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Bis[(diphenylphosphino)acetato-κ^2O,P]palladium(II) dichloromethane solvate

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The title compound, $[Pd(C_{14}H_{12}O_2P)_2]\cdot CH_2Cl_2$, consists of mononuclear $[(Ph_2PCH_2COO)_2Pd]$ complexes and dichloromethane molecules in a 1:1 molar ratio. The two (diphenylphosphino)acetate ligands are coordinated to the central Pd atom by their P and O atoms, forming five-membered chelate rings. The Pd^{II} atom has a distorted square-planar configuration [Pd—O = 2.076 (3) and 2.082 (3) Å, and Pd—P = 2.214 (1) and 2.216 (1) Å].

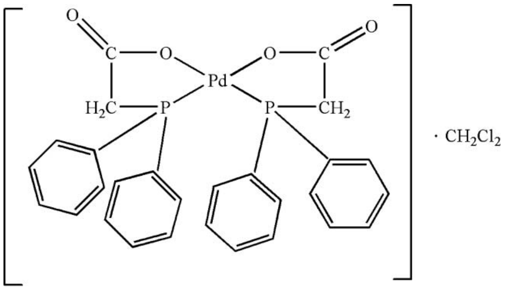
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.007 Å
R factor = 0.048
wR factor = 0.097
Data-to-parameter ratio = 14.9

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

Comment

The palladium-catalyzed Suzuki cross-coupling reaction is one of the most efficient methods for the construction of C_{aryl}—C_{aryl} bonds and has found widespread use in organic synthesis as well as pharmaceutical and agricultural chemistry (Baudoin *et al.*, 2002; Hoeck *et al.*, 2000) There has recently been considerable interest in the synthesis of new high activity palladium-based catalysts that can be used in low concentration in the Suzuki reaction since such catalysts have the potential to be used in industrial systems (Bedford & Welch, 2001). In particular, palladacyclic catalysts in which a ligand coordinates to the metal center through both a donor atom and a metallated carbon have shown considerable promise. Some palladated phosphine complexes have shown good activity (Beller *et al.*, 1995; Zim *et al.*, 1999). We recently found some *P*,*O*-bidentate phosphine palladacycles to act as excellent catalysts in the Suzuki reaction. In this paper, we report the crystal structure of one of these palladacyles.



(I)

In the title compound, (I), the Pd atom is chelated by two (diphenylphosphino)acetate ligands in a *cis* arrangement. The Pd^{II} ion has a square-planar coordination geometry. The two (diphenylphosphino)acetate ligands are each bonded to palladium in a bidentate mode, forming five-membered chelate rings. The Pd—O bond distances of 2.076 (3) and 2.082 (3) Å are slightly longer than those found in other O-coordinated Pd^{II} complexes (Johansson & Oskarsson, 2001; Kirik *et al.*, 2004). The Pd—P bond distances of 2.214 (1) and

2.216 (1) Å are in accordance with the values usually encountered in related compounds (Fernandez-Rivas *et al.*, 2001; Civiš *et al.*, 1980).

The molecular packing is stabilized by intermolecular C—H···O interactions (Table 2).

Experimental

The title compound was prepared by reaction of sodium diphenylphosphine carboxylate (2 mmol) with Na_2PdCl_4 (1 mmol) at 298 K in almost quantitative yield. Single crystals suitable for X-ray measurements were obtained by recrystallization from dichloromethane at room temperature.

Crystal data

$[Pd(C_{14}H_{12}O_2P)_2]\cdot CH_2Cl_2$
$M_r = 677.74$
Monoclinic, $P2_1/c$
$a = 10.658$ (2) Å
$b = 17.943$ (4) Å
$c = 16.618$ (6) Å
$\beta = 116.72$ (2)°
$V = 2838.6$ (13) Å^3
$Z = 4$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7367 reflections
$\theta = 4$–26°
$\mu = 0.99$ mm^{-1}
$T = 273$ (2) K
Block, red
0.27 × 0.22 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.775$, $T_{max} = 0.879$
14955 measured reflections
5119 independent reflections
4508 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.2°$
$h = -10 \rightarrow 12$
$k = -21 \rightarrow 21$
$l = -19 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.098$
$S = 1.17$
5119 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 2.9516P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—O4	2.076 (3)	Pd1—P1	2.2141 (14)
Pd1—O2	2.082 (3)	Pd1—P2	2.2161 (11)
O4—Pd1—O2	92.18 (10)	O4—Pd1—P2	84.20 (8)
O4—Pd1—P1	174.15 (8)	O2—Pd1—P2	174.11 (8)
O2—Pd1—P1	82.74 (8)	P1—Pd1—P2	100.61 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*A*···O2^{i}	0.97	2.43	3.269 (5)	144
C27—H27*A*···O1^{ii}	0.93	2.58	3.241 (5)	129
C29—H29*A*···O1^{i}	0.97	2.24	3.186 (6)	165

Symmetry codes: (i) $-x+1, -y-1, -z$; (ii) $-x, y+\frac{1}{2}, -z-\frac{1}{2}$.

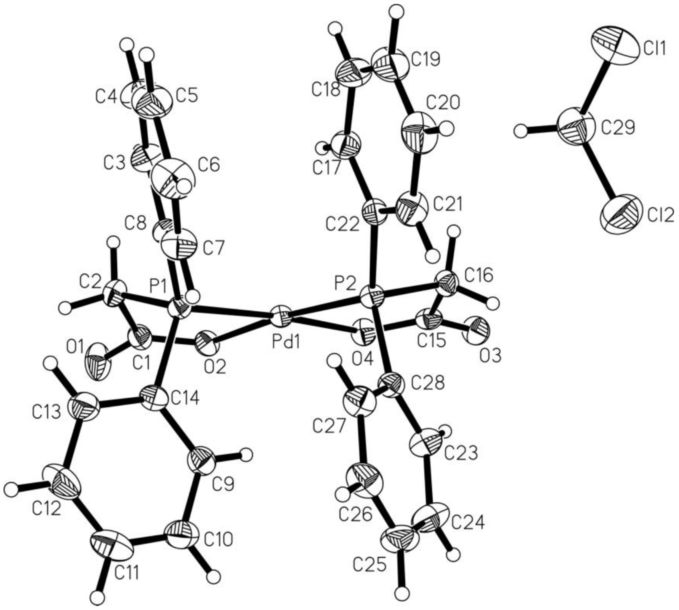


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The H atoms were positioned geometrically and treated as riding on the parent C atoms, with C—H distances in the range 0.93–0.97 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXTL/PC*.

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