Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Di-μ-phenylselenolato-bis[chloro(triphenylphosphine)palladium(II)]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.094
Data-to-parameter ratio = 19.4

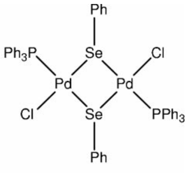
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 January 2004
Accepted 13 January 2004
Online 23 January 2004

The title compound, $[Pd_2Cl_2(\mu\text{-}C_6H_5Se)_2(C_{18}H_{15}P)_2]$, crystallizes as a centrosymmetric phenylselenolato-bridged dinuclear palladium complex. The Pd atom shows a slightly distorted square-planar coordination geometry, with the Pd—Se distance *trans* to the terminal Cl^- ligand at 2.4174 (6) Å and that *trans* to the triphenyl phospine at 2.4748 (7) Å. These values are consistent with the relative *trans* influence of Cl^- and PPh_3. The terminal Cl^- ligands are in *trans* positions with respect to each other.

Comment

Organic selenolate and tellurolate complexes of palladium and platinum can conveniently be prepared by the treatment of halogenated metal complexes with RE^- (where E is Se or Te, and R is alkyl or aryl) obtained through the reduction of R_2E_2, or by the oxidative addition of R_2E_2 to zero-valent palladium or platinum (Alyea *et al.*, 1998; Hannu *et al.*, 2000; Hannu-Kuure, Komulainen *et al.*, 2003; Oilunkaniemi *et al.*, 1999, 2001, and references therein). The product distribution is dependent on the molar ratio of the reagents. In this paper, we report the crystal and molecular structure of dinuclear $[Pd_2Cl_2(\mu\text{-SePh})_2(PPh_3)_2]$, (I), which was formed in small amounts by the reaction of $[PdCl_2(PPh_3)_2]$ and NaSePh in dichloromethane. The structure determination of (I) completes this series of structures of dinuclear palladium selenolate complexes.



(I)

The crystal structure of the centrosymmetric complex, (I), is shown in Fig. 1, together with the atomic numbering scheme. Selected bond distances and angles are listed in Table 1. The Pd atoms show a slightly distorted square-planar coordination geometry (the sum of the bond angles is 359.8°). The Pd—Se bond lengths are 2.4174 (6) Å (*trans* to Cl^-) and 2.4748 (7) Å (*trans* to Ph_3P). These bond distances are consistent with the relative *trans* influence of the Cl^- and triphenylphosphine ligands. They can be compared with the Pd—Se bond lengths of 2.4092 (9) and 2.4376 (7) Å in $[Pd_2Cl_2(\mu\text{-Cl})(\mu\text{-SeTh})(PPh_3)_2]$ (Th is 2-thienyl; Oilunkaniemi *et al.*, 2003), and with the bridging Pd—Se distances of 2.466 (1) Å in $[Pd_2(\mu\text{-SeTh})_2(SeTh)_2(PPh_3)_2]$ (Oilunkaniemi *et al.*, 1999) and 2.463 (1) and 2.494 (1) Å in $[Pd_2(\mu\text{-SePh})_2(SePh)_2(PPh_3)_2]$

(Oilunkaniemi *et al.*, 2001). The Pd—Cl bond length in (I) is 2.338 (1) Å, slightly shorter than the terminal Pd—Cl bond lengths of 2.388 (1) and 2.346 (1) Å in $[Pd_2Cl_2(\mu\text{-}Cl)(\mu\text{-}SeTh)(PPh_3)_2]$ (Oilunkaniemi *et al.*, 2003).

The packing of the molecules of (I) is shown in Fig. 2. The closest contact, of 2.794 Å between atoms Cl1 and H134, links the complex molecules into a two-dimensional network.

Experimental

A small number of orange crystals of $[Pd_2Cl_2(\mu\text{-}SePh)_2(PPh_3)_2]$, (I), were formed by the reaction of $[PdCl_2(PPh_3)_2]$ with NaSePh in a molar ratio of 2:1 in dichloromethane. The synthetic procedure was recently published by Hannu-Kuure, Palda'n *et al.* (2003). A well formed crystal of (I) was selected for the crystal structure determination.

Crystal data

$[Pd_2Cl_2(C_6H_5Se)_2(C_{18}H_{15}P)_2]$
$M_r = 1120.36$
Monoclinic, $P2_1/n$
$a = 8.878$ (2) Å
$b = 17.533$ (4) Å
$c = 14.398$ (3) Å
$\beta = 105.60$ (3)°
$V = 2158.7$ (9) Å^3
$Z = 2$

$D_x = 1.724$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4136 reflections
$\theta = 3.8$–27.5°
$\mu = 2.75$ mm^{-1}
$T = 150$ (2) K
Plate, orange
0.25 × 0.15 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: ψ scan (*SHELXTL*; Bruker, 2001)
$T_{min} = 0.546$, $T_{max} = 0.810$
23 990 measured reflections

4914 independent reflections
4136 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -22 \rightarrow 22$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.094$
$S = 1.09$
4914 reflections
253 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 3.9989P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.91$ e Å^{-3}
$\Delta\rho_{min} = -0.94$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—P1	2.3075 (10)	Se1—C1	1.935 (4)
Pd1—Cl1	2.3383 (11)	P1—C111	1.806 (4)
Pd1—Se1	2.4174 (6)	P1—C121	1.823 (4)
Pd1—Se1^i	2.4748 (7)	P1—C131	1.825 (4)
P1—Pd1—Cl1	91.24 (4)	Se1—Pd1—Se1^i	83.098 (18)
P1—Pd1—Se1	97.03 (3)	C1—Se1—Pd1	94.73 (11)
Cl1—Pd1—Se1	167.67 (3)	C1—Se1—Pd1^i	102.07 (12)
P1—Pd1—Se1^i	178.69 (3)	Pd1—Se1—Pd1^i	96.902 (18)
Cl1—Pd1—Se1^i	88.43 (3)		

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

H atoms were treated as riding, with C—H distances of 0.95 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

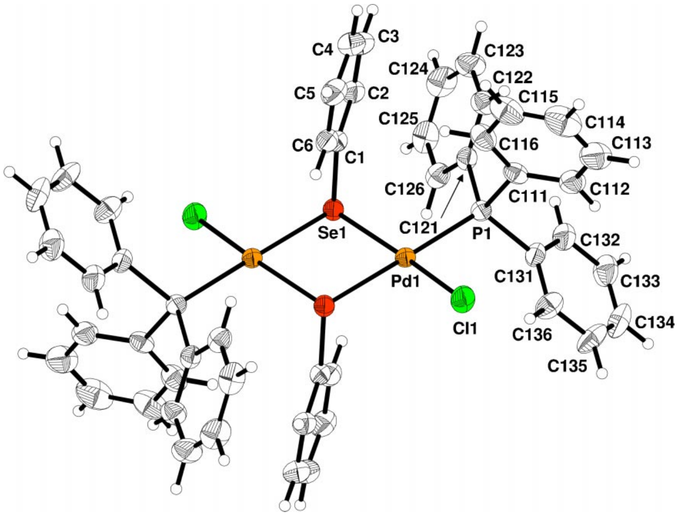


Figure 1
The molecular structure of (I), with the atom-numbering scheme of the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

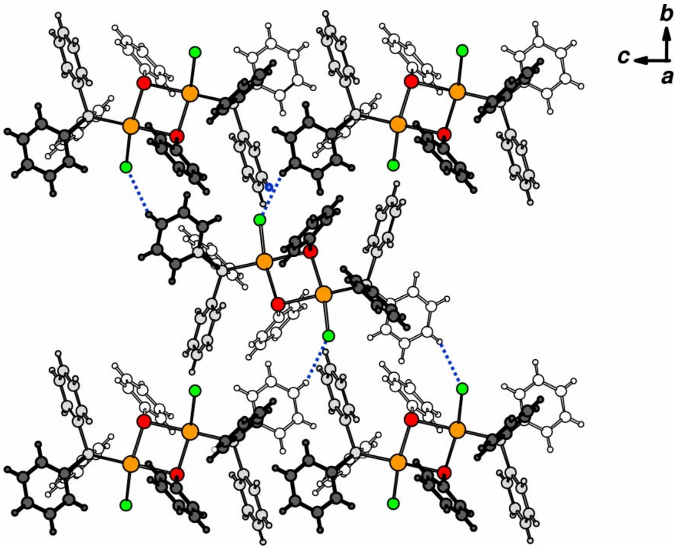


Figure 2
The packing of molecules of (I).

Financial support from the Academy of Finland, the Finnish Cultural Foundation, the Tauno Tönning Foundation and the Emil Aaltonen Foundation is gratefully acknowledged.

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