

Di- μ -diphenylphosphido-bis{chlorido[(mesitylmethyl)diphenylphosphine- κ P]}palladium(II)}

Ludovic Chahen, Bruno Therrien and Georg Süss-Fink

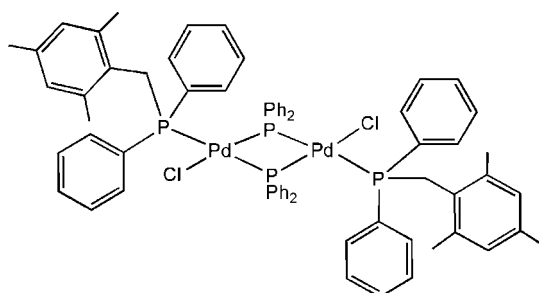
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In the title dinuclear palladium complex, $[\text{Pd}_2(\text{C}_{12}\text{H}_{10}\text{P})_2\text{Cl}_2(\text{C}_{22}\text{H}_{23}\text{P})_2]$, the terminal ligands adopt a trans configuration with a Pd...Pd distance of 3.6111 (4) Å. A crystallographic twofold rotation axis runs perpendicular to the Pd_2P_2 ring. The Cl atom lies slightly out of the plane of the Pd and three P atoms in each half of the molecule.

Related literature

For similar dinuclear palladium complexes, see: Brandon & Dixon (1981); Carty *et al.* (1982); Gebauer *et al.* (1992); Zhuravel *et al.* (2000). For the synthesis of $\text{PPh}_2\text{CH}_2(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)$, see: Chahen *et al.* (2006).



Experimental

Crystal data

$[\text{Pd}_2(\text{C}_{12}\text{H}_{10}\text{P})_2\text{Cl}_2(\text{C}_{22}\text{H}_{23}\text{P})_2]$

$M_r = 1290.83$

Orthorhombic, *Pccn*

$a = 12.0793$ (7) Å

$b = 22.6156$ (14) Å

$c = 22.5499$ (14) Å

$V = 6160.2$ (6) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.81$ mm⁻¹

$T = 173$ (2) K

$0.31 \times 0.24 \times 0.17$ mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
46413 measured reflections

6011 independent reflections
3498 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.089$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.064$

$S = 0.78$

6011 reflections

346 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.53$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.98$ e Å⁻³

Data collection: *EXPOSE* in *IPDS Software* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Version 1.4.1; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

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References

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supplementary materials

supplementary materials

Di- μ -diphenylphosphido-bis{chlorido[(mesitylmethyl)diphenylphosphine- κ -P]}palladium(II)

L. Chahen, B. Therrien and G. Süss-Fink

Comment

The title compound, $[\text{PdCl}(\mu_2\text{-PPh}_2)\{\text{PPh}_2\text{CH}_2(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)\}]_2$ (I), is prepared in a similar manner as the isoelectronic dinuclear palladium complex $[\text{PdCl}(\mu_2\text{-PPh}_2)(\text{PPh}_3)]_2$ (Brandon & Dixon, 1981). The triphenylphosphine being replaced by (mesitylmethyl)(diphenyl)phosphine (Chahen *et al.*, 2006). The $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum gives rise to two multiplets centred at 7.5 and -142.6 p.p.m. respectively. The signals are consistent with an AA'BB' spin system (Brandon & Dixon, 1981). The molecular structure of (I) shows the palladium atoms to be surrounded by two phosphido-bridged ligands, a chloride, and a $\text{PPh}_2\text{CH}_2(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)$ ligand. The complex adopts the *trans*-symmetrical isomer, see scheme.

The two halves of the dinuclear complex are related by a crystallography twofold rotation axis, see Fig. 1. The Pd atoms are in a slightly distorted square-planar geometry. The plane formed by the palladium, the chloride and the three P atoms is mainly planar, with a maximum deviation from the mean plane of $0.0770(4)\text{Å}$ for one bridging P atom. However, the angle between the two square-planar units is $12.63(1)^\circ$, giving rise to a slightly bent conformation for the dimeric unit, see Fig. 2. Otherwise, the bond distances and angles are similar to those found in $[\text{PtCl}(\mu_2\text{-PPh}_2)(\text{HPPH}_2)]_2$ (Carty *et al.*, 1982), $[\text{PdCl}(\mu_2\text{-PPh}_2)(\text{HPPH}_2)]_2$ (Gebauer *et al.*, 1992) and $[\text{PdI}(\mu_2\text{-PPh}_2)(\text{HPPH}_2)]_2$ (Zhuravel *et al.*, 2000).

Experimental

The synthesis of $[\text{PdCl}(\mu_2\text{-PPh}_2)\{\text{PPh}_2\text{CH}_2(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)\}]_2$ is identical to the synthesis of $[\text{PdCl}(\mu_2\text{-PPh}_2)(\text{PPh}_3)]_2$ (Brandon & Dixon, 1981). The yellow product is purified by column chromatography on silica gel (dichloromethane:acetone, 1:2).

^1H NMR (CDCl_3): 6.9–6.2 (m, 44H), 2.76 (m, 4H), 1.58 (s, 12H), 1.34 (m, 6H). ^{31}P NMR (CDCl_3): 7.5 and -142.6 p.p.m.. MS (ESI, m/z): 1291; $[\text{C}_{68}\text{H}_{66}\text{Cl}_2\text{P}_4\text{Pd}_2]$ Calcd. for $\text{C}_{68}\text{H}_{66}\text{Cl}_2\text{P}_4\text{Pd}_2$: C, 63.27; H, 5.15 Found: C, 62.87; H, 5.02.

$[\text{PdCl}(\mu_2\text{-PPh}_2)\{\text{PPh}_2\text{CH}_2(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)\}]_2$ is dissolved in chloroform, and crystals suitable for X-ray diffraction analysis are obtained by slow evaporation of the chloroform solution.

Refinement

The H atoms were included in calculated positions and refined using a riding model, with $\text{C—H} = 0.93\text{--}0.96\text{Å}$ and with $U_{\text{iso}}(\text{H}) = 1.2$ (1.5 for methyl groups) times $U_{\text{eq}}(\text{C})$.

supplementary materials

Figures

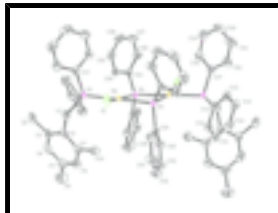


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.



Fig. 2. Bent conformation adopted by the title compound.

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$M_r = 1290.83$

Orthorhombic, $Pccn$

Hall symbol: $-P\ 2ab\ 2ac$

$a = 12.0793\ (7)\ \text{\AA}$

$b = 22.6156\ (14)\ \text{\AA}$

$c = 22.5499\ (14)\ \text{\AA}$

$V = 6160.2\ (6)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 2640$

$D_x = 1.392\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 8000 reflections

$\theta = 2.1\text{--}25.9^\circ$

$\mu = 0.81\ \text{mm}^{-1}$

$T = 173\ (2)\ \text{K}$

Plate, yellow

$0.31 \times 0.24 \times 0.17\ \text{mm}$

Data collection

Stoe IPDS
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: $0.81\ \text{pixels mm}^{-1}$

$T = 173\ (2)\ \text{K}$

ϕ oscillation scans

Absorption correction: none

46413 measured reflections

6011 independent reflections

3498 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.089$

$\theta_{\text{max}} = 26.0^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -14 \rightarrow 14$

$k = -27 \rightarrow 27$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

supplementary materials

$wR(F^2) = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2]$
$S = 0.78$	where $P = (F_o^2 + 2F_c^2)/3$
6011 reflections	$(\Delta/\sigma)_{\max} = 0.002$
346 parameters	$\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.98 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Experimental. A crystal was mounted at 173 K on a Stoe Image Plate Diffraction System (Stoe, 2000) using Mo $K\alpha$ graphite monochromated radiation. Image plate distance 70 mm, φ oscillation scans 0 – 200°, step $\Delta\varphi = 1.0^\circ$, 3 minutes per frame.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3394 (2)	0.68728 (15)	0.14638 (15)	0.0288 (8)
H1A	0.3333	0.7299	0.1498	0.035*
H1B	0.2726	0.6729	0.1273	0.035*
C2	0.3467 (2)	0.66113 (15)	0.20757 (16)	0.0313 (8)
C3	0.2996 (3)	0.60533 (16)	0.21955 (18)	0.0348 (9)
C4	0.3181 (3)	0.57908 (17)	0.27389 (19)	0.0446 (10)
H4	0.2867	0.5423	0.2813	0.054*
C5	0.3810 (3)	0.60503 (18)	0.31752 (19)	0.0467 (10)
C6	0.4210 (3)	0.66176 (18)	0.30728 (18)	0.0442 (10)
H6	0.4601	0.6810	0.3371	0.053*
C7	0.4040 (3)	0.69051 (15)	0.25328 (16)	0.0332 (8)
C8	0.2300 (3)	0.57336 (17)	0.17367 (18)	0.0455 (10)
H8A	0.2078	0.5356	0.1890	0.068*
H8B	0.2726	0.5678	0.1382	0.068*
H8C	0.1654	0.5965	0.1648	0.068*
C9	0.4049 (4)	0.5717 (2)	0.3747 (2)	0.0758 (14)
H9A	0.3390	0.5517	0.3876	0.114*
H9B	0.4282	0.5991	0.4047	0.114*
H9C	0.4625	0.5433	0.3679	0.114*
C10	0.4471 (3)	0.75305 (17)	0.24475 (16)	0.0434 (9)
H10A	0.3857	0.7799	0.2420	0.065*
H10B	0.4899	0.7550	0.2090	0.065*
H10C	0.4927	0.7638	0.2779	0.065*

supplementary materials

C11	0.4922 (2)	0.59215 (13)	0.11434 (15)	0.0257 (8)
C12	0.4717 (3)	0.54607 (15)	0.07458 (17)	0.0335 (9)
H12	0.4377	0.5538	0.0384	0.040*
C13	0.5023 (3)	0.48856 (15)	0.08905 (17)	0.0379 (10)
H13	0.4872	0.4579	0.0628	0.045*
C14	0.5544 (3)	0.47669 (17)	0.14176 (19)	0.0409 (10)
H14	0.5772	0.4384	0.1504	0.049*
C15	0.5733 (3)	0.52168 (16)	0.18205 (18)	0.0364 (9)
H15	0.6068	0.5135	0.2183	0.044*
C16	0.5420 (2)	0.57898 (15)	0.16824 (17)	0.0304 (8)
H16	0.5545	0.6091	0.1955	0.036*
C17	0.4122 (2)	0.67027 (14)	0.02220 (15)	0.0279 (8)
C18	0.4917 (3)	0.67252 (16)	-0.02190 (16)	0.0372 (9)
H18	0.5662	0.6716	-0.0117	0.045*
C19	0.4618 (3)	0.67611 (17)	-0.08118 (17)	0.0443 (10)
H19	0.5163	0.6770	-0.1103	0.053*
C20	0.3531 (3)	0.67830 (17)	-0.09702 (19)	0.0508 (11)
H20	0.3329	0.6810	-0.1367	0.061*
C21	0.2743 (3)	0.6765 (2)	-0.0539 (2)	0.0614 (13)
H21	0.2000	0.6780	-0.0646	0.074*
C22	0.3022 (3)	0.67225 (18)	0.00542 (18)	0.0475 (11)
H22	0.2469	0.6708	0.0341	0.057*
C23	0.7748 (2)	0.63475 (14)	0.04978 (15)	0.0256 (8)
C24	0.7492 (3)	0.57485 (15)	0.05565 (16)	0.0324 (8)
H24	0.7251	0.5605	0.0921	0.039*
C25	0.7593 (3)	0.53672 (18)	0.00822 (18)	0.0466 (10)
H25	0.7424	0.4969	0.0130	0.056*
C26	0.7940 (3)	0.5572 (2)	-0.0457 (2)	0.0540 (12)
H26	0.8001	0.5314	-0.0777	0.065*
C27	0.8196 (3)	0.6159 (2)	-0.05260 (18)	0.0527 (11)
H27	0.8432	0.6297	-0.0894	0.063*
C28	0.8109 (3)	0.65508 (17)	-0.00562 (16)	0.0378 (9)
H28	0.8289	0.6947	-0.0108	0.045*
C29	0.7848 (2)	0.65141 (14)	0.17945 (16)	0.0262 (8)
C30	0.8636 (3)	0.60701 (15)	0.18551 (18)	0.0357 (9)
H30	0.9022	0.5934	0.1526	0.043*
C31	0.8840 (3)	0.58332 (18)	0.2410 (2)	0.0500 (11)
H31	0.9355	0.5530	0.2450	0.060*
C32	0.8293 (3)	0.6039 (2)	0.2902 (2)	0.0550 (12)
H32	0.8440	0.5875	0.3272	0.066*
C33	0.7531 (4)	0.64862 (19)	0.28494 (17)	0.0513 (11)
H33	0.7170	0.6631	0.3183	0.062*
C34	0.7303 (3)	0.67193 (16)	0.22955 (16)	0.0375 (9)
H34	0.6778	0.7018	0.2258	0.045*
Cl1	0.46039 (6)	0.81271 (4)	0.08771 (4)	0.0368 (2)
P3	0.76040 (6)	0.68822 (3)	0.10901 (4)	0.02197 (18)
P5	0.46032 (6)	0.66918 (4)	0.09866 (4)	0.02419 (19)
Pd1	0.601393 (16)	0.741408 (10)	0.103340 (11)	0.02204 (7)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0208 (15)	0.033 (2)	0.033 (2)	0.0008 (14)	0.0053 (14)	-0.0005 (17)
C2	0.0270 (17)	0.035 (2)	0.032 (2)	0.0045 (15)	0.0070 (15)	0.0018 (18)
C3	0.0304 (17)	0.038 (2)	0.037 (2)	0.0009 (16)	0.0071 (16)	-0.0002 (19)
C4	0.039 (2)	0.042 (2)	0.052 (3)	-0.0029 (18)	0.012 (2)	0.010 (2)
C5	0.048 (2)	0.055 (3)	0.037 (3)	0.002 (2)	0.006 (2)	0.007 (2)
C6	0.045 (2)	0.055 (3)	0.033 (3)	0.0003 (19)	0.0029 (18)	-0.004 (2)
C7	0.0287 (16)	0.036 (2)	0.034 (2)	0.0011 (17)	0.0073 (17)	-0.0054 (17)
C8	0.042 (2)	0.046 (3)	0.048 (3)	-0.0153 (18)	0.0046 (18)	0.005 (2)
C9	0.095 (4)	0.081 (4)	0.052 (3)	-0.002 (3)	-0.010 (3)	0.024 (3)
C10	0.0496 (19)	0.045 (2)	0.036 (2)	-0.0038 (19)	0.0057 (16)	-0.010 (2)
C11	0.0238 (15)	0.0244 (17)	0.029 (2)	0.0010 (13)	0.0014 (14)	0.0013 (16)
C12	0.0353 (18)	0.033 (2)	0.033 (2)	-0.0068 (15)	-0.0013 (16)	0.0033 (17)
C13	0.048 (2)	0.0225 (19)	0.043 (3)	-0.0054 (16)	0.0112 (18)	-0.0054 (18)
C14	0.046 (2)	0.029 (2)	0.048 (3)	0.0036 (16)	0.0113 (19)	0.007 (2)
C15	0.0339 (19)	0.038 (2)	0.038 (3)	0.0032 (15)	0.0048 (16)	0.0130 (19)
C16	0.0228 (16)	0.032 (2)	0.037 (2)	0.0005 (14)	0.0033 (15)	0.0003 (18)
C17	0.0299 (17)	0.0249 (18)	0.029 (2)	-0.0019 (15)	-0.0042 (15)	-0.0012 (15)
C18	0.0399 (19)	0.040 (2)	0.031 (2)	-0.0046 (17)	0.0001 (17)	0.0013 (19)
C19	0.062 (3)	0.042 (2)	0.029 (2)	-0.0030 (19)	0.0041 (19)	-0.0003 (19)
C20	0.070 (3)	0.053 (2)	0.029 (3)	0.0086 (19)	-0.016 (2)	0.000 (2)
C21	0.046 (3)	0.090 (4)	0.048 (3)	0.011 (2)	-0.024 (2)	-0.006 (3)
C22	0.0335 (19)	0.070 (3)	0.039 (3)	0.0002 (19)	-0.0039 (18)	-0.001 (2)
C23	0.0205 (17)	0.030 (2)	0.026 (2)	0.0043 (13)	-0.0024 (13)	-0.0059 (16)
C24	0.0297 (15)	0.032 (2)	0.036 (2)	-0.0042 (15)	0.0036 (19)	-0.0080 (18)
C25	0.046 (2)	0.042 (2)	0.052 (3)	-0.0046 (18)	0.001 (2)	-0.019 (2)
C26	0.059 (2)	0.061 (3)	0.042 (3)	0.002 (2)	-0.002 (2)	-0.028 (2)
C27	0.063 (3)	0.076 (3)	0.019 (2)	0.007 (2)	0.002 (2)	-0.005 (2)
C28	0.047 (2)	0.041 (2)	0.026 (2)	0.0048 (17)	-0.0022 (18)	-0.0015 (19)
C29	0.0283 (17)	0.0248 (19)	0.026 (2)	-0.0061 (14)	-0.0016 (14)	0.0050 (16)
C30	0.0361 (19)	0.036 (2)	0.035 (2)	0.0031 (15)	-0.0041 (16)	0.0025 (18)
C31	0.055 (2)	0.043 (2)	0.052 (3)	0.010 (2)	-0.018 (2)	0.014 (2)
C32	0.064 (3)	0.068 (3)	0.032 (3)	-0.006 (2)	-0.013 (2)	0.019 (2)
C33	0.059 (2)	0.070 (3)	0.025 (2)	-0.003 (2)	0.005 (2)	0.007 (2)
C34	0.041 (2)	0.039 (2)	0.032 (2)	0.0016 (16)	-0.0012 (16)	0.0034 (19)
C11	0.0234 (4)	0.0269 (5)	0.0601 (7)	0.0045 (3)	-0.0014 (4)	0.0051 (4)
P3	0.0207 (4)	0.0218 (4)	0.0234 (5)	0.0006 (3)	0.0003 (4)	0.0009 (4)
P5	0.0200 (4)	0.0234 (4)	0.0291 (5)	-0.0016 (3)	0.0004 (4)	0.0007 (4)
Pd1	0.01932 (10)	0.02174 (12)	0.02506 (13)	0.00082 (11)	-0.00015 (11)	0.00017 (13)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.504 (5)	C18—C19	1.387 (5)
C1—P5	1.860 (3)	C18—H18	0.9300
C1—H1A	0.9700	C19—C20	1.362 (5)
C1—H1B	0.9700	C19—H19	0.9300

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C2—C7	1.408 (5)	C20—C21	1.361 (6)
C2—C3	1.410 (5)	C20—H20	0.9300
C3—C4	1.380 (5)	C21—C22	1.382 (6)
C3—C8	1.517 (5)	C21—H21	0.9300
C4—C5	1.375 (5)	C22—H22	0.9300
C4—H4	0.9300	C23—C24	1.396 (4)
C5—C6	1.390 (5)	C23—C28	1.401 (5)
C5—C9	1.521 (6)	C23—P3	1.810 (3)
C6—C7	1.396 (5)	C24—C25	1.379 (5)
C6—H6	0.9300	C24—H24	0.9300
C7—C10	1.519 (5)	C25—C26	1.368 (6)
C8—H8A	0.9600	C25—H25	0.9300
C8—H8B	0.9600	C26—C27	1.373 (6)
C8—H8C	0.9600	C26—H26	0.9300
C9—H9A	0.9600	C27—C28	1.385 (5)
C9—H9B	0.9600	C27—H27	0.9300
C9—H9C	0.9600	C28—H28	0.9300
C10—H10A	0.9600	C29—C34	1.388 (5)
C10—H10B	0.9600	C29—C30	1.390 (4)
C10—H10C	0.9600	C29—P3	1.817 (3)
C11—C16	1.388 (5)	C30—C31	1.383 (5)
C11—C12	1.397 (5)	C30—H30	0.9300
C11—P5	1.819 (3)	C31—C32	1.372 (6)
C12—C13	1.391 (5)	C31—H31	0.9300
C12—H12	0.9300	C32—C33	1.373 (6)
C13—C14	1.372 (5)	C32—H32	0.9300
C13—H13	0.9300	C33—C34	1.383 (5)
C14—C15	1.383 (5)	C33—H33	0.9300
C14—H14	0.9300	C34—H34	0.9300
C15—C16	1.385 (5)	C11—Pd1	2.3718 (8)
C15—H15	0.9300	P3—Pd1	2.2699 (8)
C16—H16	0.9300	P3—Pd1 ⁱ	2.3101 (8)
C17—C18	1.383 (5)	P5—Pd1	2.3629 (8)
C17—C22	1.383 (4)	Pd1—P3 ⁱ	2.3101 (8)
C17—P5	1.819 (3)		
C2—C1—P5	113.5 (2)	C20—C19—C18	120.4 (4)
C2—C1—H1A	108.9	C20—C19—H19	119.8
P5—C1—H1A	108.9	C18—C19—H19	119.8
C2—C1—H1B	108.9	C21—C20—C19	119.0 (4)
P5—C1—H1B	108.9	C21—C20—H20	120.5
H1A—C1—H1B	107.7	C19—C20—H20	120.5
C7—C2—C3	118.7 (3)	C20—C21—C22	121.5 (4)
C7—C2—C1	121.0 (3)	C20—C21—H21	119.2
C3—C2—C1	120.3 (3)	C22—C21—H21	119.2
C4—C3—C2	119.3 (4)	C21—C22—C17	120.1 (4)
C4—C3—C8	119.4 (3)	C21—C22—H22	120.0
C2—C3—C8	121.3 (3)	C17—C22—H22	120.0
C5—C4—C3	122.8 (4)	C24—C23—C28	118.2 (3)

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C5—C4—H4	118.6	C24—C23—P3	123.9 (3)
C3—C4—H4	118.6	C28—C23—P3	117.9 (3)
C4—C5—C6	117.9 (4)	C25—C24—C23	120.9 (4)
C4—C5—C9	120.0 (4)	C25—C24—H24	119.5
C6—C5—C9	122.1 (4)	C23—C24—H24	119.5
C5—C6—C7	121.6 (4)	C26—C25—C24	120.3 (4)
C5—C6—H6	119.2	C26—C25—H25	119.8
C7—C6—H6	119.2	C24—C25—H25	119.8
C6—C7—C2	119.4 (3)	C25—C26—C27	119.8 (4)
C6—C7—C10	119.6 (3)	C25—C26—H26	120.1
C2—C7—C10	121.0 (3)	C27—C26—H26	120.1
C3—C8—H8A	109.5	C26—C27—C28	121.0 (4)
C3—C8—H8B	109.5	C26—C27—H27	119.5
H8A—C8—H8B	109.5	C28—C27—H27	119.5
C3—C8—H8C	109.5	C27—C28—C23	119.8 (4)
H8A—C8—H8C	109.5	C27—C28—H28	120.1
H8B—C8—H8C	109.5	C23—C28—H28	120.1
C5—C9—H9A	109.5	C34—C29—C30	119.1 (3)
C5—C9—H9B	109.5	C34—C29—P3	118.8 (3)
H9A—C9—H9B	109.5	C30—C29—P3	121.9 (3)
C5—C9—H9C	109.5	C31—C30—C29	119.4 (4)
H9A—C9—H9C	109.5	C31—C30—H30	120.3
H9B—C9—H9C	109.5	C29—C30—H30	120.3
C7—C10—H10A	109.5	C32—C31—C30	120.9 (4)
C7—C10—H10B	109.5	C32—C31—H31	119.5
H10A—C10—H10B	109.5	C30—C31—H31	119.5
C7—C10—H10C	109.5	C31—C32—C33	120.2 (4)
H10A—C10—H10C	109.5	C31—C32—H32	119.9
H10B—C10—H10C	109.5	C33—C32—H32	119.9
C16—C11—C12	118.6 (3)	C32—C33—C34	119.4 (4)
C16—C11—P5	117.9 (3)	C32—C33—H33	120.3
C12—C11—P5	123.5 (3)	C34—C33—H33	120.3
C13—C12—C11	120.0 (3)	C33—C34—C29	120.9 (4)
C13—C12—H12	120.0	C33—C34—H34	119.5
C11—C12—H12	120.0	C29—C34—H34	119.5
C14—C13—C12	120.5 (3)	C23—P3—C29	108.87 (16)
C14—C13—H13	119.7	C23—P3—Pd1	113.18 (10)
C12—C13—H13	119.7	C29—P3—Pd1	115.42 (11)
C13—C14—C15	120.1 (4)	C23—P3—Pd1 ⁱ	110.50 (10)
C13—C14—H14	120.0	C29—P3—Pd1 ⁱ	104.28 (10)
C15—C14—H14	120.0	Pd1—P3—Pd1 ⁱ	104.08 (3)
C14—C15—C16	119.7 (4)	C11—P5—C17	105.36 (15)
C14—C15—H15	120.2	C11—P5—C1	105.35 (15)
C16—C15—H15	120.2	C17—P5—C1	107.13 (15)
C15—C16—C11	121.1 (3)	C11—P5—Pd1	120.04 (10)
C15—C16—H16	119.5	C17—P5—Pd1	105.25 (10)
C11—C16—H16	119.5	C1—P5—Pd1	112.86 (11)
C18—C17—C22	118.0 (3)	P3—Pd1—P3 ⁱ	75.55 (3)

supplementary materials

C18—C17—P5	117.4 (2)	P3—Pd1—P5	104.26 (3)
C22—C17—P5	124.5 (3)	P3 ⁱ —Pd1—P5	179.37 (4)
C17—C18—C19	121.0 (3)	P3—Pd1—C11	167.50 (3)
C17—C18—H18	119.5	P3 ⁱ —Pd1—C11	93.37 (3)
C19—C18—H18	119.5	P5—Pd1—C11	86.87 (3)
P5—C1—C2—C7	85.3 (3)	C31—C32—C33—C34	1.2 (6)
P5—C1—C2—C3	-92.2 (3)	C32—C33—C34—C29	-1.1 (6)
C7—C2—C3—C4	-4.9 (5)	C30—C29—C34—C33	-0.3 (5)
C1—C2—C3—C4	172.6 (3)	P3—C29—C34—C33	-174.6 (3)
C7—C2—C3—C8	175.6 (3)	C24—C23—P3—C29	31.2 (3)
C1—C2—C3—C8	-6.9 (5)	C28—C23—P3—C29	-150.5 (2)
C2—C3—C4—C5	0.0 (5)	C24—C23—P3—Pd1	-98.5 (3)
C8—C3—C4—C5	179.5 (3)	C28—C23—P3—Pd1	79.7 (2)
C3—C4—C5—C6	4.3 (6)	C24—C23—P3—Pd1 ⁱ	145.2 (2)
C3—C4—C5—C9	-175.6 (4)	C28—C23—P3—Pd1 ⁱ	-36.6 (3)
C4—C5—C6—C7	-3.7 (6)	C34—C29—P3—C23	-149.4 (3)
C9—C5—C6—C7	176.2 (4)	C30—C29—P3—C23	36.5 (3)
C5—C6—C7—C2	-1.1 (5)	C34—C29—P3—Pd1	-20.9 (3)
C5—C6—C7—C10	178.6 (3)	C30—C29—P3—Pd1	165.0 (2)
C3—C2—C7—C6	5.5 (5)	C34—C29—P3—Pd1 ⁱ	92.6 (3)
C1—C2—C7—C6	-172.1 (3)	C30—C29—P3—Pd1 ⁱ	-81.5 (3)
C3—C2—C7—C10	-174.2 (3)	C16—C11—P5—C17	173.5 (2)
C1—C2—C7—C10	8.3 (5)	C12—C11—P5—C17	-5.0 (3)
C16—C11—C12—C13	-0.8 (5)	C16—C11—P5—C1	-73.4 (3)
P5—C11—C12—C13	177.6 (2)	C12—C11—P5—C1	108.1 (3)
C11—C12—C13—C14	-1.2 (5)	C16—C11—P5—Pd1	55.2 (3)
C12—C13—C14—C15	2.5 (5)	C12—C11—P5—Pd1	-123.2 (2)
C13—C14—C15—C16	-1.8 (5)	C18—C17—P5—C11	-83.7 (3)
C14—C15—C16—C11	-0.3 (5)	C22—C17—P5—C11	100.0 (3)
C12—C11—C16—C15	1.5 (4)	C18—C17—P5—C1	164.5 (3)
P5—C11—C16—C15	-177.0 (2)	C22—C17—P5—C1	-11.8 (3)
C22—C17—C18—C19	-0.6 (5)	C18—C17—P5—Pd1	44.1 (3)
P5—C17—C18—C19	-177.1 (3)	C22—C17—P5—Pd1	-132.2 (3)
C17—C18—C19—C20	0.9 (6)	C2—C1—P5—C11	40.5 (3)
C18—C19—C20—C21	-0.5 (6)	C2—C1—P5—C17	152.3 (2)
C19—C20—C21—C22	-0.2 (7)	C2—C1—P5—Pd1	-92.3 (2)
C20—C21—C22—C17	0.5 (7)	C23—P3—Pd1—P3 ⁱ	-126.54 (12)
C18—C17—C22—C21	-0.1 (6)	C29—P3—Pd1—P3 ⁱ	107.09 (11)
P5—C17—C22—C21	176.2 (3)	Pd1 ⁱ —P3—Pd1—P3 ⁱ	-6.53 (5)
C28—C23—C24—C25	0.0 (5)	C23—P3—Pd1—P5	54.08 (13)
P3—C23—C24—C25	178.3 (3)	C29—P3—Pd1—P5	-72.29 (12)
C23—C24—C25—C26	-0.4 (6)	Pd1 ⁱ —P3—Pd1—P5	174.09 (3)
C24—C25—C26—C27	0.5 (6)	C23—P3—Pd1—C11	-98.4 (2)
C25—C26—C27—C28	-0.1 (6)	C29—P3—Pd1—C11	135.22 (18)
C26—C27—C28—C23	-0.3 (6)	Pd1 ⁱ —P3—Pd1—C11	21.60 (18)
C24—C23—C28—C27	0.4 (5)	C11—P5—Pd1—P3	11.84 (14)

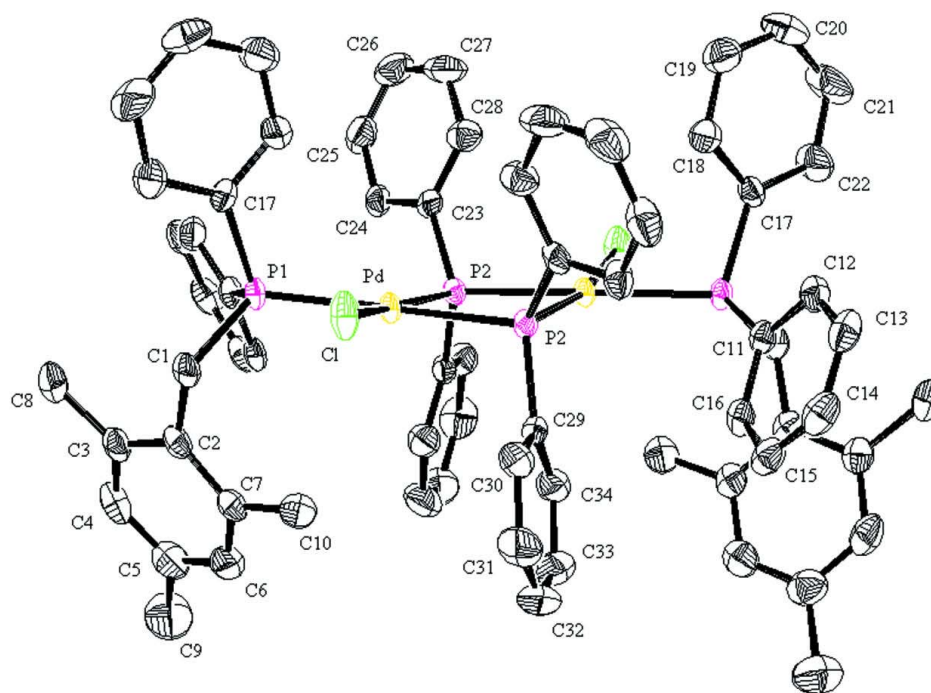
supplementary materials

P3—C23—C28—C27	-178.0 (3)	C17—P5—Pd1—P3	-106.51 (11)
C34—C29—C30—C31	1.5 (5)	C1—P5—Pd1—P3	136.98 (12)
P3—C29—C30—C31	175.6 (3)	C11—P5—Pd1—C11	-173.91 (14)
C29—C30—C31—C32	-1.4 (6)	C17—P5—Pd1—C11	67.74 (11)
C30—C31—C32—C33	0.0 (6)	C1—P5—Pd1—C11	-48.76 (12)

Symmetry codes: (i) $-x+3/2, -y+3/2, z$.

supplementary materials

Fig. 1



supplementary materials

Fig. 2

