

Dibromidodimethyldipyridine-platinum(IV)

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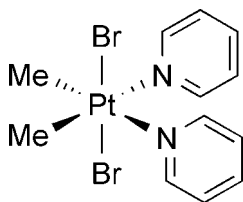
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 Key indicators: single-crystal X-ray study; $T = 220$ K; mean $\sigma(\text{C}-\text{C}) = 0.010$ Å; R factor = 0.037; wR factor = 0.091; data-to-parameter ratio = 18.2.

In the title complex, $[\text{PtBr}_2(\text{CH}_3)_2(\text{C}_5\text{H}_5\text{N})_2]$, the Pt^{IV} metal centre lies on a twofold rotation axis and adopts a slightly distorted octahedral coordination geometry. The structure displays weak intramolecular $\text{C}-\text{H}\cdots\text{Br}$ hydrogen-bonding interactions.

Related literature

For the crystal structures of related compounds, see: Brammer *et al.* (2001); Burton *et al.* (1983); Canty *et al.* (1990); Clark *et al.* (1983); Contreras *et al.* (2001); Hall & Swile (1971); Hindmarch *et al.* (1997); Hughes *et al.* (2001); Kaluderović *et al.* (2007); Kelly, Gómez-Ruiz, Kluge *et al.* (2008); Kelly, Gómez-Ruiz, Schmidt *et al.* (2008); Kelly, Dietrich *et al.* (2008); Klingler *et al.* (1982). For bond-length data, see: Allen (2002).



Experimental

Crystal data

$[\text{PtBr}_2(\text{CH}_3)_2(\text{C}_5\text{H}_5\text{N})_2]$	$V = 1490.1$ (5) Å ³
$M_r = 543.18$	$Z = 4$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation
$a = 13.297$ (2) Å	$\mu = 14.76$ mm ⁻¹
$b = 8.2906$ (15) Å	$T = 220$ (2) K
$c = 13.516$ (3) Å	$0.40 \times 0.34 \times 0.30$ mm

Data collection

Stoe IPDS diffractometer	10568 measured reflections
Absorption correction: numerical (<i>IPDS</i> ; Stoe & Cie, 1999)	1453 independent reflections
$T_{\text{min}} = 0.024$, $T_{\text{max}} = 0.069$	1166 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.144$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	80 parameters
$wR(F^2) = 0.091$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 1.71$ e Å ⁻³
1453 reflections	$\Delta\rho_{\text{min}} = -1.68$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C6}-\text{H8}\cdots\text{Br}^{\text{i}}$	0.93	2.92	3.412 (6)	115

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

Data collection: *IPDS Software* (Stoe & Cie, 1999); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: RZ2250).

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supplementary materials

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Comment

The structure of the title compound is one of a relatively small number of structures with the PtBr₂Me₂ moiety (Contreras *et al.*, 2001; Kaluderović *et al.*, 2007; Kelly, Gómez-Ruiz, Kluge *et al.*, 2008; Kelly, Gómez-Ruiz, Schmidt *et al.*, 2008; Kelly, Dietrich *et al.*, 2008). The compound crystallizes in the orthorhombic space group *Pbcn* and half the molecule is generated by a twofold crystallographic axis bisecting the C—Pt—N axis as illustrated in Fig. 1. The ligating atoms have an approximate octahedral arrangement around the platinum atom. The Pt—N bond length (2.226 (5) Å) is slightly longer than expected for a platinum(IV)—N bond *trans*-configured to a ligating carbon atom (median: 2.156 Å; lower/upper quartile: 2.135/2.194 Å for 402 entries in the Cambridge Structural Database; CSD, Version 5.28, August 2007; Allen, 2002). The Pt—Br bond length (2.461 (1) Å) and the Pt—C bond length (2.053 (7) Å) are typical for bonds of these types (Clark *et al.*, 1983; Klingler *et al.*, 1982; Burton *et al.*, 1983; Hughes *et al.*, 2001; Cauty *et al.*, 1990; Hindmarch *et al.*, 1997; Kelly, Gómez-Ruiz, Kluge *et al.*, 2008; Kelly, Gómez-Ruiz, Schmidt *et al.*, 2008; Kelly, Dietrich *et al.*, 2008). A short intramolecular distance between the C6 carbon atom of the pyridine ligand and a bromo ligand of the same molecule is found, indicating the presence of weak C—H...Br interactions (Brammer *et al.*, 2001).

Experimental

The title compound was prepared by dissolving [(PtBr₂Me₂)_n] in an excess of pyridine (Hall & Swile, 1971). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a chloroform solution.

Refinement

H atoms were positioned geometrically and treated as riding, with C—H bonding lengths constrained to 0.93–0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The poor quality of the crystal may account for the rather high R_{int} value.

Figures

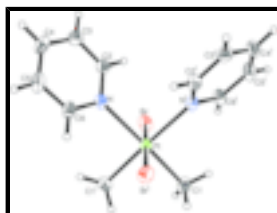


Fig. 1. A view of the title complex with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) $-x, y, -z + 3/2z$].

Dibromidodimethyldipyridineplatinum(IV)

Crystal data

[PtBr₂(CH₃)₂(C₅H₅N)₂]

$M_r = 543.18$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 13.297 (2) \text{ \AA}$

$b = 8.2906 (15) \text{ \AA}$

$c = 13.516 (3) \text{ \AA}$

$V = 1490.1 (5) \text{ \AA}^3$

$Z = 4$

$F_{000} = 1000$

$D_x = 2.421 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8000 reflections

$\theta = 2.2\text{--}25.9^\circ$

$\mu = 14.76 \text{ mm}^{-1}$

$T = 220 (2) \text{ K}$

Block, orange

$0.40 \times 0.34 \times 0.30 \text{ mm}$

Data collection

Stoe IPDS
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 220(2) \text{ K}$

area detector scans

Absorption correction: numerical
(IPDS; Stoe & Cie, 1999)

$T_{\min} = 0.024$, $T_{\max} = 0.069$

10568 measured reflections

1453 independent reflections

1166 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.144$

$\theta_{\text{max}} = 26.0^\circ$

$\theta_{\text{min}} = 2.9^\circ$

$h = -16 \rightarrow 16$

$k = -10 \rightarrow 10$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.091$

$S = 1.01$

1453 reflections

80 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2]$$

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

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 1.71 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -1.68 \text{ e \AA}^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0012 (2)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0667 (6)	-0.0872 (9)	0.6693 (6)	0.0487 (17)
H3	0.1207	-0.1336	0.7069	0.058*
H1	0.0928	-0.0434	0.6087	0.058*
H2	0.0179	-0.1690	0.6544	0.058*
C2	-0.1238 (5)	0.4045 (8)	0.7975 (5)	0.0412 (15)
H4	-0.1268	0.4089	0.7288	0.049*
C3	-0.1703 (5)	0.5237 (9)	0.8514 (6)	0.0479 (17)
H5	-0.2038	0.6070	0.8191	0.057*
C4	-0.1673 (5)	0.5196 (10)	0.9542 (6)	0.0526 (19)
H6	-0.1976	0.6001	0.9918	0.063*
C5	-0.1182 (5)	0.3930 (10)	0.9987 (6)	0.057 (2)
H7	-0.1161	0.3847	1.0673	0.068*
C6	-0.0716 (4)	0.2771 (9)	0.9396 (4)	0.0423 (15)
H8	-0.0373	0.1928	0.9699	0.051*
N	-0.0747 (3)	0.2833 (7)	0.8400 (3)	0.0355 (11)
Br	-0.14288 (5)	0.08616 (9)	0.63441 (5)	0.0465 (2)
Pt	0.0000	0.09312 (4)	0.7500	0.03167 (18)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.055 (4)	0.047 (4)	0.044 (4)	-0.004 (3)	0.007 (3)	-0.012 (3)
C2	0.039 (3)	0.045 (4)	0.039 (4)	0.002 (3)	-0.001 (3)	0.003 (3)
C3	0.036 (3)	0.044 (4)	0.064 (5)	0.000 (3)	0.004 (3)	0.003 (4)
C4	0.035 (3)	0.062 (5)	0.061 (5)	-0.001 (3)	0.010 (3)	-0.023 (4)
C5	0.045 (4)	0.087 (7)	0.038 (4)	0.002 (3)	0.006 (3)	-0.010 (4)
C6	0.034 (3)	0.063 (4)	0.030 (3)	0.007 (3)	-0.002 (2)	-0.004 (3)
N	0.029 (2)	0.047 (3)	0.030 (3)	0.000 (2)	0.0029 (19)	0.003 (2)
Br	0.0385 (3)	0.0661 (5)	0.0347 (4)	-0.0103 (3)	-0.0087 (3)	0.0010 (3)
Pt	0.0312 (2)	0.0397 (2)	0.0242 (3)	0.000	-0.00189 (11)	0.000

supplementary materials

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

C1—Pt	2.053 (7)	C4—H6	0.9300
C1—H3	0.9600	C5—C6	1.394 (10)
C1—H1	0.9600	C5—H7	0.9300
C1—H2	0.9600	C6—N	1.347 (7)
C2—N	1.329 (8)	C6—H8	0.9300
C2—C3	1.375 (10)	N—Pt	2.226 (5)
C2—H4	0.9300	Br—Pt	2.4605 (7)
C3—C4	1.391 (10)	Pt—C1 ⁱ	2.053 (7)
C3—H5	0.9300	Pt—N ⁱ	2.226 (5)
C4—C5	1.375 (11)	Pt—Br ⁱ	2.4605 (7)
Pt—C1—H3	109.5	C5—C6—H8	118.9
Pt—C1—H1	109.5	C2—N—C6	118.4 (6)
H3—C1—H1	109.5	C2—N—Pt	121.2 (4)
Pt—C1—H2	109.5	C6—N—Pt	120.4 (5)
H3—C1—H2	109.5	C1—Pt—C1 ⁱ	86.5 (4)
H1—C1—H2	109.5	C1—Pt—N	178.4 (2)
N—C2—C3	122.4 (7)	C1 ⁱ —Pt—N	91.9 (3)
N—C2—H4	118.8	C1—Pt—N ⁱ	91.9 (3)
C3—C2—H4	118.8	C1 ⁱ —Pt—N ⁱ	178.4 (2)
C2—C3—C4	119.9 (7)	N—Pt—N ⁱ	89.8 (3)
C2—C3—H5	120.0	C1—Pt—Br ⁱ	89.2 (2)
C4—C3—H5	120.0	C1 ⁱ —Pt—Br ⁱ	88.8 (2)
C5—C4—C3	117.9 (7)	N—Pt—Br ⁱ	90.80 (12)
C5—C4—H6	121.0	N ⁱ —Pt—Br ⁱ	91.11 (12)
C3—C4—H6	121.0	C1—Pt—Br	88.8 (2)
C4—C5—C6	119.1 (7)	C1 ⁱ —Pt—Br	89.2 (2)
C4—C5—H7	120.4	N—Pt—Br	91.11 (12)
C6—C5—H7	120.4	N ⁱ —Pt—Br	90.80 (12)
N—C6—C5	122.2 (7)	Br ⁱ —Pt—Br	177.31 (4)
N—C6—H8	118.9		
N—C2—C3—C4	-0.3 (10)	C5—C6—N—Pt	-179.4 (5)
C2—C3—C4—C5	-0.9 (11)	C2—N—Pt—N ⁱ	49.9 (4)
C3—C4—C5—C6	1.6 (11)	C6—N—Pt—N ⁱ	-130.6 (6)
C4—C5—C6—N	-1.3 (11)	C2—N—Pt—Br ⁱ	141.0 (4)
C3—C2—N—C6	0.7 (9)	C6—N—Pt—Br ⁱ	-39.5 (5)
C3—C2—N—Pt	-179.8 (5)	C2—N—Pt—Br	-40.9 (4)
C5—C6—N—C2	0.1 (10)	C6—N—Pt—Br	138.6 (5)

Symmetry codes: (i) $-x, y, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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C6—H8···Brⁱ

0.93

2.92

3.412 (6)

115

Symmetry codes: (i) $-x, y, -z+3/2$.

Fig. 1

