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trans-Bromohydridobis(triphenylphosphine)-platinum toluene hemisolvate

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In the crystal structure of $[PtBrH(C_{18}H_{15}P)_2]\cdot 0.5C_7H_8$, the Pt atom has a distorted square-planar environment, with the coordination sphere comprising hydride, bromide and mutually *trans* triphenylphosphine ligands.

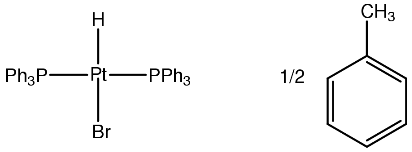
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
H-atom completeness 89%
Disorder in solvent or counterion
R factor = 0.032
wR factor = 0.071
Data-to-parameter ratio = 20.4

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

Comment

The title complex, *trans*-$(Ph_3P)_2Pt(H)Br$, initially reported by Carr *et al.* (1985), was prepared in this case by hydrolysis of the platinum(II) boryl complex *trans*-$(Ph_3P)_2Pt[B(Mes)Br]Br$ (Mes = 2,4,6-$Me_3C_6H_2$) (Coombs & Aldridge, 2003).



(I)

The crystal structure of *trans*-$(Ph_3P)_2Pt(H)Br\cdot 0.5C_6H_5CH_3$, (I), at 150 K consists of discrete molecular units. The Pt atom has a distorted square-planar environment [sum of angles at Pt = 360 (4)°] and a coordination sphere comprising hydride, bromide and mutually *trans* triphenylphosphine ligands. The Pt—P [mean 2.278 (1) Å] and Pt—Br [2.535 (1) Å] distances are within the expected ranges for similar platinum(II) complexes (Owston *et al.*, 1960; Bender *et al.*, 1984), and the bending of the P1—Pt1—P2 angle from linearity [172.73 (3)°] presumably reflects the differing steric requirements of the bromide and hydride ligands. The Pt—H distance is among the shortest reported to date, being significantly shorter than that found in *trans*-$(^{i}Pr_3P)_2Pt(H)Cl$ (1.846 Å; Robertson *et al.*, 1986), although comparable to that reported for the triazacyclononane complex $[(tacn)PtMe_2H]^+$ (1.380 Å; Prokopchuk *et al.*, 1999).

Experimental

The title compound was prepared by the hydrolysis of a toluene solution of *trans*-$(Ph_3P)_2Pt[B(Mes)Br]Br$ on exposure to air. A solution containing 137 mg (0.13 mmol) of *trans*-$(Ph_3P)_2Pt[B(Mes)Br]Br$ in *ca* 10 ml of toluene was exposed to air for a period of 9 d, at which point monitoring of the reaction mixture by ^{31}P NMR revealed that the sole phosphorus-containing product in solution was *trans*-$(Ph_3P)_2Pt(H)Br$. Removal of solvent *in vacuo* and recrystallization from toluene/hexane (50:50) led to the isolation of the title compound as colourless crystals suitable for X-ray diffraction. Further synthetic details are recorded in the CIF.

Crystal data

$[PtBrH(C_{18}H_{15}P)_2]\cdot 0.5C_7H_8$
$M_r = 846.61$
Monoclinic, $P2_1/c$
$a = 11.514$ (2) Å
$b = 13.447$ (3) Å
$c = 22.449$ (5) Å
$\beta = 97.01$ (3)°
$V = 3449.8$ (12) Å^3
$Z = 4$

$D_x = 1.630$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 55 096 reflections
$\theta = 2.9$–27.5°
$\mu = 5.35$ mm^{-1}
$T = 150$ (2) K
Prism, colourless
0.20 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.325$, $T_{\max} = 0.461$
55 828 measured reflections
7893 independent reflections

6645 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.083$
$\theta_{\max} = 27.5°$
$h = -14 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.071$
$S = 1.02$
7893 reflections
386 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 8.05P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.08$ e Å^{-3}
$\Delta\rho_{\min} = -0.97$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00126 (7)

Table 1
Selected geometric parameters (Å, °).

Pt1—P2	2.2749 (12)	Pt1—Br1	2.5347 (8)
Pt1—P1	2.2805 (12)	Pt1—H99	1.38 (6)
P2—Pt1—P1	172.73 (3)	P2—Pt1—H99	88 (2)
P2—Pt1—Br1	92.10 (3)	P1—Pt1—H99	85 (2)
P1—Pt1—Br1	94.57 (3)	Br1—Pt1—H99	177 (2)

The H atom, H99, attached to the Pt1 centre was located in a difference Fourier synthesis and refined isotropically with no restraints. Phenyl H atoms were placed in idealized positions (C—H = 0.95 Å) and refined using a riding model with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. A disordered molecule of toluene was found in the crystal lattice, residing on a center of symmetry (0.5 molecule per asymmetric unit). The disorder was successfully modelled and atoms C37–C41 were refined isotropically without attached H atoms. The maximum electron-density peak is located near the Pt1 atom.

Data collection: *COLLECT* (Hooft, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

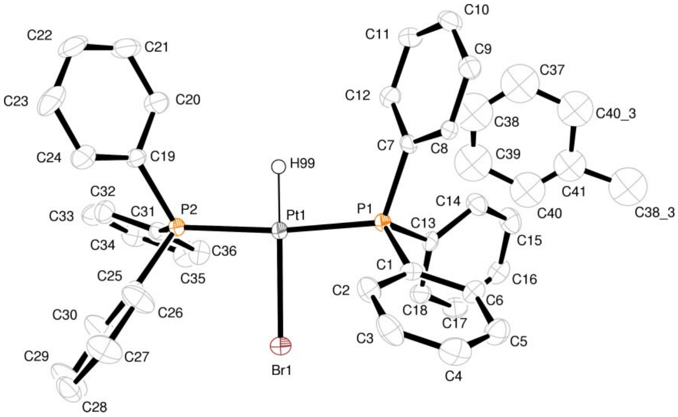


Figure 1
View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. The H atoms of the triphenylphosphine ligands have been omitted for clarity.

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