N(3)—C(8)—C(9)	113.8 (7)	111.0 (4)
C(8)—C(9)—C(10)	111.5 (7)	113.6 (5)
N(4)—C(10)—C(9)	112.1 (6)	112.8 (4)
N(1)—C(1)—C(2)—C(3)	−63.9 (6)	61.5 (4)
C(1)—C(2)—C(3)—N(2)	66.8 (6)	−65.0 (4)
C(4)—N(2)—C(3)—C(2)	173.9 (9)	−173.2 (5)
C(3)—N(2)—C(4)—C(5)	168.7 (8)	−166.0 (5)
N(2)—C(4)—C(5)—N(3)	−47.6 (4)	53.5 (3)
C(8)—N(3)—C(5)—C(4)	172.0 (8)	−176.9 (5)
C(5)—N(3)—C(8)—C(9)	172.0 (9)	−172.8 (5)
N(3)—C(8)—C(9)—C(10)	66.3 (7)	−69.6 (4)
C(8)—C(9)—C(10)—N(4)	−66.2 (7)	61.9 (4)

For both structures, *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

The authors thank the National Science Council for support under grants NSC82-0208-M007-119 and NSC82-0208-M007-032. They are also indebted to Ms Shu-Fang Tung for collecting the X-ray diffraction data.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and possible hydrogen bonds have been deposited with the IUCr (Reference: AS1127). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Di-μ-chloro-bis[chloro(tributylphosphine)palladium]

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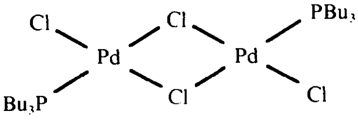
(*Received* 31 *May* 1994; *accepted* 8 *September* 1994)

Abstract

The structure of *trans*-di-μ-chloro-bis[chloro(tributylphosphine)palladium], [$Pd_2Cl_4(C_{12}H_{27}P)_2$], is planar with an inversion centre between the metal atoms. Bond lengths are typical of related species.

Comment

The title complex (I) was prepared (Hartley, 1970) as a precursor to palladium complexes of diaziridines. The structures of a range of related platinum complexes have been determined. In [$Pt_2(\mu$-$Cl)_2Cl_2(PEt_3)_2$] (Blake, Gould, Marr, Rankin & Schröder, 1989) the terminal metal–chlorine bonds were 2.282 (3) Å in length, compared with 2.270 (1) Å here. Bonds between the Pt atoms and bridging halogens were 2.318 (3) and 2.431 (1) Å (the longer bond being for the halogen *trans* to the Cl atoms, as would be expected from the *trans* effect), compared with 2.314 (1) and 2.439 (1) Å here. Angles at the Pt and Cl atoms were also similar. In [$PdPt(\mu$-$Cl)_2Cl_2(PEt_3)_2$] (Clark, Ferguson, Jain & Parvez, 1985) the two metals were disordered and the complex possessed a centre of symmetry. Related complexes which have been structurally characterized include the platinum derivative of di-*tert*-



(I)

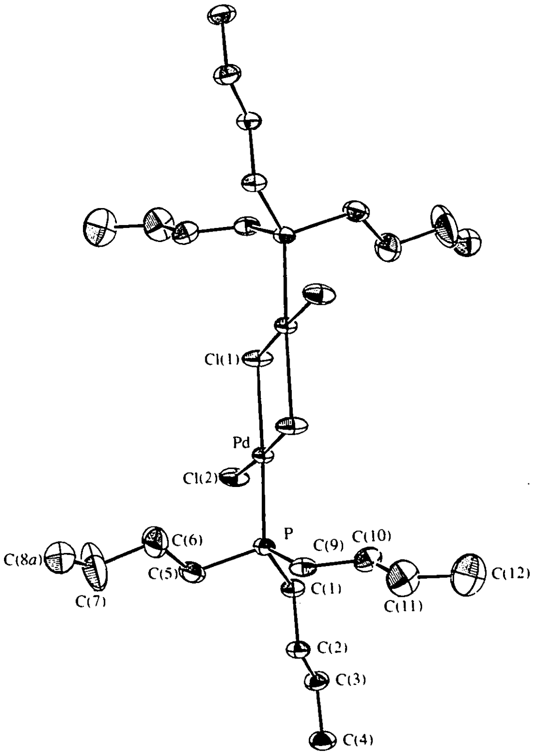


Fig. 1. Molecular structure and numbering scheme (*ORTEP*; Johnson, 1965). The displacement ellipsoids are drawn at the 50% probability level.

butylcyclopropylphosphine (Simms, Shang, Lu, Youngs & Ibers, 1987), tripropylphosphine (Black, Mais & Owston, 1969) and $(CF_3)_2P{=}N{=}PPh_3$ (Ang, Cai, Kwik & Rheingold, 1990).

Experimental

Crystal data

$[Pd_2Cl_4(C_{12}H_{27}P)_2]$
$M_r = 762.3$
Monoclinic
$C2/c$
$a = 21.728\ (9)$ Å
$b = 12.624\ (6)$ Å
$c = 13.838\ (7)$ Å
$\beta = 113.80\ (4)°$
$V = 3473$ Å^3
$Z = 4$
$D_x = 1.46$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 7–10°$
$\mu = 1.44$ mm^{-1}
$T = 293$ K
Block
0.15 × 0.15 × 0.15 mm
Orange
Crystal source: recrystallized from hot absolute ethanol

Data collection

Enraf-Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: empirical
$T_{\min} = 0.90$, $T_{\max} = 1.00$
3272 measured reflections
3194 independent reflections
2463 observed reflections $[|F^2| > 2\sigma(F^2)]$

$R_{\text{int}} = 0.02$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 15$
$l = -16 \rightarrow 16$
2 standard reflections
frequency: 60 min
intensity decay: 0.6%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.046$
$S = 1.4$
2463 reflections
148 parameters
H-atom parameters not refined, $U_{\text{iso}} = 1.3 \times U_{\text{eq}}$ of parent

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.09$
$\Delta\rho_{\max} = 0.46$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *MolEN* (Fair, 1990)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for disordered C(8); $U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	$U_{\text{iso}}/U_{\text{eq}}$
Pd	0.26155 (2)	0.15327 (3)	0.59556 (2)	0.048 (1)
Cl(1)	0.22038 (8)	0.16603 (9)	0.40388 (8)	0.075 (1)
Cl(2)	0.23913 (8)	−0.02301 (10)	0.58586 (9)	0.080 (1)
P	0.29443 (6)	0.14227 (10)	0.76911 (8)	0.052 (1)
C(1)	0.3468 (2)	0.0287 (4)	0.8296 (3)	0.058 (2)
C(2)	0.3739 (2)	0.0258 (4)	0.9495 (3)	0.063 (2)
C(3)	0.4124 (3)	−0.0732 (4)	0.9979 (4)	0.073 (3)
C(4)	0.4397 (3)	−0.0724 (5)	1.1168 (4)	0.094 (3)
C(5)	0.2209 (3)	0.1284 (5)	0.7999 (4)	0.084 (3)
C(6)	0.1688 (3)	0.2080 (7)	0.7545 (6)	0.122 (4)
C(7)	0.1106 (4)	0.2040 (9)	0.7873 (7)	0.204 (7)
C(8*a*)	0.0730 (10)	0.2897 (19)	0.7842 (16)	0.113 (7)
C(8*b*)	0.0884 (12)	0.124 (2)	0.8029 (19)	0.131 (8)
C(8*c*)	0.1057 (11)	0.2084 (19)	0.8538 (16)	0.115 (7)
C(9)	0.3408 (2)	0.2564 (4)	0.8400 (3)	0.064 (2)
C(10)	0.4100 (3)	0.2703 (4)	0.8411 (4)	0.075 (3)
C(11)	0.4469 (4)	0.3651 (5)	0.9013 (6)	0.124 (4)
C(12)	0.5151 (5)	0.3794 (7)	0.9106 (7)	0.157 (6)

Table 2. *Geometric parameters* (Å, °)

Pd—Cl(1)	2.439 (1)	Pd—Cl(1′)	2.314 (1)
Pd—Cl(2)	2.270 (1)	Pd—P	2.216 (1)
P—C(1)	1.811 (4)	P—C(5)	1.820 (7)
P—C(9)	1.804 (5)	C(1)—C(2)	1.521 (6)
C(2)—C(3)	1.502 (7)	C(3)—C(4)	1.506 (7)
C(5)—C(6)	1.454 (9)	C(6)—C(7)	1.506 (13)
C(7)—C(8*a*)	1.35 (3)	C(7)—C(8*b*)	1.17 (3)
C(7)—C(8*c*)	0.97 (3)	C(9)—C(10)	1.508 (8)
C(10)—C(11)	1.492 (8)	C(11)—C(12)	1.447 (14)
Cl(1)—Pd—Cl(1′)	85.74 (4)	Cl(1)—Pd—Cl(2)	91.41 (4)
Cl(1)—Pd—P	177.53 (6)	Cl(1′)—Pd—Cl(2)	176.79 (4)
Cl(1′)—Pd—P	94.48 (4)	Cl(2)—Pd—P	88.30 (4)
Pd—Cl(1)—Pd	94.26 (4)	Pd—P—C(1)	114.7 (2)
Pd—P—C(5)	109.3 (2)	Pd—P—C(9)	113.8 (2)
C(1)—P—C(5)	105.2 (3)	C(1)—P—C(9)	105.8 (2)
C(5)—P—C(9)	107.5 (3)	P—C(1)—C(2)	114.7 (3)
C(1)—C(2)—C(3)	114.1 (4)	C(2)—C(3)—C(4)	112.7 (5)
P—C(5)—C(6)	115.9 (5)	C(5)—C(6)—C(7)	116.7 (7)
C(6)—C(7)—C(8*a*)	122 (1)	C(6)—C(7)—C(8*b*)	123 (2)
C(6)—C(7)—C(8*c*)	135 (1)	C(8*a*)—C(7)—C(8*b*)	114 (2)
C(8*a*)—C(7)—C(8*c*)	72 (2)	C(8*b*)—C(7)—C(8*c*)	70 (2)
P—C(9)—C(10)	115.8 (4)	C(9)—C(10)—C(11)	114.1 (6)
C(10)—C(11)—C(12)	116.7 (7)		

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

C(8) is disordered over three sites with equal occupancy and these were refined with isotropic displacement parameters. H atoms on C(8) and C(7) were omitted. Only one of the sites for the disordered C(8) atom is shown in the *ORTEP* (Johnson, 1965) drawing. The molecule lies across a crystallographic inversion centre.

Computaions were performed using the Enraf–Nonius *MolEN* package (Fair, 1990).

We thank Johnson Matthey PLC for a generous loan of palladium salts.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: HU1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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