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trans-Dichloro(2-chloroaniline-κN)(triphenylphosphine-κP)palladium(II) dichloromethane solvate

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In the title compound, $[PdCl_2(C_6H_5ClN)(C_{18}H_{15}P)]\cdot CH_2Cl_2$, the four-coordinated Pd^{II} complex exhibits a nearly square-planar geometry. The Pd—N, Pd—P and two Pd—Cl bond lengths are 2.170 (3), 2.2322 (9) and 2.2910 (9)/2.3104 (9) Å, respectively, and the angles at Pd^{II} lie in the range 86.85 (3)–93.58 (4)°.

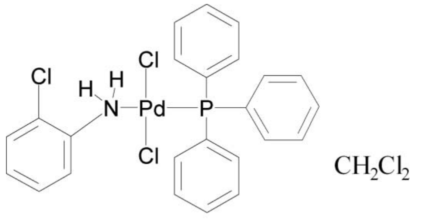
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.044
wR factor = 0.116
Data-to-parameter ratio = 21.0

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

Comment

Palladium(II) complexes are of current interest due to their antitumor (Faraglia *et al.*, 2001) and catalytic activity (Ali *et al.*, 1996), similar to Pt^{II} complexes (Loehrer & Einhorn, 1984). For that reason, a variety of palladium(II) complexes containing N- and S-donor ligands, such as Pd(2,3-diaminotoluene)Cl_2, Pd(4,5-diaminoxylene)Cl_2 (Perez-Cabre *et al.*, 2004) and Pd(2-benzoylpyridine thiosemicarbazone) have been extensively investigated (Rebolledo *et al.*, 2005). In addition, various palladium complexes coordinated to diaminocyclohexane-containing ligands have proved to be catalytically active in Heck-type reactions (Bravo *et al.*, 2002).



(I)

The Pd^{II}–phosphine complexes *cis*-$Pd(H_2O)_2(PPh_3)$(*p*-$CH_3C_6H_4SO_3)\cdot 2H_2O$ and *cis*-$Pd(H_2O)_2(PPh_3)(CH_3SO_3)_2\cdot 2CH_2Cl_2$ are efficient catalysts for carbonylation of olefins (Cavinato *et al.*, 2004).

Here, in continuation of our previous work (Parvez *et al.*, 2004), we report a new convenient synthesis and the crystal structure of a palladium(II) complex containing phosphine and aniline ligands. Palladium(II) complexes are unique due to a strong preference of Pd^{II} for square-planar coordination in which the ligand framework is proven to be highly stable (Porai-Koshits, 1987). In the title compound, the planar environment of the Pd^{II} atom is a slightly distorted square. The sum of the bond angles around atom Pd1 (Table 1) is 360.0°. 2-Chloroaniline and triphenylphosphine are *trans* to each other, with N1—Pd1—P1 and Cl2—Pd1—Cl1 angles of 174.79 (9) and 179.16 (4)°, respectively. The metal–ligand Pd—N, Pd—P and Pd—Cl bond lengths (Table 1) are in good agreement with those found in $Pd(PPh_3)$(indoline-κN)Cl_2 (Chen *et al.*, 1997).

Experimental

Palladium(II) chloride (0.5 g, 2.82 mmol; E. Merck) was dissolved completely in distilled water (20 ml) by adding 2–3 drops of dilute HCl. A solution of triphenylphosphine (0.74 g, 2.82 mmol) in acetone was added dropwise with constant stirring. The reaction mixture was stirred overnight at room temperature. The resulting yellow precipitate of $[PdCl_2(PPh_3)(H_2O)]$ was filtered off, washed with diethyl ether and dried under vacuum (0.23 ml, 2.20 mmol). 2-Chloroaniline was added dropwise to a suspension of $[PdCl_2(PPh_3)(H_2O)]$ (0.97 g, 2.20 mmol) in CH_2Cl_2 (20 ml) and the resulting solution refluxed for 1 h, resulting in a clear solution. Dark-orange crystals were obtained after slow evaporation of the solvent at room temperature.

Crystal data

$[PdCl_2(C_6H_5ClN)(C_{18}H_{15}P)]\cdot CH_2Cl_2$
$M_r = 652.06$
Triclinic, $P\overline{1}$
$a = 10.0120$ (2) Å
$b = 10.3890$ (2) Å
$c = 14.2220$ (4) Å
$\alpha = 104.6190$ (10)°
$\beta = 89.9230$ (10)°
$\gamma = 112.7541$ (12)°
$V = 1312.30$ (5) Å^3
$Z = 2$
$D_x = 1.650$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 621 reflections
$\theta = 2.2$–30.0°
$\mu = 1.29$ mm^{-1}
$T = 298$ (2) K
Prism, orange
0.32 × 0.29 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*HKL2000*; Otwinowski & Minor, 1997)
$T_{min} = 0.680$, $T_{max} = 0.740$
10268 measured reflections
6247 independent reflections
4057 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 30.0°$
$h = -13 \rightarrow 12$
$k = -12 \rightarrow 14$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.116$
$S = 1.01$
6247 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—N1	2.170 (3)	Pd1—Cl1	2.3104 (9)
Pd1—P1	2.2322 (9)	Cl3—C6	1.724 (4)
Pd1—Cl2	2.2910 (9)	N1—C1	1.427 (5)
N1—Pd1—P1	174.79 (9)	N1—Pd1—Cl1	88.06 (9)
N1—Pd1—Cl2	91.52 (9)	P1—Pd1—Cl1	86.85 (3)
P1—Pd1—Cl2	93.58 (4)	Cl2—Pd1—Cl1	179.16 (4)

All H atoms were initially located in a difference Fourier map and were refined as riding, with N—H = 0.90 Å, C—H = 0.93–0.97 Å and $U_{iso} = 1.2$–$1.5U_{eq}$(parent atom).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL/SCALEPACK*; program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MAXUS* (Mackay *et al.*, 1998); software used to prepare material for publication: *SHELXL97*.

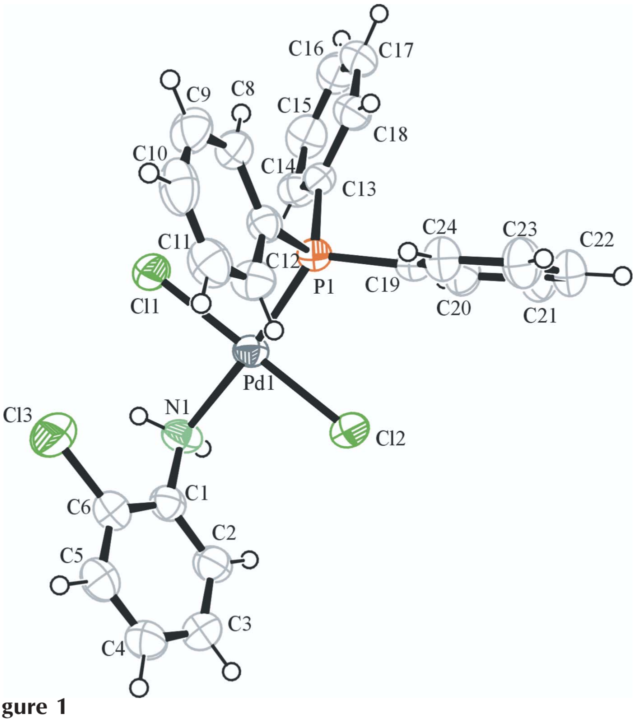


Figure 1
View of the title Pd^{II} complex, showing the atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. The dichloromethane solvent molecule has been omitted for clarity.

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