cis-Diiodido(N,N,N′,N′-tetramethyl-ethylenediamine-κ²N,N′)platinum(II)

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In the title complex, cis-[PtI$_2$(C$_6$H$_{16}$N$_2$)], the Pt atom lies on a crystallographic twofold axis and the molecule exhibits C_2 symmetry. The Pt atom is four-coordinated by a chelating N,N,N′,N′-tetramethylethylenediamine ligand [Pt—N = 2.081 (9) Å] and two iodide ligands [Pt—I = 2.589 (2) Å], forming a distorted square-planar geometry.

Related literature

For related literature, see: Connick & Gray (1994); Deacon *et al.* (1991); Hughes *et al.* (2004); Preut *et al.* (1991); Łakomska *et al.* (2004, 2007).
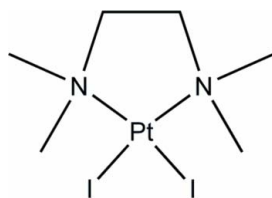
Experimental

Crystal data

[PtI$_2$(C$_6$H$_{16}$N$_2$)]
$M_r = 565.10$
Monoclinic, $C2/c$
$a = 7.937$ (7) Å
$b = 14.733$ (14) Å
$c = 10.430$ (10) Å
$\beta = 107.513$ (17)°

$V = 1163.1$ (19) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 17.32$ mm^{-1}
$T = 100$ (2) K
$0.14 \times 0.10 \times 0.03$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.145$, $T_{max} = 0.593$

3585 measured reflections
1359 independent reflections
1175 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.105$
$S = 1.07$
1359 reflections
53 parameters

12 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 3.38$ e Å^{-3}
$\Delta\rho_{min} = -2.98$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—N1	2.081 (9)	Pt1—I1	2.5891 (18)
N1—Pt1—N1^i	84.1 (5)	I1—Pt1—I1^i	88.02 (8)
N1^i—Pt1—I1	94.0 (3)		

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

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supplementary materials

cis-Diiodido(*N*,*N*,*N'*,*N'*-tetramethylethylenediamine-$\kappa^2 N$,*N'*)platinum(II)

I. Lakomska and L. Dobrzanska

Comment

During the course of our ongoing studies on the design of new antitumor prodrugs related to cisplatin Łakomska *et al.*, 2007;Łakomska *et al.*, 2004) we have isolated the title compound (I) (Fig. 1). The asymmetric unit comprises a half of the molecule as the Pt atom lies on a crystallographic twofold axis. The crystal structure displays a distorted square-planar geometry around Pt(II) which is coordinated by a chelating *N*,*N*,*N'*,*N'*-tetramethylethylenediamine tmeda ligand (dihedral angle of N—C—C—N = −53.1 (12)°) and two iodo ligands. The largest deviation from the ideal geometry is imposed by N1—Pt—N1[i] angle of 84.1 (5)° (symmetry code:(i) −*x*, *y*, −*z* + 3/2), which reflects geometric constrains introduced by the bite angle of the ligand. This value is similar to the corresponding ones of 85.0 (5) and 84.0 (3)° in the cationic complex *cis*-[Pt(1-methylcytosine)$_2$(tmeda)]$^{2+}$ (Preut *et al.*, 1991) and *cis*-[Pt(pentafluorophenyl)$_2$(tmeda)] (Deacon *et al.*, 1991), respectively. The coordinated ligand atoms and Pt(II) are coplanar within the limits of experimentl errors: I1 and N1 are displaced from the least square plane defined by five atoms (Pt I$_2$N$_2$) by −0.003 (2) and −0.004 (2) Å, respectively. Pt—I, Pt—N and the diamine ring distances compare well with the previously reported values (Hughes *et al.*, 2004; Connick & Gray, 1994). The crystal structure of (I) is defined by the van der Waals interactions (Fig. 2).

Experimental

To a solution of K$_2$PtCl$_4$ (1.605 g; 3.9 mmol) in 20 cm^3 of water, an aqueous solution of KI (3.212 g, 19.3 mmol) was added and the reaction mixture was stirred at room temperature for 1 h. Then 0.88 cm^3 (5.9 mmol) of *N*,*N*,*N'*,*N'*-tetramethyl-ethylenediamine (tmeda) was added dropwise, with vigorous stirring. The yellow-brown precipitate was filtered, washed several times with water and ethanol and dried under vacuum. Yield 1.938 g (88%). Analysis: calcd/exp. for C$_6$H$_{16}$I$_2$N$_2$Pt: C 12.8/12.7; N 5.0/4.9; H, 2.9/2.8. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from a water:methanol:2-propanol mixture (2:1:1).

Refinement

H atoms were positioned geometrically (C—H = 0.98 and 0.99 Å) and constrained to ride on their parent atoms; U_{iso}(H) values were fixed at 1.2 times U_{eq}(C) (1.5 times for methyl H atoms).
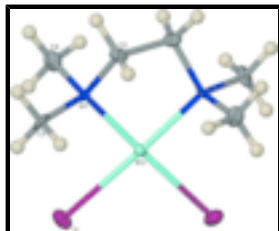
supplementary materials

Figures

Fig. 1. The molecular structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to the labelled atoms by the symmetry operation $-x, y, -z + 3/2$. H atoms are shown as spheres of arbitrary radii.
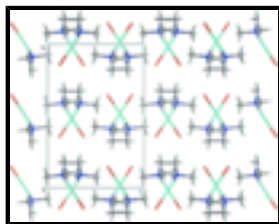

Fig. 2. Packing diagram of (I) viewed along [100].

cis-**Diiodido(*N,N,N',N'*-tetramethylethylenediamine-κ^2*N,N'*)platinum(II)**

Crystal data

[PtI$_2$(C$_6$H$_{16}$N$_2$)]	$Z = 4$
$M_r = 565.10$	$F_{000} = 1000$
Monoclinic, $C2/c$	$D_x = 3.227$ Mg m^{-3}
Hall symbol: -C 2yc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.937$ (7) Å	$\mu = 17.32$ mm^{-1}
$b = 14.733$ (14) Å	$T = 100$ (2) K
$c = 10.430$ (10) Å	Plate, colourless
$\beta = 107.513$ (17)º	$0.14 \times 0.10 \times 0.03$ mm
$V = 1163.1$ (19) Å^3	

Data collection

Bruker APEX CCD area-detector diffractometer	1359 independent reflections
Radiation source: fine-focus sealed tube	1175 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.051$
$T = 100(2)$ K	$\theta_{max} = 28.3º$
ω scans	$\theta_{min} = 2.8º$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$h = -10 \rightarrow 10$
$T_{min} = 0.145$, $T_{max} = 0.593$	$k = -19 \rightarrow 16$
3585 measured reflections	$l = -13 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.105$

$S = 1.07$

1359 reflections

53 parameters

12 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 24.7364P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 3.38$ e Å^{-3}

$\Delta\rho_{min} = -2.98$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pt1	0.0000	0.01766 (4)	0.7500	0.01035 (18)
I1	0.21455 (11)	0.14405 (5)	0.87331 (8)	0.0226 (2)
N1	−0.1649 (12)	−0.0872 (6)	0.6530 (9)	0.0125 (19)
C2	−0.0594 (15)	−0.1730 (8)	0.6785 (12)	0.018 (2)
H2A	0.0124	−0.1773	0.6158	0.021*
H2B	−0.1396	−0.2260	0.6632	0.021*
C4	−0.3189 (16)	−0.0968 (9)	0.7077 (12)	0.022 (3)
H4A	−0.3890	−0.1500	0.6673	0.033*
H4B	−0.3928	−0.0423	0.6859	0.033*
H4C	−0.2753	−0.1044	0.8055	0.033*
C3	−0.2406 (15)	−0.0778 (8)	0.5058 (12)	0.020 (3)
H3A	−0.1448	−0.0725	0.4650	0.031*
H3B	−0.3147	−0.0233	0.4853	0.031*
H3C	−0.3124	−0.1313	0.4694	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.0099 (3)	0.0105 (3)	0.0116 (3)	0.000	0.0048 (2)	0.000
I1	0.0261 (5)	0.0206 (4)	0.0206 (5)	−0.0096 (3)	0.0061 (4)	−0.0026 (3)
N1	0.012 (2)	0.011 (2)	0.014 (2)	−0.0021 (18)	0.0025 (18)	−0.0021 (17)

C2	0.019 (3)	0.016 (3)	0.018 (3)	0.0004 (19)	0.0054 (19)	0.0004 (19)
C4	0.021 (6)	0.032 (7)	0.012 (6)	0.003 (6)	0.005 (5)	0.001 (5)
C3	0.015 (6)	0.023 (6)	0.021 (6)	−0.013 (5)	0.003 (5)	0.000 (5)

Geometric parameters (Å, °)

Pt1—N1	2.081 (9)	C2—H2A	0.9900
Pt1—N1^i	2.081 (9)	C2—H2B	0.9900
Pt1—I1	2.5891 (18)	C4—H4A	0.9800
Pt1—I1^i	2.5891 (18)	C4—H4B	0.9800
N1—C3	1.478 (15)	C4—H4C	0.9800
N1—C2	1.495 (14)	C3—H3A	0.9800
N1—C4	1.503 (14)	C3—H3B	0.9800
C2—C2^i	1.51 (2)	C3—H3C	0.9800
N1—Pt1—N1^i	84.1 (5)	C2^i—C2—H2B	109.9
N1—Pt1—I1	177.9 (3)	H2A—C2—H2B	108.3
N1^i—Pt1—I1	94.0 (3)	N1—C4—H4A	109.5
N1^i—Pt1—I1^i	177.9 (3)	N1—C4—H4B	109.5
I1—Pt1—I1^i	88.02 (8)	H4A—C4—H4B	109.5
C3—N1—C2	107.2 (9)	N1—C4—H4C	109.5
C3—N1—C4	106.3 (9)	H4A—C4—H4C	109.5
C2—N1—C4	109.2 (9)	H4B—C4—H4C	109.5
C3—N1—Pt1	115.8 (7)	N1—C3—H3A	109.5
C2—N1—Pt1	107.7 (7)	N1—C3—H3B	109.5
C4—N1—Pt1	110.5 (7)	H3A—C3—H3B	109.5
N1—C2—C2^i	109.1 (7)	N1—C3—H3C	109.5
N1—C2—H2A	109.9	H3A—C3—H3C	109.5
C2^i—C2—H2A	109.9	H3B—C3—H3C	109.5
N1—C2—H2B	109.9		
N1^i—Pt1—N1—C3	−133.8 (9)	I1^i—Pt1—N1—C4	−74.1 (7)
I1^i—Pt1—N1—C3	46.8 (8)	C3—N1—C2—C2^i	164.6 (10)
N1^i—Pt1—N1—C2	−13.9 (5)	C4—N1—C2—C2^i	−80.7 (13)
I1^i—Pt1—N1—C2	166.7 (6)	Pt1—N1—C2—C2^i	39.4 (12)
N1^i—Pt1—N1—C4	105.3 (8)		

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

Fig. 1

Fig. 2