

tance of 1.50 Å for the second anion. This suggests the presence of some disorder in the PF_6^- groups.

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cis-Bis(cyclopropylamine)diiodoplatinum(II)

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Abstract. $[\text{PtI}_2(\text{C}_3\text{H}_7\text{N})_2]$, $M_r = 563.1$, monoclinic, $C2/c$, $a = 23.252$ (3), $b = 8.577$ (1), $c = 15.456$ (2) Å, $\beta = 128.38$ (1)°, $V = 2416$ (1) Å³, $Z = 8$, $D_x = 3.09$ g cm⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 173$ cm⁻¹, $F(000) = 1984$, $T = 298$ K, $R = 0.027$ for 1044 unique observed reflections. The structure consists of monomeric molecules with the four ligand atoms and the central metal ion in an essentially *cis*-planar arrangement. One of the two cyclopropane rings is partly disordered with one C atom occupying two positions.

Introduction. Studies on oligomeric platinum complexes raised a question about the effect of amine-ligand size on the tendency towards oligomerization. Traditionally research has been carried out with ammonia ligands, and various coloured species have been reported (Rosenberg, van Camp, Trosko & Mansour, 1969; Shimura, Tomohiro, Laitalainen, Moriyama, Uemura & Okuno, 1988; Laitalainen, Okuno & Tomohiro, 1987). The cyclopropylamine ligand forms blue oligomeric complexes (Laitalainen & Oksanen, 1989). The title compound was prepared, isolated and characterized for our studies on oligomerization reactions of platinum.

Experimental. The crystals were prepared by dissolving K_2PtCl_4 (0.1245 g, 0.300 mmol) and KI (0.1992 g, 1.200 mmol) in water (1.26 ml) with ultrasonication at room temperature. The reaction mixture was heated to 313 K and aqueous cyclopropylamine solution (14%, 250 μl , 0.613 mmol) was added dropwise within 10 minutes, the mixture was kept at 313 K under magnetic stirring for 20 additional minutes, then cooled on ice. A yellow powdery precipitate was filtered off. Very small crystals slowly formed in the filtrate. The whole synthesis was carried out in the dark.

A small crystal (0.02 × 0.06 × 0.10 mm) was measured with a CAD-4 diffractometer. The unit-cell parameters were determined on the basis of 25 well centred reflections in the angular range $24 < 2\theta < 34^\circ$. The intensity data were collected in the $\omega/2\theta$ scan mode, with the scan speed 0.9–16.5° min⁻¹ in the range $4.0 < 2\theta < 60^\circ$. The absorption correction was calculated with the DIFABS program (Walker & Stuart, 1983); the minimum/maximum corrections were 0.78/1.22. Intensities were collected up to $(\sin\theta)/\lambda = 0.7035$ Å⁻¹. Five standard reflections were measured once every hour. The intensities decreased 15% during the measurements and the data were

corrected for decay. A total of 2282 reflections ($0 < h < 27$, $0 < k < 10$, $-18 < l < 13$) were measured giving 1044 unique reflections; $R_{\text{int}} = 0.031$. 1238 reflections were unobserved according to the criterion $I < 3\sigma(I)$. The structure was solved by *SHELXS86* program (Sheldrick, 1986). Full-matrix least-squares refinement was performed using *XTAL2.2* program system (Hall & Stewart, 1987) which minimized the function $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F_o)$. The disordered C(6) atom occupying two positions was refined isotropically. All the other non-H atoms were refined anisotropically. Owing to the disordering, H atoms were not included in the calculations. Final cycle of least-squares refinement with 101 parameters gave $R = 0.027$, $wR = 0.024$, $S = 2.26$, $(\Delta/\sigma)_{\text{max}} = 0.09$, $\Delta\rho_{\text{max}} = 0.61$, $\Delta\rho_{\text{min}} = -0.88 \text{ e } \text{Å}^{-3}$. Neutral-atom scattering factors were those included in the program systems and the corrections for anomalous dispersion for all atoms were taken from *International Tables for X-ray Crystallography* (1974).*

Discussion. The structure consists of monomeric molecules with the four ligand atoms and the central metal ion in an essentially *cis*-planar arrangement. The numbering system and the configuration of the molecule are shown in Fig. 1. The positional and isotropic thermal parameters are given in Table 1 and the bond distances and angles in Table 2.

The Pt—N bond distances of 2.08 (1) and 2.092 (9) Å are normal (Raudaschl-Sieber, Lippert, Britten & Beauchamp, 1986), as are the Pt—I distances of 2.598 (1) and 2.586 (1) Å (Raudaschl-Sieber *et al.*, 1986; Mégnamisi-Bélombé & Endres, 1985). The intramolecular I(1)⋯I(2) separation of 3.780 (2) Å is reflected in the I(1)—Pt—I(2) angle value of 94.26 (4)°, which is significantly greater than 90°. The configuration of the title compound is quite

* Lists of structure factors, anisotropic thermal parameters and least-squares-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51938 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

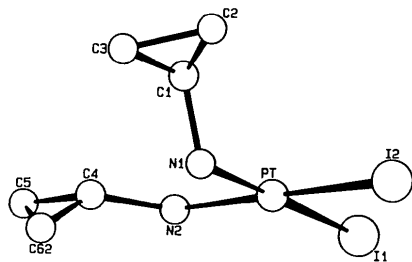


Fig. 1. The molecular structure of $[\text{PtI}_2(\text{C}_3\text{H}_5\text{NH}_2)_2]$ showing the atom numbering. C(61) has been omitted for clarity.

Table 1. *Positional and isotropic thermal parameters for $[\text{PtI}_2(\text{C}_3\text{H}_5\text{NH}_2)_2]$*

Pt	x	y	z	U_{eq}^* or $U_{\text{iso}}(\text{Å}^2)$
Pt	0.50452 (3)	0.10340 (8)	0.61011 (5)	0.0428 (4)
I(1)	0.41305 (5)	-0.1238 (1)	0.55134 (8)	0.0623 (7)
I(2)	0.40285 (6)	0.3064 (2)	0.48452 (8)	0.0708 (8)
N(1)	0.5802 (5)	0.280 (1)	0.6603 (7)	0.040 (7)
N(2)	0.5869(5)	-0.062 (1)	0.7092 (8)	0.047 (8)
C(1)	0.6021 (8)	0.357 (2)	0.765 (1)	0.07 (1)
C(2)	0.586 (1)	0.525 (2)	0.757 (1)	0.10 (1)
C(3)	0.665 (1)	0.476 (2)	0.814 (1)	0.11 (2)
C(4)	0.668 (1)	-0.016 (1)	0.781 (1)	0.10 (1)
C(5)	0.7250(7)	-0.121 (2)	0.849 (1)	0.09 (1)
C(61)†	0.704 (2)	0.061 (4)	0.869 (2)	0.07 (1)
C(62)‡	0.712 (1)	0.024 (4)	0.767 (2)	0.06 (1)

$$* U_{\text{eq}} = \frac{1}{3}[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)].$$

† Occupancy 0.51 (2).

‡ Occupancy 0.49 (2).

Table 2. *Selected bond lengths (Å) and angles (°) for $[\text{PtI}_2(\text{C}_3\text{H}_5\text{NH}_2)_2]$*

Pt—N(1)	2.08 (1)	N(1)—Pt—N(2)	92.5 (4)
Pt—N(2)	2.092 (9)	I(1)—Pt—I(2)	94.26 (4)
Pt—I(1)	2.598 (1)	N(1)—Pt—I(2)	87.5 (3)
Pt—I(2)	2.586 (1)	N(2)—Pt—I(1)	85.8 (3)
N(1)—C(1)	1.51 (2)	Pt—N(1)—C(1)	111 (1)
N(2)—C(4)	1.52 (2)	Pt—N(2)—C(4)	120.8 (9)
C(1)—C(2)	1.48 (2)	N(1)—C(1)—C(2)	116 (1)
C(1)—C(3)	1.54 (3)	N(1)—C(1)—C(3)	114 (2)
C(2)—C(3)	1.53 (3)	N(2)—C(4)—C(5)	124 (2)
C(4)—C(5)	1.40 (2)	C(1)—C(2)—C(3)	62 (1)
C(4)—C(61)	1.24 (3)	C(2)—C(3)—C(1)	58 (1)
C(5)—C(61)	1.72 (4)	C(3)—C(1)—C(2)	61 (1)
C(4)—C(62)	1.25 (5)		
C(5)—C(62)	1.67 (4)		

different from that of the chloride analogue reported earlier (Howard-Lock, Lock, Turner & Zvagulis, 1981). In the chloride compound both N—C bonds are oriented away from the coordination plane, whereas in the title compound only the N(1)—C(1) bond is oriented in this way, and the N(2)—C(4) bond is in the coordination plane. The dihedral angles N(2)—Pt—N(1)—C(1) and N(1)—Pt—N(2)—C(4) are $-86.8 (9)$ and $0 (1)^\circ$. The orientation of the N(2)—C(4) bond results in a short intramolecular N(1)⋯C(4) contact of 3.06 (2) Å. The N(2)—Pt—I(1) and Pt—N(2)—C(4) angles of 85.8 (3) and 120.8 (9)° probably reflect the tendency to reduce the repulsion between the N(1) and C(4) atoms. The C(5) atom lies approximately in the coordination plane. The disordered C(6) atom occupies two positions, one on either side of the coordination plane. The dihedral angle between the coordination plane and the plane through the C(1), C(2) and C(3) atoms of one cyclopropyl ring is 19 (1)°, which can be compared with the corresponding values of 3.9 and 26.0° reported for the chloride compound.

Packing of the molecules is shown in Fig. 2. The shortest distance between non-H atoms of neighbouring molecules is fairly long, 3.62 (2) Å, indicating lack

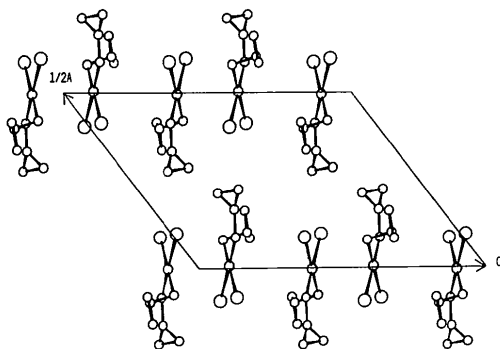


Fig. 2. The packing of the $[\text{PtI}_2(\text{C}_3\text{H}_5\text{NH}_2)_2]$ molecules viewed down the b axis. C(61) has been omitted for clarity.

of intermolecular steric interactions between the cyclopropane rings. Moreover, the shortest Pt...Pt distance of 3.727 (1) Å is evidently too long for metal-metal interactions between the neighbouring molecules. There are no hydrogen bonds, but the structure is held together through weak van der Waals interactions.

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Structure du Composé $[\text{As}(\text{C}_6\text{H}_5)_4]\text{HgCl}_3$

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Abstract. Tetraphenylarsenic(I) trichloromercurate(II), $M_r = 689.71$, triclinic, $P\bar{1}$, $a = 12.96$ (3), $b = 9.83$ (4), $c = 9.76$ (1) Å, $\alpha = 100.2$ (3), $\beta = 94.1$ (2), $\gamma = 108.2$ (2)°, $V = 1152.78$ Å³, $Z = 2$, $D_m = 1.95$, $D_x = 1.98$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.7107$ Å, $\mu = 7.11$ mm⁻¹, $F(000) = 636$, $T = 300$ K, $R = 0.052$, $wR = 0.051$ for 1711 reflections with $I > \sigma(I)$. The structure consists of isolated bitetrahedral $(\text{Hg}_2\text{Cl}_6)^{2-}$ units sharing one edge, with two short bonds of 2.365 (5) and 2.370 (5) Å, and two long bonds of 2.614 (5) and 2.589 (5) Å. The organic cation

$[\text{As}(\text{C}_6\text{H}_5)_4]^+$ is located between the $(\text{Hg}_2\text{Cl}_6)^{2-}$ units.

Introduction. Différents types de coordination ont été observés par l'étude structurale des complexes du mercure(II), une étude détaillée a été réalisée par Grdenic (1960), Sandström (1978) et Ben Salah (1984) montrant les différentes coordinations du mercure. Dans les composés du type $R^1M^{\text{II}}X_3$ où R^1 est un cation monovalent, M^{II} un métal divalent, la structure est du type CsNiCl_3 formant des chaînes

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