Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

trans-Dibromidobis[diphenyl(*p*-tolyl)-phosphine]palladium(II)

Leo Kirsten,* Gideon Steyl and Andreas Roodt

Department of Chemistry, University of the Free State, PO Box 339, Bloemfontein 9300, South Africa
Correspondence e-mail: leokirsten@gmail.com

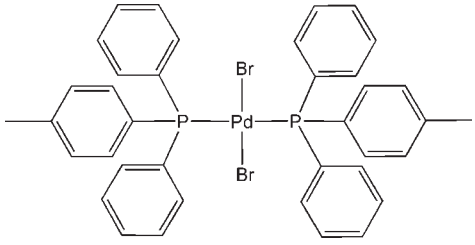
Received 22 October 2009; accepted 29 October 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.021; wR factor = 0.051; data-to-parameter ratio = 20.3.

In the title compound, $[PdBr_2(C_{19}H_{17}P)_2]$, the Pd^{II} ion resides on a centre of symmetry and is coordinated by two Br anions [Pd—Br = 2.4266 (2) Å] and two P-donor ligands [Pd—P = 2.3462 (5) Å] in a slightly distorted square-planar geometry [P—Pd—Br = 93.528 (12)°]. Weak intermolecular C—H···Br hydrogen bonds link molecules into chains extended in [1$\bar{1}$0].

Related literature

For the isostructural compound, *trans*-$[PdCl_2\{P((Ph)_2(p\text{-}Tol))\}_2]$, in which the Pd centers are coordinated by Cl anions instead of Br, see: Steyl *et al.* (2006).



Experimental

Crystal data

$[PdBr_2(C_{19}H_{17}P)_2]$
M_r = 818.81
Triclinic, $P\bar{1}$
a = 10.0321 (4) Å
b = 10.0521 (4) Å
c = 10.2967 (4) Å
α = 70.876 (2)°
β = 68.288 (2)°
γ = 60.312 (2)°
V = 824.54 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 3.11 mm^{-1}
T = 100 K
0.33 × 0.11 × 0.09 mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.427, T_{max} = 0.767
25915 measured reflections
3982 independent reflections
3621 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.051
S = 1.04
3982 reflections
196 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C33—H33···Br^{i}	0.93	2.88	3.7498 (19)	157
C22—H22···Br^{ii}	0.93	2.71	3.501 (2)	144

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *SHELXL97*.

Financial assistance from the University of the Free State is gratefully acknowledged. Part of this material is based on work supported by the South African National Research Foundation (NRF) under grant No. GUN 2068915.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2640).

References

Brandenburg, K. & Putz, H. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (1998). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *SAINT-Plus* (including *XPREP*). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Steyl, G., Kirsten, L. & Roodt, A. (2006). *Acta Cryst.* E**62**, m1705–m1707.

supporting information

Acta Cryst. (2009). E**65**, m1564 [doi:10.1107/S1600536809045401]

trans-Dibromidobis[diphenyl(*p*-tolyl)phosphine]palladium(II)

Leo Kirsten, Gideon Steyl and Andreas Roodt

S1. Comment

This study is aimed at expanding the knowledge of *trans* square-planar palladium complexes containing phosphine donor ligands and bromido or chlorido anions as coordinating atoms.

The title compound is centrosymmetric with a slightly distorted-square-planar geometry, as seen by the *cis* angle P—Pd—Br of 93.528 (12)°. The packing of the title compound is stabilized by two weak intermolecular C—H···Br hydrogen bonds (Table 1).

The corresponding chloro complex (Steyl *et al.*, 2006) is iso-structural to the title complex when comparing the geometrical parameters as well as the crystallization mode. The RMS error of 0.061 Å also indicate the iso-structurality of the two complexes (the title complex superimposed with the corresponding dichloro-palladium complex (Steyl *et al.*, 2006) including the Pd, Cl(Br), P and first C atoms of the phenyl rings).

S2. Experimental

The title complex was synthesized by the addition of 2.2 equivalents of diphenyl(*p*-tolyl)phosphine (16 mg, 0.059 mmol) to [Pd(COD)Br_2] (10 mg, 0.026 mmol) in 10 ml of dichloromethane while stirring for 5 minutes. Slow evaporation of the solvent resulted in orange crystals suitable for X-Ray diffraction (yield 71%, 16 mg).

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.95 or 0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = (1.2 or 1.5) times U_{eq} of the parent atom, respectively. The s.u.'s on all the Cell Axes and all the Cell Angles are equal as calculated from the unit cell determination.

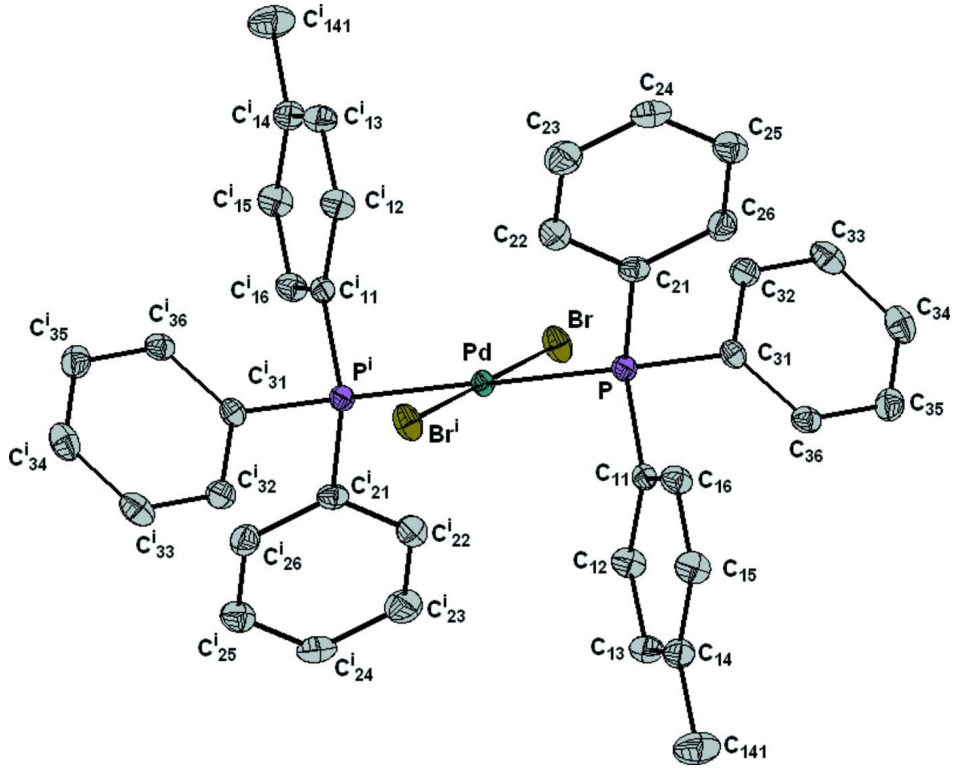


Figure 1

A sketch of the title compound showing the atomic numbering and 50% probability displacement ellipsoids [symmetry code: (i) 1 - *x*,1 - *y*,1 - *z*]. For the phenyl rings the first digit refers to ring number and the second digit to the atom in the ring. Hydrogen atoms have been omitted for clarity.

trans-Dibromidobis[diphenyl(*p*-tolyl)phosphine]palladium(II)

Crystal data

$[PdBr_2(C_{19}H_{17}P)_2]$
M_r = 818.81
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 10.0321 (4) Å
b = 10.0521 (4) Å
c = 10.2967 (4) Å
α = 70.876 (2)°
β = 68.288 (2)°
γ = 60.312 (2)°
V = 824.54 (6) Å^3
Z = 1
F(000) = 408
D_x = 1.649 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6740 reflections
θ = 2.4–28.3°
μ = 3.11 mm^{-1}
T = 100 K
Cuboid, orange
0.33 × 0.11 × 0.09 mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 512 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.427, T_{max} = 0.767
25915 measured reflections
3982 independent reflections
3621 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

θ_{max} = 28.0°, θ_{min} = 2.4°
h = −13→13
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.051
S = 1.04
3982 reflections
196 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: riding model
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 0.7898P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 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}
Pd	0.5000	0.5000	0.5000	0.01341 (5)
Br	0.39230 (2)	0.78196 (2)	0.45123 (2)	0.02035 (6)
P	0.28802 (5)	0.49477 (5)	0.69762 (5)	0.01335 (9)
C11	0.3682 (2)	0.3708 (2)	0.84911 (18)	0.0139 (3)
C12	0.4892 (2)	0.3878 (2)	0.8690 (2)	0.0189 (4)
H12	0.5284	0.4573	0.8035	0.023*
C13	0.5507 (2)	0.3014 (2)	0.9859 (2)	0.0195 (4)
H13	0.6301	0.3149	0.9986	0.023*
C14	0.4963 (2)	0.1951 (2)	1.0847 (2)	0.0195 (4)
C15	0.3776 (2)	0.1772 (2)	1.0628 (2)	0.0210 (4)
H15	0.3408	0.1053	1.1267	0.025*
C16	0.3131 (2)	0.2648 (2)	0.9470 (2)	0.0178 (4)
H16	0.2327	0.2523	0.9351	0.021*
C141	0.5660 (3)	0.1007 (3)	1.2101 (2)	0.0332 (5)
H14A	0.5145	0.0339	1.2670	0.050*
H14B	0.5504	0.1693	1.2662	0.050*
H14C	0.6774	0.0386	1.1772	0.050*
C21	0.1532 (2)	0.4290 (2)	0.68471 (19)	0.0160 (3)
C22	0.2059 (2)	0.3178 (2)	0.6032 (2)	0.0235 (4)
H22	0.3122	0.2743	0.5549	0.028*
C23	0.1014 (3)	0.2714 (3)	0.5934 (2)	0.0281 (5)
H23	0.1386	0.1952	0.5404	0.034*
C24	−0.0579 (2)	0.3376 (2)	0.6621 (2)	0.0229 (4)

H24	−0.1280	0.3071	0.6543	0.027*
C25	−0.1120 (2)	0.4490 (2)	0.7422 (2)	0.0225 (4)
H25	−0.2192	0.4945	0.7878	0.027*
C26	−0.0077 (2)	0.4938 (2)	0.7553 (2)	0.0202 (4)
H26	−0.0449	0.5673	0.8114	0.024*
C31	0.1493 (2)	0.6787 (2)	0.75882 (19)	0.0144 (3)
C32	0.0568 (2)	0.8003 (2)	0.6700 (2)	0.0181 (4)
H32	0.0697	0.7872	0.5801	0.022*
C33	−0.0535 (2)	0.9396 (2)	0.7151 (2)	0.0218 (4)
H33	−0.1149	1.0196	0.6558	0.026*
C34	−0.0728 (2)	0.9604 (2)	0.8489 (2)	0.0224 (4)
H34	−0.1464	1.0545	0.8788	0.027*
C35	0.0175 (2)	0.8410 (2)	0.9375 (2)	0.0202 (4)
H35	0.0041	0.8547	1.0274	0.024*
C36	0.1286 (2)	0.7006 (2)	0.89275 (19)	0.0160 (3)
H36	0.1892	0.6208	0.9527	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd	0.01211 (9)	0.01332 (9)	0.01158 (9)	−0.00500 (7)	−0.00154 (7)	−0.00073 (7)
Br	0.01631 (9)	0.01435 (9)	0.02261 (10)	−0.00509 (7)	−0.00119 (7)	−0.00071 (7)
P	0.0124 (2)	0.0147 (2)	0.0114 (2)	−0.00587 (17)	−0.00238 (16)	−0.00109 (16)
C11	0.0122 (8)	0.0143 (8)	0.0127 (8)	−0.0038 (7)	−0.0030 (6)	−0.0027 (6)
C12	0.0188 (9)	0.0186 (9)	0.0207 (9)	−0.0112 (7)	−0.0061 (7)	0.0014 (7)
C13	0.0189 (9)	0.0206 (9)	0.0229 (10)	−0.0094 (8)	−0.0088 (7)	−0.0030 (8)
C14	0.0199 (9)	0.0182 (9)	0.0178 (9)	−0.0057 (7)	−0.0071 (7)	−0.0016 (7)
C15	0.0210 (9)	0.0222 (10)	0.0191 (9)	−0.0130 (8)	−0.0051 (7)	0.0035 (7)
C16	0.0158 (8)	0.0197 (9)	0.0183 (9)	−0.0096 (7)	−0.0046 (7)	−0.0003 (7)
C141	0.0392 (13)	0.0362 (13)	0.0271 (11)	−0.0181 (11)	−0.0190 (10)	0.0065 (10)
C21	0.0177 (9)	0.0173 (9)	0.0138 (8)	−0.0090 (7)	−0.0068 (7)	0.0021 (7)
C22	0.0188 (9)	0.0258 (10)	0.0261 (10)	−0.0065 (8)	−0.0061 (8)	−0.0095 (8)
C23	0.0297 (11)	0.0280 (11)	0.0338 (12)	−0.0112 (9)	−0.0117 (9)	−0.0119 (9)
C24	0.0278 (10)	0.0268 (10)	0.0216 (10)	−0.0166 (9)	−0.0132 (8)	0.0028 (8)
C25	0.0198 (9)	0.0285 (10)	0.0198 (10)	−0.0140 (8)	−0.0048 (8)	0.0008 (8)
C26	0.0202 (9)	0.0249 (10)	0.0169 (9)	−0.0113 (8)	−0.0024 (7)	−0.0053 (8)
C31	0.0111 (8)	0.0160 (8)	0.0155 (8)	−0.0073 (7)	−0.0009 (6)	−0.0024 (7)
C32	0.0161 (8)	0.0214 (9)	0.0151 (9)	−0.0082 (7)	−0.0039 (7)	−0.0007 (7)
C33	0.0158 (9)	0.0179 (9)	0.0265 (10)	−0.0063 (7)	−0.0061 (8)	0.0015 (8)
C34	0.0168 (9)	0.0174 (9)	0.0306 (11)	−0.0067 (8)	−0.0014 (8)	−0.0079 (8)
C35	0.0194 (9)	0.0238 (10)	0.0213 (9)	−0.0115 (8)	−0.0015 (7)	−0.0087 (8)
C36	0.0142 (8)	0.0187 (9)	0.0169 (9)	−0.0095 (7)	−0.0041 (7)	−0.0012 (7)

Geometric parameters (Å, º)

Pd—P	2.3462 (5)	C21—C26	1.399 (3)
Pd—P^{i}	2.3462 (5)	C22—C23	1.387 (3)
Pd—Br^{i}	2.4266 (2)	C22—H22	0.9300

Pd—Br	2.4266 (2)	C23—C24	1.384 (3)
P—C11	1.8150 (18)	C23—H23	0.9300
P—C31	1.8217 (18)	C24—C25	1.379 (3)
P—C21	1.8331 (19)	C24—H24	0.9300
C11—C16	1.387 (3)	C25—C26	1.388 (3)
C11—C12	1.399 (2)	C25—H25	0.9300
C12—C13	1.385 (3)	C26—H26	0.9300
C12—H12	0.9300	C31—C36	1.391 (3)
C13—C14	1.389 (3)	C31—C32	1.401 (3)
C13—H13	0.9300	C32—C33	1.383 (3)
C14—C15	1.392 (3)	C32—H32	0.9300
C14—C141	1.505 (3)	C33—C34	1.389 (3)
C15—C16	1.391 (3)	C33—H33	0.9300
C15—H15	0.9300	C34—C35	1.383 (3)
C16—H16	0.9300	C34—H34	0.9300
C141—H14A	0.9600	C35—C36	1.391 (3)
C141—H14B	0.9600	C35—H35	0.9300
C141—H14C	0.9600	C36—H36	0.9300
C21—C22	1.390 (3)		

P—Pd—P^{i}	180.0	C22—C21—C26	118.53 (17)
P—Pd—Br^{i}	86.472 (12)	C22—C21—P	121.58 (14)
P^{i}—Pd—Br^{i}	93.528 (12)	C26—C21—P	119.87 (14)
P—Pd—Br	93.528 (12)	C23—C22—C21	120.60 (19)
P^{i}—Pd—Br	86.472 (12)	C23—C22—H22	119.7
Br^{i}—Pd—Br	180.0	C21—C22—H22	119.7
C11—P—C31	103.51 (8)	C24—C23—C22	120.5 (2)
C11—P—C21	107.56 (8)	C24—C23—H23	119.8
C31—P—C21	101.59 (8)	C22—C23—H23	119.8
C11—P—Pd	108.36 (6)	C25—C24—C23	119.51 (19)
C31—P—Pd	116.57 (6)	C25—C24—H24	120.2
C21—P—Pd	118.01 (6)	C23—C24—H24	120.2
C16—C11—C12	118.91 (16)	C24—C25—C26	120.46 (19)
C16—C11—P	123.69 (14)	C24—C25—H25	119.8
C12—C11—P	117.36 (14)	C26—C25—H25	119.8
C13—C12—C11	120.18 (17)	C25—C26—C21	120.41 (18)
C13—C12—H12	119.9	C25—C26—H26	119.8
C11—C12—H12	119.9	C21—C26—H26	119.8
C12—C13—C14	121.38 (17)	C36—C31—C32	118.96 (17)
C12—C13—H13	119.3	C36—C31—P	122.06 (14)
C14—C13—H13	119.3	C32—C31—P	118.94 (14)
C13—C14—C15	118.01 (17)	C33—C32—C31	120.42 (18)
C13—C14—C141	120.66 (18)	C33—C32—H32	119.8
C15—C14—C141	121.32 (18)	C31—C32—H32	119.8
C16—C15—C14	121.24 (18)	C32—C33—C34	120.13 (18)
C16—C15—H15	119.4	C32—C33—H33	119.9
C14—C15—H15	119.4	C34—C33—H33	119.9
C11—C16—C15	120.26 (17)	C35—C34—C33	119.91 (18)

C11—C16—H16	119.9	C35—C34—H34	120.0
C15—C16—H16	119.9	C33—C34—H34	120.0
C14—C141—H14A	109.5	C34—C35—C36	120.19 (18)
C14—C141—H14B	109.5	C34—C35—H35	119.9
H14A—C141—H14B	109.5	C36—C35—H35	119.9
C14—C141—H14C	109.5	C31—C36—C35	120.38 (17)
H14A—C141—H14C	109.5	C31—C36—H36	119.8
H14B—C141—H14C	109.5	C35—C36—H36	119.8
Br^{i}—Pd—P—C11	54.51 (6)	C11—P—C21—C26	90.47 (16)
Br—Pd—P—C11	−125.49 (6)	C31—P—C21—C26	−17.89 (17)
Br^{i}—Pd—P—C31	170.70 (7)	Pd—P—C21—C26	−146.67 (13)
Br—Pd—P—C31	−9.30 (7)	C26—C21—C22—C23	−0.6 (3)
Br^{i}—Pd—P—C21	−67.94 (7)	P—C21—C22—C23	−179.11 (16)
Br—Pd—P—C21	112.06 (7)	C21—C22—C23—C24	1.5 (3)
C31—P—C11—C16	97.53 (16)	C22—C23—C24—C25	−0.9 (3)
C21—P—C11—C16	−9.49 (18)	C23—C24—C25—C26	−0.6 (3)
Pd—P—C11—C16	−138.11 (14)	C24—C25—C26—C21	1.5 (3)
C31—P—C11—C12	−80.59 (15)	C22—C21—C26—C25	−0.9 (3)
C21—P—C11—C12	172.39 (14)	P—C21—C26—C25	177.67 (15)
Pd—P—C11—C12	43.78 (15)	C11—P—C31—C36	1.98 (17)
C16—C11—C12—C13	−1.0 (3)	C21—P—C31—C36	113.45 (15)
P—C11—C12—C13	177.24 (15)	Pd—P—C31—C36	−116.86 (14)
C11—C12—C13—C14	0.9 (3)	C11—P—C31—C32	−175.85 (14)
C12—C13—C14—C15	0.1 (3)	C21—P—C31—C32	−64.39 (16)
C12—C13—C14—C141	179.2 (2)	Pd—P—C31—C32	65.30 (15)
C13—C14—C15—C16	−1.1 (3)	C36—C31—C32—C33	−0.1 (3)
C141—C14—C15—C16	179.83 (19)	P—C31—C32—C33	177.80 (14)
C12—C11—C16—C15	0.0 (3)	C31—C32—C33—C34	0.4 (3)
P—C11—C16—C15	−178.07 (15)	C32—C33—C34—C35	−0.6 (3)
C14—C15—C16—C11	1.0 (3)	C33—C34—C35—C36	0.5 (3)
C11—P—C21—C22	−91.04 (17)	C32—C31—C36—C35	0.0 (3)
C31—P—C21—C22	160.60 (16)	P—C31—C36—C35	−177.80 (14)
Pd—P—C21—C22	31.82 (18)	C34—C35—C36—C31	−0.2 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32···Br	0.93	2.99	3.2380 (18)	97
C33—H33···Br^{ii}	0.93	2.88	3.7498 (19)	157
C22—H22···Br^{i}	0.93	2.71	3.501 (2)	144

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1.