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trans-Dichloro(dicyclohexylamine)(triphenylphosphine)palladium(II)

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Key indicators

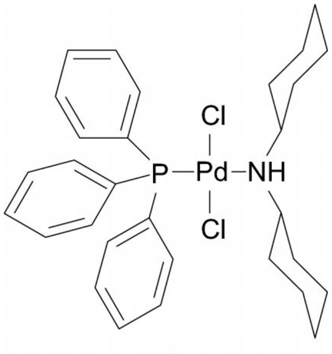
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.068
Data-to-parameter ratio = 20.5

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

In the crystal structure of the title compound, [$PdCl_2(C_{12}H_{23}N)(C_{18}H_{15}P)$], the Pd atom is four-coordinate in a nearly square-planar environment, with Pd—Cl = 2.2974 (6) and 2.3055 (7) Å, Pd—N = 2.1573 (17) Å and Pd—P = 2.2504 (7) Å. Both cyclohexyl rings adopt chair conformations.

Comment

Palladium(II) preferentially forms complexes with nitrogen-, phosphorus- and sulfur-donor ligands, while it shows a small affinity towards oxygen. Palladium(II) complexes with amines (Cabre *et al.*, 2004) and sulfur-containing thiones (Krischner *et al.*, 1966) are known to exhibit antibacterial and antitumor activity, while palladium(II)–phosphine complexes are important from a catalytic point of view (Tsuji, 1995). Some examples of palladium(II) complexes used as catalysts are: [$Pd(PPh_3)_2Cl_2$] (Nicholas, 1987), [$Pd(PPh_3)_4$]$^{2+}$ (Bumagin *et al.*, 1984), orthometallated palladium(II) complexes derived from (1*R*,2*R*)-1,2-diaminocyclohexane (Bravo *et al.*, 2002) and [$(PCy_3)_2Pd(H)(H_2O)$]BF_4 (Cy is cyclohexyl; Ali *et al.*, 1996). The palladium(II) complexes of tertiary phosphines and amines have been studied extensively (Okeya *et al.*, 1981; Belykh *et al.*, 2002). A number of mixed-ligand complexes of phosphines and amines have also been reported, *e.g.* *trans*-[$Pd(PBu_3)(PhNH_2)Cl_2$] (Romm *et al.*, 1995), *trans*-[$Pd(PPh_3)(CH_3NHCH_2Ph)Cl_2$] (Jones *et al.*, 2000) and *trans*-[$Pd\{P(o\text{-}CH_3Ph)_3\}(NHMe_2)Cl_2$] (Paul *et al.*, 1994). In this paper, we report the synthesis and crystal structure of *trans*-dichloro(dicyclohexylamine)(triphenylphosphine)palladium(II), (I).



(I)

The crystal structure of (I) contains discrete molecules (Fig. 1), in which the amino H atom is involved in an intermolecular interaction with Cl2 of a symmetry-related molecule, as well as an intramolecular interaction with Cl2, with H···Cl distances of 2.64 and 2.68 Å, respectively (Fig. 2). The

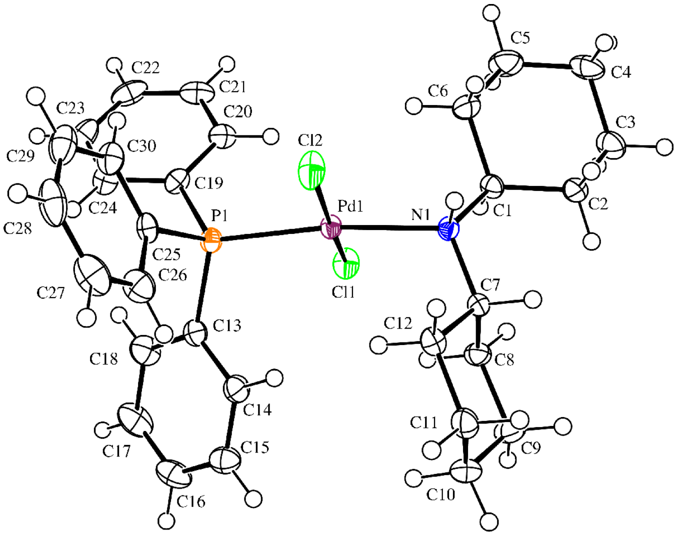


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), with displacement ellipsoids plotted at the 30% probability level.

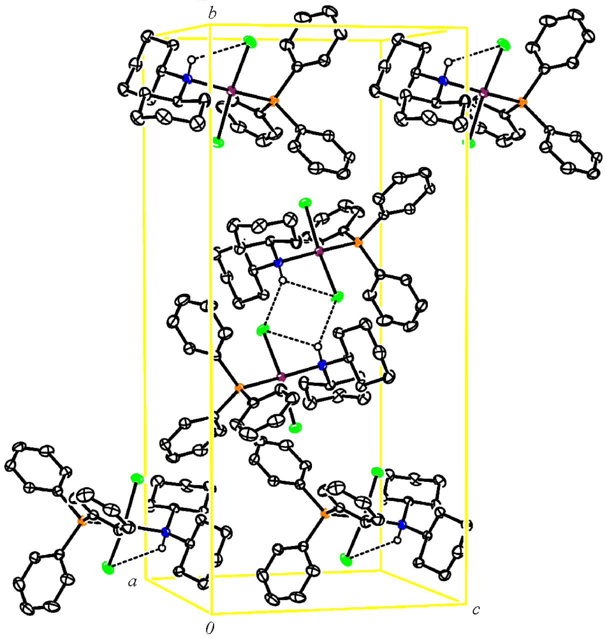


Figure 2
Please supply caption.

Pd atom is four-coordinate in a nearly square-planar environment slightly distorted towards tetrahedral; deviations of the atoms from the $PdCl_2PN$ mean plane are: Pd1 0.0033 (4), P1 0.1441 (5), Cl1 −0.1472 (4), Cl2 −0.1469 (4) and N1 0.1466 (5) Å. The bond lengths at palladium (Table 1) agree well with values reported for the corresponding distances in a handful of Pd complexes in which the Pd atom is coordinated in an environment similar to (I) (Cambridge Structural Database, Version 5.25, 2003 release; Allen, 2002). The remaining dimensions [mean P—C = 1.823 (5) Å, N—C = 1.503 (1) Å, aromatic C—C = 1.386 (3) Å and aliphatic C—C = 1.525 (3) Å] are normal and in accord with expected values. Cyclohexyl rings C1—C6 and C7—C12 adopt classical chair conformations, with puckering parameters (Cremer & Pople, 1975) Q = 0.573 (2) and 0.581 (2) Å, θ = 1.8 (2) and 0.9 (2)° and φ = 71 (6) and 87 (7)°, respectively.

Experimental

Palladium(II) chloride (0.236 g, 2.19 mmol, from Merck) was dissolved in water (30 ml) and a solution of triphenylphosphine (0.33 g, 2.19 mmol) in acetone was added dropwise with constant stirring. The reaction mixture was stirred overnight at room temperature. The resulting yellow precipitate was collected, washed with diethyl ether and dried under vacuum. This precipitate (0.57 g, 1.29 mmol) was dissolved in tetrahydrofuran (20 ml) and dicyclohexylamine (0.26 ml, 1.29 mmol) was added dropwise. The resulting reaction mixture was refluxed for 1 h at 323 K with constant stirring. Stirring was continued overnight at room temperature. The yellow precipitate thus obtained was washed with diethyl ether and *n*-hexane. The product was dissolved in acetone and light-yellow crystals of (I) were obtained on slow evaporation.

Crystal data

$[PdCl_2(C_{12}H_{23}N)(C_{18}H_{15}P)]$
M_r = 620.88
Monoclinic, $P2_1/n$
a = 10.326 (2) Å
b = 24.760 (4) Å
c = 11.178 (2) Å
β = 96.274 (7)°
V = 2840.8 (9) Å^3
Z = 4

D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 408 reflections
θ = 3.7–27.5°
μ = 0.92 mm^{-1}
T = 173 (2) K
Prism, yellow
0.14 × 0.12 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.882, T_{max} = 0.898
12 408 measured reflections
6483 independent reflections

5185 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -13 \rightarrow 13$
$k = -32 \rightarrow 31$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.068$
S = 1.02
6483 reflections
316 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 0.6275P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—N1	2.1573 (17)	P1—C25	1.823 (2)
Pd1—P1	2.2504 (7)	P1—C13	1.828 (2)
Pd1—Cl2	2.2974 (6)	N1—C1	1.502 (2)
Pd1—Cl1	2.3055 (6)	N1—C7	1.503 (2)
P1—C19	1.818 (2)		
N1—Pd1—P1	172.60 (4)	C25—P1—C13	103.92 (10)
N1—Pd1—Cl2	87.59 (5)	C19—P1—Pd1	115.70 (7)
P1—Pd1—Cl2	93.13 (2)	C25—P1—Pd1	114.25 (7)
N1—Pd1—Cl1	94.26 (5)	C13—P1—Pd1	108.83 (7)
P1—Pd1—Cl1	86.00 (2)	C1—N1—C7	115.37 (15)
Cl2—Pd1—Cl1	172.39 (2)	C1—N1—Pd1	114.31 (12)
C19—P1—C25	104.67 (10)	C7—N1—Pd1	113.53 (12)
C19—P1—C13	108.72 (10)		

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
N1—H1N$\cdots$Cl2^{i}	0.93	2.64	3.471 (2)	148

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

All H atoms were located in difference Fourier syntheses and were included in the refinement in geometrically idealized positions, with N—H = 0.93 Å and C—H = 0.95, 0.99 and 1.00 Å, and $U_{\rm iso} = 1.2U_{\rm eq}$(C,N). The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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