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trans-Diiodobis(triphenylphosphine)palladium(II) bis(tetrahydrofuran) solvate

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Key indicators

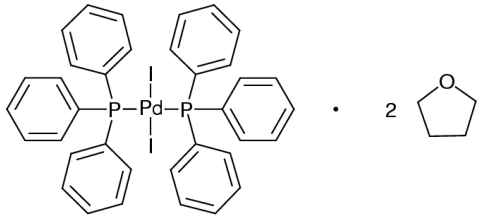
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.036
wR factor = 0.073
Data-to-parameter ratio = 20.6

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

The title compound, *trans*-$[PdI_2(PPh_3)_2]\cdot 2C_4H_8O$, has a square-planar coordination of the Pd atom, which lies at an inversion centre. The crystal is isostructural with that of the CH_2Cl_2 solvate.

Comment

During an attempted synthesis of $[PdI(CO_2Me)(PPh_3)_2]$, we obtained, as an incidental by-product, *trans*-$[PdI_2(PPh_3)_2]\cdot 2THF$, where THF is tetrahydrofuran, (I), the crystal structure of which is reported herein. The Pd atom occupies a crystallographic inversion centre, hence its tetragonal coordination is rigorously planar (Fig. 1). The tetrahydrofuran molecule of crystallization in (I) occupies a general position. Its C2 atom is disordered between positions *A* and *B* in a 0.6:0.4 ratio.



(I)

Compound (I) can be described as a pseudo-polymorph of $[PdI_2(PPh_3)_2]\cdot 2CH_2Cl_2$ (Debaerdemaeker *et al.*, 1973) and $[PtI_2(PPh_3)_2]\cdot 2CH_2Cl_2$ (Boag *et al.*, 1991) which contain different solvents of crystallization, but have similar crystal lattice parameters and molecular structures. In these two structures, as well as in solvent-free *trans*-$[PdI_2(PPh_3)_2]$ (Cave *et al.*, 1999), the metal atom also lies at an inversion centre. However, in the crystal structure of *trans*-$[PdI_2(PPh_3)_2]\cdot CHCl_3$ (Kubota *et al.*, 1991), the Pd atom lies on a crystallographic twofold axis and the I—Pd—I angle is reduced to 168.9 (1)°, although the P—Pd—P angle of 178.8 (1)° remains close to 180°. Thus, the planarity of the metal coordination can be easily distorted under the influence of the crystal packing. An even stronger distortion (of a distinctly tetrahedral character) was observed in *trans*-$[PdI_2(PPhMe_2)_2]$ (Bailey & Mason, 1968).

The axial coordination sites of the Pd atom in (I) (as in its analogues, studied earlier) are masked by the β-H atoms of the phenyl rings, namely those at C26, C36 and their inversion equivalents. The Pd···H distances of 3.01 and 3.16 Å (for the calculated H-atom positions) are too long for any bonding interactions. The angles between the Pd···H vectors and the PdI_2P_2 coordination plane amount to 56 and 59°.

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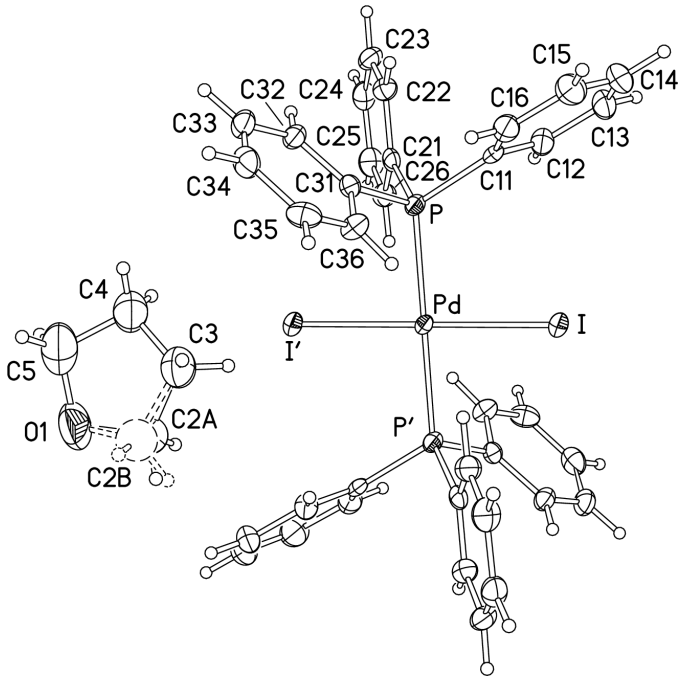

Figure 1
Molecules in the structure of *trans*-$[PdI_2(PPh_3)_2]\cdot 2THF$, (I). Displacement ellipsoids are drawn at the 50% probability level. Primed atoms are symmetrically dependent *via* the inversion centre.

Experimental

Solid $[PdCl(CO_2Me)(PPh_3)]$ (0.2 g, 0.27 mmol) and $AgBF_4$ (0.53 g, 0.27 mmol) were dissolved in dry degassed acetonitrile (40 ml), added to $[PdCl(CO_2Me)(dppe)]$ (0.18 g, 0.30 mmol), and the resulting solution stirred for 2 h at room temperature in a flask protected from light. The precipitated AgCl was filtered off from the orange solution, KI (0.043 g, 0.26 mmol) was added to the filtrate at 273 K and the mixture was stirred for 1 h. The colourless precipitate (KBF_4) was filtered off, the solution was reduced in volume and then dried *in vacuo*, the residue was washed with ether and recrystallized from THF. Found: C 49.48, H 3.35%. $C_{36}H_{30}I_2P_2Pd$ requires: C 48.87, H 3.42%. $^{31}P\{^1H\}$ NMR in MeCN: δ = 19.9 p.p.m. (s). ^1H NMR in $CDCl_3$: 7.2–7.9 p.p.m. (30H, *m*, Ph).

Crystal data

$[PdI_2(C_{18}H_{15}P)_2]\cdot 2C_4H_8O$
M_r = 1028.95
Monoclinic, $P2_1/c$
a = 12.011 (3) Å
b = 20.234 (4) Å
c = 8.625 (2) Å
β = 90.35 (1)°
V = 2096.1 (8) Å^3
Z = 2
D_x = 1.630 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 512 reflections
θ = 12–20°
μ = 2.03 mm^{-1}
T = 150 (2) K
Block, orange
0.20 × 0.15 × 0.12 mm

Data collection

SMART 1 K CCD area-detector diffractometer
ω scans
Absorption correction: by integration (*XPREP SHELXTL*; Siemens, 1995), R_{int} = 0.061 before correction
T_{min} = 0.709, T_{max} = 0.795
15 358 measured reflections
4806 independent reflections
4029 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 27.5°
$h = -11 \rightarrow 16$
$k = -25 \rightarrow 28$
$l = -12 \rightarrow 12$
Intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.073
S = 1.14
4806 reflections
233 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 5.8022P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0007 (1)

Table 1
Selected geometric parameters (Å, °).

I—Pd	2.6081 (5)	P—C21	1.824 (4)
Pd—P	2.3384 (11)	P—C11	1.840 (4)
P—C31	1.820 (4)		
P^{i}—Pd—I	87.25 (3)	C21—P—C11	100.76 (19)
P—Pd—I	92.75 (3)	C31—P—Pd	108.96 (14)
C31—P—C21	109.51 (18)	C21—P—Pd	114.00 (14)
C31—P—C11	103.56 (19)	C11—P—Pd	119.24 (13)

Symmetry code: (i) $1-x, 1-y, 1-z$.

Pseudo-orthorhombic twinning with the $(\bar{1}00,0\bar{1}0,001)$ law was observed, the contribution of the second crystal was refined to 3.25 (4)%, reducing $R[F^2 > 2\sigma(F^2)]$ from 0.053 to 0.036. All H atoms were treated as riding, with Csp^2—H and Csp^3—H bond distances of 0.95 and 0.99 Å, respectively.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL* (Siemens, 1995); software used to prepare material for publication: *SHELXTL*.

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