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(Diphenylmethoxyphosphane-κP)(diphenylphosphanito-κP)(3,5,7-tribromotropolonato-$\kappa^2 O,O'$)palladium(II) methanol solvate

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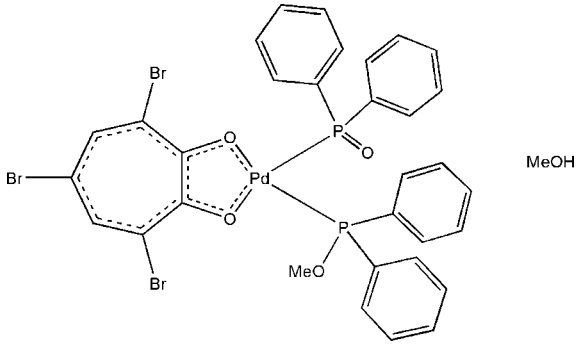
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 18.9.

The title compound, $[Pd(C_7H_2Br_3)(C_{12}H_{10}OP)(C_{13}H_{13}OP)]\cdot CH_3OH$, the by-product of an attempted Suzuki-coupling reaction, has two different diphenylphosphane groups bonded to Pd and is the first structural example of a metal complex having a diphenylphosphanite anion [–P(O)Ph_2] and a diphenylmethoxyphosphane molecule (MeOPPh_2) coordinated to the same metal centre. Molecules are linked by hydrogen bonds, Br···Br interactions [3.729 (1) Å] and π–π stacking interactions parallel to the a axis [interplanar distance 3.496 (8) Å and centroid-to-centroid distance 3.507 (1) Å].

Related literature

Common examples of the class of Pd—P-bonded compounds represented by the title compound have phosphinate and phosphane hydroxide ligands with a hydrogen-bond interaction between the oxo and hydroxo groups; see: Gebauer *et al.* (1992, 1995); Pryjomska *et al.* (2006).



Experimental

Crystal data

$[Pd(C_7H_2Br_3)(C_{12}H_{10}OP)(C_{13}H_{13}OP)]\cdot CH_4O$
M_r = 913.63
Triclinic, $P\overline{1}$
a = 9.125 (3) Å
b = 11.326 (4) Å
c = 17.137 (3) Å
α = 87.652 (8)°
β = 78.285 (7)°
γ = 72.121 (8)°
V = 1650.0 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.33 mm^{-1}
T = 100 (2) K
0.19 × 0.14 × 0.04 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.493, T_{max} = 0.846
33856 measured reflections
7543 independent reflections
6131 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.09
7543 reflections
399 parameters
13 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.92 e Å^{-3}
$\Delta\rho_{min}$ = −0.91 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd—O1	2.076 (3)	Pd—P2	2.2055 (12)
Pd—O2	2.140 (3)	Pd—P1	2.2267 (12)
O1—Pd—O2	75.61 (11)	O1—Pd—P1	92.64 (9)
O1—Pd—P2	178.23 (9)	O2—Pd—P1	168.03 (9)
O2—Pd—P2	102.66 (9)	P2—Pd—P1	89.07 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···Br3	0.95	2.95	3.818 (5)	152
O5—H5···O3	0.84	2.01	2.740 (7)	144
C20—H20B···O2	0.98	2.30	3.143 (6)	144
C12—H12···O5^{i}	0.95	2.56	3.435 (9)	154
C31—H31···Br1^{ii}	0.95	2.99	3.920 (6)	168

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

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *SHELXL97*.

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2068915. Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the NRF.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2311).

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supplementary materials

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(Diphenylmethoxyphosphane-κP)(diphenylphosphanito-κP)(3,5,7-tribromotropolonato-$\kappa^2 O,O'$)palladium(II) methanol solvate

G. Steyl

Comment

In the current paper the title compound, (I), is presented as an example of a by-product formed during the catalytic cycle. It should be noted that both a diphenylphosphanito and a diphenylmethoxyphosphane, Fig. 1, was formed from the initial starting diphenylnaphtolphosphane. In either case, the diphenylnaphtolphosphane moiety decomposed and in the one instance a Suzuki coupling occurred to form the diphenylmethoxyphosphane derivative. This product is in contrast to the more readily observed hydroxyl derivative.

The Pd—O and Pd—P bond distances only differ slightly, Table 1. This relative similarity in bond distances (Pd—P) might be due to the hydrogen bonding observed between the methanol solvate and the diphenylphosphaneoxide oxygen atom, Table 2. The P—O(Me) and P═O bond distances are significantly different indicating the bond order in which the oxygen atoms bond with the phosphorous atom.

Weak intra- and intermolecular interactions is observed in the solid state, see Table 2. The role of the bromo-atoms on the solid state arrangement can be observed from the intermolecular distances between $Br_3\cdots Br_7$ [$1-x$, $1-y$, $1-z$] and $Br_5\cdots Pd$ [$1-x$, $1-y$, $1-z$] in the order of 3.729 (1) and 3.717 (1) Å, respectively. This interaction is further enhanced through π–π stacking of the cycloheptatriene rings systems of the bromo moieties with an interplanar distance of 3.496 (8) Å and a centroid-to-centroid distance of 3.507 (1) Å. The ordering of the solid state can be observed as stacking along the *a* axis, see Figure 2.

Experimental

The title compound was obtained unintentionally as the product of a Suzuki coupling reaction of diphenylnaphtolphosphane and bis(3,5,7-tribromotropolonato)palladium(II) in methanol (10 ml) solution. On evaporation of the solvent; crystals suitable for X-Ray crystallography was obtained.

Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 and 0.98 Å and with U_{iso}(H) = 1.2 times U_{eq}(C aromatic) and U_{iso}(H) = 1.2 times U_{eq}(C methyl). The final difference Fourier map had a large peak near Pd.

Figures

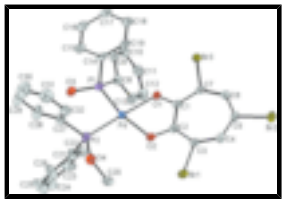

Fig. 1. Representation of the title compound (I), showing the numbering scheme and displacement ellipsoids (50% probability). For the carbon rings, first digit refers to ring number, second digit to atom in the ring. Hydrogen atoms omitted for clarity.

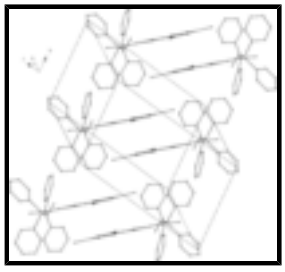

Fig. 2. Fraction of the unit cell showing the stacking pattern [symmetry codes: (*a*) *x*, −*y*, *z* − 1/2; (*b*) −*x*, −*y*, −*z*].

(Diphenylmethoxyphosphane-κ*P*)(diphenylphosphanito-κ*P*)(3,5,7- tribromotropolonato-κ^2*O*,*O*')palladium(II) methanol solvate

Crystal data

$[Pd(C_7H_2Br_3)(C_{12}H_{10}OP)(C_{13}H_{13}OP)]\cdot CH_4O$	$Z = 2$
$M_r = 913.63$	$F_{000} = 896$
Triclinic, $P\overline{1}$	$D_x = 1.839$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.125$ (3) Å	Cell parameters from 5022 reflections
$b = 11.326$ (4) Å	$\theta = 2.4$–27.7º
$c = 17.137$ (3) Å	$\mu = 4.33$ mm^{-1}
$\alpha = 87.652$ (8)º	$T = 100$ (2) K
$\beta = 78.285$ (7)º	Plate, yellow
$\gamma = 72.121$ (8)º	0.19 × 0.14 × 0.04 mm
$V = 1650.0$ (8) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	7543 independent reflections
Radiation source: fine-focus sealed tube	6131 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.041$
Detector resolution: 512 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 100(2)$ K	$\theta_{min} = 1.2$º
φ and ω scans	$h = -11\rightarrow11$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$k = -14\rightarrow14$
$T_{min} = 0.493$, $T_{max} = 0.846$	$l = -22\rightarrow22$
33856 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 2.8917P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
7543 reflections	$\Delta\rho_{max} = 1.92$ e Å^{-3}
399 parameters	$\Delta\rho_{min} = -0.91$ e Å^{-3}
13 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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. The solvent molecule in the asymmetric unit were were identified from the difference Fourier map. Refinement of the MeOH moiety was done with the *DFIX* command to place the C and O atoms at an idealized distance from each other and the ISOR command was used to attempt to restrain the C atom within a certain range.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd	0.79443 (4)	0.29918 (3)	0.25916 (2)	0.01933 (9)
Br1	0.35292 (5)	0.69319 (4)	0.31393 (3)	0.02862 (12)
Br2	0.05373 (5)	0.55669 (4)	0.59648 (3)	0.02661 (12)
Br3	0.57021 (5)	0.15579 (4)	0.51586 (3)	0.02441 (11)
P1	0.97836 (13)	0.11498 (10)	0.24798 (7)	0.0202 (2)
P2	0.92127 (13)	0.35250 (10)	0.14639 (7)	0.0224 (2)
O1	0.6711 (3)	0.2547 (3)	0.36606 (19)	0.0240 (6)
O2	0.6024 (3)	0.4642 (3)	0.29453 (18)	0.0236 (6)
O3	1.1000 (3)	0.0763 (3)	0.17293 (19)	0.0255 (7)
O4	0.8531 (4)	0.4997 (3)	0.1320 (2)	0.0294 (7)
O5	1.3757 (8)	−0.1108 (7)	0.1698 (5)	0.109 (2)
H5	1.2994	−0.0506	0.1907	0.164*
C1	0.5507 (5)	0.3391 (4)	0.4014 (3)	0.0205 (9)
C2	0.5083 (5)	0.4569 (4)	0.3591 (3)	0.0198 (8)

C3	0.3716 (5)	0.5589 (3)	0.3854 (3)	0.0199 (8)
C4	0.2530 (5)	0.5765 (4)	0.4518 (3)	0.0224 (9)
H4	0.1712	0.6531	0.4550	0.027*
C5	0.2366 (5)	0.4976 (4)	0.5147 (3)	0.0216 (9)
C6	0.3344 (5)	0.3816 (4)	0.5269 (3)	0.0220 (9)
H6	0.3041	0.3436	0.5753	0.026*
C7	0.4715 (5)	0.3140 (4)	0.4769 (3)	0.0196 (8)
C8	0.8745 (5)	−0.0001 (4)	0.2719 (3)	0.0228 (9)
C9	0.9531 (5)	−0.1131 (4)	0.2999 (3)	0.0257 (9)
H9	1.0514	−0.1237	0.3145	0.031*
C10	0.8907 (6)	−0.2109 (4)	0.3071 (3)	0.0292 (10)
H10	0.9466	−0.2885	0.3258	0.035*
C11	0.7459 (6)	−0.1955 (5)	0.2870 (3)	0.0329 (11)
H11	0.7038	−0.2630	0.2905	0.039*
C12	0.6649 (6)	−0.0832 (5)	0.2620 (3)	0.0367 (12)
H12	0.5641	−0.0717	0.2502	0.044*
C13	0.7280 (5)	0.0145 (5)	0.2537 (3)	0.0321 (11)
H13	0.6709	0.0920	0.2354	0.038*
C14	1.0716 (5)	0.1175 (4)	0.3310 (3)	0.0222 (9)
C15	1.2223 (5)	0.1309 (4)	0.3165 (3)	0.0254 (9)
H15	1.2759	0.1347	0.2632	0.030*
C16	1.2936 (5)	0.1386 (4)	0.3793 (3)	0.0286 (10)
H16	1.3966	0.1461	0.3691	0.034*
C17	1.2151 (5)	0.1353 (4)	0.4570 (3)	0.0261 (10)
H17	1.2636	0.1415	0.5001	0.031*
C18	1.0648 (5)	0.1230 (4)	0.4721 (3)	0.0256 (9)
H18	1.0110	0.1205	0.5255	0.031*
C19	0.9936 (5)	0.1143 (4)	0.4091 (3)	0.0228 (9)
H19	0.8909	0.1061	0.4196	0.027*
C20	0.6949 (6)	0.5537 (5)	0.1206 (3)	0.0340 (11)
H20A	0.6740	0.6432	0.1132	0.051*
H20B	0.6214	0.5392	0.1675	0.051*
H20C	0.6811	0.5157	0.0734	0.051*
C21	0.9153 (5)	0.2799 (4)	0.0560 (3)	0.0267 (10)
C22	0.8548 (6)	0.1815 (4)	0.0572 (3)	0.0305 (10)
H22	0.8160	0.1496	0.1065	0.037*
C23	0.8513 (7)	0.1303 (5)	−0.0138 (3)	0.0376 (12)
H23	0.8115	0.0620	−0.0133	0.045*
C24	0.9044 (8)	0.1771 (5)	−0.0842 (3)	0.0484 (15)
H24	0.8996	0.1421	−0.1327	0.058*
C25	0.9657 (8)	0.2754 (5)	−0.0864 (3)	0.0516 (16)
H25	1.0032	0.3074	−0.1360	0.062*
C26	0.9716 (7)	0.3266 (5)	−0.0155 (3)	0.0404 (13)
H26	1.0142	0.3934	−0.0161	0.048*
C27	1.1224 (5)	0.3462 (4)	0.1445 (3)	0.0290 (10)
C28	1.2481 (6)	0.2606 (5)	0.0974 (4)	0.0435 (14)
H28	1.2306	0.2047	0.0631	0.052*
C29	1.4008 (6)	0.2571 (6)	0.1008 (4)	0.0552 (17)
H29	1.4879	0.1987	0.0686	0.066*

C30	1.4252 (7)	0.3370 (6)	0.1501 (4)	0.0560 (17)
H30	1.5297	0.3339	0.1518	0.067*
C31	1.3020 (7)	0.4221 (6)	0.1976 (4)	0.0453 (14)
H31	1.3212	0.4774	0.2317	0.054*
C32	1.1494 (6)	0.4265 (5)	0.1954 (3)	0.0328 (11)
H32	1.0633	0.4843	0.2286	0.039*
C33	1.460 (3)	−0.071 (2)	0.0948 (10)	0.289 (12)
H33A	1.4047	0.0150	0.0844	0.434*
H33B	1.4650	−0.1249	0.0504	0.434*
H33C	1.5675	−0.0775	0.1002	0.434*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd	0.01660 (16)	0.01855 (16)	0.02193 (18)	−0.00428 (12)	−0.00401 (13)	0.00306 (12)
Br1	0.0284 (2)	0.0192 (2)	0.0348 (3)	−0.00220 (17)	−0.0076 (2)	0.00673 (18)
Br2	0.0233 (2)	0.0228 (2)	0.0304 (3)	−0.00586 (17)	0.00139 (18)	−0.00407 (17)
Br3	0.0233 (2)	0.0216 (2)	0.0265 (2)	−0.00448 (16)	−0.00538 (18)	0.00626 (17)
P1	0.0175 (5)	0.0191 (5)	0.0250 (6)	−0.0055 (4)	−0.0072 (4)	0.0017 (4)
P2	0.0193 (5)	0.0220 (5)	0.0258 (6)	−0.0083 (4)	−0.0019 (5)	0.0016 (4)
O1	0.0190 (15)	0.0222 (15)	0.0245 (16)	−0.0007 (12)	0.0000 (12)	0.0047 (12)
O2	0.0202 (15)	0.0210 (14)	0.0268 (17)	−0.0029 (12)	−0.0046 (13)	0.0053 (12)
O3	0.0226 (16)	0.0240 (15)	0.0305 (18)	−0.0069 (12)	−0.0067 (13)	−0.0017 (13)
O4	0.0295 (17)	0.0222 (15)	0.0352 (19)	−0.0089 (13)	−0.0025 (15)	0.0026 (13)
O5	0.082 (4)	0.099 (5)	0.135 (6)	−0.005 (3)	−0.031 (4)	0.008 (4)
C1	0.019 (2)	0.0187 (19)	0.026 (2)	−0.0058 (16)	−0.0092 (17)	0.0024 (16)
C2	0.018 (2)	0.021 (2)	0.024 (2)	−0.0087 (16)	−0.0070 (17)	0.0011 (16)
C3	0.022 (2)	0.0127 (18)	0.027 (2)	−0.0057 (15)	−0.0083 (18)	0.0041 (16)
C4	0.019 (2)	0.019 (2)	0.029 (2)	−0.0058 (16)	−0.0054 (18)	−0.0037 (17)
C5	0.017 (2)	0.024 (2)	0.025 (2)	−0.0079 (16)	−0.0027 (17)	−0.0052 (17)
C6	0.024 (2)	0.023 (2)	0.021 (2)	−0.0102 (17)	−0.0062 (18)	0.0015 (17)
C7	0.019 (2)	0.0162 (18)	0.025 (2)	−0.0045 (15)	−0.0087 (17)	0.0042 (16)
C8	0.022 (2)	0.022 (2)	0.025 (2)	−0.0072 (17)	−0.0034 (18)	−0.0029 (17)
C9	0.022 (2)	0.024 (2)	0.030 (3)	−0.0060 (17)	−0.0029 (19)	−0.0011 (18)
C10	0.034 (3)	0.024 (2)	0.030 (3)	−0.0102 (19)	−0.006 (2)	0.0024 (19)
C11	0.034 (3)	0.040 (3)	0.031 (3)	−0.023 (2)	−0.003 (2)	0.001 (2)
C12	0.026 (2)	0.046 (3)	0.043 (3)	−0.019 (2)	−0.007 (2)	0.005 (2)
C13	0.024 (2)	0.033 (2)	0.041 (3)	−0.0101 (19)	−0.007 (2)	0.004 (2)
C14	0.020 (2)	0.0159 (18)	0.031 (2)	−0.0030 (15)	−0.0105 (18)	0.0004 (17)
C15	0.021 (2)	0.024 (2)	0.031 (3)	−0.0063 (17)	−0.0060 (19)	−0.0037 (18)
C16	0.022 (2)	0.030 (2)	0.037 (3)	−0.0094 (18)	−0.012 (2)	−0.001 (2)
C17	0.027 (2)	0.021 (2)	0.035 (3)	−0.0075 (17)	−0.017 (2)	0.0042 (18)
C18	0.031 (2)	0.0168 (19)	0.031 (3)	−0.0073 (17)	−0.012 (2)	0.0045 (17)
C19	0.020 (2)	0.0189 (19)	0.030 (2)	−0.0054 (16)	−0.0083 (18)	0.0054 (17)
C20	0.032 (3)	0.031 (2)	0.036 (3)	−0.006 (2)	−0.008 (2)	0.013 (2)
C21	0.030 (2)	0.026 (2)	0.024 (2)	−0.0110 (18)	0.0017 (19)	−0.0039 (18)
C22	0.035 (3)	0.032 (2)	0.031 (3)	−0.017 (2)	−0.012 (2)	0.004 (2)
C23	0.048 (3)	0.035 (3)	0.038 (3)	−0.017 (2)	−0.020 (3)	0.002 (2)

C24	0.080 (5)	0.041 (3)	0.028 (3)	−0.024 (3)	−0.012 (3)	−0.008 (2)
C25	0.087 (5)	0.044 (3)	0.025 (3)	−0.029 (3)	0.000 (3)	0.001 (2)
C26	0.062 (4)	0.034 (3)	0.027 (3)	−0.024 (3)	0.003 (2)	−0.004 (2)
C27	0.025 (2)	0.029 (2)	0.035 (3)	−0.0136 (19)	−0.002 (2)	0.003 (2)
C28	0.028 (3)	0.042 (3)	0.059 (4)	−0.014 (2)	0.004 (3)	−0.012 (3)
C29	0.022 (3)	0.056 (4)	0.081 (5)	−0.010 (3)	0.003 (3)	−0.012 (3)
C30	0.039 (3)	0.049 (4)	0.088 (5)	−0.028 (3)	−0.010 (3)	0.003 (3)
C31	0.046 (3)	0.055 (3)	0.050 (4)	−0.030 (3)	−0.020 (3)	0.002 (3)
C32	0.031 (3)	0.041 (3)	0.029 (3)	−0.015 (2)	−0.007 (2)	0.003 (2)
C33	0.295 (15)	0.303 (15)	0.274 (15)	−0.101 (10)	−0.053 (9)	0.001 (9)

Geometric parameters (Å, °)

Pd—O1	2.076 (3)	C14—C15	1.401 (6)
Pd—O2	2.140 (3)	C15—C16	1.386 (6)
Pd—P2	2.2055 (12)	C15—H15	0.9500
Pd—P1	2.2267 (12)	C16—C17	1.383 (7)
Br1—C3	1.902 (4)	C16—H16	0.9500
Br2—C5	1.907 (4)	C17—C18	1.392 (6)
Br3—C7	1.904 (4)	C17—H17	0.9500
P1—O3	1.499 (3)	C18—C19	1.389 (6)
P1—C14	1.806 (4)	C18—H18	0.9500
P1—C8	1.823 (4)	C19—H19	0.9500
P2—O4	1.618 (3)	C20—H20A	0.9800
P2—C21	1.801 (5)	C20—H20B	0.9800
P2—C27	1.809 (5)	C20—H20C	0.9800
O1—C1	1.273 (5)	C21—C26	1.380 (7)
O2—C2	1.273 (5)	C21—C22	1.384 (6)
O4—C20	1.435 (6)	C22—C23	1.378 (7)
O5—C33	1.488 (9)	C22—H22	0.9500
O5—H5	0.8400	C23—C24	1.357 (8)
C1—C7	1.412 (6)	C23—H23	0.9500
C1—C2	1.475 (6)	C24—C25	1.387 (8)
C2—C3	1.422 (6)	C24—H24	0.9500
C3—C4	1.376 (6)	C25—C26	1.385 (8)
C4—C5	1.385 (6)	C25—H25	0.9500
C4—H4	0.9500	C26—H26	0.9500
C5—C6	1.378 (6)	C27—C28	1.383 (7)
C6—C7	1.384 (6)	C27—C32	1.393 (7)
C6—H6	0.9500	C28—C29	1.395 (8)
C8—C9	1.381 (6)	C28—H28	0.9500
C8—C13	1.394 (6)	C29—C30	1.358 (9)
C9—C10	1.384 (6)	C29—H29	0.9500
C9—H9	0.9500	C30—C31	1.372 (9)
C10—C11	1.391 (7)	C30—H30	0.9500
C10—H10	0.9500	C31—C32	1.386 (7)
C11—C12	1.362 (7)	C31—H31	0.9500
C11—H11	0.9500	C32—H32	0.9500
C12—C13	1.386 (7)	C33—H33A	0.9800

C12—H12	0.9500	C33—H33B	0.9800
C13—H13	0.9500	C33—H33C	0.9800
C14—C19	1.387 (6)		
O1—Pd—O2	75.61 (11)	C16—C15—C14	120.3 (4)
O1—Pd—P2	178.23 (9)	C16—C15—H15	119.8
O2—Pd—P2	102.66 (9)	C14—C15—H15	119.8
O1—Pd—P1	92.64 (9)	C17—C16—C15	120.1 (4)
O2—Pd—P1	168.03 (9)	C17—C16—H16	120.0
P2—Pd—P1	89.07 (5)	C15—C16—H16	120.0
O3—P1—C14	110.12 (19)	C16—C17—C18	120.0 (4)
O3—P1—C8	109.78 (19)	C16—C17—H17	120.0
C14—P1—C8	105.7 (2)	C18—C17—H17	120.0
O3—P1—Pd	120.99 (13)	C19—C18—C17	120.0 (4)
C14—P1—Pd	102.86 (14)	C19—C18—H18	120.0
C8—P1—Pd	106.28 (15)	C17—C18—H18	120.0
O4—P2—C21	104.4 (2)	C14—C19—C18	120.4 (4)
O4—P2—C27	97.64 (19)	C14—C19—H19	119.8
C21—P2—C27	108.3 (2)	C18—C19—H19	119.8
O4—P2—Pd	111.14 (13)	O4—C20—H20A	109.5
C21—P2—Pd	117.19 (15)	O4—C20—H20B	109.5
C27—P2—Pd	115.78 (17)	H20A—C20—H20B	109.5
C1—O1—Pd	117.4 (3)	O4—C20—H20C	109.5
C2—O2—Pd	115.1 (3)	H20A—C20—H20C	109.5
C20—O4—P2	120.7 (3)	H20B—C20—H20C	109.5
C33—O5—H5	109.5	C26—C21—C22	120.4 (4)
O1—C1—C7	118.5 (4)	C26—C21—P2	117.7 (4)
O1—C1—C2	115.8 (4)	C22—C21—P2	121.9 (4)
C7—C1—C2	125.7 (4)	C23—C22—C21	119.5 (5)
O2—C2—C3	119.6 (4)	C23—C22—H22	120.2
O2—C2—C1	115.9 (4)	C21—C22—H22	120.2
C3—C2—C1	124.5 (4)	C24—C23—C22	120.3 (5)
C4—C3—C2	131.8 (4)	C24—C23—H23	119.9
C4—C3—Br1	115.6 (3)	C22—C23—H23	119.9
C2—C3—Br1	112.6 (3)	C23—C24—C25	120.9 (5)
C3—C4—C5	128.8 (4)	C23—C24—H24	119.6
C3—C4—H4	115.6	C25—C24—H24	119.6
C5—C4—H4	115.6	C26—C25—C24	119.3 (5)
C6—C5—C4	129.1 (4)	C26—C25—H25	120.3
C6—C5—Br2	114.9 (3)	C24—C25—H25	120.3
C4—C5—Br2	116.0 (3)	C21—C26—C25	119.6 (5)
C5—C6—C7	127.7 (4)	C21—C26—H26	120.2
C5—C6—H6	116.1	C25—C26—H26	120.2
C7—C6—H6	116.1	C28—C27—C32	119.9 (5)
C6—C7—C1	132.1 (4)	C28—C27—P2	122.0 (4)
C6—C7—Br3	114.3 (3)	C32—C27—P2	118.1 (4)
C1—C7—Br3	113.6 (3)	C27—C28—C29	119.3 (5)
C9—C8—C13	118.4 (4)	C27—C28—H28	120.3
C9—C8—P1	118.2 (3)	C29—C28—H28	120.3
C13—C8—P1	122.9 (3)	C30—C29—C28	120.1 (6)

C8—C9—C10	120.9 (4)	C30—C29—H29	119.9
C8—C9—H9	119.5	C28—C29—H29	119.9
C10—C9—H9	119.5	C29—C30—C31	121.4 (6)
C9—C10—C11	119.9 (4)	C29—C30—H30	119.3
C9—C10—H10	120.1	C31—C30—H30	119.3
C11—C10—H10	120.1	C30—C31—C32	119.3 (5)
C12—C11—C10	119.6 (4)	C30—C31—H31	120.3
C12—C11—H11	120.2	C32—C31—H31	120.3
C10—C11—H11	120.2	C31—C32—C27	119.9 (5)
C11—C12—C13	120.6 (5)	C31—C32—H32	120.0
C11—C12—H12	119.7	C27—C32—H32	120.0
C13—C12—H12	119.7	O5—C33—H33A	109.5
C12—C13—C8	120.5 (5)	O5—C33—H33B	109.5
C12—C13—H13	119.8	H33A—C33—H33B	109.5
C8—C13—H13	119.8	O5—C33—H33C	109.5
C19—C14—C15	119.2 (4)	H33A—C33—H33C	109.5
C19—C14—P1	121.4 (3)	H33B—C33—H33C	109.5
C15—C14—P1	119.2 (4)		
O1—Pd—P1—O3	170.90 (17)	Pd—P1—C8—C13	33.4 (4)
O2—Pd—P1—O3	−178.3 (4)	C13—C8—C9—C10	2.3 (7)
P2—Pd—P1—O3	−9.57 (15)	P1—C8—C9—C10	−169.7 (4)
O1—Pd—P1—C14	−65.82 (17)	C8—C9—C10—C11	−0.9 (7)
O2—Pd—P1—C14	−55.0 (4)	C9—C10—C11—C12	−1.6 (8)
P2—Pd—P1—C14	113.71 (15)	C10—C11—C12—C13	2.5 (8)
O1—Pd—P1—C8	44.99 (18)	C11—C12—C13—C8	−1.0 (8)
O2—Pd—P1—C8	55.8 (4)	C9—C8—C13—C12	−1.4 (7)
P2—Pd—P1—C8	−135.49 (16)	P1—C8—C13—C12	170.2 (4)
O2—Pd—P2—O4	9.33 (17)	O3—P1—C14—C19	−160.9 (3)
P1—Pd—P2—O4	−168.29 (14)	C8—P1—C14—C19	−42.4 (4)
O2—Pd—P2—C21	−110.6 (2)	Pd—P1—C14—C19	68.8 (3)
P1—Pd—P2—C21	71.77 (18)	O3—P1—C14—C15	23.2 (4)
O2—Pd—P2—C27	119.52 (19)	C8—P1—C14—C15	141.7 (3)
P1—Pd—P2—C27	−58.09 (18)	Pd—P1—C14—C15	−107.1 (3)
O2—Pd—O1—C1	−1.4 (3)	C19—C14—C15—C16	1.1 (6)
P1—Pd—O1—C1	176.3 (3)	P1—C14—C15—C16	177.2 (3)
O1—Pd—O2—C2	−1.1 (3)	C14—C15—C16—C17	−1.2 (7)
P2—Pd—O2—C2	179.3 (3)	C15—C16—C17—C18	0.7 (6)
P1—Pd—O2—C2	−12.3 (6)	C16—C17—C18—C19	−0.2 (6)
C21—P2—O4—C20	62.7 (4)	C15—C14—C19—C18	−0.6 (6)
C27—P2—O4—C20	174.0 (4)	P1—C14—C19—C18	−176.6 (3)
Pd—P2—O4—C20	−64.6 (4)	C17—C18—C19—C14	0.2 (6)
Pd—O1—C1—C7	−175.5 (3)	O4—P2—C21—C26	46.2 (5)
Pd—O1—C1—C2	3.4 (5)	C27—P2—C21—C26	−57.1 (5)
Pd—O2—C2—C3	−176.2 (3)	Pd—P2—C21—C26	169.6 (4)
Pd—O2—C2—C1	3.2 (4)	O4—P2—C21—C22	−133.0 (4)
O1—C1—C2—O2	−4.4 (6)	C27—P2—C21—C22	123.7 (4)
C7—C1—C2—O2	174.4 (4)	Pd—P2—C21—C22	−9.5 (5)
O1—C1—C2—C3	174.9 (4)	C26—C21—C22—C23	0.1 (8)
C7—C1—C2—C3	−6.3 (7)	P2—C21—C22—C23	179.2 (4)

O2—C2—C3—C4	−179.3 (4)	C21—C22—C23—C24	−1.0 (8)
C1—C2—C3—C4	1.4 (7)	C22—C23—C24—C25	1.2 (10)
O2—C2—C3—Br1	1.0 (5)	C23—C24—C25—C26	−0.3 (10)
C1—C2—C3—Br1	−178.3 (3)	C22—C21—C26—C25	0.8 (9)
C2—C3—C4—C5	2.1 (8)	P2—C21—C26—C25	−178.4 (5)
Br1—C3—C4—C5	−178.3 (4)	C24—C25—C26—C21	−0.6 (10)
C3—C4—C5—C6	0.0 (8)	O4—P2—C27—C28	−129.7 (5)
C3—C4—C5—Br2	179.8 (4)	C21—P2—C27—C28	−21.7 (5)
C4—C5—C6—C7	−2.4 (8)	Pd—P2—C27—C28	112.3 (4)
Br2—C5—C6—C7	177.7 (4)	O4—P2—C27—C32	53.5 (4)
C5—C6—C7—C1	−0.8 (8)	C21—P2—C27—C32	161.6 (4)
C5—C6—C7—Br3	−179.2 (4)	Pd—P2—C27—C32	−64.4 (4)
O1—C1—C7—C6	−175.0 (4)	C32—C27—C28—C29	−0.9 (9)
C2—C1—C7—C6	6.2 (7)	P2—C27—C28—C29	−177.5 (5)
O1—C1—C7—Br3	3.3 (5)	C27—C28—C29—C30	0.1 (10)
C2—C1—C7—Br3	−175.5 (3)	C28—C29—C30—C31	0.2 (11)
O3—P1—C8—C9	72.5 (4)	C29—C30—C31—C32	0.1 (10)
C14—P1—C8—C9	−46.2 (4)	C30—C31—C32—C27	−0.9 (8)
Pd—P1—C8—C9	−155.0 (3)	C28—C27—C32—C31	1.2 (8)
O3—P1—C8—C13	−99.1 (4)	P2—C27—C32—C31	178.0 (4)
C14—P1—C8—C13	142.2 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19···Br3	0.95	2.95	3.818 (5)	152
O5—H5···O3	0.84	2.01	2.740 (7)	144
C20—H20B···O2	0.98	2.30	3.143 (6)	144
C12—H12···O5^{i}	0.95	2.56	3.435 (9)	154
C31—H31···Br1^{ii}	0.95	2.99	3.920 (6)	168

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

Fig. 1

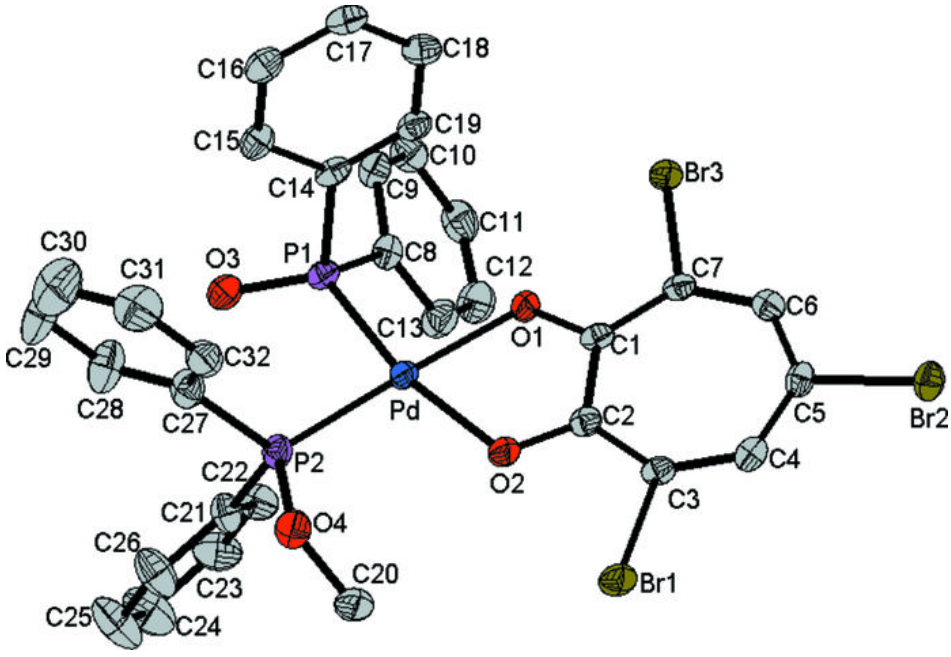
C17
C18
C16
C19
C15
C10
C14
C9
Br3
C11
C30
O3
P1
C8
C12
C7
C31
C6
C13
O1
C1
C29
C32
Br2
Pd
C5
C28
C27
C2
P2
C4
C22
O2
C3
C21
O4
C26
C20
C23
Br1
C25
C24

Fig. 2