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trans-Dichloridobis(triisopropylphosphine-κP)palladium(II)

Aleksandra Wisniewska, Katarzyna Baranowska* and Jerzy Pikies

Department of Inorganic Chemistry, Gdańsk University of Technology, 11/12 G. Narutowicz Street, 80952-PL Gdańsk, Poland
Correspondence e-mail: kasiab29@wp.pl

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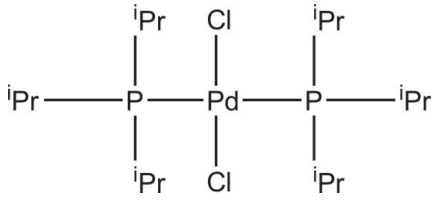
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.090; data-to-parameter ratio = 19.4.

The title compound, $[PdCl_2(C_9H_{21}P)_2]$, is a centrosymmetric mononuclear palladium(II) complex. The Pd^{II} atom, which lies on an inversion center, is in a square-planar geometry.

Related literature

For *trans*-dichlorido-bis(triphenylphosphine)palladium(II), see: Ferguson *et al.* (1982). For *trans*-dichlorido-bis[diphenyl(cyclohexyl)phosphine]palladium(II), see: Meij *et al.* (2003). For *trans*-dichlorido-bis[diphenyl(*p*-tolyl)phosphine]palladium(II), see: Steyl *et al.* (2006). For related literature, see: Baum *et al.* (2006); Bedford *et al.* (2003); Schultz *et al.* (1992).



Experimental

Crystal data

$[PdCl_2(C_9H_{21}P)_2]$ M_r = 497.76
Monoclinic, $P2_1/c$
a = 8.0919 (3) Å
b = 8.9176 (4) Å
c = 16.1920 (6) Å
β = 92.552 (3)°
V = 1167.26 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 120 (2) K
0.15 × 0.09 × 0.02 mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.791, T_{max} = 0.955
7043 measured reflections
2175 independent reflections
1985 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.089
S = 1.13
2175 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2618).

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supporting information

Acta Cryst. (2008). E**64**, m967 [doi:10.1107/S1600536808018904]

trans-Dichloridobis(triisopropylphosphine-*κP*)palladium(II)

Aleksandra Wisniewska, Katarzyna Baranowska and Jerzy Pikies

S1. Comment

Expanding our work upon the reactivity of $[(R_3P)_2MCl_2]$ (M = Ni, Pd, Pt) towards ^{t}Bu$_2$P–PLi–P^{t}Bu$_2$ (Baum *et al.*, 2006), we have studied the reaction of ^{t}Bu$_2$P-PLi-P(^{t}Bu)(SiMe$_3$).2THF with [*trans*-(^{i}Pr$_3$P)$_2$PdCl$_2$] in a 1:1 molar ratio in THF. Unreacted [*trans*-(^{i}Pr$_3$P)$_2$PdCl$_2$] was isolated from the toluene solution of reaction product as yellow crystals.

The molecular structure of the title compound is shown in Fig.1. The mononuclear complex is centrosymmetric, with the Pd^{II} atom lying on an inversion centre. The geometry around the Pd^{II} atom is strictly square-planar. The Pd—P [2.3603 (6) Å] and Pd—Cl [2.3030 (6) Å] distances and P—Pd—Cl [89.92 (2)° and 90.18 (2)°] angles are typical for [*trans*-(R_3P)$_2$PdCl$_2$] (Ferguson *et al.*, 1982; Meij *et al.*, 2003; Steyl *et al.*, 2006; Bedford *et al.*, 2003). The distances in [*cis*-(R_3P)$_2$PdCl$_2$] differ significantly from those reported for [*trans*-(R_3P)$_2$PdCl$_2$]. For [*cis*-(Me$_3$P)$_2$PdCl$_2$], the related distances are 2.374 (3) Å (Pd—Cl, mean value) and 2.258 (2) Å (Pd—P, mean value) (Schultz *et al.*, 1992). The elongation of Pd—Cl distances in *cis* isomers compared to *trans* isomers is due to a strong *trans* effect of PR_3 ligand in a position *trans* to Cl ligand. The shortening of Pd—P distances in *cis* isomers are caused by a lack of a second phosphine ligand in the *trans* position. The Cl ligand exerts only weak *trans* effect.

S2. Experimental

A solution of ^{t}Bu$_2$P-PLi-P(^{t}Bu)(SiMe$_3$).2THF (139 mg, 0.285 mmol) in tetrahydrofuran (THF, 2 mL) was added dropwise to a suspension of yellow powder of [(^{i}Pr$_3$P)$_2$PdCl$_2$] (139 mg, 0.28 mmol) in THF (2 ml) at room temperature. The mixture turned red. After allowed to stand at room temperature for 1 d, the mixture was dried under vacuum at 1 mTorr for 1 h, and the residue was dissolved in toluene (4 ml) and filtered. The solution was kept at 277 K for 2d to obtain small yellow crystals of [*trans*-(^{i}Pr$_3$P)$_2$PdCl$_2$].

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C–H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The highest residual electron-density peak is located 0.86 Å from atom Cl1.

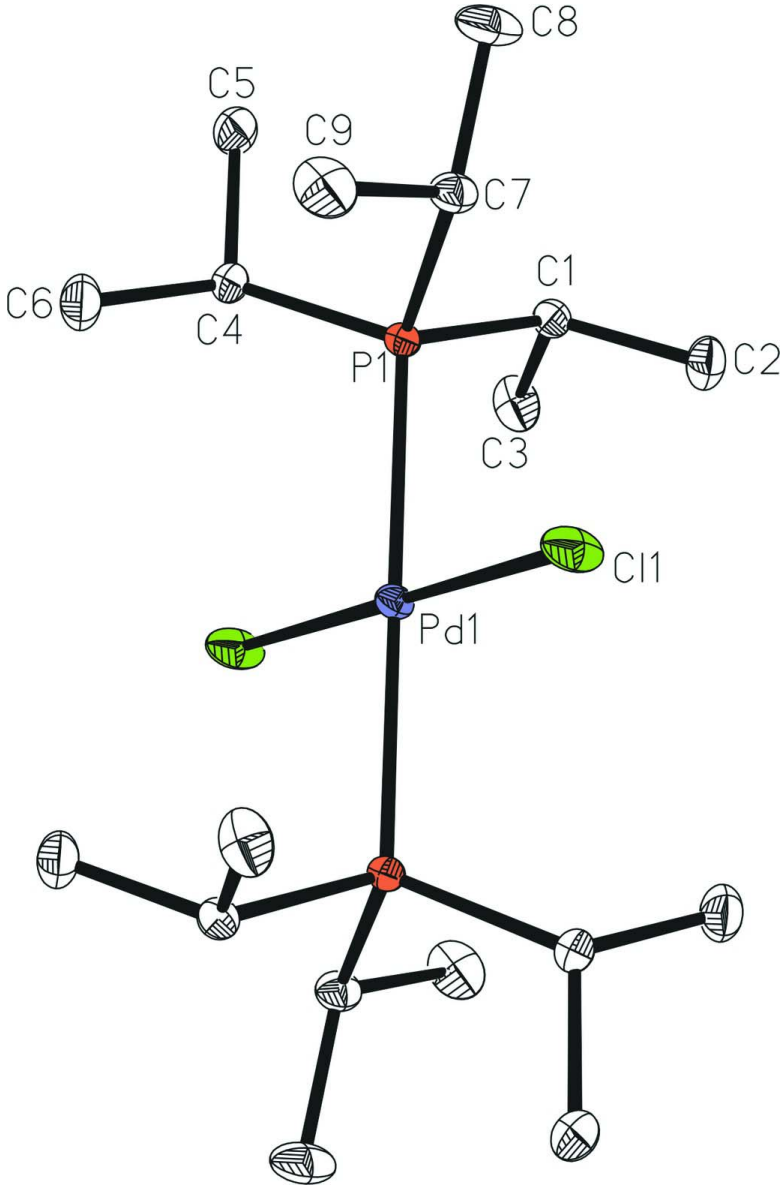

Figure 1

A view of the title molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. Unlabelled atoms are related to labelled atoms by the symmetry operation (1-x, 1-y, -z).

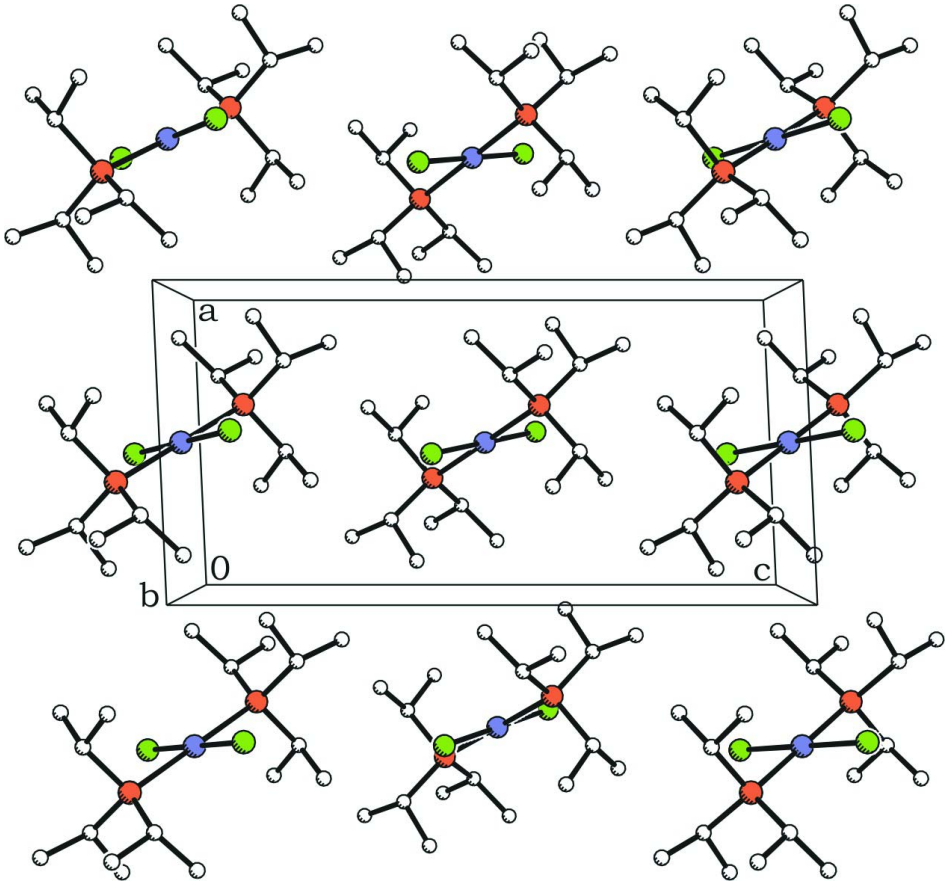

Figure 2

Crystal packing of the title compound, viewed approximately along the *b* axis.

trans-Dichloridobis(triisopropylphosphine-κP)palladium(II)

Crystal data

$[PdCl_2(C_9H_{21}P)_2]$
$M_r = 497.76$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.0919\ (3)$ Å
$b = 8.9176\ (4)$ Å
$c = 16.1920\ (6)$ Å
$\beta = 92.552\ (3)°$
$V = 1167.26\ (8)$ Å^3
$Z = 2$
$F(000) = 520$
$D_x = 1.416$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7947 reflections
$\theta = 2.3–32.5°$
$\mu = 1.16$ mm^{-1}
$T = 120$ K
Prism, colourless
0.15 × 0.09 × 0.02 mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer
Graphite monochromator
Detector resolution: 8.1883 pixels mm^{-1}
0.75° wide ω scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.791$, $T_{max} = 0.955$
7043 measured reflections
2175 independent reflections
1985 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.5°$
$h = -9\rightarrow9$
$k = -10\rightarrow6$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.089$
$S = 1.13$
2175 reflections
112 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.2267P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.44$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.5	0.5	0	0.01758 (14)
P1	0.62758 (8)	0.33350 (7)	0.09656 (4)	0.01777 (17)
Cl1	0.53892 (10)	0.69501 (7)	0.09206 (4)	0.0358 (2)
C1	0.4699 (3)	0.2487 (3)	0.16092 (14)	0.0218 (5)
H1	0.529	0.1858	0.204	0.026*
C2	0.3743 (3)	0.3692 (3)	0.20534 (17)	0.0322 (6)
H2A	0.3178	0.4347	0.1645	0.048*
H2B	0.4512	0.4288	0.2404	0.048*
H2C	0.2925	0.3217	0.2397	0.048*
C3	0.3525 (4)	0.1470 (3)	0.11119 (18)	0.0352 (7)
H3A	0.2641	0.1132	0.1462	0.053*
H3B	0.4134	0.0599	0.0916	0.053*
H3C	0.3043	0.2023	0.0637	0.053*
C4	0.7288 (3)	0.1727 (3)	0.04740 (15)	0.0222 (5)
H4	0.639	0.1221	0.0134	0.027*
C5	0.7990 (4)	0.0510 (3)	0.10597 (17)	0.0336 (6)
H5A	0.8222	−0.0396	0.0742	0.05*
H5B	0.7182	0.0277	0.1474	0.05*
H5C	0.9015	0.0871	0.1337	0.05*
C6	0.8569 (4)	0.2198 (3)	−0.01420 (17)	0.0355 (7)
H6A	0.9591	0.2505	0.0159	0.053*
H6B	0.8136	0.304	−0.0475	0.053*
H6C	0.8802	0.1352	−0.0505	0.053*
C7	0.7729 (3)	0.4281 (3)	0.17166 (15)	0.0248 (5)
H7	0.7088	0.512	0.1959	0.03*

C8	0.8372 (4)	0.3339 (3)	0.24483 (18)	0.0393 (7)
H8A	0.9122	0.2566	0.2254	0.059*
H8B	0.7439	0.2861	0.2711	0.059*
H8C	0.8968	0.3985	0.285	0.059*
C9	0.9156 (5)	0.5020 (3)	0.1284 (2)	0.0403 (9)
H9A	0.9727	0.5725	0.1663	0.06*
H9B	0.8723	0.5562	0.0794	0.06*
H9C	0.9936	0.4249	0.1116	0.06*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0209 (2)	0.0166 (2)	0.01496 (18)	−0.00014 (9)	−0.00173 (12)	−0.00049 (8)
P1	0.0189 (3)	0.0187 (3)	0.0156 (3)	0.0011 (2)	−0.0004 (2)	0.0009 (2)
Cl1	0.0575 (5)	0.0223 (3)	0.0259 (3)	0.0064 (3)	−0.0155 (3)	−0.0074 (3)
C1	0.0226 (12)	0.0224 (11)	0.0205 (12)	0.0007 (10)	0.0030 (10)	0.0034 (10)
C2	0.0311 (14)	0.0334 (14)	0.0329 (14)	0.0026 (13)	0.0120 (12)	−0.0013 (12)
C3	0.0326 (15)	0.0392 (16)	0.0344 (15)	−0.0152 (13)	0.0068 (12)	−0.0051 (12)
C4	0.0226 (12)	0.0216 (12)	0.0225 (12)	0.0035 (10)	0.0023 (10)	−0.0014 (10)
C5	0.0391 (17)	0.0292 (14)	0.0329 (15)	0.0120 (14)	0.0053 (13)	0.0000 (13)
C6	0.0354 (16)	0.0381 (16)	0.0341 (15)	0.0051 (13)	0.0129 (13)	0.0002 (12)
C7	0.0252 (13)	0.0262 (13)	0.0225 (12)	−0.0011 (11)	−0.0049 (10)	−0.0019 (10)
C8	0.0494 (18)	0.0365 (15)	0.0301 (15)	0.0017 (14)	−0.0196 (13)	−0.0004 (12)
C9	0.0307 (19)	0.052 (2)	0.0378 (19)	−0.0199 (12)	−0.0029 (16)	−0.0036 (12)

Geometric parameters (Å, º)

Pd1—Cl1^{i}	2.3030 (6)	C4—C5	1.533 (4)
Pd1—Cl1	2.3030 (6)	C4—H4	1
Pd1—P1	2.3603 (6)	C5—H5A	0.98
Pd1—P1^{i}	2.3603 (6)	C5—H5B	0.98
P1—C1	1.845 (2)	C5—H5C	0.98
P1—C4	1.849 (2)	C6—H6A	0.98
P1—C7	1.856 (2)	C6—H6B	0.98
C1—C3	1.518 (4)	C6—H6C	0.98
C1—C2	1.523 (3)	C7—C8	1.525 (4)
C1—H1	1	C7—C9	1.528 (4)
C2—H2A	0.98	C7—H7	1
C2—H2B	0.98	C8—H8A	0.98
C2—H2C	0.98	C8—H8B	0.98
C3—H3A	0.98	C8—H8C	0.98
C3—H3B	0.98	C9—H9A	0.98
C3—H3C	0.98	C9—H9B	0.98
C4—C6	1.529 (3)	C9—H9C	0.98
Cl1^{i}—Pd1—Cl1	180.00 (3)	C6—C4—H4	105.2
Cl1^{i}—Pd1—P1	89.82 (2)	C5—C4—H4	105.2
Cl1—Pd1—P1	90.18 (2)	P1—C4—H4	105.2

Cl1^{i}—Pd1—P1^{i}	90.18 (2)	C4—C5—H5A	109.5
Cl1—Pd1—P1^{i}	89.82 (2)	C4—C5—H5B	109.5
P1—Pd1—P1^{i}	180	H5A—C5—H5B	109.5
C1—P1—C4	104.84 (11)	C4—C5—H5C	109.5
C1—P1—C7	104.47 (11)	H5A—C5—H5C	109.5
C4—P1—C7	110.77 (12)	H5B—C5—H5C	109.5
C1—P1—Pd1	109.81 (8)	C4—C6—H6A	109.5
C4—P1—Pd1	113.07 (8)	C4—C6—H6B	109.5
C7—P1—Pd1	113.18 (8)	H6A—C6—H6B	109.5
C3—C1—C2	110.7 (2)	C4—C6—H6C	109.5
C3—C1—P1	112.16 (17)	H6A—C6—H6C	109.5
C2—C1—P1	110.86 (17)	H6B—C6—H6C	109.5
C3—C1—H1	107.6	C8—C7—C9	110.8 (2)
C2—C1—H1	107.6	C8—C7—P1	116.30 (18)
P1—C1—H1	107.6	C9—C7—P1	111.43 (19)
C1—C2—H2A	109.5	C8—C7—H7	105.8
C1—C2—H2B	109.5	C9—C7—H7	105.8
H2A—C2—H2B	109.5	P1—C7—H7	105.8
C1—C2—H2C	109.5	C7—C8—H8A	109.5
H2A—C2—H2C	109.5	C7—C8—H8B	109.5
H2B—C2—H2C	109.5	H8A—C8—H8B	109.5
C1—C3—H3A	109.5	C7—C8—H8C	109.5
C1—C3—H3B	109.5	H8A—C8—H8C	109.5
H3A—C3—H3B	109.5	H8B—C8—H8C	109.5
C1—C3—H3C	109.5	C7—C9—H9A	109.5
H3A—C3—H3C	109.5	C7—C9—H9B	109.5
H3B—C3—H3C	109.5	H9A—C9—H9B	109.5
C6—C4—C5	110.8 (2)	C7—C9—H9C	109.5
C6—C4—P1	113.17 (18)	H9A—C9—H9C	109.5
C5—C4—P1	116.20 (17)	H9B—C9—H9C	109.5

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