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trans-Bromohydridobis(triphenylphosphine)-platinum(II)

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Key indicators

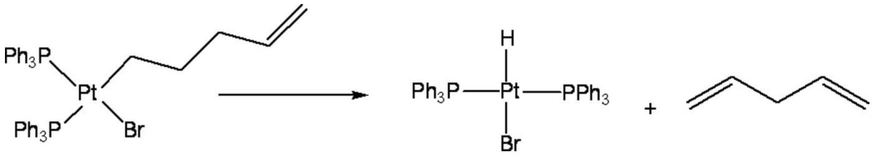
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.006 Å
R factor = 0.027
wR factor = 0.051
Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [PtBrH($C_{18}H_{15}P$)$_2$], has a square-planar environment around the Pt atom, with the hydride and bromide ligands being exactly collinear with Pt since they all lie on a crystallographic twofold rotation axis, and with mutually *trans* triphenylphosphine ligands with a P—Pt—P bond angle that is slightly bent towards the hydride [P—Pt—P = 170.81 (5)°]. The Pt—H distance (1.610 Å) is in good agreement with those found in structures determined by neutron diffraction.

Comment

The preparation of the title compound, *trans*-[HPtBr(PPh_3)$_2$], (I), was reported initially by Carr *et al.* (1985). It has also been prepared from the hydrolysis of either the platinum(II) boryl complex, *trans*-Pt(PPh_3)$_2$[B(Mes)Br]Br (Mes = 2,4,6-$Me_3C_6H_2$) (Aldridge *et al.*, 2003), or the alkoxycarbonyl, [PtX(COOR)(PPh_3)$_2$] (where R = Me or Et, X = halide), in the presence of a salt catalyst (NH_4X or KX, X = halide), giving *trans*-[PtHX(PPh_3)$_2$] (Clark *et al.*, 1969). The X-ray single-crystal structures of the toluene monosolvated title compound (Habereder & Nöth, 2003) and of the toluene hemisolvated title compound (Aldridge *et al.*, 2003) were reported recently. We have now prepared and characterized the unsolvated title compound, (I) (Fig. 1), from decomposition of (PPh_3)$_2$Pt(1-pentenyl) (see equation below), and present the results here.



(I)

Compound (I) crystallizes in the space group $C2/c$ with Z = 4. The molecule is located at a special position at Wyckoff position e, with a twofold rotation axis running through the linear Br—Pt—H group. This results in *trans*-oriented triphenylphosphine ligands, with P—Pt—P [bond angle 170.81 (5)°] being slightly bent towards the hydride. The Pt—P distance of 2.2827 (9) Å and the Pt—Br distance of 2.5229 (6) Å are within the accepted ranges for similar platinum(II) complexes (Orpen *et al.*, 1989). The Pt—H distance is 1.61 Å. A search of the Cambridge Structural Database (CSD, Version 5.27 with August 2006 updates; Allen, 2002) for platinum(II) complexes with terminal hydrides gave rise to 65 hits. For all 65 complexes, Pt—H(terminal) distances range from 1.00 to 2.153 Å, with a median value of 1.626 Å and a mean value of 1.628 Å. In a search of all the platinum

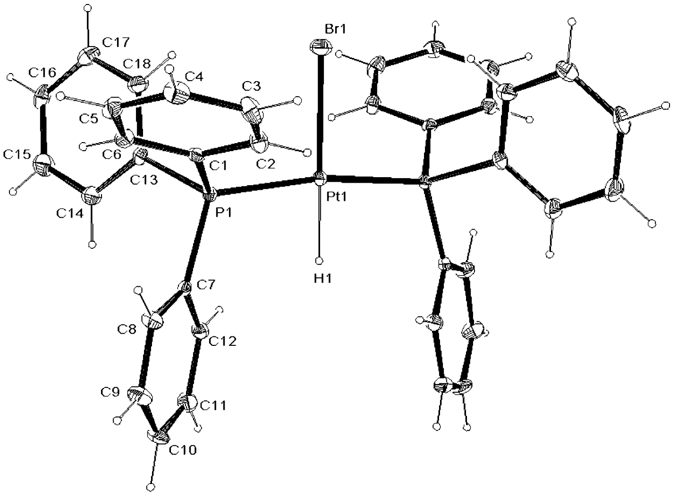


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary size.

complexes in the CSD, two structures with terminal hydrides had been determined by neutron diffraction and their Pt—H(terminal) distances are 1.549 Å (CSD refcode WAZPUI01; Albinati *et al.*, 1997) and 1.610 Å (CSD refcode CAKNEH01; Chiang *et al.*, 1984), respectively. Therefore, the Pt—H distance of 1.61 Å found for structure (I) is very reasonable. The salient bond lengths and angles are listed in Table 1.

Experimental

Compound (I) was prepared from a dichloromethane solution of *cis*-[Pt(1-pentenyl)Br(PPh$_3$)$_2$] on exposure to air (Sivaramakrishna & Moss, 2006). This is probably due to the breaking of the *M*—C bond through β-hydride elimination to form the Pt—H bond, and also isomerization from *cis* to *trans* (see scheme). ^{1}H NMR showed the Pt—H signals as a triplet at −14.8 p.p.m. with platinum satellites ($J_{\rm Pt-H}$ = 1240 Hz and $J_{\rm P-H}$ = 25.6 Hz). ^{31}P NMR indicated a singlet at 28.8 p.p.m. with platinum satellites ($J_{\rm Pt-P}$ = 2988 Hz). A solution containing *cis*-(PPh$_3$)$_2$Pt(CH$_2$CH$_2$CH$_2$CH=CH$_2$)Br (0.168 g, 0.193 mmol) in CH$_2$Cl$_2$ (20 ml) was exposed to air for three weeks. Removal of the solvent in high vacuum and recrystallization from a mixture of CH$_2$Cl$_2$ and *n*-hexane (1:1 *v*/*v*) led to the isolation of the title compound, (I), as colourless crystals (0.098 g, 63%).

Crystal data

[PtBrH(C$_{18}$H$_{15}$P)$_2$]
M_r = 800.55
Monoclinic, $C2/c$
a = 15.2835 (2) Å
b = 9.5241 (1) Å
c = 22.5045 (4) Å
β = 106.618 (1)°
V = 3138.97 (8) Å^3
Z = 4
D_x = 1.694 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 5.87 mm^{-1}
T = 173 (2) K
Needle, colourless
0.08 × 0.07 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\rm min}$ = 0.651, $T_{\rm max}$ = 0.799
37736 measured reflections
3048 independent reflections
2705 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.081
$\theta_{\rm max}$ = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.051
S = 1.07
3048 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0152P)^2 + 11.9998P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 1.27 e Å^{-3}
$\Delta\rho_{\rm min}$ = −1.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.00053 (4)

Table 1
Selected geometric parameters (Å, °).

Pt1—P1	2.2827 (9)	P1—C1	1.817 (4)
Pt1—Br1	2.5229 (6)	P1—C7	1.833 (4)
Pt1—H1	1.61	P1—C13	1.825 (4)
P1—Pt1—P1$^{\rm i}$	170.81 (5)	C7—P1—Pt1	113.4 (1)
Br1—Pt1—H1	180	C13—P1—Pt1	115.5 (1)
P1—Pt1—Br1	94.60 (2)	C1—P1—C13	104.5 (2)
P1—Pt1—H1	85	C1—P1—C7	104.8 (2)
C1—P1—Pt1	113.3 (1)	C13—P1—C7	104.2 (2)

Symmetry code: (i) $-x, y, -z + \frac{3}{2}$.

Aromatic H atoms were placed in idealized positions in a riding model, with C—H = 0.95 Å, and refined with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The hydride atom (H1) was located in a difference electron-density map and refined with the constraint $U_{\rm iso}$(H1) = $U_{\rm eq}$(Pt1). The highest peak is located 0.65 Å from atom Pt1 and the deepest hole 0.03 Å from Pt1.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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supporting information

Acta Cryst. (2007). E**63**, m244–m245   [doi:10.1107/S160053680605358X]

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S1. Comment

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S2. Experimental

Compound (I) was prepared by the hydrolysis of a dichloromethane solution of *cis*-[Pt(1-pentenyl)Br(PPh_3)$_2$] on exposure to air (Sivaramakrishna & Moss, 2006). This is probably due to the breaking of the *M*—C bond through *β*-hydride elimination to form the Pt—H bond, and also isomerization from *cis* to *trans* (see scheme). ^{1}H NMR showed the Pt—H signals as a triplet at −14.8 p.p.m. with platinum satellites ($J_{Pt—H}$ = 1240 Hz and $J_{P—H}$ = 25.6 Hz). ^{31}P NMR indicated a singlet at 28.8 p.p.m. with platinum satellites ($J_{Pt—P}$ = 2988 Hz). A solution containing *cis*-(PPh_3)$_2$Pt($CH_2CH_2CH_2CH{=}CH_2$)Br (0.168 g, 0.193 mmol) in CH_2Cl_2 (20 ml) was exposed to air for 3 weeks. Removal of the solvent in high vacuum and recrystallization from a mixture of CH_2Cl_2 and n-hexane (1:1 *v*/*v*) led to the isolation of the title compound, (I), as colourless crystals (0.098 g, 63%). Analysis, calculated for $C_{36}H_{31}BrP_2Pt$: C 54.01, H 3.90, C 53.82, H 3.84%; **Actual results?**; M^+ = 800, Pt(PPh_3)$_2^+$ = 719.

S3. Refinement

Aromatic H atoms were placed in idealized positions in a riding model, with C—H = 0.95 Å, and refined with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The hydride atom (H1) was located in a difference electron-density map and refined with $U_{\rm iso}$(H1) = $U_{\rm eq}$(Pt1).

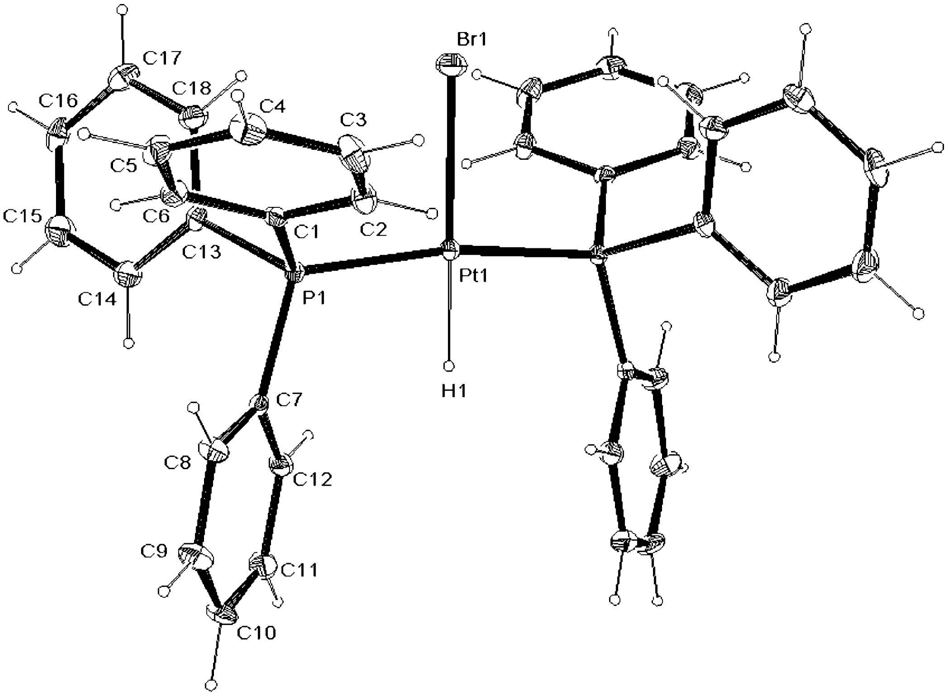

Figure 1

The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary size.

trans-Bromohydridobis(triphenylphosphine)platinum(II)

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c = 22.5045 (4) Å
β = 106.618 (1)°
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Z = 4
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T = 173 K
Needle, colourless
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3048 independent reflections
2705 reflections with *I* > 2*σ*(*I*)
$R_{\rm int}$ = 0.081
$\theta_{\rm max}$ = 26.0°, $\theta_{\rm min}$ = 3.4°
h = −18→18
k = −11→11
l = −27→27

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.028$	$w = 1/[\sigma^2(F_o^2) + (0.0152P)^2 + 11.9998P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.051$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 1.27$ e Å^{-3}
3048 reflections	$\Delta\rho_{min} = -1.21$ e Å^{-3}
183 parameters	Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.00053 (4)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Experimental. Half sphere of data collected using *COLLECT* strategy (Nonius, 2000). Crystal to detector distance = 30 mm; combination of φ and ω scans of 1.2°, 30 s per °, 2 iterations.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pt1	0.0000	0.01637 (2)	0.7500	0.01499 (9)
Br1	0.0000	−0.24853 (6)	0.7500	0.03449 (17)
P1	0.11219 (6)	0.03558 (9)	0.84173 (4)	0.0140 (2)
C7	0.1313 (2)	0.2163 (4)	0.87055 (16)	0.0155 (8)
C12	0.1362 (3)	0.3214 (4)	0.82916 (18)	0.0206 (9)
H12	0.1344	0.2974	0.7878	0.025*
C2	−0.0015 (3)	−0.0986 (4)	0.90009 (17)	0.0216 (9)
H2	−0.0489	−0.0743	0.8640	0.026*
C6	0.1570 (3)	−0.1000 (4)	0.95768 (17)	0.0198 (8)
H6	0.2187	−0.0769	0.9610	0.024*
C1	0.0882 (2)	−0.0625 (4)	0.90455 (16)	0.0154 (8)
C4	0.0466 (3)	−0.2061 (4)	1.00064 (18)	0.0269 (10)
H4	0.0323	−0.2555	1.0333	0.032*
C8	0.1358 (3)	0.2530 (4)	0.93120 (18)	0.0223 (9)
H8	0.1346	0.1820	0.9606	0.027*
C5	0.1358 (3)	−0.1705 (4)	1.00553 (18)	0.0236 (9)
H5	0.1829	−0.1944	1.0419	0.028*
C9	0.1421 (3)	0.3932 (4)	0.9488 (2)	0.0282 (10)
H9	0.1442	0.4177	0.9901	0.034*
C3	−0.0222 (3)	−0.1697 (4)	0.94788 (19)	0.0289 (10)
H3	−0.0838	−0.1936	0.9446	0.035*

C13	0.2250 (2)	−0.0249 (4)	0.84063 (15)	0.0167 (8)
C10	0.1453 (3)	0.4967 (4)	0.90715 (19)	0.0286 (9)
H10	0.1486	0.5925	0.9194	0.034*
C11	0.1439 (3)	0.4613 (4)	0.8472 (2)	0.0286 (10)
H11	0.1481	0.5324	0.8186	0.034*
C18	0.2372 (3)	−0.1661 (4)	0.82942 (19)	0.0259 (10)
H18	0.1868	−0.2287	0.8224	0.031*
C14	0.2997 (3)	0.0649 (4)	0.8499 (2)	0.0292 (10)
H14	0.2930	0.1619	0.8574	0.035*
C17	0.3213 (3)	−0.2163 (4)	0.82838 (19)	0.0280 (10)
H17	0.3288	−0.3133	0.8212	0.034*
C16	0.3943 (3)	−0.1264 (4)	0.83772 (19)	0.0282 (10)
H16	0.4522	−0.1612	0.8369	0.034*
C15	0.3835 (3)	0.0132 (5)	0.8481 (2)	0.0360 (11)
H15	0.4340	0.0752	0.8542	0.043*
H1	0.0000	0.1854	0.7500	0.015*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.01540 (12)	0.01460 (13)	0.01317 (12)	0.000	0.00123 (8)	0.000
Br1	0.0440 (4)	0.0169 (3)	0.0312 (4)	0.000	−0.0074 (3)	0.000
P1	0.0142 (5)	0.0144 (5)	0.0125 (5)	0.0004 (4)	0.0023 (4)	0.0001 (4)
C7	0.0106 (18)	0.0151 (18)	0.020 (2)	0.0009 (15)	0.0028 (16)	0.0013 (16)
C12	0.024 (2)	0.018 (2)	0.016 (2)	0.0000 (16)	0.0009 (17)	−0.0009 (16)
C2	0.020 (2)	0.028 (2)	0.016 (2)	−0.0023 (17)	0.0038 (17)	−0.0014 (17)
C6	0.020 (2)	0.020 (2)	0.020 (2)	0.0008 (16)	0.0064 (17)	0.0006 (16)
C1	0.018 (2)	0.0125 (17)	0.0154 (19)	−0.0010 (15)	0.0044 (16)	−0.0028 (14)
C4	0.037 (3)	0.025 (2)	0.022 (2)	−0.0045 (19)	0.014 (2)	0.0047 (18)
C8	0.028 (2)	0.019 (2)	0.021 (2)	−0.0038 (16)	0.0073 (18)	−0.0026 (16)
C5	0.029 (2)	0.023 (2)	0.017 (2)	0.0047 (18)	0.0048 (18)	0.0050 (16)
C9	0.036 (3)	0.024 (2)	0.027 (2)	−0.0051 (19)	0.012 (2)	−0.0097 (18)
C3	0.027 (2)	0.037 (2)	0.024 (2)	−0.0137 (19)	0.010 (2)	−0.0016 (19)
C13	0.0176 (19)	0.0220 (19)	0.0092 (17)	0.0030 (16)	0.0018 (14)	0.0026 (15)
C10	0.029 (2)	0.015 (2)	0.040 (3)	−0.0067 (18)	0.0069 (19)	−0.0074 (19)
C11	0.029 (2)	0.020 (2)	0.031 (2)	−0.0041 (17)	0.0008 (19)	0.0062 (18)
C18	0.026 (2)	0.018 (2)	0.034 (3)	0.0017 (17)	0.009 (2)	0.0008 (17)
C14	0.019 (2)	0.024 (2)	0.043 (3)	−0.0001 (17)	0.007 (2)	−0.0056 (19)
C17	0.029 (2)	0.026 (2)	0.031 (2)	0.0096 (19)	0.013 (2)	0.0018 (19)
C16	0.017 (2)	0.044 (3)	0.024 (2)	0.0111 (19)	0.0073 (18)	0.0023 (19)
C15	0.019 (2)	0.036 (3)	0.054 (3)	−0.003 (2)	0.011 (2)	−0.007 (2)

Geometric parameters (Å, º)

Pt1—P1	2.2827 (9)	C8—C9	1.389 (5)
Pt1—Br1	2.5229 (6)	C8—H8	0.9500
Pt1—H1	1.610	C5—H5	0.9500
P1—C1	1.817 (4)	C9—C10	1.370 (6)

P1—C7	1.833 (4)	C9—H9	0.9500
P1—C13	1.825 (4)	C3—H3	0.9500
Pt1—P1^{i}	2.2827 (9)	C13—C18	1.391 (5)
C7—C12	1.384 (5)	C13—C14	1.393 (5)
C7—C8	1.391 (5)	C10—C11	1.384 (6)
C12—C11	1.388 (5)	C10—H10	0.9500
C12—H12	0.9500	C11—H11	0.9500
C2—C3	1.382 (5)	C18—C17	1.378 (6)
C2—C1	1.388 (5)	C18—H18	0.9500
C2—H2	0.9500	C14—C15	1.384 (6)
C6—C5	1.383 (5)	C14—H14	0.9500
C6—C1	1.394 (5)	C17—C16	1.375 (6)
C6—H6	0.9500	C17—H17	0.9500
C4—C5	1.379 (6)	C16—C15	1.368 (6)
C4—C3	1.387 (6)	C16—H16	0.9500
C4—H4	0.9500	C15—H15	0.9500

P1—Pt1—P1^{i}	170.81 (5)	C4—C5—C6	120.2 (4)
Br1—Pt1—H1	180.0	C4—C5—H5	119.9
P1—Pt1—Br1	94.60 (2)	C6—C5—H5	119.9
P1—Pt1—H1	85.4	C10—C9—C8	120.7 (4)
C1—P1—Pt1	113.3 (1)	C10—C9—H9	119.6
C7—P1—Pt1	113.4 (1)	C8—C9—H9	119.6
C13—P1—Pt1	115.5 (1)	C2—C3—C4	120.1 (4)
C1—P1—C13	104.5 (2)	C2—C3—H3	119.9
C1—P1—C7	104.8 (2)	C4—C3—H3	119.9
C13—P1—C7	104.2 (2)	C18—C13—C14	118.3 (4)
P1^{i}—Pt1—Br1	94.60 (2)	C18—C13—P1	118.9 (3)
P1^{i}—Pt1—H1	85.4	C14—C13—P1	122.8 (3)
C12—C7—C8	118.8 (3)	C9—C10—C11	119.8 (4)
C12—C7—P1	118.3 (3)	C9—C10—H10	120.1
C8—C7—P1	122.7 (3)	C11—C10—H10	120.1
C7—C12—C11	120.9 (4)	C10—C11—C12	119.7 (4)
C7—C12—H12	119.6	C10—C11—H11	120.1
C11—C12—H12	119.6	C12—C11—H11	120.1
C3—C2—C1	120.4 (4)	C17—C18—C13	120.8 (4)
C3—C2—H2	119.8	C17—C18—H18	119.6
C1—C2—H2	119.8	C13—C18—H18	119.6
C5—C6—C1	120.3 (4)	C15—C14—C13	120.2 (4)
C5—C6—H6	119.8	C15—C14—H14	119.9
C1—C6—H6	119.8	C13—C14—H14	119.9
C2—C1—C6	119.0 (3)	C16—C17—C18	120.1 (4)
C2—C1—P1	119.1 (3)	C16—C17—H17	119.9
C6—C1—P1	121.9 (3)	C18—C17—H17	119.9
C5—C4—C3	119.8 (4)	C15—C16—C17	119.9 (4)
C5—C4—H4	120.1	C15—C16—H16	120.0
C3—C4—H4	120.1	C17—C16—H16	120.0
C9—C8—C7	120.0 (4)	C16—C15—C14	120.6 (4)

C9—C8—H8	120.0	C16—C15—H15	119.7
C7—C8—H8	120.0	C14—C15—H15	119.7

Br1—Pt1—P1—C1	−53.40 (13)	C3—C4—C5—C6	−0.8 (6)
Br1—Pt1—P1—C13	67.11 (13)	C1—C6—C5—C4	1.0 (6)
Br1—Pt1—P1—C7	−172.67 (13)	C7—C8—C9—C10	−1.0 (6)
C1—P1—C7—C12	−169.4 (3)	C1—C2—C3—C4	−0.3 (6)
C13—P1—C7—C12	81.1 (3)	C5—C4—C3—C2	0.4 (6)
Pt1—P1—C7—C12	−45.3 (3)	C1—P1—C13—C18	61.1 (3)
C1—P1—C7—C8	6.9 (4)	C7—P1—C13—C18	170.8 (3)
C13—P1—C7—C8	−102.6 (3)	Pt1—P1—C13—C18	−64.1 (3)
Pt1—P1—C7—C8	131.0 (3)	C1—P1—C13—C14	−119.6 (3)
C8—C7—C12—C11	−1.2 (6)	C7—P1—C13—C14	−10.0 (4)
P1—C7—C12—C11	175.3 (3)	Pt1—P1—C13—C14	115.2 (3)
C3—C2—C1—C6	0.5 (6)	C8—C9—C10—C11	−1.1 (6)
C3—C2—C1—P1	−178.2 (3)	C9—C10—C11—C12	2.0 (6)
C5—C6—C1—C2	−0.8 (5)	C7—C12—C11—C10	−0.9 (6)
C5—C6—C1—P1	177.8 (3)	C14—C13—C18—C17	0.9 (6)
C13—P1—C1—C2	−147.8 (3)	P1—C13—C18—C17	−179.8 (3)
C7—P1—C1—C2	102.9 (3)	C18—C13—C14—C15	−0.3 (6)
Pt1—P1—C1—C2	−21.3 (3)	P1—C13—C14—C15	−179.6 (3)
C13—P1—C1—C6	33.6 (3)	C13—C18—C17—C16	−0.8 (6)
C7—P1—C1—C6	−75.7 (3)	C18—C17—C16—C15	0.1 (6)
Pt1—P1—C1—C6	160.1 (3)	C17—C16—C15—C14	0.5 (7)
C12—C7—C8—C9	2.1 (6)	C13—C14—C15—C16	−0.4 (7)
P1—C7—C8—C9	−174.1 (3)		

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