

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# A second polymorph of [1,2-bis(di-tert-butylphosphino)ethane]dichlorido-platinum(II)

Ahmet Gunay, William W. Brennessel and William D. Jones\*

Department of Chemistry, University of Rochester, Box 270216, Rochester, NY 14627-0216, USA

Correspondence e-mail: jones@chem.rochester.edu

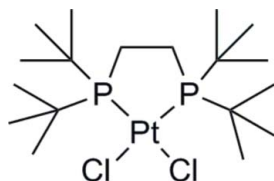
Received 18 December 2007; accepted 7 January 2008

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.060; data-to-parameter ratio = 38.6.

The title complex,  $[\text{PtCl}_2(\text{C}_{18}\text{H}_{40}\text{P}_2)]$ , contains a  $\text{Pt}^{\text{II}}$  center in an approximately square-planar geometry [ $\text{cis}$  angle range =  $88.09(3)$ – $91.39(3)^\circ$ ; twist angle =  $1.19(5)^\circ$ ]. The Pt–P bond lengths of 2.2536(8) and 2.2513(8) Å and the Pt–Cl bond lengths of 2.3750(8) and 2.3588(8) Å are normal. This crystal form is a polymorph of a structure reported previously [Harada, Kai, Yasuoka & Kasai (1976). *Bull. Chem. Soc. Jpn*, **49**, 3472–3477].

## Related literature

For related literature, see: Crascall & Spencer (1990); Green *et al.* (1977); McDermott *et al.* (1976); Ogoshi *et al.* (2004).



## Experimental

### Crystal data

$[\text{PtCl}_2(\text{C}_{18}\text{H}_{40}\text{P}_2)]$   
 $M_r = 584.43$   
 Monoclinic,  $P2_1/n$   
 $a = 11.0981(10)$  Å  
 $b = 15.3242(13)$  Å  
 $c = 14.5413(13)$  Å  
 $\beta = 109.287(1)^\circ$

$V = 2334.2(4)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 6.38$  mm<sup>-1</sup>  
 $T = 100.0(1)$  K  
 $0.20 \times 0.14 \times 0.08$  mm

### Data collection

Bruker SMART APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2007)  
 $T_{\text{min}} = 0.342$ ,  $T_{\text{max}} = 0.600$

20415 measured reflections  
 8022 independent reflections  
 6312 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.060$   
 $S = 1.01$   
 8022 reflections

208 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 1.11$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.81$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Pt1–P2	2.2513 (8)	Pt1–Cl2	2.3588 (8)
Pt1–P1	2.2536 (8)	Pt1–Cl1	2.3750 (8)
P2–Pt1–P1	89.70 (3)	P2–Pt1–Cl1	178.84 (3)
P2–Pt1–Cl2	90.82 (3)	P1–Pt1–Cl1	91.39 (3)
P1–Pt1–Cl2	178.77 (3)	Cl2–Pt1–Cl1	88.09 (3)

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

We thank the US Department of Energy for support (grant FG02–86ER13569).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2062).

## References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bruker (2000). *SHELXTL*. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2006). *APEX2* (Version 2.1-0) and *SAINT* (Version 7.34A). Bruker AXS Inc., Madison, Wisconsin, USA.
- Crascall, L. E. & Spencer, J. L. (1990). *Inorg. Synth.* **28**, 126–129.
- Green, M., Howard, J. A. K., Spencer, J. L. & Stone, F. G. A. (1977). *J. Chem. Soc. Dalton Trans.* pp. 271–277.
- Harada, M., Kai, Y., Yasuoka, N. & Kasai, N. (1976). *Bull. Chem. Soc. Jpn*, **49**, 3472–3477.
- McDermott, J. X., White, J. F. & Whitesides, G. M. (1976). *J. Am. Chem. Soc.* **98**, 6521–6528.
- Ogoshi, S., Morita, M., Inoue, K. & Kurosawa, H. (2004). *J. Org. Chem.* **689**, 662–665.
- Sheldrick, G. M. (2007). *SADABS*. Version 2007/2. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

*Acta Cryst.* (2008). E64, m454 [ doi:10.1107/S1600536808000603 ]

## A second polymorph of [1,2-bis(di-*tert*-butylphosphino)ethane]dichloridoplatinum(II)

A. Gunay, W. W. Brennessel and W. D. Jones

### Comment

One of the most commonly used Pt(0) precursors, Pt(COD)<sub>2</sub>, COD = 1,5-cyclooctadiene, is generally synthesized by the reduction of platinumdichlorides, like Pt(COD)Cl<sub>2</sub>, with Li<sub>2</sub>(COT), COT = cyclooctatetraene (Green *et al.*, 1977; Crascall & Spencer, 1990), or with SmI<sub>2</sub> (Ogoshi *et al.*, 2004). The latter reduction with 20 equivalents of SmI<sub>2</sub> afforded Pt(COD)<sub>2</sub> in moderate yields (45% average). After addition of chelating ligand 1,2-bis(di-*tert*-butylphosphino)ethane (dtbpe) to the SmI<sub>2</sub> reduction product, it was observed that some Pt<sup>II</sup> remained, based on the formation of the title compound, Pt(dtbppe)Cl<sub>2</sub> (I). An independent synthesis of (I) was performed to support these observations, in which dtbpe was added directly to Pt(COD)Cl<sub>2</sub> (see experimental section). The resulting pure product in 88% yield was characterized by <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P NMR spectroscopies and by single-crystal X-ray diffraction.

### Experimental

Pt(COD)Cl<sub>2</sub>, COD = 1,5-cyclooctadiene, was synthesized according to the published procedure (McDermott *et al.*, 1976). Under an atmosphere of dinitrogen, bis(di-*tert*-butylphosphino)ethane (dtbpe) (212 mg, 0.67 mmol) was added to a light yellow suspension of Pt(COD)Cl<sub>2</sub> (250 mg, 0.67 mmol) in THF (25 ml). The reaction mixture was heated with stirring for 12 h at 373 K. After complete conversion to (I) was verified by <sup>31</sup>P NMR spectroscopy, the volatiles (THF, COD) were removed *in vacuo*, leaving the white powdery product (343.4 mg, 0.59 mmol) in 88% yield. Crystals of (I) were grown by vapor diffusion of hexanes into THF.

### Refinement

The H-atoms were included in the refinements at geometrically idealized positions with C—H distances 0.98 and 0.99 Å for CH<sub>3</sub> and CH<sub>2</sub> type H-atoms, respectively; *U*<sub>iso</sub> values were 1.5*U*<sub>eq</sub> and 1.2*U*<sub>eq</sub> of the carrier atoms for the methyl and CH<sub>2</sub> groups, respectively. The final difference map showed a residual electron density in the vicinity of H31A atom and was chemically meaningless.

### Figures

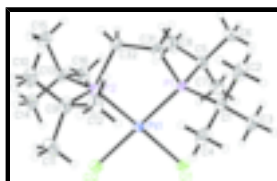


Fig. 1. Displacement ellipsoid (50% probability) drawing of (I) with H atoms omitted.

## [1,2-bis(di-*tert*-butylphosphino)ethane]dichloridoplatinum(II)

### Crystal data

[PtCl<sub>2</sub>(C<sub>18</sub>H<sub>40</sub>P<sub>2</sub>)]

$M_r = 584.43$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 11.0981 (10) \text{ \AA}$

$b = 15.3242 (13) \text{ \AA}$

$c = 14.5413 (13) \text{ \AA}$

$\beta = 109.287 (1)^\circ$

$V = 2334.2 (4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 1160$

$D_x = 1.663 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4040 reflections

$\theta = 3.0\text{--}32.9^\circ$

$\mu = 6.38 \text{ mm}^{-1}$

$T = 100.0 (1) \text{ K}$

Block, colorless

$0.20 \times 0.14 \times 0.08 \text{ mm}$

### Data collection

Bruker SMART APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0(1) \text{ K}$

area detector,  $\omega$  scans per  $\phi$

Absorption correction: multi-scan  
(SADABS; Sheldrick, 2007)

$T_{\min} = 0.342$ ,  $T_{\max} = 0.600$

20415 measured reflections

8022 independent reflections

6312 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\text{max}} = 32.0^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -15 \rightarrow 16$

$k = -22 \rightarrow 22$

$l = -19 \rightarrow 21$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.060$

$S = 1.02$

8022 reflections

208 parameters

Primary atom site location: structure-invariant direct  
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring  
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 1.11 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.81 \text{ e \AA}^{-3}$

Extinction correction: none

*Special details*

**Experimental.**  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 20 °C):  $\delta$  1.5 (d,  $^3J_{\text{HP}} = 14.1$  Hz, 36 H,  $-(\text{CH}_3)_3$ ), 1.9 (d,  $^2J_{\text{HP}} = 16$  Hz, 4 H,  $-\text{CH}_2-$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 20 °C):  $\delta$  24.5 (d,  $^1J_{\text{CP}} = 33$  Hz,  $-\text{CH}_2-$ ), 30.4 (s,  $-(\text{CH}_3)_3$ ), 37.6 (d,  $^1J_{\text{CP}} = 30$  Hz,  $-\text{C}-$ );  $^{31}\text{P}$  NMR ( $\text{CDCl}_3$ , 20 °C):  $\delta$  75.7 (s, with platinum satellites  $^1J_{\text{Ppt}} = 3643.2$  Hz).

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and

goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based

on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of

$F^2 > 2\text{sigma}(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is

not relevant to the choice of reflections for refinement.  $R$ -factors based

on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -

factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.268368 (10)	0.706474 (7)	0.781256 (8)	0.01505 (3)
Cl1	0.17379 (7)	0.80168 (5)	0.86681 (6)	0.02422 (16)
Cl2	0.44219 (8)	0.80401 (5)	0.81687 (6)	0.02415 (16)
P1	0.09956 (7)	0.61552 (5)	0.74524 (5)	0.01603 (14)
P2	0.36188 (7)	0.61746 (5)	0.70173 (6)	0.01776 (15)
C1	0.0577 (3)	0.5718 (2)	0.8529 (2)	0.0227 (6)
C2	-0.0053 (4)	0.4814 (2)	0.8323 (3)	0.0323 (8)
H2A	-0.0253	0.4613	0.8896	0.048*
H2B	0.0533	0.4400	0.8177	0.048*
H2C	-0.0842	0.4851	0.7763	0.048*
C3	-0.0326 (3)	0.6333 (2)	0.8823 (3)	0.0287 (7)
H3A	-0.0520	0.6085	0.9380	0.043*
H3B	-0.1120	0.6402	0.8273	0.043*
H3C	0.0083	0.6903	0.9002	0.043*
C4	0.1821 (3)	0.5634 (2)	0.9392 (2)	0.0308 (8)
H4A	0.1632	0.5409	0.9960	0.046*

## supplementary materials

---

H4B	0.2227	0.6208	0.9547	0.046*
H4C	0.2400	0.5231	0.9221	0.046*
C5	-0.0478 (3)	0.6578 (2)	0.6491 (2)	0.0232 (7)
C6	-0.1592 (3)	0.5927 (2)	0.6247 (3)	0.0302 (8)
H6A	-0.2334	0.6177	0.5744	0.045*
H6B	-0.1812	0.5807	0.6834	0.045*
H6C	-0.1342	0.5383	0.6006	0.045*
C7	-0.0919 (3)	0.7459 (2)	0.6749 (3)	0.0316 (8)
H7A	-0.1686	0.7645	0.6224	0.047*
H7B	-0.0240	0.7891	0.6833	0.047*
H7C	-0.1113	0.7406	0.7357	0.047*
C8	-0.0127 (3)	0.6711 (3)	0.5569 (3)	0.0388 (9)
H8A	-0.0871	0.6932	0.5046	0.058*
H8B	0.0143	0.6154	0.5370	0.058*
H8C	0.0573	0.7133	0.5700	0.058*
C9	0.5007 (3)	0.5527 (2)	0.7849 (2)	0.0246 (7)
C10	0.5670 (4)	0.4957 (2)	0.7285 (3)	0.0349 (9)
H10A	0.6377	0.4637	0.7747	0.052*
H10B	0.5999	0.5329	0.6874	0.052*
H10C	0.5053	0.4541	0.6874	0.052*
C11	0.5985 (3)	0.6126 (2)	0.8564 (3)	0.0340 (8)
H11A	0.6690	0.5774	0.8984	0.051*
H11B	0.5573	0.6440	0.8967	0.051*
H11C	0.6317	0.6546	0.8199	0.051*
C12	0.4469 (3)	0.4907 (2)	0.8451 (3)	0.0298 (8)
H12A	0.5166	0.4561	0.8890	0.045*
H12B	0.3840	0.4516	0.8011	0.045*
H12C	0.4058	0.5249	0.8834	0.045*
C13	0.4076 (3)	0.6704 (2)	0.5996 (2)	0.0241 (7)
C14	0.5394 (3)	0.7143 (2)	0.6356 (3)	0.0292 (7)
H14A	0.5579	0.7404	0.5802	0.044*
H14B	0.6046	0.6707	0.6667	0.044*
H14C	0.5396	0.7600	0.6829	0.044*
C15	0.4083 (4)	0.6035 (2)	0.5199 (3)	0.0374 (9)
H15A	0.4322	0.6329	0.4687	0.056*
H15B	0.3230	0.5780	0.4919	0.056*
H15C	0.4702	0.5572	0.5489	0.056*
C16	0.3071 (3)	0.7402 (2)	0.5518 (3)	0.0286 (7)
H16A	0.3286	0.7684	0.4988	0.043*
H16B	0.3055	0.7839	0.6006	0.043*
H16C	0.2229	0.7126	0.5256	0.043*
C31	0.1465 (3)	0.5180 (2)	0.6924 (2)	0.0216 (6)
H31A	0.1853	0.4755	0.7452	0.026*
H31B	0.0690	0.4908	0.6465	0.026*
C32	0.2411 (3)	0.5363 (2)	0.6385 (2)	0.0211 (6)
H32A	0.1938	0.5577	0.5721	0.025*
H32B	0.2842	0.4813	0.6320	0.025*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.01739 (5)	0.01316 (5)	0.01369 (5)	0.00118 (5)	0.00389 (4)	-0.00008 (4)
C11	0.0232 (4)	0.0226 (4)	0.0254 (4)	0.0034 (3)	0.0061 (3)	-0.0079 (3)
C12	0.0275 (4)	0.0195 (4)	0.0271 (4)	-0.0052 (3)	0.0112 (3)	-0.0035 (3)
P1	0.0182 (4)	0.0177 (4)	0.0132 (3)	-0.0006 (3)	0.0066 (3)	-0.0002 (3)
P2	0.0202 (4)	0.0158 (4)	0.0199 (4)	-0.0004 (3)	0.0102 (3)	-0.0011 (3)
C1	0.0277 (16)	0.0264 (16)	0.0185 (15)	0.0043 (13)	0.0137 (13)	0.0043 (12)
C2	0.046 (2)	0.0275 (18)	0.0333 (19)	-0.0032 (16)	0.0261 (17)	0.0057 (15)
C3	0.0365 (19)	0.0297 (18)	0.0269 (17)	0.0024 (15)	0.0201 (15)	0.0022 (14)
C4	0.0348 (19)	0.041 (2)	0.0186 (16)	0.0045 (16)	0.0116 (14)	0.0099 (15)
C5	0.0185 (15)	0.0330 (18)	0.0165 (15)	-0.0029 (13)	0.0034 (12)	0.0040 (13)
C6	0.0208 (16)	0.037 (2)	0.0302 (19)	-0.0070 (14)	0.0053 (14)	-0.0058 (15)
C7	0.0222 (17)	0.0323 (19)	0.034 (2)	0.0028 (14)	0.0009 (15)	0.0085 (16)
C8	0.0208 (17)	0.071 (3)	0.0212 (18)	-0.0038 (17)	0.0018 (14)	0.0117 (18)
C9	0.0239 (16)	0.0186 (15)	0.0326 (18)	0.0045 (12)	0.0109 (14)	0.0015 (13)
C10	0.0326 (19)	0.0228 (17)	0.056 (2)	0.0042 (15)	0.0233 (18)	-0.0002 (16)
C11	0.0236 (17)	0.0309 (18)	0.041 (2)	0.0022 (15)	0.0021 (15)	0.0048 (16)
C12	0.0287 (18)	0.0263 (17)	0.036 (2)	0.0079 (14)	0.0126 (15)	0.0104 (15)
C13	0.0296 (17)	0.0250 (16)	0.0238 (17)	-0.0025 (14)	0.0172 (14)	-0.0011 (13)
C14	0.0272 (17)	0.0292 (18)	0.0363 (19)	0.0001 (14)	0.0173 (15)	0.0008 (15)
C15	0.050 (2)	0.042 (2)	0.0306 (19)	-0.0120 (19)	0.0282 (18)	-0.0071 (17)
C16	0.0315 (18)	0.0327 (18)	0.0255 (18)	-0.0035 (15)	0.0148 (15)	0.0084 (14)
C31	0.0270 (16)	0.0180 (15)	0.0243 (16)	-0.0022 (12)	0.0144 (13)	-0.0048 (12)
C32	0.0244 (15)	0.0202 (15)	0.0221 (15)	-0.0033 (12)	0.0125 (12)	-0.0046 (12)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Pt1—P2	2.2513 (8)	C8—H8B	0.9800
Pt1—P1	2.2536 (8)	C8—H8C	0.9800
Pt1—C12	2.3588 (8)	C9—C11	1.535 (5)
Pt1—C11	2.3750 (8)	C9—C10	1.542 (5)
P1—C31	1.833 (3)	C9—C12	1.540 (5)
P1—C5	1.881 (3)	C10—H10A	0.9800
P1—C1	1.897 (3)	C10—H10B	0.9800
P2—C32	1.837 (3)	C10—H10C	0.9800
P2—C9	1.895 (3)	C11—H11A	0.9800
P2—C13	1.903 (3)	C11—H11B	0.9800
C1—C4	1.534 (5)	C11—H11C	0.9800
C1—C3	1.535 (4)	C12—H12A	0.9800
C1—C2	1.537 (5)	C12—H12B	0.9800
C2—H2A	0.9800	C12—H12C	0.9800
C2—H2B	0.9800	C13—C16	1.536 (5)
C2—H2C	0.9800	C13—C14	1.536 (5)
C3—H3A	0.9800	C13—C15	1.549 (5)
C3—H3B	0.9800	C14—H14A	0.9800
C3—H3C	0.9800	C14—H14B	0.9800

## supplementary materials

---

C4—H4A	0.9800	C14—H14C	0.9800
C4—H4B	0.9800	C15—H15A	0.9800
C4—H4C	0.9800	C15—H15B	0.9800
C5—C7	1.525 (5)	C15—H15C	0.9800
C5—C8	1.528 (5)	C16—H16A	0.9800
C5—C6	1.536 (4)	C16—H16B	0.9800
C6—H6A	0.9800	C16—H16C	0.9800
C6—H6B	0.9800	C31—C32	1.529 (4)
C6—H6C	0.9800	C31—H31A	0.9900
C7—H7A	0.9800	C31—H31B	0.9900
C7—H7B	0.9800	C32—H32A	0.9900
C7—H7C	0.9800	C32—H32B	0.9900
C8—H8A	0.9800		
P2—Pt1—P1	89.70 (3)	C5—C8—H8C	109.5
P2—Pt1—C12	90.82 (3)	H8A—C8—H8C	109.5
P1—Pt1—C12	178.77 (3)	H8B—C8—H8C	109.5
P2—Pt1—C11	178.84 (3)	C11—C9—C10	110.2 (3)
P1—Pt1—C11	91.39 (3)	C11—C9—C12	107.7 (3)
C12—Pt1—C11	88.09 (3)	C10—C9—C12	107.1 (3)
C31—P1—C5	105.46 (15)	C11—C9—P2	111.3 (2)
C31—P1—C1	103.85 (14)	C10—C9—P2	112.8 (2)
C5—P1—C1	110.23 (14)	C12—C9—P2	107.4 (2)
C31—P1—Pt1	105.77 (10)	C9—C10—H10A	109.5
C5—P1—Pt1	114.31 (11)	C9—C10—H10B	109.5
C1—P1—Pt1	115.98 (11)	H10A—C10—H10B	109.5
C32—P2—C9	105.70 (15)	C9—C10—H10C	109.5
C32—P2—C13	103.69 (14)	H10A—C10—H10C	109.5
C9—P2—C13	110.56 (15)	H10B—C10—H10C	109.5
C32—P2—Pt1	106.44 (10)	C9—C11—H11A	109.5
C9—P2—Pt1	113.88 (11)	C9—C11—H11B	109.5
C13—P2—Pt1	115.45 (11)	H11A—C11—H11B	109.5
C4—C1—C3	108.6 (3)	C9—C11—H11C	109.5
C4—C1—C2	108.2 (3)	H11A—C11—H11C	109.5
C3—C1—C2	108.1 (3)	H11B—C11—H11C	109.5
C4—C1—P1	107.8 (2)	C9—C12—H12A	109.5
C3—C1—P1	111.9 (2)	C9—C12—H12B	109.5
C2—C1—P1	112.1 (2)	H12A—C12—H12B	109.5
C1—C2—H2A	109.5	C9—C12—H12C	109.5
C1—C2—H2B	109.5	H12A—C12—H12C	109.5
H2A—C2—H2B	109.5	H12B—C12—H12C	109.5
C1—C2—H2C	109.5	C16—C13—C14	108.5 (3)
H2A—C2—H2C	109.5	C16—C13—C15	107.8 (3)
H2B—C2—H2C	109.5	C14—C13—C15	107.8 (3)
C1—C3—H3A	109.5	C16—C13—P2	107.9 (2)
C1—C3—H3B	109.5	C14—C13—P2	113.0 (2)
H3A—C3—H3B	109.5	C15—C13—P2	111.7 (2)
C1—C3—H3C	109.5	C13—C14—H14A	109.5
H3A—C3—H3C	109.5	C13—C14—H14B	109.5
H3B—C3—H3C	109.5	H14A—C14—H14B	109.5

C1—C4—H4A	109.5	C13—C14—H14C	109.5
C1—C4—H4B	109.5	H14A—C14—H14C	109.5
H4A—C4—H4B	109.5	H14B—C14—H14C	109.5
C1—C4—H4C	109.5	C13—C15—H15A	109.5
H4A—C4—H4C	109.5	C13—C15—H15B	109.5
H4B—C4—H4C	109.5	H15A—C15—H15B	109.5
C7—C5—C8	107.0 (3)	C13—C15—H15C	109.5
C7—C5—C6	109.4 (3)	H15A—C15—H15C	109.5
C8—C5—C6	107.7 (3)	H15B—C15—H15C	109.5
C7—C5—P1	113.1 (2)	C13—C16—H16A	109.5
C8—C5—P1	106.7 (2)	C13—C16—H16B	109.5
C6—C5—P1	112.6 (2)	H16A—C16—H16B	109.5
C5—C6—H6A	109.5	C13—C16—H16C	109.5
C5—C6—H6B	109.5	H16A—C16—H16C	109.5
H6A—C6—H6B	109.5	H16B—C16—H16C	109.5
C5—C6—H6C	109.5	C32—C31—P1	113.8 (2)
H6A—C6—H6C	109.5	C32—C31—H31A	108.8
H6B—C6—H6C	109.5	P1—C31—H31A	108.8
C5—C7—H7A	109.5	C32—C31—H31B	108.8
C5—C7—H7B	109.5	P1—C31—H31B	108.8
H7A—C7—H7B	109.5	H31A—C31—H31B	107.7
C5—C7—H7C	109.5	C31—C32—P2	112.3 (2)
H7A—C7—H7C	109.5	C31—C32—H32A	109.2
H7B—C7—H7C	109.5	P2—C32—H32A	109.2
C5—C8—H8A	109.5	C31—C32—H32B	109.2
C5—C8—H8B	109.5	P2—C32—H32B	109.2
H8A—C8—H8B	109.5	H32A—C32—H32B	107.9
P2—Pt1—P1—C31	8.74 (11)	C1—P1—C5—C6	-48.9 (3)
C11—Pt1—P1—C31	-170.88 (11)	Pt1—P1—C5—C6	178.4 (2)
P2—Pt1—P1—C5	-106.82 (12)	C32—P2—C9—C11	-169.3 (2)
C11—Pt1—P1—C5	73.55 (12)	C13—P2—C9—C11	79.1 (3)
P2—Pt1—P1—C1	123.22 (11)	Pt1—P2—C9—C11	-52.8 (3)
C11—Pt1—P1—C1	-56.40 (11)	C32—P2—C9—C10	66.3 (3)
P1—Pt1—P2—C32	9.20 (11)	C13—P2—C9—C10	-45.3 (3)
C12—Pt1—P2—C32	-169.68 (11)	Pt1—P2—C9—C10	-177.3 (2)
P1—Pt1—P2—C9	-106.85 (12)	C32—P2—C9—C12	-51.6 (2)
C12—Pt1—P2—C9	74.27 (12)	C13—P2—C9—C12	-163.2 (2)
P1—Pt1—P2—C13	123.65 (12)	Pt1—P2—C9—C12	64.9 (2)
C12—Pt1—P2—C13	-55.24 (12)	C32—P2—C13—C16	81.9 (2)
C31—P1—C1—C4	83.6 (2)	C9—P2—C13—C16	-165.2 (2)
C5—P1—C1—C4	-163.8 (2)	Pt1—P2—C13—C16	-34.1 (3)
Pt1—P1—C1—C4	-31.9 (3)	C32—P2—C13—C14	-158.1 (2)
C31—P1—C1—C3	-157.1 (2)	C9—P2—C13—C14	-45.2 (3)
C5—P1—C1—C3	-44.5 (3)	Pt1—P2—C13—C14	85.9 (2)
Pt1—P1—C1—C3	87.4 (2)	C32—P2—C13—C15	-36.4 (3)
C31—P1—C1—C2	-35.4 (3)	C9—P2—C13—C15	76.5 (3)
C5—P1—C1—C2	77.2 (3)	Pt1—P2—C13—C15	-152.4 (2)
Pt1—P1—C1—C2	-151.0 (2)	C5—P1—C31—C32	91.5 (2)
C31—P1—C5—C7	-172.7 (2)	C1—P1—C31—C32	-152.5 (2)

## supplementary materials

---

C1—P1—C5—C7	75.7 (3)	Pt1—P1—C31—C32	-30.0 (2)
Pt1—P1—C5—C7	-57.0 (3)	P1—C31—C32—P2	39.6 (3)
C31—P1—C5—C8	-55.3 (3)	C9—P2—C32—C31	91.6 (2)
C1—P1—C5—C8	-166.8 (2)	C13—P2—C32—C31	-152.0 (2)
Pt1—P1—C5—C8	60.4 (3)	Pt1—P2—C32—C31	-29.8 (2)
C31—P1—C5—C6	62.6 (3)		

Fig. 1

