

trans-Tetrachloridobis(pyrazine- κ N)-platinum(IV)

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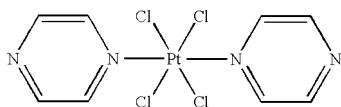
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.053; wR factor = 0.154; data-to-parameter ratio = 17.6.

The molecule of the title compound, $[\text{PtCl}_4(\text{C}_4\text{H}_4\text{N}_2)_2]$, possesses mmm symmetry. The Pt^{IV} atom is six-coordinated in an octahedral configuration by two N atoms of two pyrazine rings and four Cl atoms. In the crystal structure, there are $\pi-\pi$ interactions between the pyrazine rings [the closest distance between adjacent rings is 3.6485 (7) Å].

Related literature

For related literature, see: Hedin (1886); Joergensen (1900); Bajusz et al. (1989); Vorobevdesyatovskii et al. (1991); Junicke et al. (1997); Khripun et al. (2006); Witkowski et al. (1997); Kuduk-Jaworska et al. (1988, 1990); Bokach et al. (2003); Kukushkin et al. (1998); Garnovskii et al. (2001); Luzyanin, Kukushkin et al. (2002); Gonzalez et al. (2002); Luzyanin, Haukka et al. (2002); Gaballa et al. (2003); Casas et al. (2005); Hambley (1986); Hafizovic et al. (2006); Crowder et al. (2004).

**Experimental***Crystal data*

$[\text{PtCl}_4(\text{C}_4\text{H}_4\text{N}_2)_2]$

$M_r = 497.06$

Orthorhombic, $Cmca$

$a = 7.2107$ (7) Å

$b = 14.6318$ (18) Å

$c = 11.8392$ (11) Å

$V = 1249.1$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 12.07$ mm⁻¹

$T = 120$ (2) K

$0.25 \times 0.25 \times 0.08$ mm

Data collection

Stoe IPDSII diffractometer

Absorption correction: numerical

[shape of crystal determined

optically (X -RED; Stoe & Cie, 2005)]

$T_{\text{min}} = 0.070$, $T_{\text{max}} = 0.380$

6750 measured reflections
898 independent reflections

883 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.099$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.154$

$S = 1.38$

898 reflections

51 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 4.88$ e Å⁻³

$\Delta\rho_{\text{min}} = -3.22$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pt1—Cl1	2.3186 (10)	N1—Pt1	2.047 (5)
Cl1 ⁱ —Pt1—Cl1 ⁱⁱ	90.38 (5)	N1—Pt1—Cl1	90.52 (10)
Cl1—Pt1—Cl1 ⁱⁱⁱ	89.62 (5)	Cl1 ⁱ —Pt1—Cl1	180
Cl1 ⁱⁱ —Pt1—Cl1 ⁱⁱⁱ	180	N1—Pt1—N1 ⁱ	180
N1—Pt1—Cl1 ⁱ	89.48 (10)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, -y, -z$; (iii) $-x, y, z$.

Data collection: X -AREA (Stoe & Cie, 2005); cell refinement: X -AREA; data reduction: X -RED (Stoe & Cie, 2005); program(s) used to solve structure: $SHELXS97$ (Sheldrick, 1997); program(s) used to refine structure: $SHELXL97$ (Sheldrick, 1997); molecular graphics: $ORTEP-3$ for Windows (Farrugia, 1997); software used to prepare material for publication: $WinGX$ (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2345).

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supplementary materials

Acta Cryst. (2007). E63, m2869-m2870 [doi:10.1107/S1600536807052555]

***trans*-Tetrachloridobis(pyrazine- κ N)platinum(IV)**

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Comment

Amine platinum(IV) complexes have been known since the end of the last century (Hedin, 1886; Joergensen, 1900). Some of them have cancerostatic properties from which new interest aroused in these complexes (Bajusz *et al.*, 1989; Vorobevdesyatovskii *et al.*, 1991). Due to the kinetic inertness of hexachloro-platinate(IV), *cis*- and *trans*-[PtCl₄L₂] complexes (*L* = N, O, P, S donor ligands) were mainly prepared by oxidation reactions of the corresponding platinum(II) complexes [PtCl₂L₂], (Hedin, 1886; Joergensen, 1900). Several Pt^{IV} complexes, with formula, [PtCl₄L₂], such as *cis*- and *trans*-[PtCl₄(py)₂], (II), (Junicke *et al.*, 1997), *cis*- and *trans*-[PtCl₄(PzH)₂], (III), (Khripun *et al.*, 2006), *trans*-[PtCl₄(NH₃)₂](1-Mu), (IV), (Witkowski *et al.*, 1997), *trans*-[PtCl₄(1-Prim)₂], (V), (Kuduk-Jaworska *et al.*, 1988), *cis*-[PtCl₄(1-Etim)₂], (VI), (Kuduk-Jaworska *et al.*, 1990), *trans*-[PtCl₄{NH=C(NMe₂)OH}₂], (VII), (Bokach *et al.*, 2003), *trans*-[PtCl₄{NH=C(Me)ON=CMe₂}₂], (VIII), (Kukushkin *et al.*, 1998), *cis*-[PtCl₄{NH=C(Et)N=CPh₂}₂], (IX), (Garnovskii *et al.*, 2001), *trans*-[PtCl₄{NH=C(Et)ON=C(OH)Ph}₂].2DMSO, (X), (Luzyanin, Kukushkin *et al.*, 2002), *trans*-[PtCl₄{NH=C(OMe)Bu^t}₂], (XI), (Gonzalez *et al.*, 2002) and *trans*-[PtCl₄{NH=C(OH)Et}₂], (XII), (Luzyanin, Haukka *et al.*, 2002) [where PzH is pyrazole, 1-Mu is 1-methyluracil, 1-Prim is 1-propylimidazole and 1-Etim is 1-ethylimidazolyl] have been synthesized and characterized by single-crystal X-ray diffraction methods.

There are also several Pt^{IV} complexes, with formula, [PtCl₄(N—N)], such as [PtCl₄(bipy)], (XIII), (Gaballa *et al.*, 2003), [PtCl₄(Me₂bim)], (XIV), (Casas *et al.*, 2005), [PtCl₄(bipy)], (XV), (Hambley, 1986), [PtCl₄(dcbipy)].H₂O, (XVI), (Hafizovic *et al.*, 2006) and [PtCl₄(dpk)], (XVII), (Crowder *et al.*, 2004) [where bipy is 2,2'-bipyrimidinyl, Me₂bim is 1,1'-dimethyl-2,2'-bi-imidazolyl, bipy is 2,2'-bipyridine, dcbipy is 2,2'-bipyridine-5,5'-dicarboxylic acid and dpk is bis(2-pyridyl)ketone] have been synthesized and characterized by single-crystal X-ray diffraction methods. We report herein the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1), contains one pyrazine and one chlorido-platinate ligands. The Pt^{IV} atom is six-coordinated in octahedral configuration by two N atoms of two pyrazine and four Cl atoms of tetrachloridoplatinate ligands. The Pt—Cl and Pt—N bond lengths and angles (Table 1) are in good agreement with the corresponding values in (II) and (VI).

In the crystal structure (Fig. 2), there are some π - π interactions between the pyrazine rings, in which they may be effective in the stabilization of the structure. The distance between the adjacent rings is 3.6485 (7) Å [symmetry codes: $-1/2 - x, y, 1/2 - z$; $1/2 - x, y, 1/2 - z$; $-1/2 + x, y, 1/2 - z$; $1/2 + x, y, 1/2 - z$].

Experimental

For the preparation of the title compound, (I), a solution of pyrazine (60 mg, 0.74 mmol) in methanol (10 ml) was added to a solution of H₂PtCl₆.2H₂O (200 mg, 0.37 mmol) in methanol (10 ml) at room temperature. The suitable crystals for

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X-ray analysis were obtained by methanol diffusion in a solution of the yellow precipitate in DMSO, after one week (yield; 150 mg, 81.6%).

Refinement

H atoms were positioned geometrically, with C—H = 0.93 Å, for aromatic H atoms and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

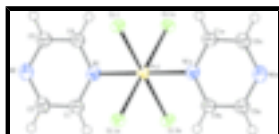


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry codes: (a) $-x, -y, -z$; (b) $x, -y, -z$; (c) $-x, y, z$].

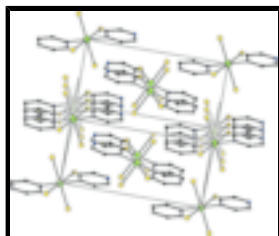


Fig. 2. A packing diagram of (I).

trans-Tetrachloridobis(pyrazine- κ N)platinum(IV)

Crystal data

[PtCl₄(C₄H₄N₂)₂]

$M_r = 497.06$

Orthorhombic, *Cmca*

Hall symbol: -C 2bc 2

$a = 7.2107 (7) \text{ \AA}$

$b = 14.6318 (18) \text{ \AA}$

$c = 11.8392 (11) \text{ \AA}$

$V = 1249.1 (2) \text{ \AA}^3$

$Z = 4$

$F_{000} = 920$

$D_x = 2.643 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 2500 reflections

$\theta = 3.3\text{--}29.2^\circ$

$\mu = 12.07 \text{ mm}^{-1}$

$T = 120 (2) \text{ K}$

Plate, yellow

$0.25 \times 0.25 \times 0.08 \text{ mm}$

Data collection

Stoe IPDSII
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0.15 mm pixels mm^{-1}

$T = 120(2) \text{ K}$

rotation method scans

Absorption correction: numerical

898 independent reflections

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

$R_{\text{int}} = 0.099$

$\theta_{\text{max}} = 29.2^\circ$

$\theta_{\text{min}} = 3.3^\circ$

$h = -9 \rightarrow 9$

$k = -20 \rightarrow 20$

[shape of crystal determined optically (X-RED; Stoe & Cie, 2005)]

$T_{\min} = 0.070$, $T_{\max} = 0.380$

$l = -16 \rightarrow 16$

6750 measured reflections

Refinement

Refinement on F^2

Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.053$

$$w = 1/[\sigma^2(F_o^2) + (0.0791P)^2 + 1.5933P]$$

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

$wR(F^2) = 0.154$

$(\Delta/\sigma)_{\max} = 0.004$

$S = 1.38$

$\Delta\rho_{\max} = 4.88 \text{ e } \text{\AA}^{-3}$

898 reflections

$\Delta\rho_{\min} = -3.22 \text{ e } \text{\AA}^{-3}$

51 parameters

Extinction correction: SHELXL97 (Sheldrick, 1997),

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

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0156 (16)

Secondary atom site location: difference Fourier map

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.0000	0.0000	0.0000	0.0186 (4)
Cl1	0.22812 (15)	0.08739 (7)	0.08595 (9)	0.0236 (4)
N1	0.0000	-0.0885 (3)	0.1339 (4)	0.0219 (11)
N2	0.0000	-0.2037 (4)	0.3202 (5)	0.0284 (12)
C1	0.0000	-0.1797 (4)	0.1203 (5)	0.0242 (12)
H1	0.0000	-0.2050	0.0482	0.029*
C2	0.0000	-0.2356 (5)	0.2146 (6)	0.0266 (13)
H2	0.0000	-0.2985	0.2039	0.032*
C3	0.0000	-0.1117 (4)	0.3307 (6)	0.0254 (12)
H3	0.0000	-0.0863	0.4027	0.030*
C4	0.0000	-0.0545 (4)	0.2386 (5)	0.0237 (12)
H4	0.0000	0.0085	0.2493	0.028*

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Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.0162 (4)	0.0201 (5)	0.0194 (5)	0.000	0.000	-0.00249 (8)
Cl1	0.0211 (6)	0.0257 (7)	0.0239 (7)	-0.0031 (3)	-0.0027 (3)	-0.0028 (4)
N1	0.016 (2)	0.026 (2)	0.024 (3)	0.000	0.000	-0.0021 (19)
N2	0.026 (2)	0.031 (3)	0.028 (3)	0.000	0.000	0.005 (2)
C1	0.021 (2)	0.024 (3)	0.029 (3)	0.000	0.000	-0.004 (2)
C2	0.025 (3)	0.024 (3)	0.031 (4)	0.000	0.000	0.004 (3)
C3	0.023 (3)	0.027 (3)	0.026 (3)	0.000	0.000	-0.002 (3)
C4	0.022 (2)	0.023 (3)	0.026 (3)	0.000	0.000	-0.003 (2)

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

Pt1—Cl1 ⁱ	2.3186 (10)	C1—H1	0.9300
Pt1—Cl1	2.3186 (10)	C2—N2	1.335 (8)
Pt1—Cl1 ⁱⁱ	2.3186 (10)	C2—H2	0.9300
Pt1—Cl1 ⁱⁱⁱ	2.3186 (10)	C3—N2	1.352 (8)
N1—Pt1	2.047 (5)	C3—C4	1.375 (9)
Pt1—N1 ⁱ	2.047 (5)	C3—H3	0.9300
C1—N1	1.344 (7)	C4—N1	1.336 (8)
C1—C2	1.384 (9)	C4—H4	0.9300
Cl1 ⁱ —Pt1—Cl1 ⁱⁱ	90.38 (5)	N1—C1—H1	120.3
Cl1—Pt1—Cl1 ⁱⁱ	89.62 (5)	C2—C1—H1	120.3
Cl1 ⁱ —Pt1—Cl1 ⁱⁱⁱ	89.62 (5)	N2—C2—C1	123.3 (6)
Cl1—Pt1—Cl1 ⁱⁱⁱ	90.38 (5)	N2—C2—H2	118.3
Cl1 ⁱⁱ —Pt1—Cl1 ⁱⁱⁱ	180	C1—C2—H2	118.3
N1—Pt1—Cl1 ⁱ	89.48 (10)	N2—C3—C4	122.3 (6)
N1 ⁱ —Pt1—Cl1 ⁱ	90.52 (10)	N2—C3—H3	118.9
N1—Pt1—Cl1	90.52 (10)	C4—C3—H3	118.9
N1 ⁱ —Pt1—Cl1	89.48 (10)	N1—C4—C3	120.6 (6)
Cl1 ⁱ —Pt1—Cl1	180	N1—C4—H4	119.7
N1—Pt1—Cl1 ⁱⁱ	89.48 (10)	C3—C4—H4	119.7
N1 ⁱ —Pt1—Cl1 ⁱⁱ	90.52 (10)	C4—N1—C1	118.8 (6)
N1—Pt1—N1 ⁱ	180	C4—N1—Pt1	118.9 (4)
N1—Pt1—Cl1 ⁱⁱⁱ	90.52 (10)	C1—N1—Pt1	122.4 (4)
N1 ⁱ —Pt1—Cl1 ⁱⁱⁱ	89.48 (10)	C2—N2—C3	115.7 (6)
N1—C1—C2	119.3 (6)		
C4—N1—Pt1—Cl1 ⁱ	-134.81 (3)	N1—C1—C2—N2	0.0
C1—N1—Pt1—Cl1 ⁱ	45.19 (3)	N2—C3—C4—N1	0.0
C4—N1—Pt1—Cl1	45.19 (3)	C3—C4—N1—C1	0.0
C1—N1—Pt1—Cl1	-134.81 (3)	C3—C4—N1—Pt1	180.0
C4—N1—Pt1—Cl1 ⁱⁱ	134.81 (3)	C2—C1—N1—C4	0.0

C1—N1—Pt1—C11 ⁱⁱ	-45.19 (3)	C2—C1—N1—Pt1	180.0
C4—N1—Pt1—C11 ⁱⁱⁱ	-45.19 (3)	C1—C2—N2—C3	0.0
C1—N1—Pt1—C11 ⁱⁱⁱ	134.81 (3)	C4—C3—N2—C2	0.0

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, -y, -z$; (iii) $-x, y, z$.

Fig. 1

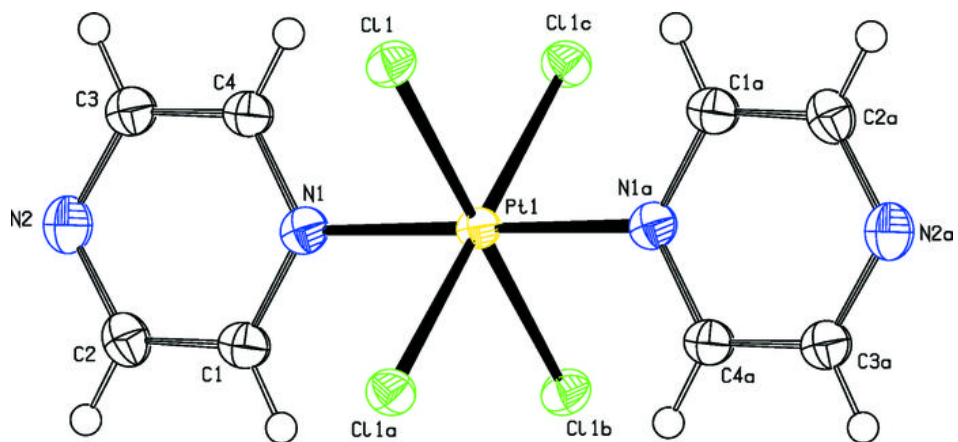


Fig. 2

