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Chloro[(*N*,*N*-dimethylamino)methylphenyl-κ^2C^2,*N*](triphenylstibine)palladium(**II**)

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Key indicators

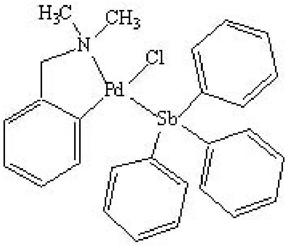
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.023
wR factor = 0.044
Data-to-parameter ratio = 17.7

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

The molecular structure of the title compound, $[PdCl(C_9H_{12}N)(C_{18}H_{15}Sb)]$, obtained by reaction of $[Pd(dmba)(\mu\text{-}Cl)]_2$ [dmba = $N(CH_3)_2CH_2C_6H_5$] with triphenylstibine in dichloromethane, shows a slightly distorted square planar geometry about Pd, which is bonded to C, N, Cl and Sb atoms.

Comment

Benzylamines undergo cyclometallation reactions with appropriate palladium(II) compounds (Cope & Friedrich, 1968; Albert *et al.*, 2003; Fuchita *et al.*, 1999; Fuchita & Tsuchiya, 2004). Carbon-to-metal σ-bonds also arise from the reaction of Pd^{II} and variously substituted Schiff base ligands (Fernández *et al.*, 2002; López-Torres *et al.*, 2002). This paper describes the crystal structure of chloro[(*N*,*N*-dimethylamino)methylphenyl-κ^2C^2,N](triphenylstibine)palladium(II), (I) (Fig. 1), which was grown from CH_2Cl_2 and petroleum ether (313–333 K) at room temperature.



(I)

In (I), the Pd—C bond length [1.991 (3) Å] *trans* to chlorine is shorter than corresponding bond lengths in related complexes, *e.g.* *trans*-$[PdClPh(SbPh_3)_2]$ [2.016 (7) Å; Mentes *et al.*, 1997] and $[Pd(dmba)Cl(PPh_3)]$ [2.012 (4) Å; Mentes *et al.*, 2004]. The Pd—Cl [2.4023 (9) Å], Pd—N [2.134 (2) Å] and Pd—Sb [2.5063 (3) Å] bond lengths compare well with the literature values for similar complexes of palladium (Mentes *et al.*, 1997, 1999, 2004; Albert *et al.*, 1995). The *cis* angles at Pd are close to the expected value of 90°, with the most noticeable distortion being the C—Pd—Sb angle of 99.16 (7)°, as a result of chelation (Fig. 1).

Experimental

The preparation and characterization of compound (I) are described in the literature (Mentes *et al.*, 2004). The reaction of $\{[Pd(dmba)(\mu\text{-}Cl)]_2\}$ with triphenylstibine in dichloromethane gave compound (I), which was crystallized from dichloromethane–petroleum ether.

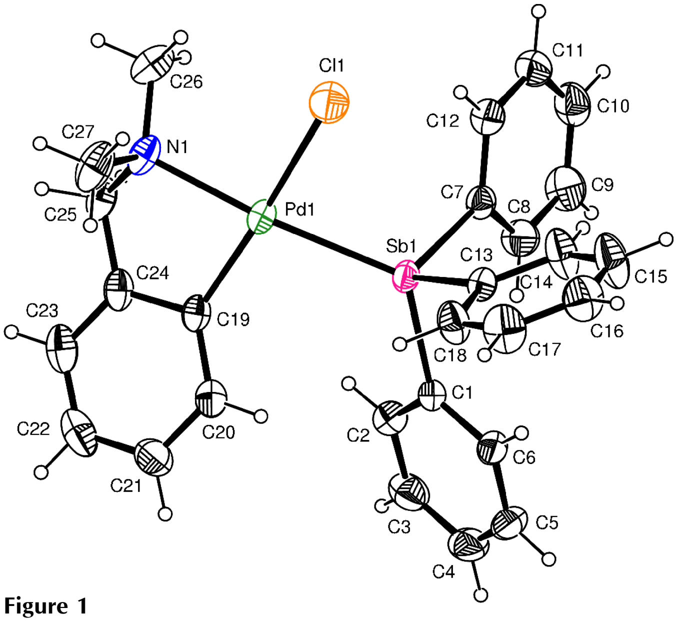


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

Crystal data

$[PdCl(C_9H_{12}N)(C_{18}H_{15}Sb)(C_9H_{12}N)]$
M_r = 629.10
Triclinic, $P\bar{1}$
a = 9.7455 (7) Å
b = 10.0846 (7) Å
c = 14.9766 (10) Å
α = 81.475 (6)°
β = 72.856 (6)°
γ = 63.805 (5)°
V = 1261.76 (15) Å^3
Z = 2
D_x = 1.656 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17979 reflections
θ = 2.3–26.0°
μ = 1.90 mm^{-1}
T = 293 K
Prism, yellow
0.29 × 0.27 × 0.25 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min}$ = 0.637, $T_{\max}$ = 0.712
27284 measured reflections
4954 independent reflections
3625 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.062
$\theta_{\max}$ = 26.0°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.044
S = 0.85
4954 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.42 e Å^{-3}
$\Delta\rho_{\min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C19—C20	1.392 (4)	N1—Pd1	2.134 (2)
C19—C24	1.407 (4)	Cl1—Pd1	2.4023 (9)
C19—Pd1	1.991 (3)	Pd1—Sb1	2.5063 (3)
C25—N1	1.484 (4)		
C19—Pd1—N1	81.97 (11)	C19—Pd1—Sb1	99.16 (7)
C19—Pd1—Cl1	171.69 (8)	N1—Pd1—Sb1	168.34 (7)
N1—Pd1—Cl1	95.20 (8)	Cl1—Pd1—Sb1	85.20 (2)

All H atoms were placed in idealized positions and refined using a riding model, with C—H = 0.93 Å ($U_{\rm iso} = 1.2U_{\rm eq}$ of the parent atom) for aromatic C atoms, C—H = 0.97 Å ($U_{\rm iso} = 1.2U_{\rm eq}$ of the parent atom) for methylene C atoms and C—H = 0.96 Å ($U_{\rm iso} = 1.5U_{\rm eq}$ of the parent atom) for methyl C atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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