

Edwin C. Constable, Catherine E. Housecroft,* Markus Neuburger, Silvia Schaffner and Ellen Shardlow

Department of Chemistry, University of Basel,
Spitalstrasse 51, CH 4056 Basel, Switzerland

Correspondence e-mail:
catherine.housecroft@unibas.ch

Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ 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>.

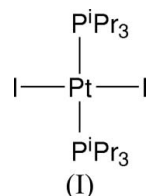
trans-Diiodobis(triisopropylphosphino)platinum(II)

In the title compound, $[\text{PtI}_2(\text{C}_9\text{H}_{21}\text{P})_2]$, 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 $\text{C}-\text{H}\cdots\text{I}$ interactions.

Received 18 July 2006
Accepted 7 August 2006

Comment

During our ongoing investigations of the formation of heterometallomacrocyclic complexes (Constable *et al.*, 2005*a,b*), we have prepared a range of *trans*- $[\text{PtI}_2\text{L}_2]$ (L = tertiary phosphine) complexes, including the title compound, (I). Although the synthesis and IR and ^1H 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

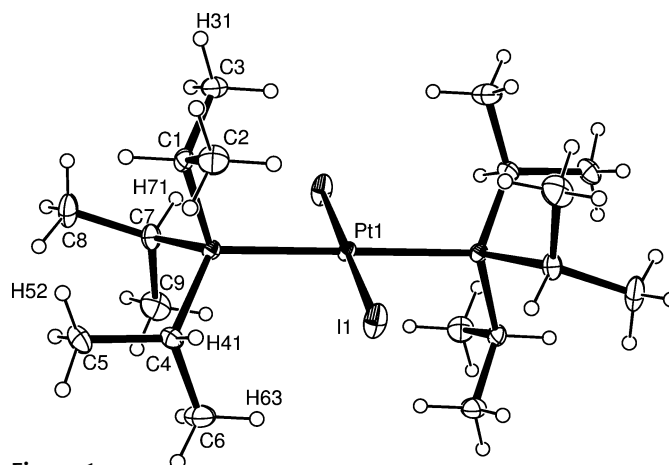


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. The Pt atom lies on an inversion centre. Unlabelled atoms are related to labelled atoms by $-x, -y, -z$.

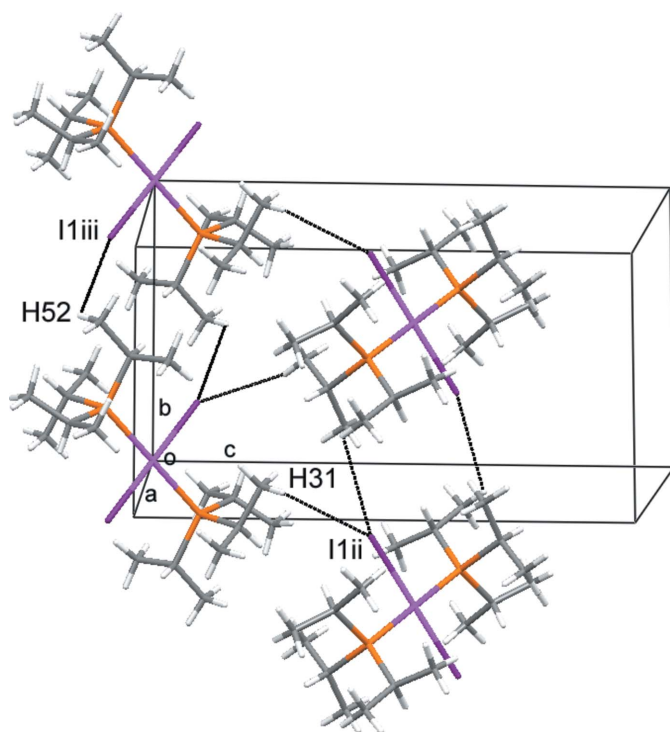


Figure 2
Short CH...I contacts between molecules of (I). Symmetry codes for intermolecular contacts: (ii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $-x, 1 - y, -z$.

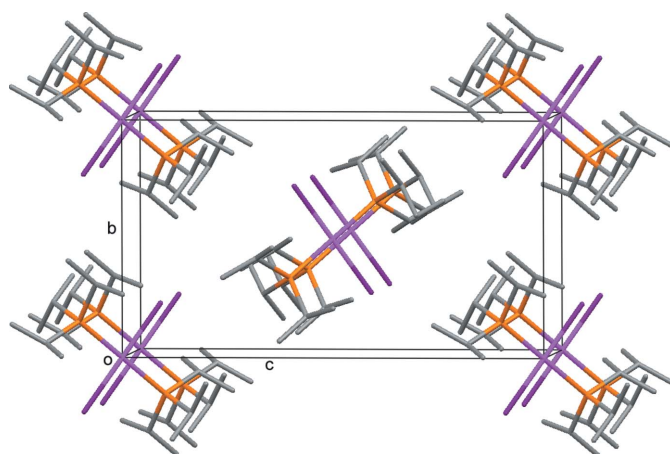


Figure 3
The packing of molecules of (I) in the unit cell, viewed approximately down the a axis. H atoms have been omitted.

chloro derivative [2.339 (1) Å; Robertson *et al.*, 1986]. The presence of an inversion centre at the Pt means that the coordinated P^iPr_3 ligands are mutually staggered. Within a molecule, there are three symmetry-related pairs of short C—H...I contacts involving the Me_2C-H hydrogens (C4/H41...I1 = 2.93 Å, C4—H41...I1 = 117.0°; C7/H71...I1ⁱ = 2.76 Å, C7—H71...I1ⁱ = 128°) and one methyl H atom [C6/H63...I1ⁱ = 2.98 Å and C6—H63...I1ⁱ = 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...I1ⁱⁱⁱ = 3.22 Å, C3—H31...I1ⁱⁱⁱ = 122°; C5/H52...I1ⁱⁱⁱ = 3.37 Å, C5—H52...I1ⁱⁱⁱ = 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

[PtI ₂ (C ₉ H ₂₁ P) ₂]	$Z = 2$
$M_r = 769.38$	$D_x = 2.068 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.0337$ (1) Å	$\mu = 8.31 \text{ mm}^{-1}$
$b = 9.2664$ (1) Å	$T = 173 \text{ K}$
$c = 16.6207$ (2) Å	Plate, orange
$\beta = 93.2395$ (8)°	$0.22 \times 0.20 \times 0.10 \text{ mm}$
$V = 1235.33$ (3) Å ³	

Data collection

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

Refinement

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

Table 1

Selected geometric parameters (Å, °).

Pt1—I1	2.6150 (2)	Pt1—C4	1.864 (3)
Pt1—P1	2.3748 (7)	Pt1—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 $U_{\text{iso}}(\text{H})$ between $1.0U_{\text{eq}}(\text{C})$ and $1.2U_{\text{eq}}(\text{C})$. 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-

tion: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

We thank the Swiss National Science Foundation and the University of Basel for financial support.

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Constable, E. C., Housecroft, C. E., Neuburger, M., Schaffner, S. & Shardlow, E. J. (2005a). *Dalton Trans.* pp. 234–235.
- Constable, E. C., Housecroft, C. E., Neuburger, M., Schaffner, S. & Shardlow, E. J. (2005b). *Inorg. Chem. Commun.* **8**, 743–745.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Leviston, P. G. & Wallbridge, M. G. H. (1976). *J. Organomet. Chem.* **110**, 271–279.
- Nonius (1997–2001). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
- Robertson, G. B., Tucker, P. A. & Wickramasinghe, W. A. (1986). *Aust. J. Chem.* **39**, 1495–1507.
- Watkin, D. (1994). *Acta Cryst.* **A50**, 411–437.