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# Solvent-free [1,2-bis(diphenylphosphino)ethane-*P*,*P*′]-diiodopalladium(II)

**Andrei S. Batsanov,* Judith A. K. Howard, Graham S. Robertson and Melvin Kilner**

Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, England

Correspondence e-mail: a.s.batsanov@durham.ac.uk

**Key indicators**

Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.009 Å
$R$ factor = 0.041
$wR$ factor = 0.080
Data-to-parameter ratio = 21.1

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

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The title complex, $[PdI_2(dppe)]$, where dppe is 1,2-bis(diphenylphosphino)ethane ($C_{26}H_{24}P_2$), has a square-planar coordination of the Pd atom with a small tetrahedral distortion and an envelope conformation of the five-membered metallocycle, similar to those observed in $[PdI_2(dppe)]\cdot CHCl_3$.

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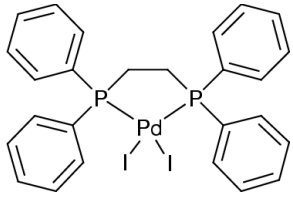
## Comment

The title complex, (I), was characterized crystallographically by Oberhauser *et al.* (1997) in the form of a 1:1 solvate with $CHCl_3$, (II). During an attempted synthesis of $[PdI(CO_2Me)(dppe)]$ we obtained, as an incidental by-product, solvent-free crystals of (I), the structure of which is reported herein.




(I)

The Pd atom in (I) (Fig. 1) has a square-planar coordination with a tetrahedral distortion: the $PdI_2$ plane is twisted by 5.6 (1)° with respect to the $PdP_2$ plane. A similar distortion was observed in (II) also. The five-membered metallocycle in (I) adopts an envelope conformation: the Pd, P1, P2 and C1 atoms are coplanar within 0.02 Å, while C2 deviates from their mean plane by 0.72 Å. Probably, this asymmetric conformation induces a small, but significant, non-equivalence of the Pd—P bonds (by 0.016 Å, or 15 s.u.) and, through *trans*-influence, of the Pd—I bonds as well (by 0.016 Å, or 28 s.u.). An asymmetric conformation of the metallacycle (described as a distorted twist conformation) and similar asymmetry of the Pd—P and Pd—I bonds were observed in structure (II) as well.

The molecular geometry of (I) is similar to that of the platinum analogue, in the crystal structure of $[PtI_2(dppe)]\cdot I_2\cdot CH_2Cl_2$ (Parkin *et al.*, 1991).

## Experimental

Degassed solid $[PdCl(CO_2Me)(dppe)]$ (0.18 g, 0.30 mmol) and $AgBF_4$ (0.30 mmol) were dissolved in dry degassed acetonitrile (30 mol) and stirred for 2 h at 313 K in a flask protected from light. The precipitated AgCl was filtered off, the filtrate was added to KI (0.05 g, 0.31 mmol), the mixture was stirred overnight, acquiring an intense orange–yellow colour. The precipitated $KBF_4$ was filtered off, the solvent was removed under reduced pressure, the residue was

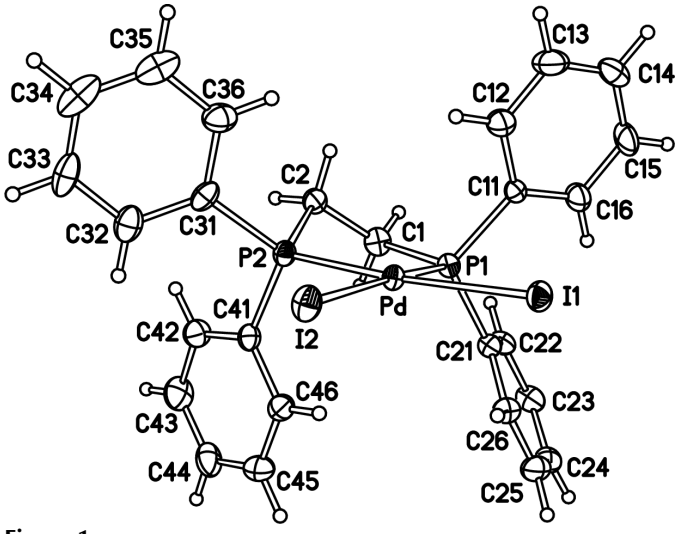



**Figure 1**
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

dried *in vacuo* and washed with ether and hexane, yielding some single crystals of X-ray quality. Found: C 40.64, H, 3.37%; $C_{26}H_{24}I_2P_2Pd$ requires: C 41.16, H 3.19%. $^{31}P\{^1H\}$ NMR in MeCN: $\delta$ = 66.2 p.p.m. (*s*). $^1H$ NMR in $CDCl_3$: $\delta$ = 1.7–1.9 (4H, *m*, $PCH_2CH_2P$), 7.2–7.5 p.p.m. (20H, *m*, Ph).

*Crystal data*

| | |
|---|---|
| $[PdI_2(C_{26}H_{24}P_2)]$ | $D_x$ = 1.962 Mg m$^{-3}$ |
| $M_r$ = 758.59 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 432 reflections |
| $a$ = 9.465 (1) Å | $\theta$ = 12–20° |
| $b$ = 19.821 (1) Å | $\mu$ = 3.26 mm$^{-1}$ |
| $c$ = 14.208 (1) Å | $T$ = 150 (2) K |
| $\beta$ = 105.51 (1)° | Plate, yellow |
| $V$ = 2568.4 (4) Å$^3$ | 0.30 × 0.12 × 0.06 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| SMART 1 K CCD area-detector diffractometer | 18 477 measured reflections |
| $\omega$ scans | 5903 independent reflections |
| Absorption correction: by integration (*XPREP SHELXTL*; Siemens, 1995), $R_{int}$ = 0.107 before correction | 4690 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.355, $T_{max}$ = 0.832 | $R_{int}$ = 0.056 |
| | $\theta_{max}$ = 27.5° |
| | $h = -12 \rightarrow 12$ |
| | $k = -27 \rightarrow 27$ |
| | $l = -20 \rightarrow 20$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0102P)^2 + 11.8040P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.041 | $(\Delta/\sigma)_{max}$ < 0.001 |
| $wR(F^2)$ = 0.080 | $\Delta\rho_{max}$ = 0.66 e Å$^{-3}$ |
| $S$ = 1.16 | $\Delta\rho_{min}$ = −1.05 e Å$^{-3}$ |
| 5903 reflections | |
| 280 parameters | |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Pd—P2 | 2.2612 (14) | P1—C21 | 1.818 (5) |
| Pd—P1 | 2.2769 (14) | P1—C1 | 1.850 (6) |
| Pd—I2 | 2.6514 (6) | P2—C41 | 1.798 (5) |
| Pd—I1 | 2.6678 (5) | P2—C31 | 1.815 (5) |
| P1—C11 | 1.815 (5) | P2—C2 | 1.834 (5) |
| P2—Pd—P1 | 85.08 (5) | C11—P1—Pd | 113.39 (17) |
| P2—Pd—I2 | 90.30 (4) | C21—P1—Pd | 117.44 (17) |
| P1—Pd—I2 | 173.71 (4) | C1—P1—Pd | 108.34 (18) |
| P2—Pd—I1 | 173.81 (4) | C41—P2—C31 | 109.8 (3) |
| P1—Pd—I1 | 90.09 (4) | C41—P2—C2 | 103.1 (3) |
| I2—Pd—I1 | 94.790 (18) | C31—P2—C2 | 103.4 (2) |
| C11—P1—C21 | 107.8 (2) | C41—P2—Pd | 110.99 (18) |
| C11—P1—C1 | 105.1 (2) | C31—P2—Pd | 121.39 (18) |
| C21—P1—C1 | 103.6 (2) | C2—P2—Pd | 106.18 (18) |

All H atoms were treated as riding, with C$sp^2$—H and C$sp^3$—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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