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cis-Dichloro(*N*,*N*,*N*′,*N*′-tetramethyl-1,2-diaminoethane)palladium(II)

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.007 Å
R factor = 0.026
wR factor = 0.061
Data-to-parameter ratio = 21.7

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

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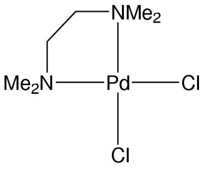
The title compound, $C_6H_{16}Cl_2N_2Pd$, exhibits slightly distorted square-planar coordination about the metal atom, with normal Pd—Cl and Pd—N distances.

Comment

The complex *cis*-dichloro(*N*,*N*,*N*′,*N*′-tetramethyl-1,2-diaminoethane)palladium(II), (I), has proved to be a versatile precursor to a variety of organometallic palladium complexes by virtue of the fact that the diamine ligand (Cusimano *et al.*, 1998; Smith & Slawin, 2000; Shoukry & Van Eldik, 1996) and the chloride ligands (Chen *et al.*, 1998; de Graaf *et al.*, 1989) can be substituted independently of one another. In the solid state, (I) shows contacts of 2.76–2.91 Å between methyl H atoms and chloride ligands. The coordination is planar to within 0.01 Å and the Pd—Cl and Pd—N distances compare well with previously reported values (Orpen *et al.*, 1989). The largest deviation from ideal square-planar geometry is in the N1—Pd—N2 angle [85.8 (1)°], which presumably reflects geometric constraints of the ligand.



(I)

Experimental

Compound (I) was prepared by a modification of the literature procedure (Meek, 1965). $PdCl_2$ (6.168 g, 34.79 mmol) was dissolved in acetonitrile and heated at 363 K for 20 min. *N*,*N*,*N*′,*N*′-Tetramethylethylenediamine (tmeda, 6 ml, 39.76 mmol) was added to the cooling red solution, whereupon (I) precipitated as a bright yellow solid. This was washed with benzene and dried overnight *in vacuo*. A portion of (I) was treated with an equivalent quantity of silver oxalate in dichloromethane to prepare Pd(oxalate)(tmeda). Following filtration to remove AgCl, the solution was allowed to evaporate slowly to dryness, forming a mixture of off-white powder and flat yellow lozenges. One of the latter was chosen for the structure determination, the result of which indicated them to be unreacted starting material.

Crystal data

$[PdCl_2(C_6H_{16}N_2)]$	D_x = 1.905 Mg m^{-3}
M_r = 293.51	Mo $K\alpha$ radiation
Monoclinic, Cc	Cell parameters from 3348 reflections
a = 15.980 (2) Å	θ = 2.8–28.3°
b = 5.9373 (6) Å	μ = 2.28 mm^{-1}
c = 11.795 (1) Å	T = 100 (2) K
β = 113.842 (2)°	Column, yellow
V = 1023.6 (2) Å^3	0.16 × 0.08 × 0.07 mm
Z = 4	

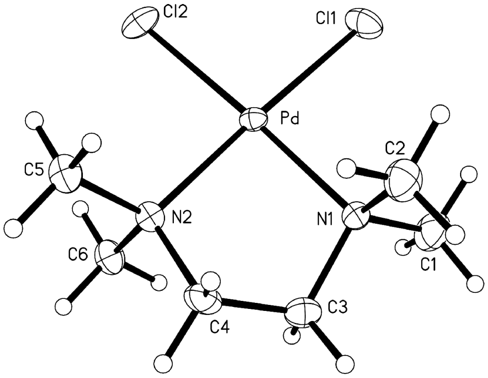


Figure 1
Perspective view of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary radius.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.737$, $T_{\max} = 0.853$
4288 measured reflections
2258 independent reflections
2168 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 27.7°$
$h = -20 \rightarrow 20$
$k = -7 \rightarrow 7$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.061$
$S = 1.12$
2258 reflections
104 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.2394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Absolute structure: Flack (1983), 1054 Friedel pairs
Flack parameter = 0.03 (6)

Table 1
Selected geometric parameters (Å, °).

Pd—N1	2.053 (3)	Pd—Cl1	2.3038 (15)
Pd—N2	2.073 (4)	Pd—Cl2	2.3059 (13)
N1—Pd—N2	85.70 (14)	N1—Pd—Cl2	176.91 (12)
N1—Pd—Cl1	92.30 (11)	N2—Pd—Cl2	91.29 (11)
N2—Pd—Cl1	177.82 (11)	Cl1—Pd—Cl2	90.72 (5)

Intensity statistics did not provide a clear choice between the possible space groups *Cc* and *C*2/*c*; the initial solution was sought in *Cc* and the refinement proceeded with no unusual features. In particular, there were no strong correlations between parameters that would be related by symmetry in *C*2/*c*. Analysis of the atomic parameters derived from refinement in *Cc* with *PLATON MISSYM*/*ADDSYM* (Spek, 2003) confirmed that no additional symmetry was present. For these reasons, we believe that the correct space group is indeed *Cc*. The highest residual electron-density peak is located 0.88 Å from the Pd atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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