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Bis(*O*,*O*′-dicyclohexyl dithiophosphato-$\kappa^2 S,S'$)platinum(II)

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Key indicators

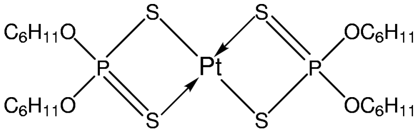
Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.009 Å
R factor = 0.034
wR factor = 0.085
Data-to-parameter ratio = 28.2

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

The monomeric title compound, $[Pt(C_{12}H_{22}O_2PS_2)_2]$, has the Pt atom located on a centre of inversion and existing within an S_4 square-planar geometry.

Comment

The structural chemistry of metal complexes containing 1,1-dithiolate ligands, *e.g.* dithiocarbamate ($^-S_2CNR_2$), xanthate (^-S_2COR) and dithiophosphate ($^-S_2P(OR)_2$), is replete with examples of structural diversity, both molecular and supramolecular, for compounds that differ only in the nature of their remote *R* groups. This is no better exemplified than in the structures of the zinc-triad dithiolates (Tiekink, 2003). In order to delineate the reasons for such a range of structural motifs, it is imperative to determine the relative coordination abilities of the dithiolate ligands themselves. In this context, systematic studies on the nickel(II) and tellurium dithiocarbamates and xanthates (Cox & Tiekink, 1999*a*,*b*) and theoretical studies of the xanthate ligands themselves (Buntine *et al.*, 2003) have shown that the *R* groups do not exert an electronic influence on the coordination ability of these ligands. As a continuation of this theme, the X-ray crystal structure of $Pt[S_2P(OCy)_2]_2$ (Cy is cyclohexyl), (I), was examined.



(I)

The structure of (I) (Fig. 1) is centrosymmetric, with the Pt atom within an S_4 donor set defined by two symmetrically chelating dithiophosphate ligands (Table 1). The Pt—S bond distances are essentially identical to those found in the other two binary platinum dithiophosphate structures available in the literature, namely *R* = Et (Gianini *et al.*, 2000) and *R* = ^{i}Pr (Tkachev & Atovmyan, 1982), thereby providing some evidence to indicate that the *R* groups in dithiophosphate ligands do not influence significantly the electronic structure of the PS_2^- donor set in dithiophosphate anions.

Experimental

Following the procedure of Gianini *et al.* (2000), a solution of K_2PtCl_4 (Agros, 0.2 g, 0.9 mmol) in CH_3CN/H_2O (1:1 *v*/*v*, 10 ml) was added dropwise to a solution of sodium cyclohexyl dithiophosphate (Cheminova, 0.6 g, 1.9 mol) in the same mixture of solvents (40 ml). After heating under reflux for 1.5 h, the resulting brown solid was filtered off, washed with CH_3CN/H_2O and vacuum dried (0.4 g, 53%).

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Crystals were prepared by the slow evaporation of a CH_2Cl_2/diethyl ether/hexane (1:1:2) mixture [m.p. 442–446 K (decomposition)]. Analysis found: C 36.78, H 5.73, S 16.49%; $C_{24}H_{44}O_4P_2S_4Pt$ requires: C 36.87, H 5.67, S 16.40%. ^1H NMR ($CDCl_3$): δ 4.71 (*m*, OCH), 2.05–1.24 (*m*, CH_2); ^{13}C NMR ($CDCl_3$): δ 78.88 (OCH), 33.34, 25.02, 23.62 (CH_2); ^{31}P ($CDCl_3$): δ 96.1 [$^2J(^{31}P–^{195}Pt)$ = 439 Hz]. IR (KBr disk): ν(C—O) 1157, ν(P—O) 977, $\nu(P—S)_{asymm}$ 646, $\nu(P—S)_{sym}$ 582 cm^{-1}.

Crystal data

$[Pt(C_{12}H_{22}O_2PS_2)_2]$
M_r = 781.86
Monoclinic, $P2_1/n$
a = 11.876 (4) Å
b = 9.379 (3) Å
c = 13.889 (5) Å
β = 96.573 (7)°
V = 1536.9 (9) $Å^3$
Z = 2
D_x = 1.690 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4456 reflections
θ = 2.4–28.4°
μ = 4.97 mm^{-1}
T = 223 (2) K
Block, orange
0.31 × 0.21 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.284, T_{max} = 0.608
12 430 measured reflections
4512 independent reflections
3533 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 30.3°
$h = -8 \rightarrow 16$
$k = -13 \rightarrow 12$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.085
S = 1.05
4512 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 2.17 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.52 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Pt—S1	2.3271 (10)	P1—O1	1.557 (2)
Pt—S2	2.3228 (12)	P1—O2	1.555 (3)
S1—P1	1.9997 (14)	O1—C1	1.466 (4)
S2—P1	2.0090 (13)	O2—C7	1.475 (4)
S1—Pt—S2	83.89 (3)	S1—P1—O2	114.83 (11)
S1—Pt—S2^i	96.11 (3)	S2—P1—O1	115.35 (11)
Pt—S1—P1	86.87 (4)	S2—P1—O2	114.78 (12)
Pt—S2—P1	86.77 (4)	O1—P1—O2	96.17 (12)
S1—P1—S2	101.67 (5)	P1—O1—C1	121.5 (2)
S1—P1—O1	114.85 (11)	P1—O2—C7	123.4 (2)

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

The H atoms were included in the riding-model approximation, with C—H = 0.99 (methine) and 0.98 Å (methylene), and $U_{iso}(H) = 1.2U_{eq}(C)$. The largest peak in the final difference Fourier map was 0.78 Å from Pt1.

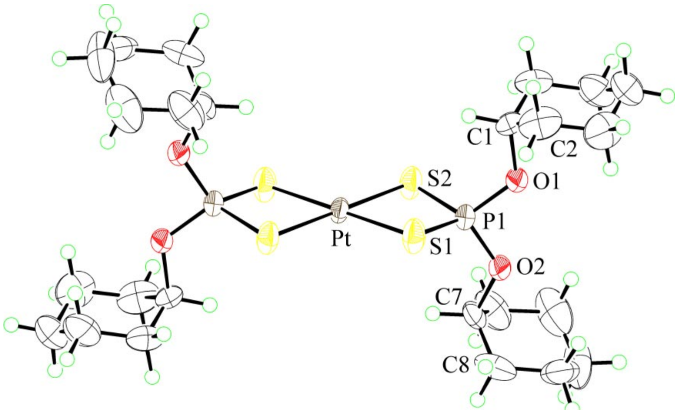

Figure 1
The molecular structure and crystallographic numbering scheme for (I); the Pt atom is located on a centre of inversion. Displacement ellipsoids are shown at the 35% probability level (Johnson, 1976). Unlabelled atoms are related to labelled atoms by $(-x, -y, -z)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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