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[1,2-Bis(diphenylphosphino)ethane-*P*,*P*′]-bis(α-toluenethiolato-*S*)palladium(II), $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2Ph)_2]$

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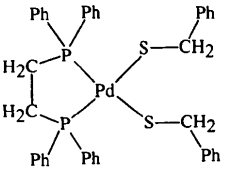
Abstract

The title compound, $[Pd(C_7H_7S)_2(C_{26}H_{24}P_2)]$, is a mononuclear palladium(II) complex. The molecule possesses a crystallographic twofold axis and the Pd atom is four-coordinated by two phosphine P atoms and two S atoms from $PhCH_2S^-$ ligands, and has a distorted square-planar geometry. The Pd—S and Pd—P distances are 2.360 (2) and 2.277 (2) Å, respectively.

Comment

Transition metal compounds with mixed sulfur and phosphine ligands have attracted much attention due to their relevance and importance to a wide variety of chemical and industrial systems. Of the nickel group metals, many nickel compounds with such mixed ligands have been reported. Surprisingly few palladium compounds, such as $[Pd_2(SC_6F_5)_2(PPh_3)_2]$ (Fenn & Segrott, 1972), have been structurally characterized. We have recently reported the palladium compounds $[Pd(SCH_2CH_2SCH_2CH_2S)(PPh_3)_2]$ and $[Pd_2(PPh_3)_2(HOC_6H_4S)_2Cl_2]$ (Cao, Hong, Jiang, Xie & Liu, 1996), $[Pd_2(PPh_3)_2(SC_2H_4S)_2]$ (Cao, Hong, Jiang & Liu, 1995) and $[Pd\{Ph_2P(CH_2)_3PPh_2\}(SC_3H_6S)].CH_3CN$ (Su, Hong, Zhou, Xue, Liu & Mak, 1996). We report here the crystal structure of a mononuclear complex, $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2Ph)_2]$, (I).



(I)

The title compound is a mononuclear palladium(II) complex, where the Pd atom is four-coordinated by two phosphine P atoms and two S atoms from two $PhCH_2S^-$ ligands, and has a distorted square-planar geometry. The molecule possesses a crystallographic twofold axis passing through the Pd atoms and the midpoint of C(1)—C(1^i) [symmetry code: (i) $1-x, -y, z$]. The Pd(1), P(1), P(1^i), S(1) and S(1^i) atoms are in a plane with displacements of 0.0000, −0.087, 0.085, 0.073 and −0.070 Å, respectively. The Pd—S and Pd—P distances are 2.360 (2) and 2.277 (2) Å, respectively. The structure of the title compound is depicted in Fig. 1.

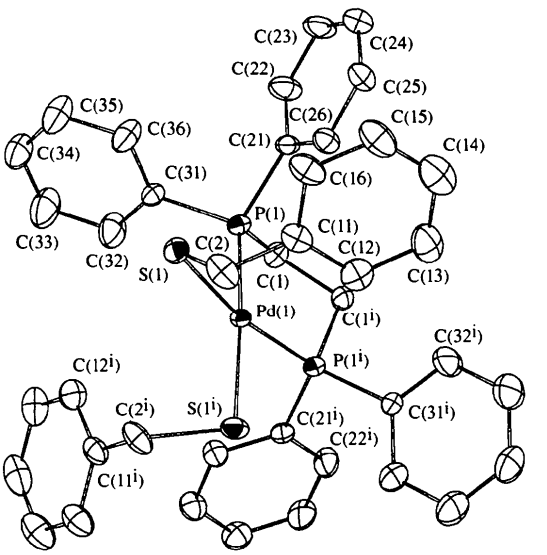


Fig. 1. The structure of $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2S)_2]$ with displacement ellipsoids at the 30% probability level.

Experimental

The title compound was obtained from the reaction of $PdCl_2$, $NaSCH_2Ph$ and $Ph_2P(CH_2)_2PPh_2$ (molar ratio 1:2:1) in MeOH, and recrystallized from CH_3CN solution.

Crystal data

$[Pd(C_7H_7S)_2(C_{26}H_{24}P_2)]$ — Mo $K\alpha$ radiation
$M_r = 751.23$ — $\lambda = 0.71073$ Å

Orthorhombic
*Aba*2
a = 18.070 (3) Å
b = 21.230 (4) Å
c = 9.021 (2) Å
V = 3460.9 (12) Å^3
Z = 4
D_x = 1.442 Mg m^{-3}
D_m not measured

Cell parameters from 20 reflections
θ = 9–12°
μ = 0.763 mm^{-1}
T = 296 (1) K
Rectangular
0.35 × 0.20 × 0.15 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.670, T_{max} = 0.923
3370 measured reflections
3334 independent reflections

2992 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.032
θ_{max} = 25°
$h = -21 \rightarrow 0$
$k = 0 \rightarrow 24$
$l = -10 \rightarrow 10$
3 standard reflections monitored every 250 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.039
wR = 0.054
S = 1.23
2992 reflections
203 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + k(F_o^2)]$

$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Extinction correction: none
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)*

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Pd(1)	1/2	0	0.5312	2.074 (8)
S(1)	0.53051 (9)	0.07872 (7)	0.3568 (2)	3.19 (3)
P(1)	0.54228 (8)	0.06336 (6)	0.7164 (2)	2.44 (2)
C(2)	0.4484 (4)	0.0792 (3)	0.2325 (7)	4.0 (1)
C(1)	0.5390 (3)	0.0179 (3)	0.8892 (7)	3.0 (1)
C(11)	0.3811 (4)	0.1055 (3)	0.3030 (7)	3.4 (1)
C(12)	0.3342 (4)	0.0675 (3)	0.3898 (9)	4.3 (2)
C(13)	0.2708 (4)	0.0921 (4)	0.458 (1)	5.4 (2)
C(14)	0.2529 (5)	0.1558 (4)	0.433 (1)	5.8 (2)
C(15)	0.2983 (5)	0.1929 (4)	0.346 (1)	5.6 (2)
C(16)	0.3625 (4)	0.1686 (3)	0.2824 (9)	4.5 (2)
C(21)	0.4924 (3)	0.1361 (3)	0.7603 (8)	2.7 (1)
C(22)	0.5189 (4)	0.1761 (3)	0.873 (1)	4.5 (1)
C(23)	0.4818 (4)	0.2334 (3)	0.899 (1)	4.7 (2)
C(24)	0.4201 (4)	0.2489 (3)	0.8198 (9)	4.2 (2)
C(25)	0.3935 (4)	0.2085 (3)	0.7107 (9)	3.8 (1)
C(26)	0.4296 (4)	0.1512 (3)	0.6817 (8)	3.2 (1)
C(31)	0.6375 (3)	0.0878 (3)	0.6913 (7)	2.7 (1)
C(32)	0.6955 (4)	0.0476 (4)	0.721 (1)	5.4 (2)
C(33)	0.7696 (4)	0.0665 (4)	0.695 (1)	7.0 (2)
C(34)	0.7820 (4)	0.1249 (4)	0.632 (1)	5.8 (2)
C(35)	0.7240 (4)	0.1659 (4)	0.596 (1)	6.1 (2)
C(36)	0.6510 (4)	0.1475 (4)	0.628 (1)	4.9 (2)

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

Pd(1)—S(1)	2.360 (2)	P(1)—C(21)	1.832 (6)
Pd(1)—P(1)	2.277 (2)	P(1)—C(31)	1.810 (6)
S(1)—C(2)	1.860 (7)	C(2)—C(11)	1.48 (1)
P(1)—C(1)	1.834 (7)	C(1)—C(1^i)	1.601 (9)
S(1)—Pd(1)—S(1^i)	96.41 (6)	C(21)—P(1)—C(31)	104.7 (3)
S(1)—Pd(1)—P(1)	89.57 (6)	S(1)—C(2)—C(11)	113.5 (5)
S(1)—Pd(1)—P(1^i)	170.27 (5)	P(1)—C(1)—C(1^i)	106.1 (4)
P(1)—Pd(1)—P(1)	85.58 (6)	C(2)—C(11)—C(12)	121.2 (6)
Pd(1)—S(1)—C(2)	102.7 (2)	C(2)—C(11)—C(16)	120.1 (6)
Pd(1)—P(1)—C(1)	107.6 (2)	P(1)—C(21)—C(22)	119.8 (5)
Pd(1)—P(1)—C(21)	119.4 (2)	P(1)—C(21)—C(26)	119.4 (5)
Pd(1)—P(1)—C(31)	113.3 (2)	P(1)—C(31)—C(32)	121.4 (5)
C(1)—P(1)—C(21)	104.1 (3)	P(1)—C(31)—C(36)	118.1 (5)
C(1)—P(1)—C(31)	106.7 (3)		

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

The structure was solved by direct methods. All non-H atoms were refined by full-matrix least-squares methods (*LSFM*; B. A. Frenz & Associates, Inc., 1985), with anisotropic displacement parameters, and the calculations included H atoms. All calculations were performed on a 486PC computer with the *MolEN* (PC version; Fair, 1990) program package.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *MULTAN*82 (Main *et al.*, 1982) in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *WP*5.1 and *GCIF* (local software).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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