

trans-Dichlorobis[diphenyl(*p*-tolyl)phosphine]-palladium(II)Gideon Steyl,* Leo Kirsten and
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Key indicators

Single-crystal X-ray study

 $T = 100$ KMean $\sigma(\text{C}-\text{C}) = 0.003$ Å

Disorder in main residue

 R factor = 0.022 wR factor = 0.054

Data-to-parameter ratio = 20.0

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

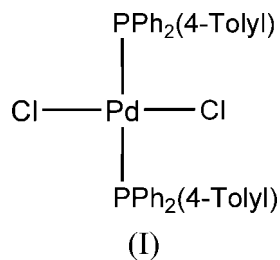
The centrosymmetric title compound, $[\text{PdCl}_2(\text{C}_{19}\text{H}_{17}\text{P})_2]$, crystallizes with a square-planar geometry about the Pd^{II} metal centre. The most important bond distances include $\text{Pd}-\text{P}$ (*trans* P) of 2.3404 (9) Å and $\text{Pd}-\text{Cl}$ (*trans* Cl) of 2.2977 (12) Å. Weak intra- and intermolecular hydrogen bonding is observed in the solid-state structure between the chloro and phenyl H atoms. This weak intermolecular hydrogen-bonding pattern forms a one-dimensional chain along the b axis.

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Comment

The application of palladium complexes as catalysts has been thoroughly investigated in the past [Cambridge Structural Database (CSD), Version 5.27 (Allen, 2002); Tsuji, 2004; Crabtree, 2001]. Recently in our group, we have started to investigate the stability of the Pd^{II} oxidation state with different mono-anionic ligands (Steyl, 2006, 2005). The effect of adding a functional group to a single phenyl ring on the triphenylphosphine ligand has not been reported for Pd^{II} complexes. The title compound, (I), has a methyl group attached at the 4-position in one of the phenyl groups of the PPh_3 ligand (Fig. 1).



Compound (I) crystallizes with a square-planar geometry about the Pd^{II} metal centre (Fig. 1). The $\text{Pd}-\text{P}$ and $\text{Pd}-\text{Cl}$ bond distances do not differ significantly from those in previously reported complexes in the CSD. The Pd^{II} metal atom lies on a centre of symmetry, which causes the 4-tolyl-diphenylphosphine groups to adopt an eclipsed configuration. This effect can also be observed from the $\text{Cl}-\text{Pd}-\text{P}-\text{C}$ torsion angle (Table 1). The H atoms of the tolyl group are disordered over two positions.

An intramolecular hydrogen bond, $\text{C12}-\text{H12}\cdots\text{Cl}$, is present in the molecule (Table 2). In the crystal structure, the molecules are linked by intermolecular $\text{C23}-\text{H23}\cdots\text{Cl}^{\text{ii}}$ bonds (symmetry code as in Table 2) to form a chain along the b axis. In addition, there is $\pi-\pi$ stacking between $\text{C11}-\text{C16}$ rings related by $(1-x, 1-y, 1-z)$, with a centroid-centroid distance of 3.712 Å.

Experimental

The title complex was synthesized by the addition of 4-tolyl-diphenylphosphine (43 mg, 0.156 mmol) to [Pd(COD)Cl₂] (COD = 1,5-cyclooctadiene) (20 mg, 0.070 mmol) in 10 ml of acetone. On evaporation of the solvent, crystals suitable for X-ray crystallographic investigation were obtained (yield 50 mg, 98%).

Crystal data

[PdCl ₂ (C ₁₉ H ₁₇ P) ₂]	<i>V</i> = 813.7 (7) Å ³
<i>M_r</i> = 729.89	<i>Z</i> = 1
Triclinic, <i>P</i> $\bar{1}$	<i>D_x</i> = 1.489 Mg m ⁻³
<i>a</i> = 9.960 (5) Å	Mo <i>K</i> α radiation
<i>b</i> = 9.985 (5) Å	<i>μ</i> = 0.86 mm ⁻¹
<i>c</i> = 10.255 (5) Å	<i>T</i> = 100 (2) K
<i>α</i> = 88.312 (5)°	Block, yellow
<i>β</i> = 68.129 (5)°	0.26 × 0.16 × 0.11 mm
<i>γ</i> = 61.045 (5)°	

Data collection

Bruker SMART CCD area-detector diffractometer	17732 measured reflections
<i>φ</i> and <i>ω</i> scans	3918 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 1998)	3644 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T_{min}</i> = 0.847, <i>T_{max}</i> = 0.905	<i>R_{int}</i> = 0.028
	<i>θ_{max}</i> = 28.0°

Refinement

Refinement on <i>F</i> ²	<i>w</i> = 1/[σ ² (<i>F_o</i> ²) + (0.0193 <i>P</i>) ² + 0.5842 <i>P</i>]
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.022	where <i>P</i> = (<i>F_o</i> ² + 2 <i>F_c</i> ²)/3
<i>wR</i> (<i>F</i> ²) = 0.054	(Δ/ <i>σ</i>) _{max} = 0.001
<i>S</i> = 1.07	Δ <i>ρ</i> _{max} = 0.47 e Å ⁻³
3918 reflections	Δ <i>ρ</i> _{min} = -0.37 e Å ⁻³
196 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

P—Pd	2.3404 (9)	Cl—Pd	2.2977 (12)
Cl—Pd—Cl ⁱ	180.00 (2)	Cl—Pd—P	86.60 (2)
Cl—Pd—P ⁱ	93.40 (2)	P ⁱ —Pd—P	180.0
C31—P—Pd—Cl	52.89 (6)	C11—P—Pd—Cl	-68.99 (6)
C21—P—Pd—Cl	169.65 (6)		

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

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C12—H12...Cl	0.93	2.64	3.440 (2)	144
C23—H23...Cl ⁱⁱ	0.93	2.80	3.675 (2)	157

Symmetry code: (ii) *x*, *y* + 1, *z*.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with *U*_{iso}(H) values of 1.2 or 1.5 times *U*_{eq}(parent atom) for aromatic and methyl H atoms, and C—H distances of 0.93 and 0.96 Å, respectively. The disordered H atoms of the methyl group were constrained to have occupancies of 0.50 for each of the two positions.

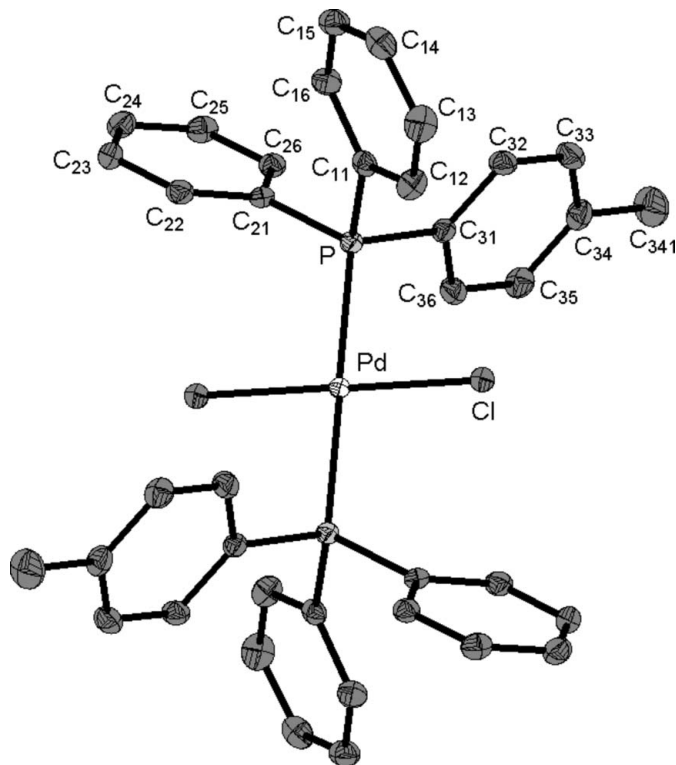


Figure 1

Representation of (I), showing the numbering scheme and displacement ellipsoids at the 50% probability level. For the carbon rings, the first digit refers to the ring number, the second digit to the atom in the ring. H atoms have been omitted for clarity. Unlabelled atoms are related to labelled atoms by (-*x*, 1 - *y*, 1 - *z*).

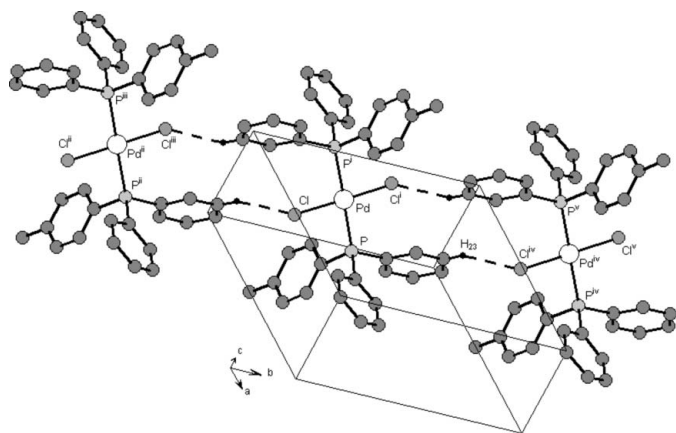


Figure 2

A packing diagram, showing the intermolecular hydrogen bonding (dashed lines) of the metal complex, forming chains along the *b* axis [symmetry codes: (i) -*x*, 1 - *y*, 1 - *z*; (ii) *x*, -1 + *y*, *z*; (iii) -*x*, -*y*, 1 - *z*; (iv) *x*, 1 + *y*, *z*; (v) -*x*, 2 - *y*, 1 - *z*].

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus and XPREP (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg & Putz, 2004); software used to prepare material for publication: SHELXL97.

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