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Key indicators

Single-crystal X-ray study T = 294 KMean $\sigma(C-C) = 0.008 \text{ Å}$ Disorder in solvent or counterion R factor = 0.032 wR factor = 0.082 Data-to-parameter ratio = 14.8

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Bis[bis(dicyclohexylphosphino)methane- $\kappa^2 P$,P']platinum(II) bis(perchlorate) 1,2-dichloroethane tetrasolvate

In the centrosymmetric title complex, $[Pt(C_{25}H_{46}P_2)_2]-(ClO_4)_2 \cdot 4C_2H_4Cl_2$, the Pt atom is four-coordinated by P atoms in a distorted square-planar coordination geometry and the bis(dicyclohexylphosphino)methane ligands form two planar four-membered chelate rings.

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Comment

Heterodinuclear d^8-d^{10} metal complexes containing d^8 Pt^{II}, Pd^{II} or Ni^{II} and d^{10} Au^I, Ag^I or Cu^I ions with a bridging ligand have attracted considerable attention due to their diverse structures and rich photoluminescent properties (Yip *et al.*, 1991, 1993, 1994; Pettijohn *et al.*,1998; Crespo *et al.*, 2000). Generally, these Pt complexes are synthesized from a highly active intermediate, for example $[Pt(dppm)_2]^{2+}$ and $[Pt(dcpm)_2]^{2+}$, where dppm is bis(diphenylphosphino)-methane and dcpm is bis(dicyclohexylphosphino)methane (Cooper *et al.*, 1984; Xia *et al.*, 2003). We report here the title complex, $[Pt(dcpm)_2](ClO_4)_2 \cdot 4ClCH_2CH_2CI$, (I).



The structure of the $[Pt(dcpm)_2]^{2+}$ cation in (I) is centrosymmetric, with the Pt atom within a P₄ donor set defined by two symmetrically chelating diphosphine ligands (Fig. 1). In the dcpm chelate rings, the Pt-P bond lengths are slightly different from each other [Pt1-P1 = 2.3450 (9) Å and Pt1 -P2 = 2.3558 (9) Å]. These values are essentially identical to those found in $[Pt(dppm)_2]^{2+}$ [2.321 (3) and 2.333 (9) Å]. The P1-Pt1-P2 bond angle is 71.68 (3), which is larger than that found in $[Pt(dppm)_2]^{2+}$ [70.46 (3)°], due to steric repulsion.

In the crystal packing, some $C-H\cdots O$ interactions are present, involving H atoms of the cyclohexyl rings and O atoms of the ClO_4^- anion (Table 1).

Experimental

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Dcpm (Acros, 97%) and K_2PtCl_4 (Kunming Institute of Precious Metals, 98%) were obtained commercially. Solvents were distilled

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using standard techniques and were saturated with nitrogen before use. A mixture of K_2PtCl_4 (41.5 mg, 0.1 mmol) and dcpm (81.7 mg, 0.2 mmol,) in ethanol (20 ml) was stirred for 8 h at room temperature under an atmosphere of N_2 , and then reduced to 5 ml. Addition of diethyl ether (20 ml) gave a yellow solid, which was filtered off and washed with water and diethyl ether. Recrystallization by diffusion of diethyl ether into a 1,2-dichloroethane solution yielded pale-yellow crystals of (I) suitable for X-ray diffraction.

V = 1822.5 (4) Å³

 $D_x = 1.464 \text{ Mg m}^{-3}$ Mo *K* α radiation $\mu = 2.43 \text{ mm}^{-1}$ T = 294 (2) K

Block, pale yellow

 $R_{\rm int} = 0.020$

 $\theta_{\rm max} = 25.0^\circ$

 $0.22 \times 0.20 \times 0.20$ mm

9358 measured reflections 6377 independent reflections 6254 reflections with $I > 2\sigma(I)$

Z = 1

Crystal data

$[Pt(C_{25}H_{46}P_2)_2](ClO_4)_2 \cdot 4C_2H_4Cl_2$
$M_r = 1606.91$
Triclinic, $P\overline{1}$
a = 11.9213 (15) Å
b = 13.7673 (16) Å
c = 13.9489 (16) Å
$\alpha = 109.137 \ (2)^{\circ}$
$\beta = 112.534 \ (2)^{\circ}$
$\gamma = 103.398 \ (2)^{\circ}$

Data collection

Bruker SMART CCD area-detector
diffractometer
φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2000)
$T_{\min} = 0.602, T_{\max} = 0.623$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_0^2) + (0.0452P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	+ 2.2251P]
$wR(F^2) = 0.082$	where $P = (F_0^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{\rm max} = 0.003$
6377 reflections	$\Delta \rho_{\rm max} = 1.25 \text{ e } \text{\AA}^{-3}$
432 parameters	$\Delta \rho_{\rm min} = -1.10 \ {\rm e} \ {\rm \AA}^{-3}$
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (Å, °).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - H \cdot \cdot \cdot A$
C7-H7···O2	0.98	2.56	3.502 (7)	161
$C13-H13B\cdots O3^{i}$	0.97	2.38	3.346 (8)	171
$C27 - H27A \cdots O1$	0.97	2.54	3.421 (19)	151

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

All H atoms were placed in calculated positions. The H atoms were then refined as riding, with C–H distances of 0.97 Å and with $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$. During the structure refinement, a region of electron density was identified as a highly disordered molecule of 1,2dichloroethane. In the final structure model, the 1,2-dichloroethane molecule was disordered over two positions. The bond distances in the minor component were restrained to the corresponding bond distances in the major component. The site occupancy factors were refined to 1.78 (1) (C26–Cl2, C27–Cl3, C26–Cl4 and C26–Cl4) and 1.52 (1) (C26–C27, C28–C29, C26'–C27' and C28'–C29') and fixed in subsequent refinement cycles. The highest peak is located within 1.03 Å of atom Cl4, and the deepest hole is 1.07 Å from the same atom.



Figure 1

The molecular structure of the cation of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (a) -x + 2, -y, -z + 1]

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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