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trans-Bis(benzylamine)dichloropalladium(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.037
wR factor = 0.137
Data-to-parameter ratio = 17.1

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

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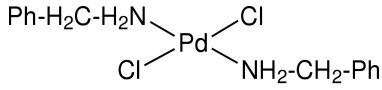
The Pd atom in the title complex, $[PdCl_2(C_7H_9N)_2]$ or *trans*-$[PdCl_2(NH_2CH_2Ph)_2]$, lies on a twofold axis. It has a distorted square-planar coordination environment formed by two benzylamine and two Cl^- ligands. The symmetry-unique *cis* angles at the Pd atom are 86.25 (11) and 93.75 (11)°.

Comment

Bis(amine) complexes of palladium have received much attention as possible intermediates in amination reactions (Widenhoefer & Buchwald, 1996; Seligson & Trogler, 1991). The title complex, (I), was prepared in the course of our studies as a precursor to other palladium compounds.



(I)

The easy access to suitable single crystals of (I) allowed us to carry out an X-ray diffraction study and compare the structural parameters of this species with those of *trans*-$[PdCl_2(NH_2CH_2Ph)_2]\cdot 2DMSO$, (II) (DMSO is dimethyl sulfoxide). In the recently published structure of (II) (Decken *et al.*, 2000), the Pd atom lies on an inversion center and hence the geometry, in the proximity of the coordination center, is different from that in (I). In (I), the CH_2 group of the benzyl moiety lies almost in the Pd coordination plane, while in (II) it is 1.293 (2) Å from the coordination plane [the Cl—Pd—N—C1 torsion angle is 1.4 (4)° in (I), *versus* 71.94 (14)° in (II)].

Experimental

The title compound was isolated as a side-product of the reaction of the dimeric complex $[(\eta^3\text{-Ind})Pd(\mu\text{-Cl})]_2$ (450 mg, 0.87 mmol; Ind is indene) with benzylamine (153 µl, 1.40 mmol) in diethyl ether (50 ml) at room temperature. After stirring for 45 min, an orange powder

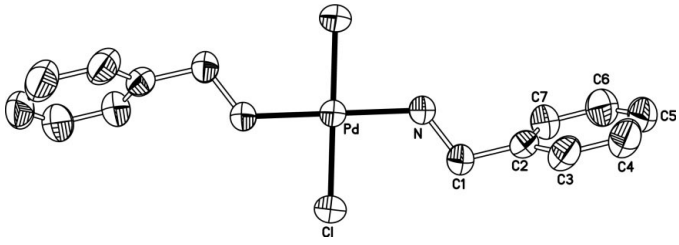


Figure 1
A view of the molecule of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. The unlabelled part of the molecule is related by the symmetry transformation $(\frac{1}{2}-x, \frac{3}{2}-y, z)$.

precipitated and was isolated after filtration. Recrystallization of a small portion of this solid from a CH_2Cl_2 solution yielded crystals of (I) suitable for an X-ray diffraction study.

Crystal data

$[PdCl_2(C_7H_9N)_2]$
M_r = 391.60
Tetragonal, $P4_2/n$
a = 15.285 (9) Å
c = 6.732 (5) Å
V = 1572.8 (15) Å^3
Z = 4
D_x = 1.654 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20.0–22.5°
μ = 12.54 mm^{-1}
T = 293 (2) K
Needle-like, orange
0.45 × 0.18 × 0.14 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: Gaussian (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.068, T_{max} = 0.260
18 272 measured reflections
1503 independent reflections
1153 reflections with $I > 2\sigma(I)$
R_{int} = 0.095
θ_{max} = 69.9°
$h = -18 \rightarrow 18$
$k = -18 \rightarrow 18$
$l = 0 \rightarrow 8$
5 standard reflections
frequency: 60 min
intensity decay: 0.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.137
S = 1.09
1503 reflections
88 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0084 (5)

Table 1
Selected geometric parameters (Å, °).

Pd—N	2.050 (4)	N—C1	1.476 (6)
Pd—Cl^{i}	2.2939 (13)	C1—C2	1.502 (6)
N—Pd—N^{i}	179.2 (2)	Cl^{i}—Pd—Cl	179.03 (6)
N—Pd—Cl^{i}	86.25 (11)	C1—N—Pd	124.2 (3)
N—Pd—Cl	93.75 (11)	N—C1—C2	112.0 (4)

Symmetry code: (i) $\frac{1}{2} - x, \frac{3}{2} - y, z$.

The H atoms were positioned geometrically (C—H = 0.94 Å and N—H = 0.90 Å) and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C, N)$.

Data collection: local program; cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: local program; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *UdMX* (local program), *PLATON* (Spek, 2003) and *NRC Crystallographic Computer Programs* (Ahmed *et al.*, 1973).

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