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{2,6-Bis[(diphenylphosphino)methyl]phenyl}chloroplatinum(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.027
wR factor = 0.041
Data-to-parameter ratio = 26.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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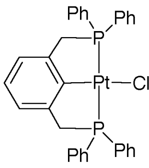
The title compound, [PtCl($C_{32}H_{27}P_2$)], has a pseudo-square-planar coordination geometry around the Pt atom with the *P,C,P′*-tridentate ligand forming two five-membered rings that are tilted with respect to the coordination plane. The two Pt—P, the Pt—Cl and the Pt—C distances are 2.2794 (9)/2.2749 (10), 2.3831 (10) and 2.002 (3) Å, respectively.

Comment

The title compound, (I), first prepared by Rimml & Venanzi (1983), belongs to a group of complexes extensively used in catalysis (Albrecht & Koten, 2001; Herrmann *et al.*, 1999). However, there is no reported crystal structure of this compound.



(I)

The coordination geometry around the Pt atom in (I) is pseudo-square-planar, with the *P,C,P′*-tridentate ligand forming two five-membered rings that are tilted with respect to the coordination plane. The largest deviation from the least-squares plane through PtP_2CCl is 0.034 (2) Å (for C1) and the angle between the plane of the C1–C6 benzene ring (including C7 and C8) and the coordination plane is 19.1 (1)°. The two Pt—P, the Pt—Cl and the Pt—C distances are 2.2794 (9)/2.2749(10), 2.3831 (10) and 2.002 (3) Å, respectively. Distances and angles are very similar to the corresponding *rac*-CHIRAPHOS complex (Gorla *et al.*, 1994). Selected bond lengths and angles are given in Table 1.

Experimental

The title compound was synthesized using a modification of a published procedure (Bennett *et al.*, 1993). A solution of *n*-BuLi in hexane (1.25 ml, 1.6 *M*, 2.12 mmol) was added at 195 K to a solution of $HPPh_2$ (1.25 ml, 2.12 mmol) in THF (11 ml). After the addition was complete, the reaction mixture was stirred at the same temperature for 1 h, and then allowed to warm to room temperature over a period of 1 h. It was then cooled to 195 K and a solution of 1,3-bis(chloromethyl)benzene (1.86 g, 1.06 mmol) in THF (4 ml) was added dropwise. The reaction mixture was allowed to warm to room temperature and was then stirred overnight. The solvent was evaporated. Without purification, mesitylene (50 ml) and $PtCl_2$(COD) (300 mg, 0.85 mmol) were added. The mixture was heated to reflux and triethylamine (0.18 ml) was added carefully. The mixture was stirred under reflux overnight. The yellow solution was

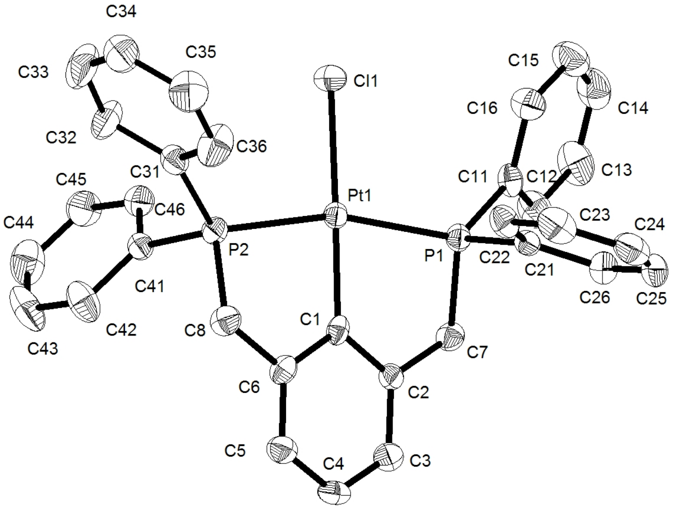


Figure 1
View of (I), with the atom numbering and 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

allowed to cool to room temperature and filtered over celite to remove a yellow precipitate and a small amount of a black solid. The filtrate was evaporated to dryness and treated with ethanol (3 × 3 ml) and *n*-hexane (1 ml). The product (260 mg, 0.39 mmol) was pale yellow (yield 44%). NMR spectra were in agreement with those published earlier (Rimml & Venanzi, 1983). Slow evaporation of a CD_2Cl_2 solution gave X-ray quality crystals of the title compound.

Crystal data

$[PtCl(C_{32}H_{27}P_2)]$
$M_r = 704.02$
Monoclinic, $P2_1/n$
$a = 10.290$ (2) Å
$b = 16.117$ (3) Å
$c = 17.173$ (3) Å
$\beta = 105.47$ (3)°
$V = 2744.8$ (10) Å^3
$Z = 4$
$D_x = 1.704$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 6134 reflections
$\theta = 2.4$–24.8°
$\mu = 5.35$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.25 × 0.20 × 0.12 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.312$, $T_{\max} = 0.526$
27 382 measured reflections
8500 independent reflections
4739 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 31.8°$
$h = -14 \rightarrow 13$
$k = -22 \rightarrow 22$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.041$
$S = 0.91$
8500 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.77$ e Å^{-3}
$\Delta\rho_{\min} = -1.10$ e Å^{-3} (0.51 Å from Pt1)

Table 1
Selected geometric parameters (Å, °).

Pt1—C1	2.002 (3)	P2—C31	1.819 (3)
Pt1—P2	2.2749 (10)	P2—C8	1.835 (3)
Pt1—P1	2.2794 (9)	P1—C11	1.816 (3)
Pt1—Cl1	2.3831 (10)	P1—C21	1.821 (3)
P2—C41	1.812 (3)	P1—C7	1.825 (3)
C1—Pt1—P2	82.18 (9)	C1—Pt1—Cl1	178.53 (9)
C1—Pt1—P1	81.04 (9)	P2—Pt1—Cl1	97.31 (4)
P2—Pt1—P1	163.11 (3)	P1—Pt1—Cl1	99.51 (3)

H atoms were constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL*97.

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