# metal-organic papers

## *trans*-Diiodobis(triisopropylphosphino)platinum(II)

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**Key indicators**

Single-crystal X-ray study
$T = 173$ K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.021
$wR$ factor = 0.025
Data-to-parameter ratio = 28.3

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

In the title compound, $[PtI_2(C_9H_{21}P)_2]$, the $Pt^{II}$ atom occupies an inversion centre and $C_i$ molecular symmetry results, with *trans* arrangements of iodo and of triisopropylphosphino ligands completing a square-planar coordination. The crystal packing reveals weak C—H···I interactions.

## Comment

During our ongoing investigations of the formation of heterometallomacrocyclic complexes (Constable *et al.*, 2005*a*,*b*), we have prepared a range of *trans*-$[PtI_2L_2]$ ($L$ = tertiary phosphine) complexes, including the title compound, (I). Although the synthesis and IR and $^1$H NMR spectroscopic properties of (I) have been reported (Leviston & Wallbridge, 1976), a search of the Cambridge Structural Database (CSD, Version 5.27; Allen, 2002; Bruno *et al.*, 2002) indicated that its structure had not previously been described.
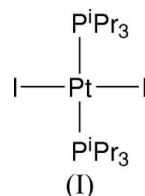


X-ray quality single crystals of (I) were grown from hot ethanol and a structure determination confirmed the *trans* arrangement of the ligands (Fig. 1). The Pt—P bond distance (Table 1) is slightly greater than the corresponding value in the
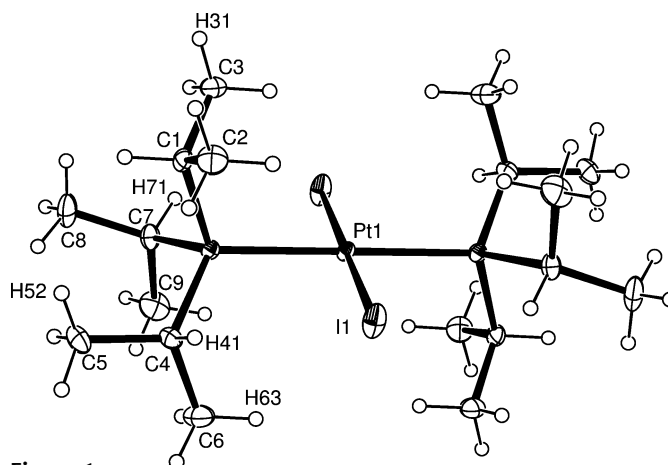


**Figure 1**
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitary radii. The Pt atom lies on an inversion centre. Unlabelled atoms are related to labelled atoms by $-x$, $-y$, $-z$.
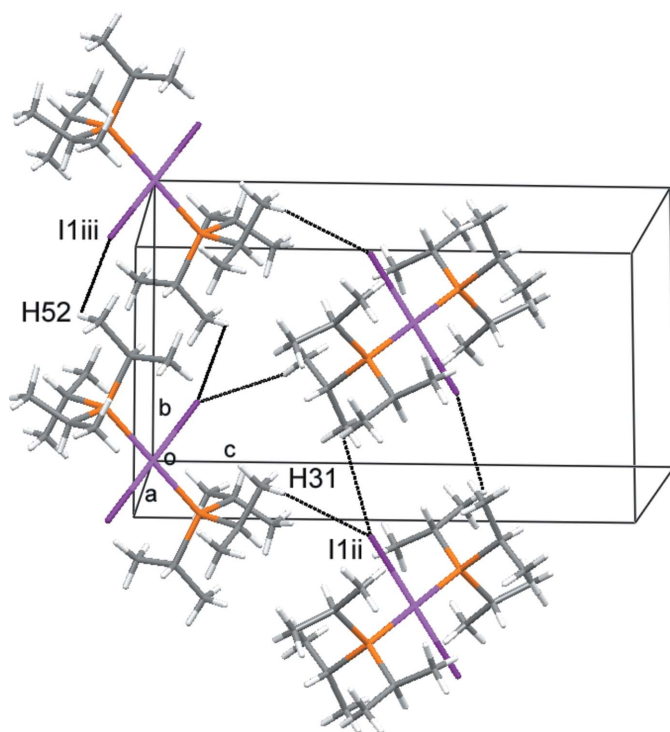
**Figure 2**
Short CH···I contacts between molecules of (I). Symmetry codes for intermolecular contacts: (ii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $-x, 1 - y, -z$.
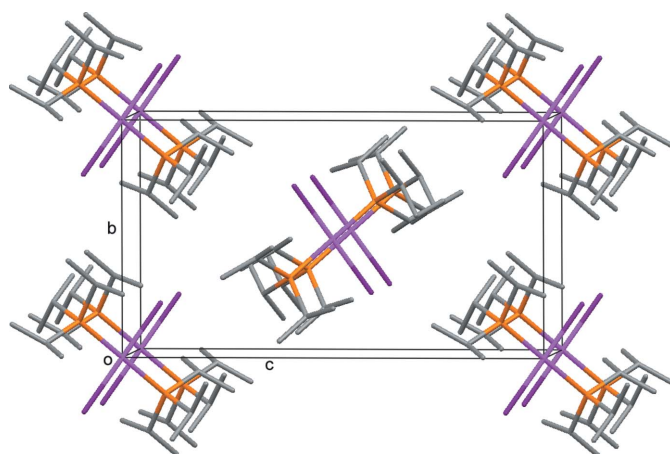


**Figure 3**
The packing of molecules of (I) in the unit cell, viewed approximately down the $a$ axis. H aoms have been omitted.

chloro derivative [2.339 (1) Å; Robertson et al., 1986]. The presence of an inversion centre at the Pt means that the coordinated P$^i$Pr₃ ligands are mutually staggered. Within a molecule, there are three symmetry-related pairs of short C—H···I contacts involving the Me₂C—H hydrogens (C4/H41···I1 = 2.93 Å, C4—H41···I1 = 117.0°; C7/H71···I1$^i$ = 2.76 Å, C7—H71···I1$^i$ = 128°) and one methyl H atom [C6/H63···I1$^i$ = 2.98 Å and C6—H63···I1$^i$ = 128°; symmetry code: (i) $-x, -y, -z$]. Molecules of (I) pack in a similar way to those of the chloro analogue (Robertson et al., 1986). Each I atom points towards two isopropyl substituents such that there are

two short contacts per I atom (Fig. 2): C3/H31···H1$^{ii}$ = 3.22 Å, C3—H31···I1$^{ii}$ = 122°; C5/H52···I1$^{iii}$ = 3.37 Å, C5—H52···I1$^{iii}$ = 113°; symmetry codes: (ii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $-x, 1 - y, -z$. The observed distances of H···I compared with the sum of the van der Waals radii of H and I of 3.35 Å. The presence of these weak C—H···I interactions contributes to the crystal packing (Fig. 3).

## Experimental

Compound (I) was prepared by a route based on that reported (Leviston & Wallbridge, 1976), using KI as the source of iodide.

*Crystal data*

| | |
|---|---|
| [PtI₂(C₉H₂₁P)₂] | $Z = 2$ |
| $M_r = 769.38$ | $D_x = 2.068$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 8.0337$ (1) Å | $\mu = 8.31$ mm$^{-1}$ |
| $b = 9.2664$ (1) Å | $T = 173$ K |
| $c = 16.6207$ (2) Å | Plate, orange |
| $\beta = 93.2395$ (8)° | $0.22 \times 0.20 \times 0.10$ mm |
| $V = 1235.33$ (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 13720 measured reflections |
| $\varphi$ and $\omega$ scans | 3616 independent reflections |
| Absorption correction: multi-scan | 3028 reflections with $I > 3\sigma(I)$ |
| DENZO/SCALEPACK | $R_{int} = 0.032$ |
| (Otwinowski & Minor, 1997) | $\theta_{max} = 30.0°$ |
| $T_{min} = 0.19$, $T_{max} = 0.44$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F$ | $w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/$ |
| $R[F > 2\sigma(F)] = 0.021$ | $[0.408T_o(x) + 0.241T_1(x) +$ |
| $wR(F) = 0.025$ | $0.196T_2(x)]$ |
| $S = 1.03$ | where $T_i$ are the Chebychev |
| 3028 reflections | polynomials and $x = F_c/F_{max}$ |
| 107 parameters | (Prince, 1982; Watkin, 1994) |
| H-atom parameters constrained | $(\Delta/\sigma)_{max} < 0.001$ |
| | $\Delta\rho_{max} = 0.78$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -1.84$ e Å$^{-3}$ |

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

| | | | |
|---|---|---|---|
| Pt1—I1 | 2.6150 (2) | P1—C4 | 1.864 (3) |
| Pt1—P1 | 2.3748 (7) | P1—C7 | 1.859 (3) |
| P1—C1 | 1.859 (3) | | |
| I1—Pt1—P1 | 88.614 (17) | Pt1—P1—C4 | 114.80 (10) |
| Pt1—P1—C1 | 109.96 (9) | Pt1—P1—C7 | 115.36 (10) |

All H atoms were treated as riding atoms, with C—H = 0.96 Å and $U_{iso}(H)$ between $1.0U_{eq}(C)$ and $1.2U_{eq}(C)$. Structure validation reports that the $U$ values of the iodine site were too large in relation to the Pt centre, causing the Hirshfeld test to fail. Assuming the iodine site to be partially contaminated by chlorine (presumably from the starting material), a disorder model has been refined using the Pt—Cl bond length from the analogous chloro compound (Robertson et al., 1986). The $U$ values passed the Hirshfeld test, but refining the occupancy to a value of less than 0.02 cannot be considered to be reliable. The deepest residual density hole is located approximately 0.6 Å from the iodine atom and about 3.0 Å from the metal centre.

Data collection: *COLLECT* (Nonius, 1997–2001).; cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduc-

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