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Powder study of *trans*-tetrachlorido-bis(isopropylamine)platinum(IV)

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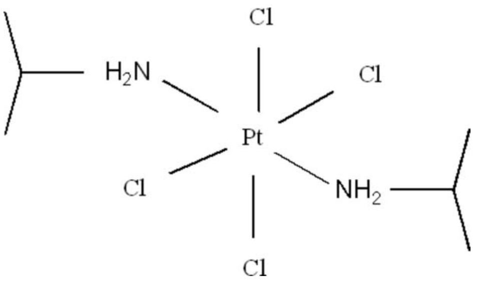
Key indicators: powder X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; R factor = 0.000; wR factor = 0.000; data-to-parameter ratio = 0.0.

The title compound, *trans*-[$PtCl_4(iso\text{-}NH_2C_3H_7)_2$], was obtained by oxidation of *trans*-[$PtCl_2(iso\text{-}NH_2C_3H_7)_2$] with chlorine gas. The crystal structure contains discrete centrosymmetric mononuclear complex molecules, with Pt^{IV} in a slightly distorted octahedral coordination environment. The slight distortion, as described by the angles at the Pt^{IV} atom, is due, in part, to intramolecular N—H···Cl hydrogen bonding [N···Cl = 2.897 (12) Å]. The Pt—N and Pt—Cl bond lengths are comparable to those in related structures. In the crystal structure, molecules are arranged in layers in the *bc* plane, with a shortest Pt···Pt distance of 6.1105 (1) Å. The molecules are organized so that isopropylamine ligands project from the layers, forming organic interlayers.

Related literature

For related literature, see: Allen (2002); Bradner *et al.* (1980); Hydes (1981); Le Bail *et al.* (1988); Milburn & Truter (1966); Rietveld (1969); Visser (1969); Wells (1984); Zhelegovskay & Fat'kin (1986); Zhelegovskay *et al.* (1991).



Experimental

Crystal data

[$PtCl_4(C_3H_9N)_2$]
M_r = 455.11
Monoclinic, $P2_1/c$
a = 8.8886 (1) Å
b = 8.9464 (2) Å
c = 8.3255 (2) Å
β = 93.868 (1)°
V = 660.54 (2) Å^3
Z = 2
Cu $K\alpha$ radiation
λ = 1.54056 Å
T = 293 K
Specimen shape: circular flate plate
20.0 × 20.0 × 0.5 mm
Specimen prepared at 101 kPa
Specimen prepared at 293 K
Particle morphology: thin powder, yellow

Data collection

DRON-4 powder diffractometer
Specimen mounting: packed powder pellet
Specimen mounted in reflection mode
$2\theta_{min}$ = 8.0, $2\theta_{max}$ = 90.0°
Increment in 2θ = 0.02°

Refinement

R_p = 0.078
R_{wp} = 0.107
R_{exp} = 0.073
R_B = 0.040
S = 1.46
Excluded region(s): none
Profile function: Pearson VII
42 parameters
H atoms treated by a mixture of independent and constrained refinement
Preferred orientation correction: March–Dollase correction

Table 1
Selected geometric parameters (Å, °).

Pt—N	2.066 (9)	Pt—Cl2	2.307 (5)
Pt—Cl1	2.331 (5)		
Cl1—Pt—Cl2	91.5 (4)	Cl2—Pt—N	86.2 (3)
Cl1—Pt—N	97.7 (3)	Pt—N—C1	123.0 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H4A···Cl2^{ii}	0.960 (12)	2.761 (12)	3.683 (12)	161.3 (9)
N—H4B···Cl2^{iii}	0.959 (12)	2.287 (12)	3.190 (12)	156.4 (9)
N—H4B···Cl1	0.959 (12)	2.728 (12)	2.897 (12)	90.3 (9)

Symmetry codes: (ii) $-x, y+\frac{1}{2}, -z-\frac{1}{2}$; (iii) $x, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *DRON-4* data collection software (unpublished); cell refinement: *POWDER* (Kirik *et al.*, 1979); data reduction: local program (Kirik, 1985); program(s) used to solve structure: modified *DBWM* (Wiles & Young, 1981); program(s) used to refine structure: modified *DBWM*; molecular graphics: *XP* (Siemens, 1989) and *PLATON* (Spek, 2003).

X-ray powder diffraction data preparation was supported by the ICDD (Grant-in-Aid 9310) and grant RFBR-KSF 07-03-96805.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2358).

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supplementary materials

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Powder study of *trans*-tetrachloridobis(isopropylamine)platinum(IV)

S. D. Kirik and **A. K. Starkov**

Comment

Asymmetric diamine dichloro platimun(II) complexes, *e.g. cis*-bis(isopropylamine) dichloro platimun(II) and related compounds (Bradner *et al.*, 1980; Hydes, 1981) can exhibit high anticancer activity. The synthesis of *cis*-(isopropylamine)amine dichloro platinum(II) has been described by Hydes (1981), Zhelegovskay & Fat'kin (1986) and Zhelegovskay *et al.* (1991). This is based on the reaction of tetrachloroplatinate(II) potassium with isopropylamine followed by refinement of the target product. The main synthetic difficulties stem from the requirements of low level of impurities because of the medical application. It justifies the interest in possible subsidiary reactions and by-products at the synthesis stage of target compounds. In the present paper, results of a synthesis and a crystal structure determination of *trans*-bis(isopropylamine) tetrachloro platimun(IV), performed using X-ray powder diffraction technique, are presented.

The crystal structure of *trans*-bis(isopropylamine) tetrachloro platimun(IV) is of the molecular type with two symmetrically equivalent molecules in unit cell. The molecular structure of the title compound presented in Fig. 2. The Pt atom lies on a center of inversion in a slightly distorted octahedral coordination environment consisting of two N and four Cl atoms. The Pt—N and Pt—Cl distances compare well to literature values (Wells, 1984, Allen, 2002). Isopropylamine as ligand induces more distortion at atom Pt in comparison to an amine as a ligand (Milburn & Truter, 1966). The N—Pt—Cl1 and N—Pt—Cl2 angles are 97.7 (3) and 86.2 (3)° respectively. The N—Cl1 distance of *ca* 2.89Å allows us to suppose that the distortion in the molecule is induced, in part, by intramolecular hydrogen bonds of the N—H···Cl1 type. In addition, isopropylamine ligands are connected to other molecules by intermolecular hydrogen bonds (see Table 2) and Van der Waals forces also contribute to the crystal packing. The molecules arrange in layers stretched along (*bc*)-plane with the shortest distances Pd···Pd within a layer about 6.1105 (2)° A. The Pt···Pt distance between layers is substantially longer *ca* 8.88° A. The molecules orientate in the layers, so that the bulky isopropylamine ligands project above and below each layer comprising organic interlayers (Fig. 3).

Experimental

Trans-Pt(iso-$NH_2C_3H_7)_2Cl_2$ (2 g) was suspended in 40 ml of water and gaseous chorine was passed through the suspension for 2 h. The yellow substance obtained was filtered, washed with ethanol and dried in air at room temperature.

Refinement

The structure determination was carried out by X-ray powder diffraction approach. The experimental data were collected using DRON-4 automatic diffractometer, equipped with a secondary flat graphite monochromator in conjunction with a scintillation detector. Cu *K*α radiation was used (λ_1=1.54056 Å, λ_2=1.54439 Å). The sample was prepared by top-loading the standard quartz sample holder with cutting the excess of well grained substance. The diffraction pattern was scanned with the step of 0.02° 2θ and counting time of 5 sec./step in the most informative angular range from 8° to 90% 2θ at ambient temperature. Corundum was used as the external standard. The powder pattern of *cis*-(isopropylamine) aminne dichloro platimun(II) is presented in Fig.1. X-ray powder diffraction data were deposited in JCPDS-ICDD PDF2 database. Cell parameters were obtained from d-spaces by indexing and refining using programs described in (Visser, 1969, Kirik *et al.*, 1979). The space group was determined from the analysis of systematic absences. The structural investigations were carried out using a full-profile structure analysis package based on a modified version of the Rietveld refinement program DBWS– 9006PC (Wiles & Young, 1981). The intensities of 50 reflections were estimated from the powder pattern by means of the full- profile fitting procedure (Le Bail *et al.*, 1988) and used in the Patterson synthesis. Atoms of Pt and Cl were located directly from the Patterson map. Positions of light atoms N and C were defined from a difference Fourier synthesis. H-atoms were not located, but they were included in the refined structure models and rigidly connected to their C and N atoms. The final refinement was carried out by Rietveld method (Rietveld, 1969).

Figures

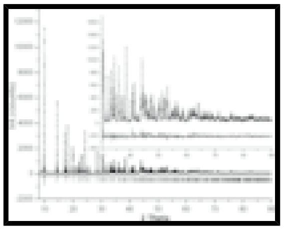


Fig. 1. Observed (dots), calculated (superimposed solid) and difference profiles after the Rietveld refinement. The reflection positions are marked by ticks.

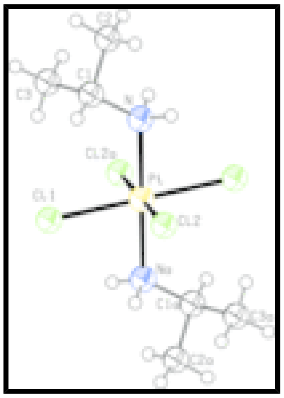


Fig. 2. Molecular structure of the title compound showing 30% isotropic dispacement ellipsoids.

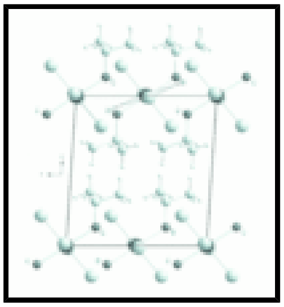


Fig. 3. Part of the crystal structure of *trans*-Pt(iso-$NH_2C_3H_7)_2Cl_4$.

trans-tetrachloridobis(isopropylamine)platinum(IV)

Crystal data

$[PtCl_4(C_3H_9N)_2]$	F_{000} = 428.0
M_r = 455.11	Cell parameters are obtained from the Rietveld refinement

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

a = 8.8886 (1) Å

b = 8.9464 (2) Å

c = 8.3255 (2) Å

β = 93.868 (1)º

V = 660.54 (2) Å^3

Z = 2

D_x = 2.288 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å

T = 293 K

Specimen shape: circular flate plate

20.0 × 20.0 × 0.5 mm

Specimen prepared at 101 kPa

Specimen prepared at 293 K

Particle morphology: thin powder, yellow

Data collection

DRON-4 powder diffractometer

Monochromator: graphite

Specimen mounting: packed powder pellet

Specimen mounted in reflection mode

Scan method: ?

T = 293 K

$2\theta_{min}$ = 8.0, $2\theta_{max}$ = 90.0º

Increment in 2θ = 0.02º

Refinement

Refinement on F^2

Least-squares matrix: full

R_p = 0.078

R_{wp} = 0.107

R_{exp} = 0.073

R_B = 0.040

S = 1.46

Wavelength of incident radiation: 1.54056 Å

Excluded region(s): none

Profile function: Pearson VII

42 parameters

H atoms treated by a mixture of independent and constrained refinement

Weighting scheme based on measured s.u.'s ?

$(\Delta/\sigma)_{max}$ = 0.1

Extinction correction: ?

Preferred orientation correction: March-Dollase correction

Special details

Refinement. R_prof-backgr = 0.078

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Pt	0.0000	0.0000	0.0000	0.0121*
Cl1	0.2013 (5)	0.0270 (12)	0.1921 (6)	0.0151*
Cl2	0.0140 (12)	−0.2571 (6)	0.0170 (12)	0.0153*
C1	0.2950 (10)	−0.0360 (10)	−0.1890 (10)	0.0256*
H1A	0.3287 (10)	−0.1102 (10)	−0.1115 (10)	0.1520*
C2	0.3450 (10)	−0.0720 (10)	−0.3560 (10)	0.0393*
H2A	0.3072 (10)	−0.1685 (10)	−0.3890 (10)	0.1520*
H2B	0.3062 (10)	0.0023 (10)	−0.4310 (10)	0.1520*
H2C	0.4531 (10)	−0.0725 (10)	−0.3533 (10)	0.1520*
C3	0.3600 (9)	0.1200 (9)	−0.1450 (9)	0.0316*
H3A	0.4681 (9)	0.1152 (9)	−0.1367 (9)	0.152*
H3B	0.3274 (9)	0.1904 (9)	−0.2271 (9)	0.152*

H3C	0.3249 (9)	0.1513 (9)	−0.0438 (9)	0.152*
N	0.1240 (10)	−0.0200 (10)	−0.2000 (10)	0.0251*
H4A	0.1007 (10)	0.0660 (10)	−0.2660 (10)	0.1520*
H4B	0.0845 (10)	−0.1050 (10)	−0.2591 (10)	0.1520*

Geometric parameters (Å, °)

Pt—N	2.066 (9)	N—H4B	0.96 (1)
Pt—Cl1	2.331 (5)	C3—H3A	0.96 (1)
Pt—Cl2	2.307 (5)	C3—H3B	0.96 (1)
C1—C3	1.545 (12)	C3—H3C	0.96 (1)
C1—C2	1.522 (12)	C2—H2A	0.96 (1)
C1—N	1.523 (13)	C2—H2B	0.96 (1)
Pt—Pt^{i}	6.1105 (1)	C2—H2C	0.96 (1)
N—H4A	0.96 (1)	C1—H1A	0.96 (1)
Cl1—Pt—Cl2	91.5 (4)	Pt—N—H4B	106.3 (6)
Cl1—Pt—N	97.7 (3)	N—C1—H1A	111.8 (8)
Cl2—Pt—N	86.2 (3)	C1—C2—H2A	109.5 (8)
C2—C1—C3	106.3 (6)	C1—C2—H2B	109.5 (8)
Pt—N—C1	123.0 (6)	C1—C2—H2C	109.5 (8)
N—C1—C2	108.5 (7)	C1—C3—H3A	109.5 (7)
N—C1—C3	106.6 (7)	C1—C3—H3B	109.5 (7)
Pt—N—H4A	106.3 (6)	C1—C3—H3C	109.5 (7)

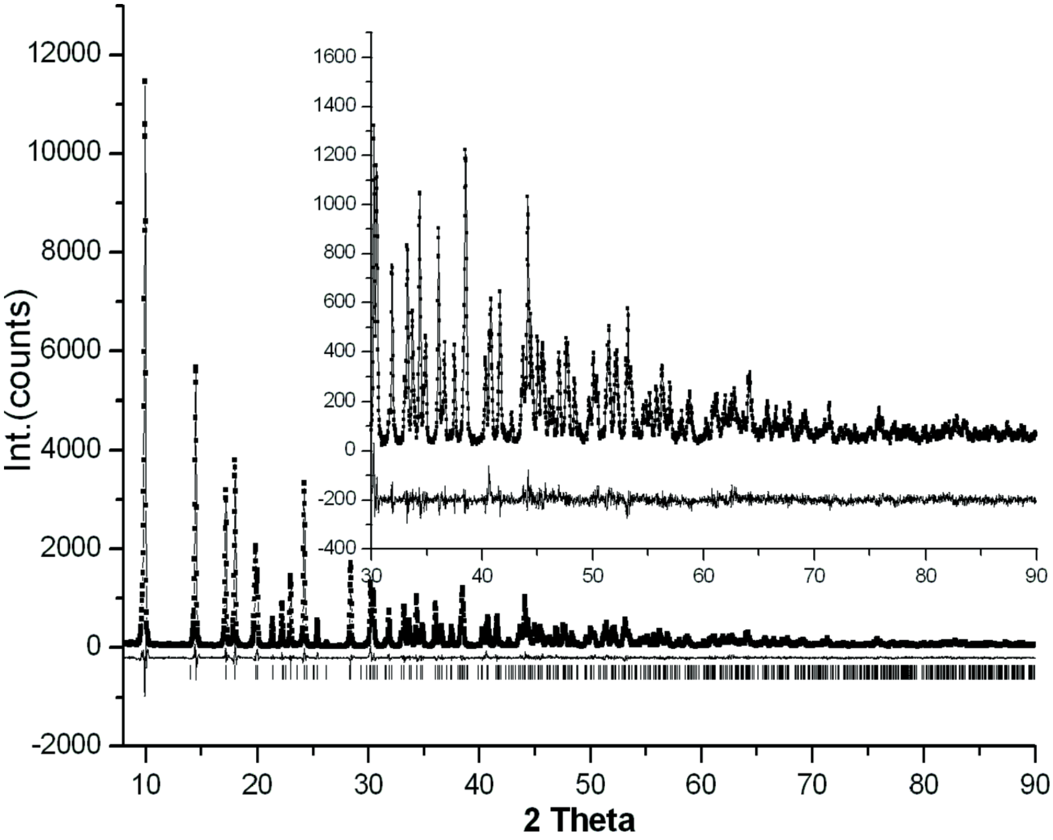
Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H4A···Cl2^{ii}	0.960 (12)	2.761 (12)	3.683 (12)	161.3 (9)
N—H4B···Cl2^{iii}	0.959 (12)	2.287 (12)	3.190 (12)	156.4 (9)

Symmetry codes: (ii) −*x*, *y*+1/2, −*z*−1/2; (iii) *x*, −*y*−1/2, *z*−1/2.

Fig. 1


Int.(counts)
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4000
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10
20
30
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50
60
70
80
90
2 Theta
1600
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1200
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800
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60
70
80
90

Fig. 2

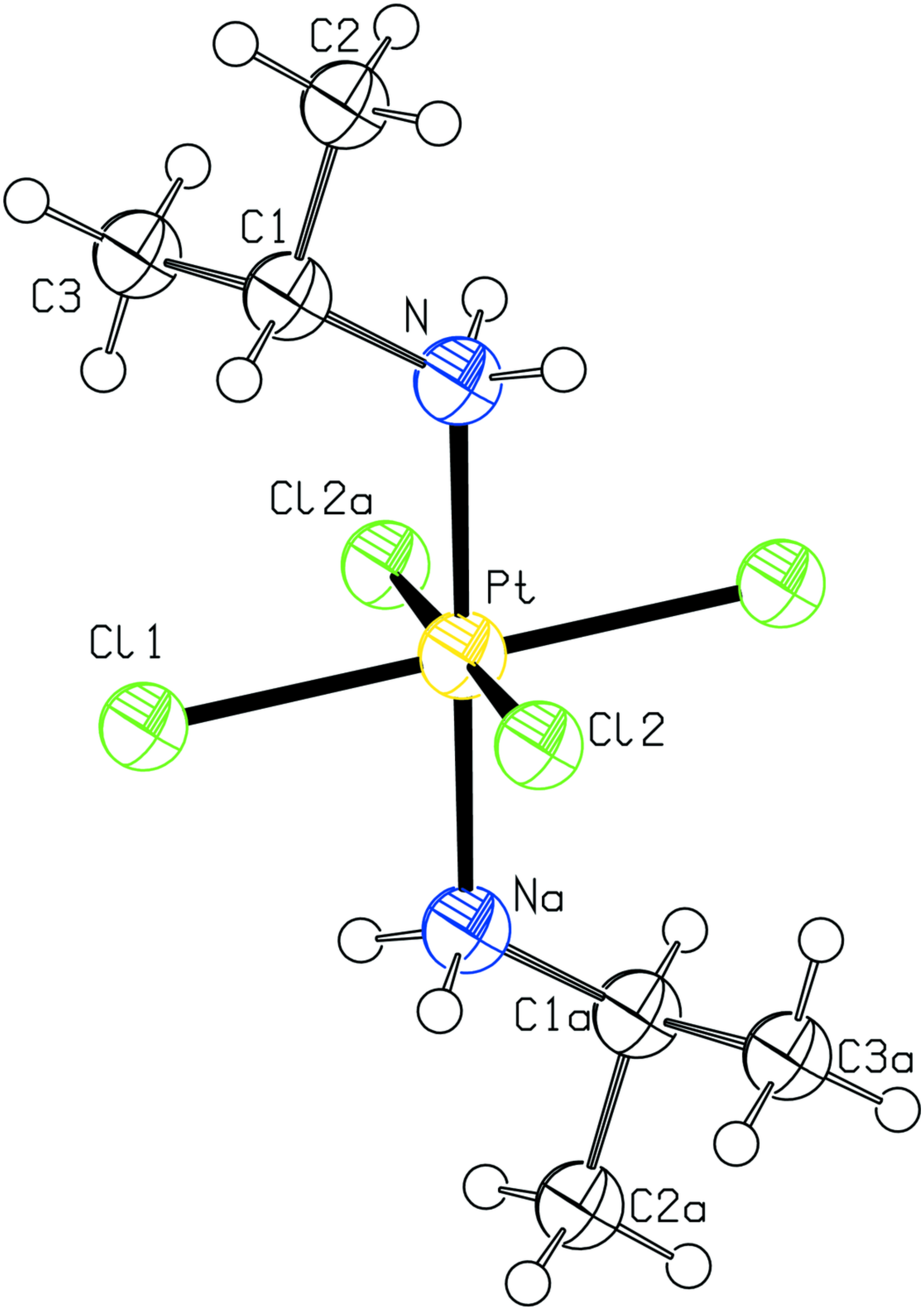

Fig. 3