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cis-[2,6-Bis(di-*tert*-butylphosphinomethyl)cyclohexyl-κ^3P,C^1,P']bromidopalladium(II)

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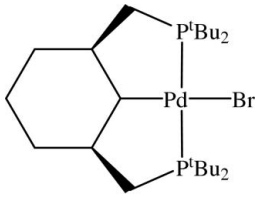
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.041; wR factor = 0.122; data-to-parameter ratio = 25.4.

The title compound, [PdBr($C_{24}H_{49}P_2$)], has a distorted square-planar coordination geometry with the *P*,*C*,*P*′-tridentate ligand forming two five-membered chelate rings. The Br atom is displaced from the square plane by 0.560 (6) Å.

Related literature

See Kuznetsov *et al.* (2006) for the rhodium complex with the same ligand, and Castonguay *et al.* (2006) and Pandarus & Zargarian (2007) for similar nickel complexes.

For related literature, see: Davis & Hassel (1963); Nilsson & Wendt (2005); Ohff *et al.* (1997); Sjövall *et al.* (2001, 2002).



Experimental

Crystal data

[PdBr($C_{24}H_{49}P_2$)]
M_r = 585.88
Monoclinic, $P2_1/c$
a = 11.4392 (2) Å
b = 15.7757 (2) Å
c = 15.6508 (2) Å
β = 97.846 (2)°
V = 2797.92 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.21 mm^{-1}
T = 293 (2) K
0.25 × 0.13 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.608, T_{max} = 0.897
22034 measured reflections
6739 independent reflections
3849 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.122
S = 0.94
6739 reflections
265 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd—C2	2.082 (4)	Pd—P1	2.3211 (11)
Pd—P2	2.3097 (11)	Pd—Br	2.5678 (5)
C2—Pd—P2	83.81 (12)	C2—Pd—Br	170.61 (13)
C2—Pd—P1	83.47 (12)	P2—Pd—Br	96.53 (3)
P2—Pd—P1	166.32 (4)	P1—Pd—Br	96.85 (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: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2286).

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supplementary materials

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cis-[2,6-Bis(di-*tert*-butylphosphinomethyl)cyclohexyl-κ^3P,C^1,P']bromidopalladium(II)

D. Olsson, J. M. Janse van Rensburg and O. F. Wendt

Comment

The title compound belongs to a family of C-sp^3-H activated PCP complexes that show interesting catalytic performance in C—C coupling reactions (Ohff *et al.*,1997; Sjövall *et al.*, 2002; Nilsson & Wendt, 2005). The iodide and trifluoroacetate analogues have previously been prepared and the crystal structure of the iodide described (Sjövall *et al.*, 2002).

The vast majority of reported palladium (PCP)-complexes are constructed with an aromatic back-bone. The aliphatic based complexes, such as the iodide compound mentioned, are studied in recent publications on PCP)-type of compounds coordinated to metals such as rhodium (Kuznetsov *et al.*, 2006) and nickel (Castonguay *et al.*, 2006; Pandarus *et al.*, 2007).

The title compound exhibits a pseudo-square-planar geometry with the phosphorus atoms positioned *trans* to each other with a P1—Pd—P2 angle of 166.32 (4) °. Coordination of the (PCP)-tridentate ligand leads to the formation of two five-membered rings. The distortion, manifested in the repulsion of the P1 and P2 from the σ-donating bromine atom, results in acute angles for the bis-chelating system.

For the coordination plane formed by Pd, P1, C2 and P2 the largest deviation from this plane is observed for Pd, at 0.0690 (7) Å (r.m.s. of fitted atoms = 0.0445). The Br lies out out of the plane by 0.560 (6) Å. There are intramolecular H···Br contacts: C113—H···Br, 156.62 °, H···Br, 2.852 Å and C211—H···Br, 154.93 °, H···Br, 2.860 Å. The mean plane of the cyclohexane ring is aligned with the palladium coordination plane owing to C—H activation in an equatorial position. Perpendicular to the coordination plane is a pseudo-mirror plane through atoms Br1, Pd1, C2 and C5. Selected bond lengths and angles are given in Table 1.

Comparison of the title compound (I) to the analogous iodo-complex indicates the expected Pd-halogen bond distance decreases by 0.2 Å. Also noted is the lower *trans*-influence of the bromide on the Pd—C2 bond distance with a *ca* 0.05 Å decrease as compared with that in the the iodine complex.

Experimental

The analogous trifluoroacetate-complex was synthesized according to a modification of a published protocol (Sjövall *et al.*, 2001 & Sjövall *et al.*, 2002). The (PCP)Pd-TFA complex (10 mg, 16 µmol) was dissolved in toluene-d_8 (0.6 ml) in a J. Young tube and an excess of NaBr added. The tube was allowed to stand over night in room temperature and after 12 h, a ^{31}P-NMR spectrum confirmed that the title complex had been quantitatively obtained. The NMR sample was first decanted and then filtered through a short pad of celite. Recrystallization in pentane at 255 K overnight resulted in colourless blocks. ^{1}H-NMR (toluene-d_8): δ 2.15–0.80 (m region, 13H, CH & CH_2), 1.35 (m, 36H, coalesced virtual triplets). ^{31}P{^{1}H} NMR (toluene-d_8): δ 70.2 (*s*). ^{13}C{^{1}H} NMR (toluene-d_8): δ 66.8 (t, 2JPC = 3.9 Hz, CHPd), 50.9 (vt, J_{PC} = 15.1 Hz, CHCHPd), 35.9 (vt, J_{PC} = 12,6 Hz, $C(CH_3)_3$), 35.2 (vt, J_{PC} = 15.1 Hz, $C(CH_3)_3$), 34.8 (vt, J_{PC} = 26.0 Hz, CH_2P), 32.9 (vt, J_{PC} = 15.8 Hz, $CH_2CHCHPd$), 30.2 (vt, J_{PC} = 5.9 Hz, $C(CH_3)_3$), 29.5 (vt, J_{PC} = 5.8 Hz, $C(CH_3)_3$), 27.0 (s, $CH_2CH_2CHCHPd$).

Refinement

The H atoms were positioned geometrically and treated as riding on their parent atoms with C–H distances of 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq} - 1.5U_{eq}$.

Figures

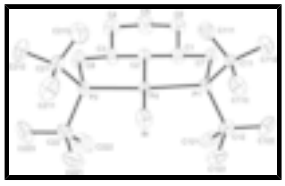


Fig. 1. The molecular structure of (I) with atom labels and 30% probability displacement ellipsoids. H-atoms were omitted for clarity.

cis-[2,6-Bis(di-*tert*-butylphosphinomethyl)cyclohexyl- κ^3P,C^1,P']bromidopalladium(II)

Crystal data

$[PdBr(C_{24}H_{49}P_2)]$	$F_{000} = 1216$
$M_r = 585.88$	$D_x = 1.391$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5773 reflections
$a = 11.4392$ (2) Å	$\theta = 2$–30º
$b = 15.7757$ (2) Å	$\mu = 2.21$ mm^{-1}
$c = 15.6508$ (2) Å	$T = 293$ (2) K
$\beta = 97.846$ (2)º	Plate, colourless
$V = 2797.92$ (7) Å^3	0.25 × 0.13 × 0.05 mm
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur diffractometer	3849 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.046$
Monochromator: graphite	$\theta_{max} = 28$º
φ and ω scans	$\theta_{min} = 2.2$º
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	$h = -15\rightarrow14$
$T_{min} = 0.608$, $T_{max} = 0.897$	$k = -20\rightarrow19$
22034 measured reflections	$l = -11\rightarrow20$
6739 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.122$

$S = 0.94$

6739 reflections

265 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.22$ e Å^{-3}

$\Delta\rho_{min} = -0.62$ e Å^{-3}

Extinction correction: none

Special details

Experimental. The intensity data were collected on a Oxford Xcalibur 3 CCD diffractometer using an exposure time of 20 s/frame. A total of 552 frames were collected with a frame width of 0.5° covering up to θ = 28.00° with 99.7% completeness accomplished. The highest difference peak in the Fourier map is located 0.58 Å from H11G.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd	0.12038 (3)	0.307166 (19)	0.294263 (19)	0.03791 (11)
Br	0.04772 (5)	0.18770 (3)	0.38524 (3)	0.06669 (18)
P1	−0.05901 (10)	0.33357 (7)	0.20991 (7)	0.0401 (3)
P2	0.31172 (10)	0.31083 (7)	0.36407 (7)	0.0434 (3)
C11	−0.1341 (4)	0.2413 (3)	0.1477 (3)	0.0489 (11)
C12	−0.1645 (4)	0.3959 (3)	0.2668 (3)	0.0563 (12)
C21	0.3996 (4)	0.2097 (3)	0.3661 (4)	0.0637 (14)
C22	0.3278 (5)	0.3646 (4)	0.4721 (3)	0.0676 (15)
C111	−0.0333 (5)	0.1887 (3)	0.1208 (3)	0.0711 (16)
H11D	0.0197	0.1726	0.1711	0.107*
H11E	0.0085	0.2216	0.0832	0.107*
H11F	−0.0648	0.1387	0.0911	0.107*
C112	−0.2143 (5)	0.2680 (4)	0.0653 (3)	0.0718 (15)
H11G	−0.2529	0.2189	0.0384	0.108*
H11H	−0.1675	0.2944	0.0262	0.108*
H11I	−0.2724	0.3075	0.0797	0.108*
C113	−0.2029 (5)	0.1875 (3)	0.2036 (3)	0.0744 (16)
H11A	−0.2735	0.2169	0.2131	0.112*
H11B	−0.1553	0.1768	0.2579	0.112*
H11C	−0.2236	0.1346	0.1751	0.112*
C121	−0.1024 (5)	0.4795 (3)	0.2942 (4)	0.0756 (16)
H12A	−0.1518	0.5128	0.3261	0.113*
H12B	−0.0876	0.5103	0.2438	0.113*
H12C	−0.0289	0.4679	0.3297	0.113*
C122	−0.2821 (5)	0.4138 (4)	0.2116 (4)	0.0853 (18)
H12D	−0.3251	0.3618	0.2007	0.128*

H12E	−0.2681	0.4384	0.1578	0.128*
H12F	−0.3271	0.4525	0.2414	0.128*
C123	−0.1849 (5)	0.3509 (4)	0.3498 (3)	0.0785 (17)
H12G	−0.24	0.3829	0.378	0.118*
H12H	−0.1115	0.3463	0.3873	0.118*
H12I	−0.2162	0.2953	0.3363	0.118*
C211	0.3732 (6)	0.1497 (4)	0.4388 (5)	0.099 (2)
H21D	0.4091	0.0956	0.4319	0.148*
H21E	0.2894	0.1428	0.4361	0.148*
H21F	0.4047	0.1735	0.4936	0.148*
C212	0.5329 (5)	0.2234 (4)	0.3741 (4)	0.0875 (19)
H21G	0.5588	0.2551	0.4256	0.131*
H21H	0.5515	0.2543	0.3249	0.131*
H21I	0.5721	0.1695	0.3769	0.131*
C213	0.3566 (6)	0.1660 (4)	0.2800 (4)	0.098 (2)
H21A	0.3774	0.1999	0.2334	0.147*
H21B	0.2725	0.1593	0.2739	0.147*
H21C	0.3932	0.1113	0.2789	0.147*
C221	0.2542 (6)	0.3207 (5)	0.5306 (4)	0.109 (3)
H22G	0.2864	0.2655	0.545	0.163*
H22H	0.1748	0.3151	0.5021	0.163*
H22I	0.2542	0.3534	0.5824	0.163*
C222	0.2813 (6)	0.4561 (4)	0.4546 (4)	0.104 (2)
H22D	0.1982	0.4545	0.4346	0.156*
H22E	0.3218	0.482	0.4115	0.156*
H22F	0.2952	0.4886	0.507	0.156*
C223	0.4555 (5)	0.3728 (5)	0.5168 (4)	0.099 (2)
H22A	0.458	0.4113	0.5645	0.149*
H22B	0.5045	0.394	0.4764	0.149*
H22C	0.4837	0.3182	0.5374	0.149*
C1	0.0980 (4)	0.4455 (3)	0.1552 (3)	0.0526 (11)
H1	0.0814	0.4912	0.1943	0.063*
C2	0.1859 (4)	0.3882 (3)	0.2071 (3)	0.0456 (10)
H2	0.2076	0.3489	0.1633	0.055*
C3	0.3007 (4)	0.4325 (3)	0.2371 (3)	0.0494 (11)
H3	0.2803	0.4787	0.2742	0.059*
C4	0.3521 (4)	0.4754 (3)	0.1645 (3)	0.0568 (12)
H4A	0.4188	0.5097	0.1888	0.068*
H4B	0.3814	0.4322	0.1287	0.068*
C5	0.2664 (5)	0.5305 (3)	0.1088 (3)	0.0678 (14)
H5A	0.3006	0.5465	0.0577	0.081*
H5B	0.2535	0.582	0.1401	0.081*
C6	0.1495 (4)	0.4881 (3)	0.0816 (3)	0.0511 (11)
H6A	0.1592	0.4459	0.0381	0.061*
H6B	0.0939	0.5301	0.0554	0.061*
C7	−0.0189 (4)	0.4034 (3)	0.1253 (3)	0.0471 (10)
H7A	−0.0133	0.3708	0.0735	0.057*
H7B	−0.0794	0.4462	0.1116	0.057*
C8	0.3874 (4)	0.3792 (3)	0.2934 (3)	0.0607 (13)

H8A	0.4432	0.4155	0.3284	0.073*
H8B	0.4311	0.3442	0.2578	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd	0.03525 (19)	0.03707 (19)	0.04042 (18)	−0.00223 (15)	0.00162 (13)	0.00658 (14)
Br	0.0634 (4)	0.0669 (4)	0.0673 (3)	−0.0170 (3)	0.0001 (3)	0.0279 (3)
P1	0.0350 (6)	0.0415 (6)	0.0425 (6)	0.0009 (5)	0.0010 (5)	0.0045 (5)
P2	0.0359 (6)	0.0443 (6)	0.0474 (6)	−0.0029 (5)	−0.0041 (5)	0.0087 (5)
C11	0.047 (3)	0.052 (3)	0.045 (2)	−0.008 (2)	−0.002 (2)	−0.004 (2)
C12	0.050 (3)	0.054 (3)	0.067 (3)	0.002 (2)	0.015 (2)	−0.004 (2)
C21	0.044 (3)	0.065 (3)	0.079 (3)	0.005 (2)	0.001 (3)	0.012 (3)
C22	0.067 (4)	0.074 (4)	0.058 (3)	−0.013 (3)	−0.005 (3)	−0.007 (3)
C111	0.074 (4)	0.064 (3)	0.071 (3)	0.003 (3)	−0.007 (3)	−0.016 (3)
C112	0.070 (4)	0.077 (4)	0.062 (3)	−0.009 (3)	−0.012 (3)	−0.005 (3)
C113	0.082 (4)	0.068 (4)	0.070 (3)	−0.034 (3)	−0.003 (3)	0.007 (3)
C121	0.082 (4)	0.055 (3)	0.096 (4)	0.008 (3)	0.035 (3)	−0.017 (3)
C122	0.056 (3)	0.088 (4)	0.112 (5)	0.033 (3)	0.014 (3)	0.003 (4)
C123	0.079 (4)	0.089 (4)	0.075 (4)	0.007 (3)	0.040 (3)	−0.003 (3)
C211	0.088 (5)	0.071 (4)	0.140 (6)	0.019 (4)	0.025 (4)	0.050 (4)
C212	0.051 (3)	0.078 (4)	0.131 (5)	0.024 (3)	0.003 (3)	0.006 (4)
C213	0.103 (5)	0.074 (4)	0.110 (5)	0.027 (4)	−0.014 (4)	−0.031 (4)
C221	0.106 (6)	0.164 (7)	0.061 (4)	−0.048 (5)	0.027 (4)	−0.029 (4)
C222	0.133 (6)	0.093 (5)	0.080 (4)	0.009 (5)	−0.003 (4)	−0.028 (4)
C223	0.084 (5)	0.120 (6)	0.082 (4)	−0.025 (4)	−0.029 (3)	−0.007 (4)
C1	0.050 (3)	0.056 (3)	0.051 (3)	−0.003 (2)	0.006 (2)	0.012 (2)
C2	0.046 (3)	0.047 (3)	0.044 (2)	−0.003 (2)	0.0064 (19)	0.011 (2)
C3	0.040 (2)	0.053 (3)	0.052 (3)	−0.011 (2)	−0.003 (2)	0.011 (2)
C4	0.052 (3)	0.049 (3)	0.071 (3)	−0.017 (2)	0.013 (2)	0.012 (2)
C5	0.075 (4)	0.064 (3)	0.064 (3)	−0.019 (3)	0.008 (3)	0.014 (3)
C6	0.056 (3)	0.044 (3)	0.052 (3)	−0.004 (2)	0.006 (2)	0.010 (2)
C7	0.039 (2)	0.046 (3)	0.055 (3)	0.001 (2)	0.003 (2)	0.008 (2)
C8	0.041 (3)	0.065 (3)	0.075 (3)	−0.009 (2)	0.003 (2)	0.014 (3)

Geometric parameters (Å, °)

Pd—C2	2.082 (4)	C211—H21D	0.96
Pd—P2	2.3097 (11)	C211—H21E	0.96
Pd—P1	2.3211 (11)	C211—H21F	0.96
Pd—Br	2.5678 (5)	C212—H21G	0.96
P1—C7	1.829 (4)	C212—H21H	0.96
P1—C12	1.873 (5)	C212—H21I	0.96
P1—C11	1.891 (4)	C213—H21A	0.96
P2—C8	1.843 (5)	C213—H21B	0.96
P2—C22	1.878 (5)	C213—H21C	0.96
P2—C21	1.883 (5)	C221—H22G	0.96
C11—C113	1.514 (6)	C221—H22H	0.96
C11—C111	1.527 (7)	C221—H22I	0.96

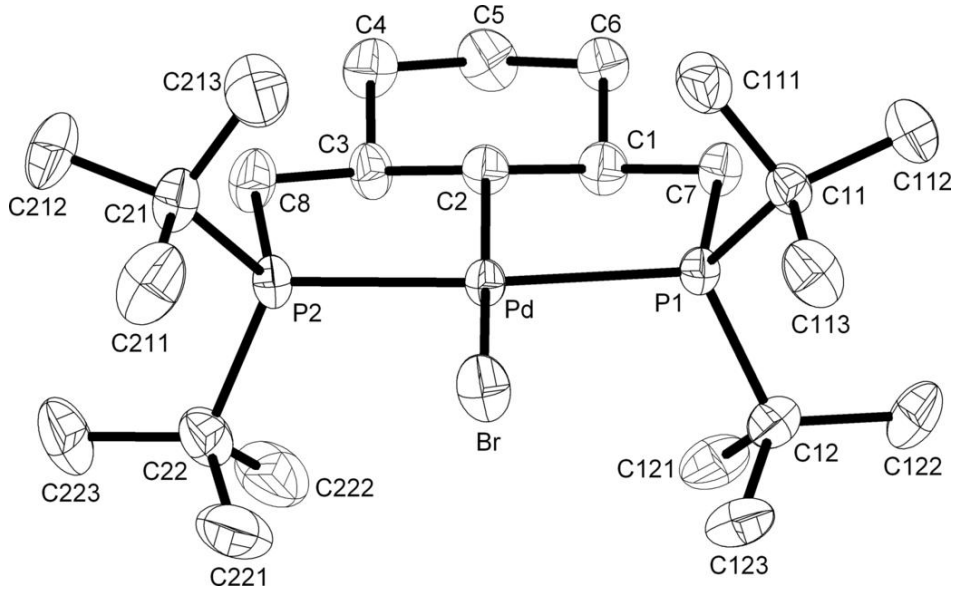
C11—C112	1.536 (6)	C222—H22D	0.96
C12—C122	1.522 (7)	C222—H22E	0.96
C12—C123	1.525 (7)	C222—H22F	0.96
C12—C121	1.532 (7)	C223—H22A	0.96
C21—C212	1.528 (7)	C223—H22B	0.96
C21—C213	1.534 (8)	C223—H22C	0.96
C21—C211	1.541 (8)	C1—C2	1.504 (6)
C22—C221	1.497 (8)	C1—C7	1.510 (6)
C22—C223	1.536 (7)	C1—C6	1.520 (6)
C22—C222	1.549 (8)	C1—H1	0.98
C111—H11D	0.96	C2—C3	1.505 (6)
C111—H11E	0.96	C2—H2	0.98
C111—H11F	0.96	C3—C8	1.493 (6)
C112—H11G	0.96	C3—C4	1.508 (6)
C112—H11H	0.96	C3—H3	0.98
C112—H11I	0.96	C4—C5	1.499 (6)
C113—H11A	0.96	C4—H4A	0.97
C113—H11B	0.96	C4—H4B	0.97
C113—H11C	0.96	C5—C6	1.504 (6)
C121—H12A	0.96	C5—H5A	0.97
C121—H12B	0.96	C5—H5B	0.97
C121—H12C	0.96	C6—H6A	0.97
C122—H12D	0.96	C6—H6B	0.97
C122—H12E	0.96	C7—H7A	0.97
C122—H12F	0.96	C7—H7B	0.97
C123—H12G	0.96	C8—H8A	0.97
C123—H12H	0.96	C8—H8B	0.97
C123—H12I	0.96		
C2—Pd—P2	83.81 (12)	C21—C211—H21F	109.5
C2—Pd—P1	83.47 (12)	H21D—C211—H21F	109.5
P2—Pd—P1	166.32 (4)	H21E—C211—H21F	109.5
C2—Pd—Br	170.61 (13)	C21—C212—H21G	109.5
P2—Pd—Br	96.53 (3)	C21—C212—H21H	109.5
P1—Pd—Br	96.85 (3)	H21G—C212—H21H	109.5
C7—P1—C12	105.6 (2)	C21—C212—H21I	109.5
C7—P1—C11	103.43 (19)	H21G—C212—H21I	109.5
C12—P1—C11	111.8 (2)	H21H—C212—H21I	109.5
C7—P1—Pd	103.31 (14)	C21—C213—H21A	109.5
C12—P1—Pd	113.64 (16)	C21—C213—H21B	109.5
C11—P1—Pd	117.32 (15)	H21A—C213—H21B	109.5
C8—P2—C22	106.2 (2)	C21—C213—H21C	109.5
C8—P2—C21	102.2 (2)	H21A—C213—H21C	109.5
C22—P2—C21	112.4 (3)	H21B—C213—H21C	109.5
C8—P2—Pd	102.91 (15)	C22—C221—H22G	109.5
C22—P2—Pd	114.03 (18)	C22—C221—H22H	109.5
C21—P2—Pd	117.12 (17)	H22G—C221—H22H	109.5
C113—C11—C111	109.0 (4)	C22—C221—H22I	109.5
C113—C11—C112	109.9 (4)	H22G—C221—H22I	109.5
C111—C11—C112	107.7 (4)	H22H—C221—H22I	109.5

C113—C11—P1	111.7 (3)	C22—C222—H22D	109.5
C111—C11—P1	104.7 (3)	C22—C222—H22E	109.5
C112—C11—P1	113.5 (3)	H22D—C222—H22E	109.5
C122—C12—C123	110.1 (4)	C22—C222—H22F	109.5
C122—C12—C121	109.8 (4)	H22D—C222—H22F	109.5
C123—C12—C121	106.3 (4)	H22E—C222—H22F	109.5
C122—C12—P1	113.9 (3)	C22—C223—H22A	109.5
C123—C12—P1	110.1 (3)	C22—C223—H22B	109.5
C121—C12—P1	106.3 (3)	H22A—C223—H22B	109.5
C212—C21—C213	109.4 (5)	C22—C223—H22C	109.5
C212—C21—C211	108.6 (5)	H22A—C223—H22C	109.5
C213—C21—C211	107.5 (5)	H22B—C223—H22C	109.5
C212—C21—P2	113.9 (4)	C2—C1—C7	113.6 (4)
C213—C21—P2	105.1 (4)	C2—C1—C6	112.0 (4)
C211—C21—P2	112.0 (4)	C7—C1—C6	112.2 (3)
C221—C22—C223	109.8 (5)	C2—C1—H1	106.1
C221—C22—C222	109.3 (5)	C7—C1—H1	106.1
C223—C22—C222	106.5 (5)	C6—C1—H1	106.1
C221—C22—P2	110.3 (4)	C1—C2—C3	112.3 (4)
C223—C22—P2	114.7 (4)	C1—C2—Pd	116.7 (3)
C222—C22—P2	105.9 (4)	C3—C2—Pd	117.2 (3)
C11—C111—H11D	109.5	C1—C2—H2	102.6
C11—C111—H11E	109.5	C3—C2—H2	102.6
H11D—C111—H11E	109.5	Pd—C2—H2	102.6
C11—C111—H11F	109.5	C8—C3—C2	113.8 (4)
H11D—C111—H11F	109.5	C8—C3—C4	113.6 (4)
H11E—C111—H11F	109.5	C2—C3—C4	112.7 (3)
C11—C112—H11G	109.5	C8—C3—H3	105.2
C11—C112—H11H	109.5	C2—C3—H3	105.2
H11G—C112—H11H	109.5	C4—C3—H3	105.2
C11—C112—H11I	109.5	C5—C4—C3	114.0 (4)
H11G—C112—H11I	109.5	C5—C4—H4A	108.7
H11H—C112—H11I	109.5	C3—C4—H4A	108.7
C11—C113—H11A	109.5	C5—C4—H4B	108.7
C11—C113—H11B	109.5	C3—C4—H4B	108.7
H11A—C113—H11B	109.5	H4A—C4—H4B	107.6
C11—C113—H11C	109.5	C4—C5—C6	113.1 (4)
H11A—C113—H11C	109.5	C4—C5—H5A	109
H11B—C113—H11C	109.5	C6—C5—H5A	109
C12—C121—H12A	109.5	C4—C5—H5B	109
C12—C121—H12B	109.5	C6—C5—H5B	109
H12A—C121—H12B	109.5	H5A—C5—H5B	107.8
C12—C121—H12C	109.5	C5—C6—C1	113.8 (4)
H12A—C121—H12C	109.5	C5—C6—H6A	108.8
H12B—C121—H12C	109.5	C1—C6—H6A	108.8
C12—C122—H12D	109.5	C5—C6—H6B	108.8
C12—C122—H12E	109.5	C1—C6—H6B	108.8
H12D—C122—H12E	109.5	H6A—C6—H6B	107.7
C12—C122—H12F	109.5	C1—C7—P1	109.9 (3)

H12D—C122—H12F	109.5	C1—C7—H7A	109.7
H12E—C122—H12F	109.5	P1—C7—H7A	109.7
C12—C123—H12G	109.5	C1—C7—H7B	109.7
C12—C123—H12H	109.5	P1—C7—H7B	109.7
H12G—C123—H12H	109.5	H7A—C7—H7B	108.2
C12—C123—H12I	109.5	C3—C8—P2	110.8 (3)
H12G—C123—H12I	109.5	C3—C8—H8A	109.5
H12H—C123—H12I	109.5	P2—C8—H8A	109.5
C21—C211—H21D	109.5	C3—C8—H8B	109.5
C21—C211—H21E	109.5	P2—C8—H8B	109.5
H21D—C211—H21E	109.5	H8A—C8—H8B	108.1
C2—Pd—P1—C7	1.49 (19)	C8—P2—C21—C211	165.7 (4)
P2—Pd—P1—C7	23.1 (3)	C22—P2—C21—C211	52.2 (5)
Br—Pd—P1—C7	−169.05 (15)	Pd—P2—C21—C211	−82.7 (4)
C2—Pd—P1—C12	−112.5 (2)	C8—P2—C22—C221	171.7 (4)
P2—Pd—P1—C12	−90.8 (2)	C21—P2—C22—C221	−77.3 (5)
Br—Pd—P1—C12	77.00 (17)	Pd—P2—C22—C221	59.1 (5)
C2—Pd—P1—C11	114.5 (2)	C8—P2—C22—C223	−63.6 (5)
P2—Pd—P1—C11	136.2 (2)	C21—P2—C22—C223	47.4 (5)
Br—Pd—P1—C11	−56.04 (16)	Pd—P2—C22—C223	−176.2 (4)
C2—Pd—P2—C8	−1.5 (2)	C8—P2—C22—C222	53.5 (4)
P1—Pd—P2—C8	−23.1 (3)	C21—P2—C22—C222	164.5 (4)
Br—Pd—P2—C8	169.09 (18)	Pd—P2—C22—C222	−59.1 (4)
C2—Pd—P2—C22	113.1 (2)	C7—C1—C2—C3	−179.1 (4)
P1—Pd—P2—C22	91.5 (3)	C6—C1—C2—C3	52.5 (5)
Br—Pd—P2—C22	−76.3 (2)	C7—C1—C2—Pd	−39.8 (5)
C2—Pd—P2—C21	−112.7 (2)	C6—C1—C2—Pd	−168.2 (3)
P1—Pd—P2—C21	−134.3 (2)	P2—Pd—C2—C1	−155.5 (3)
Br—Pd—P2—C21	57.87 (19)	P1—Pd—C2—C1	19.5 (3)
C7—P1—C11—C113	−164.7 (4)	Br—Pd—C2—C1	112.0 (7)
C12—P1—C11—C113	−51.5 (4)	P2—Pd—C2—C3	−18.2 (3)
Pd—P1—C11—C113	82.3 (4)	P1—Pd—C2—C3	156.8 (3)
C7—P1—C11—C111	77.5 (3)	Br—Pd—C2—C3	−110.7 (7)
C12—P1—C11—C111	−169.3 (3)	C1—C2—C3—C8	176.3 (4)
Pd—P1—C11—C111	−35.5 (3)	Pd—C2—C3—C8	37.2 (5)
C7—P1—C11—C112	−39.7 (4)	C1—C2—C3—C4	−52.3 (5)
C12—P1—C11—C112	73.5 (4)	Pd—C2—C3—C4	168.6 (3)
Pd—P1—C11—C112	−152.7 (3)	C8—C3—C4—C5	−178.7 (4)
C7—P1—C12—C122	67.1 (4)	C2—C3—C4—C5	49.8 (6)
C11—P1—C12—C122	−44.7 (4)	C3—C4—C5—C6	−47.3 (6)
Pd—P1—C12—C122	179.7 (3)	C4—C5—C6—C1	47.8 (6)
C7—P1—C12—C123	−168.7 (4)	C2—C1—C6—C5	−50.6 (6)
C11—P1—C12—C123	79.5 (4)	C7—C1—C6—C5	−179.7 (4)
Pd—P1—C12—C123	−56.1 (4)	C2—C1—C7—P1	38.6 (5)
C7—P1—C12—C121	−53.8 (4)	C6—C1—C7—P1	166.9 (3)
C11—P1—C12—C121	−165.7 (3)	C12—P1—C7—C1	98.3 (3)
Pd—P1—C12—C121	58.7 (4)	C11—P1—C7—C1	−144.1 (3)
C8—P2—C21—C212	41.9 (5)	Pd—P1—C7—C1	−21.3 (3)
C22—P2—C21—C212	−71.6 (5)	C2—C3—C8—P2	−36.3 (5)

Pd—P2—C21—C212	153.5 (4)	C4—C3—C8—P2	−167.2 (3)
C8—P2—C21—C213	−77.9 (4)	C22—P2—C8—C3	−99.9 (4)
C22—P2—C21—C213	168.6 (4)	C21—P2—C8—C3	142.2 (4)
Pd—P2—C21—C213	33.7 (4)	Pd—P2—C8—C3	20.3 (4)

Fig. 1


C4
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