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trans-Dicyanobis(1,3,5-triaza-7-phospha-adamantane)platinum(II), [(TPA)$_2$Pt(CN)$_2$]

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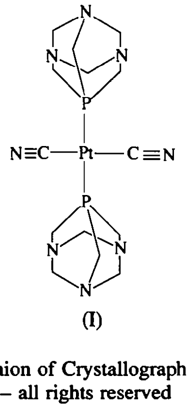
Abstract

The d^8 Pt^{II} ion in the title complex [systematic name: *trans*-dicyanobis(7-phospha-1,3,5-triazatricyclo-[3.3.1.1^{3,7}]decane-*P*)platinum(II)], [Pt(CN)$_2$(C$_6$H$_{12}$-N$_3$P)$_2$], lies on an inversion center and has a necessarily *trans*-square-planar geometry, with Pt—P and Pt—C distances of 2.305 (2) and 1.975 (9) Å, respectively, and a P—Pt—C angle of 90.6 (2)°.

Comment

The first metal complex containing bis(1,3,5-triaza-7-phosphaadamantane), TPA, was crystallographically characterized as [Mo(CO)$_5$(TPA)] (Delerno, Trefonas, Darensbourg & Majeste, 1976). The σ-donating ability of the ligand has been shown to be comparable with that of PMe$_3$. Because of the recently revived interest in the TPA ligand, metal complexes containing this ligand, such as [RuCl$_2$(TPA)$_4$] and [RhCl(TPA)$_2$].2HCl (Darensbourg, Joo, Kannisto, Katho & Reibenspies, 1992), and [Au(TPA)Cl][(TPA.HCl)AuCl] (Assefa, McBurnett, Staples, Fackler, Assmann, Angermaier & Schmidbaur, 1995), have been characterized. In all of the metal complexes characterized to date, the potentially quadridentate TPA ligand coordinates to the metal center only through the P atom. We report here the structure of the platinum complex *trans*-dicyanobis(1,3,5-triaza-7-phosphaadamantane)platinum(II), (I).



(I)

Fig. 1 shows the title molecule and the atomic labeling scheme. The Pt atom lies on the inversion center which imposes planarity and P—Pt—P and C—Pt—C angles of 180°. The two TPA ligands bound to the Pt atom are *trans* with respect to each other, with Pt—P and Pt—C distances of 2.305 (2) and 1.975 (9) Å, respectively. The P—C and C—N distances are almost identical to those found in the structure of the free ligand (Fluck, Förster, Weidlein & Hädicke, 1977). The *trans* substitution observed in this complex appears to indicate that unprotonated TPA is a stronger *trans*-director when compared with the CN$^-$ ligand. In support of this, a *cis* arrangement of the TPA ligands has been observed for (TPA)$_2$PtCl$_2$ in which the TPA ligand is protonated (Stafford, 1994).

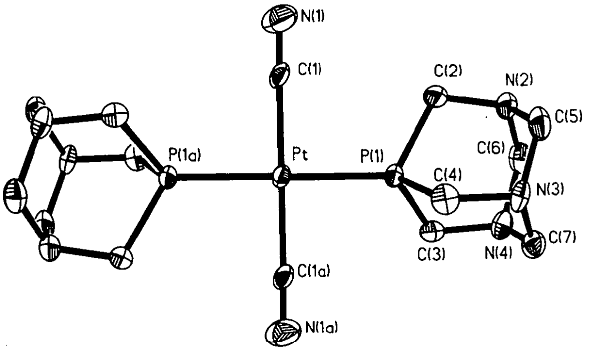


Fig. 1. A drawing of *trans*-[(TPA)$_2$Pt(CN)$_2$] showing the atomic labeling scheme, with displacement ellipsoids representing 50% probability. Atoms labels appended with *a* are symmetry equivalents (related by $-x$, $1-y$, $1-z$).

Experimental

The title compound was obtained while attempting to study the possible interaction between ionic (TPA)$_3$Au$^+$ and Pt(CN)$_4^{2-}$ centers. Aqueous solutions of (TPA)$_3$AuCl and K$_2$Pt(CN)$_4$ in 2:1 molar ratios were mixed and the resulting solution left to evaporate slowly. Two types of crystal, needle and plate, were obtained, of which the plate type was characterized as the title complex. A cell check on the needle-shaped crystals confirmed that they were crystals of (TPA)$_2$AuCl, which was also characterized and will be reported subsequently (Assefa, Staples & Fackler, 1995).

Crystal data

[Pt(CN)$_2$(C$_6$H$_{12}$N$_3$P)$_2$]
$M_r = 561.4$
Monoclinic
$P2_1/n$
$a = 7.194$ (1) Å
$b = 11.741$ (2) Å
$c = 10.630$ (2) Å
$\beta = 97.08$ (1)°

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 8.149$ mm^{-1}
$T = 298$ K
Plate

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$V = 890.97$ Å^3
$Z = 2$
$D_x = 2.093$ Mg m^{-3}

0.35 × 0.2 × 0.1 mm
Colorless

Data collection

Siemens $R3m/E$ diffractometer
Wyckoff scans
Absorption correction:
  ψ scans
  $T_{\min} = 0.362$, $T_{\max} = 1.000$
1353 measured reflections
1169 independent reflections
996 observed reflections
  $[F_o^2 > 3\sigma(F_o^2)]$

$R_{\rm int} = 0.0522$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = -11 \rightarrow 11$
3 standard reflections
  monitored every 97 reflections
  intensity decay: none

Refinement

Refinement on F
$R = 0.0331$
$wR = 0.0442$
$S = 1.13$
996 reflections
114 parameters
H atoms riding with C—H = 0.96 Å
$w = 1/[\sigma^2(F_o) + 0.0012F_o^2]$

$(\Delta/\sigma)_{\max} = -0.016$
$\Delta\rho_{\max} = 1.38$ e Å^{-3} (near Pt)
$\Delta\rho_{\min} = -2.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Pt	0	1/2	1/2	0.019 (1)
P(1)	0.1400 (2)	0.3266 (1)	0.5502 (2)	0.019 (1)
N(1)	0.3018 (10)	0.5526 (6)	0.3205 (7)	0.048 (3)
N(2)	0.3994 (8)	0.1641 (5)	0.5111 (5)	0.029 (2)
N(3)	0.3610 (8)	0.2065 (4)	0.7329 (5)	0.024 (2)
N(4)	0.1180 (8)	0.0986 (4)	0.6002 (5)	0.027 (2)
C(1)	0.190 (1)	0.5351 (7)	0.3866 (8)	0.027 (2)
C(2)	0.319 (1)	0.2707 (6)	0.4562 (7)	0.029 (2)
C(3)	−0.0027 (9)	0.1968 (6)	0.5573 (8)	0.027 (2)
C(4)	0.2747 (9)	0.3194 (6)	0.7094 (6)	0.025 (2)
C(5)	0.4897 (9)	0.1810 (6)	0.6409 (7)	0.031 (2)
C(6)	0.254 (1)	0.0750 (6)	0.5117 (7)	0.031 (2)
C(7)	0.218 (1)	0.1170 (6)	0.7262 (7)	0.029 (2)

Table 2. *Selected geometric parameters* (Å, °)

Pt—P(1)	2.305 (2)	C(1)—N(1$^{\rm i}$)	1.15 (1)
Pt—C(1)	1.975 (9)		
P(1)—Pt—P(1$^{\rm i}$)	180.0	P(1)—Pt—C(1)	90.6 (2)
C(1)—Pt—C(1$^{\rm i}$)	180.0	P(1$^{\rm i}$)—Pt—C(1)	89.4 (2)

Symmetry code: (i) $-x, 1-y, 1-z$.

The *SHELXTL-Plus* program package (Sheldrick, 1991) was used for crystallographic calculations.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1042). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexamolybdenum Cluster with One Telluride and Seven Chloride Capping Ligands

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Abstract

Tetraethylammonium hepta-μ_3-chloro-μ_3-telluro-hexachlorohexamolybdate, $(Et_4N)_3[(Mo_6Cl_7Te)Cl_6]$, has an octahedral hexamolybdenum cluster core whose faces are capped by seven Cl atoms and one Te atom. The capping telluride is disordered in the crystal. The average Mo—Mo distance is not much different from those in $[(Mo_6Cl_7Z)Cl_6]^{3-}$ (Z = S, Se).

Comment

The hexamolybdenum cluster unit has been observed in solid-state Chevrel-phase compounds, $M_xMo_6Z_8$ (M = Cu, Pb, *etc.*; Z = S, Se, Te) (Yvon, 1978). Similar Mo$_6$-cluster structures have been found in discrete halide-cluster ions, $[(Mo_6X_8)X'_6]^{2-}$ (X = X' = Cl, Br). The Chevrel-phase compounds have formal oxidation states of Mo_6^{12+} to Mo_6^{16+}, whereas the reported halide clusters are known to have only Mo_6^{12+}. The halide–chalcogenide