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trans-Di-μ-chloro-bis[chloro(triphenylphosphine)-palladium] benzene disolvate

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Key indicators

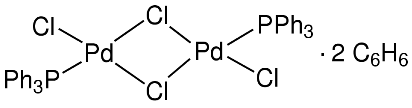
Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.089
Data-to-parameter ratio = 17.3

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

The title complex, *trans*-$[PdCl_2(PPh_3)]_2$, in the crystal structure of its benzene disolvate, $[Pd_2Cl_4(C_{18}H_{15}P)_2]\cdot 2C_6H_6$,occupies a special position in the inversion centre. The Pd atom has a distorted square-planar coordination environment formed by a triphenylphosphine [Pd—P 2.2278 (6) Å], a terminal chloride [Pd–Cl 2.2722 (7) Å], and two bridging chloride ligands. The Pd—μ-Cl bond in the *trans*-position to the phosphine ligand [2.4128 (6) Å] is considerably longer than the Pd—μ-Cl bond in the *trans*-position to the terminal chloride [2.3228 (6) Å].

Comment

The general class of $[PdCl_2P]_2$ complexes has been known since the early studies of Mann and co-workers (Mann & Purdie, 1935; Mann & Wells, 1938). The title complex, (I), was prepared as a precursor to other palladium complexes.



(I)

The dimeric molecule of the complex, *trans*-$[PdCl_2(PPh_3)]_2$, in the crystal structure of (I) (Fig. 1) is located around an inversion centre. Its geometry is similar to that observed in the structure of the unsolvated *trans*-$[PdCl_2(PPh_3)]_2$ complex, (II), published previously (Vicente *et al.*, 1997; ROQZAY in the Cambridge Structural Database, Version 5.24; Allen, 2002). The Pd atom in (I) has a distorted square-planar coordination environment formed by a triphenylphosphine [Pd1—P1 2.2278 (6) Å], a terminal chloride [Pd1—Cl2 2.2722 (7) Å], and two bridging chloride ligands. As expected, due to the higher *trans*-influence of the phosphine ligand, the Pd1—Cl1 bond in the *trans*-position to the phosphine ligand is considerably longer [2.4128 (6) Å] than the Pd1—Cl1^{i} bond [2.3228 (6) Å; symmetry code: (i) $-x, -y, 1 - z$] in the *trans*-position to the terminal chloride. The noticeable difference in the distribution of the exocylic bond angles in (I) and (II) [P1—Pd1—Cl1^{i} 95.59 (2)° in (I) *versus* 92.73° in (II); P1—Pd1—Cl2 87.38 (2)° in (I) *versus* 92.41° in (II)] is most probably due to different conformations of the PPh_3 ligands [torsion angles Cl1^{i}—Pd1—P1—C11 −7.77 (10)° and Pd1—P1—C11—C12 85.0 (2) in (I) *versus* −31.2 and 121.4°, respectively, in (II)].

Experimental

Indenyllithium (2.5 mg, 0.018 mmol) was added to a C_6D_6 solution (1 ml) of $(PhCN)_2PdCl_2$ (7 mg, 0.018 mmol) at room temperature.

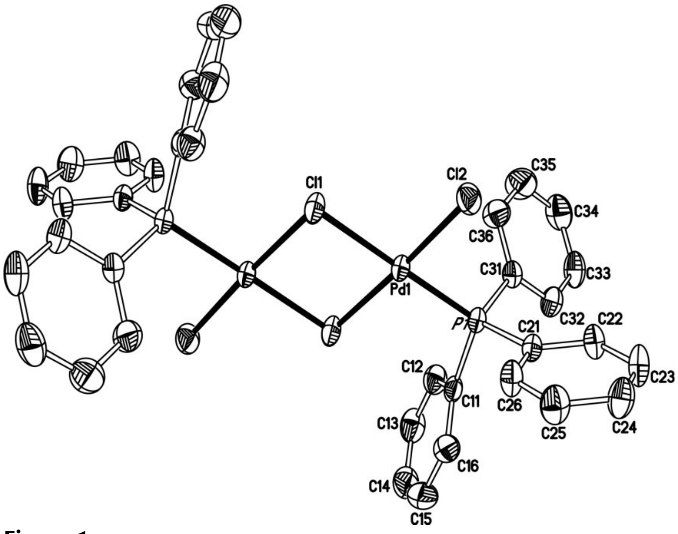


Figure 1
View of the dimeric molecule $[PdCl_2PPh_3]_2$ in the structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. The H atoms have been omitted.

After adding PPh_3 (2.3 mg, 0.009 mmol), the resulting dark red mixture was stirred for approximately 10 min. After slow evaporation under ambient conditions, the by-product of this reaction, the title compound, precipitated forming crystals suitable for X-ray diffraction study.

Crystal data

$[Pd_2Cl_4(C_{18}H_{15}P)_2]\cdot 2C_6H_6$
$M_r = 1035.40$
Monoclinic, $P2_1/c$
$a = 9.6895\ (2)$ Å
$b = 18.9619\ (3)$ Å
$c = 12.8905\ (2)$ Å
$\beta = 109.743\ (1)^\circ$
$V = 2229.17\ (7)$ Å^3
$Z = 2$

$D_x = 1.542$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 7164 reflections
$\theta = 2.3$–72.8°
$\mu = 9.64$ mm^{-1}
$T = 220\ (2)$ K
Block, dark orange–red
0.27 × 0.14 × 0.10 mm

Data collection

Bruker AXS SMART 2K/Platform diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.238$, $T_{max} = 0.382$
26449 measured reflections

4387 independent reflections
3992 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 72.8^\circ$
$h = -10 \rightarrow 11$
$k = -23 \rightarrow 23$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.089$
$S = 1.09$
4387 reflections
253 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.41$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—P1	2.2278 (6)	Pd1—Cl1^i	2.3228 (6)
Pd1—Cl2	2.2722 (7)	Pd1—Cl1	2.4128 (6)
P1—Pd1—Cl2	87.38 (2)	Cl2—Pd1—Cl1	92.04 (2)
P1—Pd1—Cl1^i	95.59 (2)	Cl1^i—Pd1—Cl1	84.99 (2)
Cl2—Pd1—Cl1^i	176.98 (2)	Pd1^i—Cl1—Pd1	95.01 (2)
P1—Pd1—Cl1	179.39 (2)		

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

In order to check for crystal decay, upon completion of the data collection the first 101 frames were recollected. Thus, 213 reflections were treated as intensity standards. No statistically significant decay was observed. The H atoms were positioned geometrically (C—H = 0.94 Å) and were included in the refinement in the riding model approximation; their displacement parameters were set at 1.2 times U_{eq} of the parent C atoms. In the final difference Fourier map, the four most prominent peaks, in the range 0.78–1.41 e Å^{-3}, were located 0.90–0.99 Å from the Pd atom. The VOID routine of *PLATON* (Spek, 1995) was used to confirm that no significant voids are present in the structure.

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

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1997). *SHELXTL*. Release 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* (Release 6.06) and *SMART* (Release 5.059). Bruker AXS Inc., Madison, Wisconsin, USA.
Mann, F. G. & Purdie, D. (1935). *J. Chem. Soc.* pp. 1549–1563.
Mann, F. G. & Wells, A. F. (1938). *J. Chem. Soc.* pp. 702–710.
Sheldrick, G. M. (1996). *SADABS*. Bruker AXS Inc., Madison, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1995). *PLATON*. Version of July 1995. University of Utrecht, The Netherlands.
Vicente, J., Lagunas, M. C., Bleuel, E. & Ramirez de Arellano, M. C. (1997). Private Communication, CCDC refcode ROQZAY. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.

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