

***trans*-Dichloridobis(triphenylphosphine)-palladium(II)<sup>1</sup>**Josefina Pons,<sup>a\*</sup> Jordi García-Antón,<sup>a</sup> Xavier Solans,<sup>b</sup> Mercè Font-Bardia<sup>b</sup> and Josep Ros<sup>a</sup><sup>a</sup>Departamento de Química, Unitat de Química Inorgànica, Universitat Autònoma de Barcelona, 08193 Bellaterra, Barcelona, Spain, and <sup>b</sup>Departament de Cristallografia, Universitat de Barcelona, Martí i Franquès, sn, E-08028 Barcelona, Spain  
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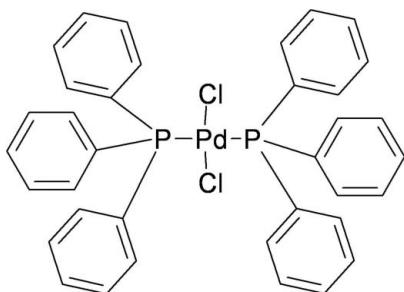
Received 17 March 2008; accepted 27 March 2008

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.077; data-to-parameter ratio = 26.2.

The title compound,  $[\text{PdCl}_2\{\text{P}(\text{C}_6\text{H}_5)_3\}_2]$ , has a slightly distorted square-planar geometry, with the chloride ligands coordinated in a *trans* configuration. The Pd atom is located on a centre of inversion.

**Related literature**

For related literature, see: Ferguson *et al.* (1982); Kitano *et al.* (1983); La Monica & Ardizzoia (1997); Montoya *et al.* (2005); Montoya *et al.* (2006); Mukherjee (2000); Oilunkaniemi *et al.* (2003); Stark *et al.* (1997); Steyl (2006); Trofimenko (1972, 1986).

**Experimental***Crystal data*

$[\text{PdCl}_2(\text{C}_{18}\text{H}_{15}\text{P})_2]$   
 $M_r = 701.84$   
 Monoclinic,  $P2_1/c$   
 $a = 9.296$  (5) Å  
 $b = 19.889$  (8) Å  
 $c = 10.621$  (6) Å  
 $\beta = 121.71$  (4)°

$V = 1670.6$  (15) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.83$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 $0.2 \times 0.17 \times 0.16$  mm

*Data collection*

Mar Research MAR345  
 diffractometer with image-plate  
 detector  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 1999)  
 $T_{\min} = 0.85$ ,  $T_{\max} = 0.87$

4898 measured reflections  
 4898 independent reflections  
 3143 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.077$   
 $S = 0.93$   
 4898 reflections  
 187 parameters

7 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.56$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.36$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Pd—Cl	2.3111 (13)	Pd—P	2.3721 (10)
Cl—Pd—P	87.62 (4)	Cl <sup>i</sup> —Pd—P	92.38 (4)
Cl—Pd—P—Cl	41.9 (2)	Cl—Pd—P—C7	163.9 (2)
Cl—Pd—P—Cl3	−75.7 (2)		

Symmetry code: (i)  $-x + 2, -y, -z + 1$ .

Data collection: *MARXDS* (Kabsch, 1988); cell refinement: *AUTOMAR* (Kabsch, 1988); data reduction: *MARSCALE* (Kabsch, 1988); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

Support by the Spanish Ministerio de Educación y Cultura (Project CTQ2007–639137) is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2685).

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<sup>1</sup> In memory of Professor Xavier Solans i Huguet, deceased September 3, 2007.

**supplementary materials**

*Acta Cryst.* (2008). E64, m621 [ doi:10.1107/S1600536808008337 ]

## ***trans*-Dichloridobis(triphenylphosphine)palladium(II)**

**J. Pons, J. García-Antón, X. Solans, M. Font-Bardia and J. Ros**

### **Comment**

The coordination chemistry of pyrazole derived ligands has been extensively studied in recent years (Trofimenko, 1972, 1986; La Monica *et al.*, 1997; Mukherjee, 2000). Recently, in our laboratory the synthesis and characterization of a family of 1,3,5-pyrazole derived ligands have been developed (Montoya *et al.* 2005) and we have studied the reactivity towards divalent metal ions. The reaction of  $[\text{PdCl}_2L^1]$  ( $L^1 = 2-(1\text{-ethyl-5-phenyl-1}H\text{-pyrazol-3-yl)pyridine}$ ) with  $\text{AgBF}_4$  followed by the addition of  $\text{PPh}_3$  and  $\text{NaBPh}_4$  yields the compound  $[\text{Pd}(L^1)(\text{PPh}_3)_2](\text{BPh}_4)_2$  (Montoya *et al.*, 2006). The title compound was obtained when the triphenylphosphine ligand was added before the precipitation of the chloride ions with  $\text{AgBF}_4$ . In this way,  $\text{PPh}_3$  ligands displace  $L^1$  to form *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]$  (**1**).

Related compounds are *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]$  (**2**) (Ferguson *et al.*, 1982), *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]\cdot\text{C}_6\text{H}_4\text{Cl}_2$  (Kitano *et al.*, 1983), *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]\cdot 2\text{CHCl}_3$  (Stark *et al.*, 1997), *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]\cdot\text{CH}_2\text{Cl}_2$  (Oilunkaniemi *et al.*, 2003), and *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]\cdot\text{C}_2\text{H}_4\text{Cl}_2$  (Steyl, 2006). There are no solvent molecules present in the structure described in this paper. The same behaviour was found for the structure described by Ferguson (**2**), but differences have been found in the crystal systems and space groups [triclinic  $\overline{P1}$  (**2**); monoclinic  $P2_1/c$ , (**1**)]. Moreover, the Pd—Cl and Pd—P bond distances (2.3111 (13) Å and 2.3721 (10) Å, respectively) in complex (**1**) are slightly longer than those found in complex (**2**) (2.290 (1) Å and 2.337 (1) Å, respectively).

### **Experimental**

Treatment of 0.14 mmol (0.060 g) of  $[\text{PdCl}_2(L^1)]$  ( $L^1 = 2-(1\text{-ethyl-5-phenyl-1}H\text{-pyrazol-3-yl)pyridine}$ ) with 0.28 mmol (0.054 g) of  $\text{PPh}_3$  in 10 ml of dichloromethane and 10 ml of methanol provokes the displacement of the pyrazolic ligand from the coordinative sphere of the metallic cation and the formation of *trans*- $[\text{PdCl}_2(\text{PPh}_3)_2]$ . This complex precipitates as a yellow solid and was filtered and dried under vacuum. Single crystals were obtained by recrystallization of the complex in dichloromethane/diethyl ether 1:1. Yield: 0.080 g (81%) -  $\text{C}_{36}\text{H}_{30}\text{Cl}_2\text{P}_2\text{Pd}$  (701.84). % C, 61.60; H, 4.30; found: C, 61.33; H, 4.42;. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu(\text{C—H})_{\text{ar}}$  3047;  $\delta(\text{C—H})_{\text{ar}}$  1437;  $\delta(\text{C—H})_{\text{oop}}$  693. IR (polyethylene,  $\text{cm}^{-1}$ ):  $\nu$  376, 358 (Pd—P),  $\nu$  (Pd—Cl).  $^1\text{H}$  NMR (250 MHz,  $[\text{D}_1]$ -chloroform solution)  $\delta = 7.71$  (m, 2H,  $\text{PPh}_3$  *ortho*), 7.44–7.35 (m, 3H,  $\text{PPh}_3$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR (63 MHz,  $[\text{D}_1]$ -chloroform solution)  $\delta = 135.5, 131.0, 130.0, 128.5$  ( $\text{PPh}_3$ ).  $^{31}\text{P}\{^1\text{H}\}$  NMR (81 MHz,  $[\text{D}_1]$ -chloroform solution)  $\delta = -21.1$  (s,  $\text{PPh}_3$ ).

## Refinement

We had serious problems growing up good crystals of reasonable size and quality and, in all cases, we obtained twinned crystals with very broad reflections (bad mosaic structure). Measurement were done in a image plate diffractometer which only measure in a single  $\theta$  angle.

All H atoms were computed and refined, using a riding model, with an isotropic temperature factor equal to 1.2 times the equivalent temperature factor of the atom which are bonded.

## Figures

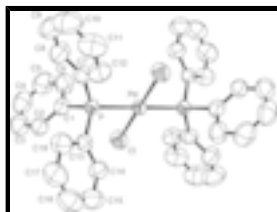


Fig. 1. *trans*-Dichlorobis(triphenylphosphine)palladium(II)

## *trans*-Dichloridobis(triphenylphosphine)palladium(II)

### Crystal data

[PdCl<sub>2</sub>(C<sub>18</sub>H<sub>15</sub>P<sub>1</sub>)<sub>2</sub>]

$M_r = 701.84$

Monoclinic,  $P2_1/c$

$a = 9.296 (5) \text{ \AA}$

$b = 19.889 (8) \text{ \AA}$

$c = 10.621 (6) \text{ \AA}$

$\beta = 121.71 (4)^\circ$

$V = 1670.6 (15) \text{ \AA}^3$

$Z = 2$

$F_{000} = 712$

$D_x = 1.395 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 26 reflections

$\theta = 3\text{--}31^\circ$

$\mu = 0.83 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Prism, yellow

$0.2 \times 0.17 \times 0.16 \text{ mm}$

### Data collection

MAR345 with image-plate detector  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2) \text{ K}$

$\varphi$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 1999)

$T_{\min} = 0.85$ ,  $T_{\max} = 0.87$

4898 measured reflections

4898 independent reflections

3143 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 33.3^\circ$

$\theta_{\text{min}} = 3.8^\circ$

$h = -14 \rightarrow 12$

$k = 0 \rightarrow 30$

$l = 0 \rightarrow 16$

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.077$	$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2]$
$S = 0.93$	where $P = (F_o^2 + 2F_c^2)/3$
4898 reflections	$(\Delta/\sigma)_{\max} = 0.002$
187 parameters	$\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$
7 restraints	$\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd	1.0000	0.0000	0.5000	0.04342 (7)
P	0.84764 (7)	0.09596 (3)	0.36120 (6)	0.04430 (13)
Cl	0.97931 (10)	0.04208 (4)	0.69271 (7)	0.06833 (18)
C1	0.6712 (3)	0.11530 (14)	0.3892 (3)	0.0580 (6)
C2	0.6470 (3)	0.17570 (17)	0.4357 (3)	0.0657 (7)
H2	0.7217	0.2106	0.4529	0.079*
C3	0.5150 (5)	0.1869 (2)	0.4584 (4)	0.0889 (10)
H3	0.5026	0.2279	0.4939	0.107*
C4	0.4034 (5)	0.1351 (2)	0.4262 (4)	0.0969 (11)
H4	0.3102	0.1429	0.4346	0.116*
C5	0.4210 (5)	0.0737 (2)	0.3832 (4)	0.0976 (11)
H5	0.3458	0.0394	0.3685	0.117*
C6	0.5576 (4)	0.06221 (17)	0.3603 (4)	0.0811 (8)
H6	0.5711	0.0207	0.3273	0.097*
C7	0.7530 (3)	0.09435 (13)	0.1606 (3)	0.0614 (6)
C8	0.5790 (4)	0.09538 (17)	0.0615 (3)	0.0835 (9)
H8	0.5064	0.0960	0.0970	0.100*

## supplementary materials

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C9	0.5128 (6)	0.0955 (2)	-0.0899 (4)	0.1180 (16)
H9	0.3962	0.0965	-0.1547	0.142*
C10	0.6163 (7)	0.0942 (2)	-0.1452 (4)	0.1208 (16)
H10	0.5706	0.0937	-0.2469	0.145*
C11	0.7918 (6)	0.0935 (2)	-0.0474 (4)	0.0996 (12)
H11	0.8631	0.0930	-0.0842	0.120*
C12	0.8601 (4)	0.09361 (17)	0.1056 (3)	0.0774 (8)
H12	0.9767	0.0932	0.1705	0.093*
C13	0.9779 (3)	0.17318 (11)	0.4177 (2)	0.0491 (5)
C14	1.1113 (3)	0.18023 (15)	0.5592 (3)	0.0659 (7)
H14	1.1381	0.1455	0.6266	0.079*
C15	1.2095 (5)	0.2397 (2)	0.6048 (4)	0.0953 (11)
H15	1.3024	0.2438	0.7007	0.114*
C16	1.1653 (5)	0.2915 (2)	0.5048 (5)	0.0961 (11)
H16	1.2282	0.3310	0.5349	0.115*
C17	1.0406 (5)	0.28661 (18)	0.3722 (5)	0.0945 (11)
H17	1.0167	0.3220	0.3067	0.113*
C18	0.9362 (4)	0.22755 (15)	0.3218 (3)	0.0761 (8)
H18	0.8419	0.2256	0.2262	0.091*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pd	0.04515 (12)	0.04507 (12)	0.04158 (11)	0.00132 (11)	0.02386 (9)	0.00015 (10)
P	0.0441 (3)	0.0449 (3)	0.0430 (3)	0.0013 (2)	0.0222 (2)	0.0019 (2)
Cl	0.0810 (4)	0.0703 (4)	0.0607 (3)	0.0072 (3)	0.0421 (3)	-0.0001 (3)
C1	0.0524 (12)	0.0642 (15)	0.0558 (12)	0.0073 (9)	0.0274 (11)	0.0050 (10)
C2	0.0627 (16)	0.0724 (19)	0.0595 (15)	0.0074 (13)	0.0304 (13)	0.0001 (11)
C3	0.088 (2)	0.104 (3)	0.081 (2)	0.018 (2)	0.0492 (18)	-0.0020 (18)
C4	0.081 (2)	0.130 (4)	0.092 (2)	0.020 (2)	0.0533 (19)	0.014 (2)
C5	0.075 (2)	0.108 (3)	0.115 (3)	-0.009 (2)	0.054 (2)	0.007 (2)
C6	0.0719 (18)	0.0721 (19)	0.111 (2)	0.0005 (13)	0.0560 (18)	0.0040 (18)
C7	0.0689 (12)	0.0598 (15)	0.0481 (11)	0.0004 (12)	0.0256 (9)	0.0019 (10)
C8	0.0704 (13)	0.094 (2)	0.0730 (14)	0.0068 (17)	0.0284 (13)	0.0058 (16)
C9	0.111 (3)	0.130 (3)	0.0689 (15)	-0.001 (3)	0.016 (2)	0.002 (2)
C10	0.148 (4)	0.126 (4)	0.070 (2)	-0.017 (3)	0.045 (3)	-0.003 (2)
C11	0.129 (3)	0.111 (3)	0.082 (2)	-0.022 (3)	0.071 (2)	-0.007 (2)
C12	0.0803 (19)	0.091 (2)	0.0613 (15)	-0.0088 (17)	0.0374 (14)	0.0021 (14)
C13	0.0493 (11)	0.0486 (12)	0.0566 (10)	0.0000 (9)	0.0328 (9)	-0.0006 (9)
C14	0.0632 (15)	0.0688 (16)	0.0605 (11)	-0.0008 (12)	0.0290 (10)	-0.0002 (11)
C15	0.088 (2)	0.090 (2)	0.100 (2)	-0.0232 (18)	0.044 (2)	-0.020 (2)
C16	0.103 (3)	0.083 (2)	0.122 (3)	-0.024 (2)	0.073 (3)	-0.019 (2)
C17	0.117 (3)	0.071 (2)	0.105 (3)	-0.008 (2)	0.065 (2)	0.0084 (19)
C18	0.088 (2)	0.0688 (19)	0.0718 (17)	-0.0061 (16)	0.0424 (16)	0.0055 (13)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Pd—Cl	2.3111 (13)	C8—C9	1.387 (5)
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Pd—Cl <sup>i</sup>	2.3111 (13)	C8—H8	0.9300
Pd—P	2.3721 (10)	C9—C10	1.366 (6)
Pd—P <sup>i</sup>	2.3721 (10)	C9—H9	0.9300
P—C7	1.829 (3)	C10—C11	1.400 (6)
P—C13	1.849 (2)	C10—H10	0.9300
P—C1	1.855 (3)	C11—C12	1.400 (4)
C1—C2	1.361 (4)	C11—H11	0.9300
C1—C6	1.408 (4)	C12—H12	0.9300
C2—C3	1.387 (4)	C13—C14	1.362 (3)
C2—H2	0.9300	C13—C18	1.394 (4)
C3—C4	1.373 (5)	C14—C15	1.416 (4)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.342 (5)	C15—C16	1.378 (5)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.432 (5)	C16—C17	1.273 (6)
C5—H5	0.9300	C16—H16	0.9300
C6—H6	0.9300	C17—C18	1.436 (5)
C7—C8	1.391 (4)	C17—H17	0.9300
C7—C12	1.394 (4)	C18—H18	0.9300
Cl—Pd—Cl <sup>i</sup>	180.0	C9—C8—C7	120.5 (4)
Cl—Pd—P	87.62 (4)	C9—C8—H8	119.8
Cl <sup>i</sup> —Pd—P	92.38 (4)	C7—C8—H8	119.8
Cl—Pd—P <sup>i</sup>	92.38 (4)	C10—C9—C8	121.0 (4)
Cl <sup>i</sup> —Pd—P <sup>i</sup>	87.62 (4)	C10—C9—H9	119.5
P—Pd—P <sup>i</sup>	180.0	C8—C9—H9	119.5
C7—P—C13	102.99 (12)	C9—C10—C11	119.4 (4)
C7—P—C1	105.43 (13)	C9—C10—H10	120.3
C13—P—C1	105.00 (12)	C11—C10—H10	120.3
C7—P—Pd	118.32 (9)	C10—C11—C12	120.2 (4)
C13—P—Pd	113.03 (8)	C10—C11—H11	119.9
C1—P—Pd	110.89 (9)	C12—C11—H11	119.9
C2—C1—C6	119.6 (3)	C7—C12—C11	119.9 (3)
C2—C1—P	124.7 (2)	C7—C12—H12	120.1
C6—C1—P	115.7 (2)	C11—C12—H12	120.1
C1—C2—C3	122.3 (3)	C14—C13—C18	117.9 (2)
C1—C2—H2	118.8	C14—C13—P	120.31 (19)
C3—C2—H2	118.8	C18—C13—P	121.59 (19)
C4—C3—C2	117.2 (4)	C13—C14—C15	120.9 (3)
C4—C3—H3	121.4	C13—C14—H14	119.6
C2—C3—H3	121.4	C15—C14—H14	119.6
C5—C4—C3	123.8 (4)	C16—C15—C14	118.9 (3)
C5—C4—H4	118.1	C16—C15—H15	120.6
C3—C4—H4	118.1	C14—C15—H15	120.6
C4—C5—C6	118.7 (4)	C17—C16—C15	121.7 (4)
C4—C5—H5	120.6	C17—C16—H16	119.2
C6—C5—H5	120.6	C15—C16—H16	119.2
C1—C6—C5	118.2 (3)	C16—C17—C18	121.3 (3)

## supplementary materials

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C1—C6—H6	120.9	C16—C17—H17	119.4
C5—C6—H6	120.9	C18—C17—H17	119.4
C8—C7—C12	119.1 (3)	C13—C18—C17	119.2 (3)
C8—C7—P	122.5 (3)	C13—C18—H18	120.4
C12—C7—P	118.4 (2)	C17—C18—H18	120.4
Cl—Pd—P—C1	41.9 (2)	Cl—Pd—P—C7	163.9 (2)
Cl—Pd—P—C13	-75.7 (2)		

Symmetry codes: (i)  $-x+2, -y, -z+1$ .



Fig. 1

