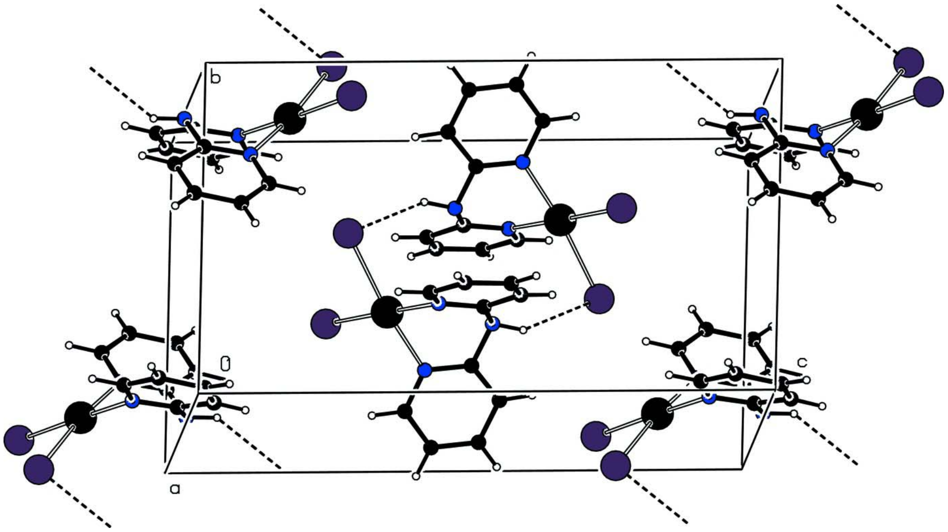


Figure 2

A view of the unit-cell content of the title complex. Intermolecular N—H···I hydrogen-bonding interactions are drawn with dashed lines.

(Di-2-pyridylamine-$\kappa^2N^2,N^{2'}$)diiodidoplatinum(II)

Crystal data

$[PtI_2(C_{10}H_9N_3)]$	Hall symbol: -P 2yn
M_r = 620.09	a = 8.2354 (6) Å
Monoclinic, $P2_1/n$	b = 9.7940 (7) Å

c = 16.4702 (12) Å
β = 102.148 (1)°
V = 1298.70 (16) Å^3
Z = 4
F(000) = 1096
D_x = 3.171 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 4677 reflections
θ = 2.4–26.0°
μ = 15.54 mm^{-1}
T = 200 K
Block, yellow
0.16 × 0.12 × 0.08 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} = 0.753, T_{max} = 1.000

7763 measured reflections
2527 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.0°, θ_{min} = 2.4°
$h = -10 \rightarrow 9$
$k = -12 \rightarrow 9$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.077$
S = 1.07
2527 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 7.7728P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.47 e Å^{-3}
$\Delta\rho_{min}$ = −1.31 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_{iso}*/U_{eq}
Pt1	0.06711 (4)	0.84628 (3)	0.151994 (17)	0.02326 (11)
I1	0.24006 (8)	1.04400 (6)	0.23202 (3)	0.04146 (18)
I2	−0.14376 (8)	0.86265 (7)	0.24634 (4)	0.04368 (18)
N1	0.2236 (8)	0.8338 (7)	0.0719 (4)	0.0272 (15)
N2	−0.0138 (9)	0.8279 (7)	−0.0353 (4)	0.0289 (15)
H2N	−0.0582	0.8189	−0.0911	0.043*
N3	−0.0752 (8)	0.7046 (7)	0.0769 (4)	0.0239 (14)
C1	0.3928 (11)	0.8266 (8)	0.0968 (6)	0.0326 (19)
H1	0.4403	0.8184	0.1543	0.039*
C2	0.4957 (11)	0.8309 (9)	0.0416 (6)	0.035 (2)

H2	0.6126	0.8244	0.0605	0.042*
C3	0.4278 (12)	0.8447 (8)	−0.0417 (6)	0.038 (2)
H3	0.4979	0.8505	−0.0806	0.046*
C4	0.2564 (11)	0.8501 (8)	−0.0688 (5)	0.0313 (19)
H4	0.2077	0.8616	−0.1260	0.038*
C5	0.1575 (10)	0.8384 (8)	−0.0103 (5)	0.0242 (17)
C6	−0.0991 (9)	0.7231 (8)	−0.0054 (5)	0.0232 (16)
C7	−0.2111 (11)	0.6449 (9)	−0.0613 (5)	0.035 (2)
H7	−0.2327	0.6645	−0.1191	0.041*
C8	−0.2908 (11)	0.5374 (9)	−0.0309 (6)	0.037 (2)
H8	−0.3666	0.4810	−0.0677	0.044*
C9	−0.2582 (11)	0.5140 (9)	0.0534 (6)	0.038 (2)
H9	−0.3101	0.4400	0.0753	0.046*
C10	−0.1502 (10)	0.5980 (9)	0.1058 (6)	0.0315 (19)
H10	−0.1278	0.5807	0.1638	0.038*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02847 (18)	0.02197 (18)	0.01700 (16)	−0.00104 (12)	−0.00054 (12)	0.00125 (12)
I1	0.0546 (4)	0.0355 (3)	0.0275 (3)	−0.0086 (3)	−0.0067 (3)	−0.0009 (3)
I2	0.0488 (4)	0.0540 (4)	0.0301 (3)	0.0015 (3)	0.0125 (3)	−0.0015 (3)
N1	0.031 (4)	0.023 (4)	0.024 (3)	−0.001 (3)	−0.001 (3)	0.001 (3)
N2	0.033 (4)	0.031 (4)	0.020 (3)	−0.004 (3)	−0.001 (3)	0.001 (3)
N3	0.023 (3)	0.023 (3)	0.024 (3)	0.003 (3)	0.002 (3)	0.002 (3)
C1	0.034 (5)	0.026 (4)	0.036 (5)	−0.002 (4)	0.002 (4)	0.000 (4)
C2	0.030 (5)	0.033 (5)	0.043 (5)	0.000 (4)	0.006 (4)	−0.002 (4)
C3	0.043 (5)	0.023 (5)	0.053 (6)	0.000 (4)	0.021 (5)	0.003 (4)
C4	0.039 (5)	0.030 (5)	0.026 (4)	0.000 (4)	0.011 (4)	−0.002 (4)
C5	0.034 (4)	0.017 (4)	0.023 (4)	−0.001 (3)	0.007 (3)	−0.002 (3)
C6	0.025 (4)	0.016 (4)	0.026 (4)	0.002 (3)	0.000 (3)	0.001 (3)
C7	0.036 (5)	0.039 (5)	0.027 (4)	−0.002 (4)	0.003 (4)	−0.004 (4)
C8	0.033 (5)	0.026 (5)	0.048 (6)	−0.005 (4)	0.000 (4)	−0.012 (4)
C9	0.042 (5)	0.023 (4)	0.048 (6)	−0.011 (4)	0.008 (4)	−0.005 (4)
C10	0.035 (5)	0.023 (4)	0.036 (5)	0.000 (4)	0.005 (4)	0.006 (4)

Geometric parameters (Å, º)

Pt1—N1	2.033 (7)	C2—H2	0.9500
Pt1—N3	2.055 (6)	C3—C4	1.390 (13)
Pt1—I2	2.5675 (7)	C3—H3	0.9500
Pt1—I1	2.5934 (7)	C4—C5	1.391 (12)
N1—C5	1.349 (10)	C4—H4	0.9500
N1—C1	1.369 (11)	C6—C7	1.389 (11)
N2—C5	1.388 (10)	C7—C8	1.388 (13)
N2—C6	1.390 (10)	C7—H7	0.9500
N2—H2N	0.9196	C8—C9	1.377 (13)
N3—C6	1.340 (10)	C8—H8	0.9500
N3—C10	1.349 (10)	C9—C10	1.375 (12)
C1—C2	1.368 (13)	C9—H9	0.9500

C1—H1	0.9500	C10—H10	0.9500
C2—C3	1.374 (13)		

N1—Pt1—N3	85.9 (3)	C4—C3—H3	120.1
N1—Pt1—I2	176.88 (18)	C3—C4—C5	118.6 (8)
N3—Pt1—I2	91.94 (18)	C3—C4—H4	120.7
N1—Pt1—I1	91.11 (18)	C5—C4—H4	120.7
N3—Pt1—I1	173.33 (18)	N1—C5—N2	117.7 (7)
I2—Pt1—I1	90.85 (2)	N1—C5—C4	121.7 (8)
C5—N1—C1	118.2 (7)	N2—C5—C4	120.5 (7)
C5—N1—Pt1	118.2 (5)	N3—C6—C7	122.1 (7)
C1—N1—Pt1	123.6 (6)	N3—C6—N2	118.7 (7)
C5—N2—C6	120.5 (6)	C7—C6—N2	119.1 (7)
C5—N2—H2N	117.9	C8—C7—C6	118.5 (8)
C6—N2—H2N	99.2	C8—C7—H7	120.7
C6—N3—C10	118.8 (7)	C6—C7—H7	120.7
C6—N3—Pt1	117.5 (5)	C9—C8—C7	118.9 (8)
C10—N3—Pt1	123.7 (6)	C9—C8—H8	120.6
C2—C1—N1	122.2 (8)	C7—C8—H8	120.6
C2—C1—H1	118.9	C10—C9—C8	119.8 (8)
N1—C1—H1	118.9	C10—C9—H9	120.1
C1—C2—C3	119.2 (8)	C8—C9—H9	120.1
C1—C2—H2	120.4	N3—C10—C9	121.7 (8)
C3—C2—H2	120.4	N3—C10—H10	119.2
C2—C3—C4	119.7 (9)	C9—C10—H10	119.2
C2—C3—H3	120.1		

N3—Pt1—N1—C5	46.4 (6)	C6—N2—C5—N1	−50.2 (10)
I1—Pt1—N1—C5	−127.7 (6)	C6—N2—C5—C4	128.4 (8)
N3—Pt1—N1—C1	−136.3 (6)	C3—C4—C5—N1	5.8 (12)
I1—Pt1—N1—C1	49.6 (6)	C3—C4—C5—N2	−172.7 (7)
N1—Pt1—N3—C6	−44.0 (6)	C10—N3—C6—C7	6.6 (12)
I2—Pt1—N3—C6	133.8 (5)	Pt1—N3—C6—C7	−170.9 (6)
N1—Pt1—N3—C10	138.6 (7)	C10—N3—C6—N2	−176.1 (7)
I2—Pt1—N3—C10	−43.6 (6)	Pt1—N3—C6—N2	6.4 (9)
C5—N1—C1—C2	3.4 (12)	C5—N2—C6—N3	52.8 (10)
Pt1—N1—C1—C2	−173.9 (6)	C5—N2—C6—C7	−129.9 (8)
N1—C1—C2—C3	0.9 (13)	N3—C6—C7—C8	−5.1 (13)
C1—C2—C3—C4	−1.9 (13)	N2—C6—C7—C8	177.6 (8)
C2—C3—C4—C5	−1.3 (12)	C6—C7—C8—C9	1.1 (13)
C1—N1—C5—N2	171.8 (7)	C7—C8—C9—C10	1.1 (14)
Pt1—N1—C5—N2	−10.8 (9)	C6—N3—C10—C9	−4.2 (12)
C1—N1—C5—C4	−6.7 (11)	Pt1—N3—C10—C9	173.1 (7)
Pt1—N1—C5—C4	170.7 (6)	C8—C9—C10—N3	0.5 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N2—H2*N*···I1^{i}	0.92	2.82	3.607 (7)	144

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