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[1,2-Bis(diphenylphosphino)ethane]- diiodidoplatinum(II) dichloromethane disolvate

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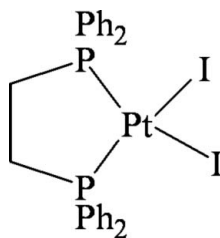
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å;
 R factor = 0.029; wR factor = 0.068; data-to-parameter ratio = 18.8.

In the title compound, $[\text{PtI}_2(\text{C}_{26}\text{H}_{24}\text{P}_2)] \cdot 2\text{CH}_2\text{Cl}_2$, the $\text{PtI}_2(\text{dppe})$ [dppe = 1,2-bis(diphenylphosphino)ethane] molecules possess twofold rotation symmetry. The Pt coordination displays a square-planar arrangement, with the sum of the angles around the Pt atom being $360.01(2)^\circ$. The Pt–I distance is $2.6484(5)$ Å. In the crystal structure, intermolecular C–H \cdots I contacts link the $\text{PtI}_2(\text{dppe})$ molecules into rows along the c axis, with a C \cdots I distance of $3.873(5)$ Å.

Related literature

For general background, see Hudson *et al.* (1968); for a related structure, see Parkin *et al.* (1991).



Experimental

Crystal data

$[\text{PtI}_2(\text{C}_{26}\text{H}_{24}\text{P}_2)] \cdot 2\text{CH}_2\text{Cl}_2$
 $M_r = 1017.13$
 Orthorhombic, $Pccn$
 $a = 12.7385(2)$ Å
 $b = 15.6542(3)$ Å
 $c = 16.7194(3)$ Å

$V = 3334.03(10)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 6.49$ mm⁻¹
 $T = 113(2)$ K
 $0.09 \times 0.08 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.616$, $T_{\max} = 0.737$

67782 measured reflections
 3156 independent reflections
 2527 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.082$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.068$
 $S = 1.12$
 3156 reflections

168 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 2.05$ e Å⁻³
 $\Delta\rho_{\min} = -0.90$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pt1–P1	2.2418 (13)	Pt1–I1	2.6484 (4)
P1–Pt1–P1 ⁱ	86.31 (6)	P1 ⁱ –Pt1–I1	90.69 (3)
P1–Pt1–I1	176.99 (3)		

Symmetry code: (i) $-x + \frac{3}{2}, -y + \frac{3}{2}, z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1A \cdots I1 ⁱⁱ	0.99	3.04	3.873 (5)	143

Symmetry code: (ii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO-SMN (Otwinowski & Minor, 1997); data reduction: DENZO-SMN; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2365).

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supporting information

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[1,2-Bis(diphenylphosphino)ethane]diiodidoplatinum(II) dichloromethane disolvate

Akella Sivaramakrishna, Hong Su and John R. Moss

S1. Comment

The preparation of the title compound, *cis*-[PtI₂(dppe)], was studied initially by Hudson *et al.* (1968). Parkin *et al.* (1991) reported the first and only crystal structure of the PtI₂(dppe) molecule as a dichloromethane solvated complex co-crystallized with an iodine molecule, namely 3[PtI₂(dppe)]·I₂·2CH₂Cl₂ (monoclinic space group *P*2₁/*n*, *a* = 8.593 (2) Å, *b* = 28.194 (16) Å, *c* = 36.206 (9) Å, *β* = 91.50 (2)°, *Z* = 12, CSD Refcode LAGBOK). However, there are no atomic coordinates available in the CSD (Version 5.28, 2007). We present here a well refined bis-dichloromethane solvated crystal structure of the title compound, [PtI₂(dppe)]·2CH₂Cl₂, (I) (Fig. 1). Here the complex crystallized in the space group *Pccn* with *Z* = 4. The molecule of PtI₂(dppe) lies on a twofold rotation axis passing through Pt atom and the midpoint of the C1—C1' bond [symmetry code: (i) 3/2 - *x*, 3/2 - *y*, *z*]. Thus, in addition to Pt on the special position *c* in Wyckoff notation, the asymmetric unit consists of one I1 atom and half of the ligand comprising P1, the ethane C1 and the C11—C16 and C21—C26 phenyl rings attached to P1 together with one CH₂Cl₂ solvent molecule which is on a general position.

The coordination of Pt is a slight tetrahedral distortion from a square-planar arrangement with the sum of angles at Pt being 360.0 (2)°. The Pt—P distance and P—Pt—P angle are 2.242 (1) Å and 86.31 (6)° respectively; the Pt—I distance and I—Pt—I angle are 2.6484 (4) Å and 92.32 (2)° respectively; other important bond lengths and angles are in Table 1. A search of *cis*-dihalide complexes of the type *M*(dppe)X₂ [where *M* = Ni, Pd and Pt; dppe = 1,2-bis(diphenylphosphino)ethane; X = Cl, Br and I] in the CSD (Version 5.28, 2007) reveals 23 entries. This resulted in the following statistics: the average distances of Pt—Cl (4 entries), Pd—Cl (3 entries) and Ni—Cl (10 entries) are 2.356 Å, 2.362 Å and 2.200 Å respectively; the average Ni—Br distances (2 entries) are 2.330 Å and the average Pd—I (2 entries) and Ni—I distances (1 entry) are 2.658 Å and 2.527 Å respectively. Intermolecular contacts of the type C(1)—H(1 A)⋯I(1) are present with a C⋯I distance of 3.873 (5) Å (details in Table 2). These interconnect the title molecules into columns propagated in the [001] direction.

S2. Experimental

Crystals of the complex I were obtained as an unexpected by-product of the reaction of Pt(dppe)Cl₂ with a Grignard reagent, IMg(CH₂)₆MgI in diethyl ether solution. The title compound was also prepared by the reaction of NaI with Pt(dppe)Cl₂ in refluxing acetone solution for 4 h. ³¹P NMR indicated a singlet at 46.2 p.p.m. with platinum satellites (*J*_{Pt—P} = 3368 Hz). A mixture containing 0.248 g (0.373 mmol) of *cis*-Pt(dppe)Cl₂ and 0.112 g (0.747 mmol) of NaI in 20 ml of acetone was refluxed for 4 h. Removal of solvent in high vacuum and recrystallize from a mixture of CH₂Cl₂ and *n*-hexane (1:1) led to the isolation of the complex I as a light yellow crystalline solid (0.296 g, 93%). Anal. Calc. for C₂₆H₂₄I₂P₂Pt: C, 36.86; H, 2.86; Found: C, 36.82; H, 2.89. Mass spectral data: *M*⁺ = 846.9; Pt(dppe)I⁺ = 719.9; Pt(dppe)⁺ = 595.

S3. Refinement

The structure was solved by the Patterson method. All H atoms were placed in idealized positions in a riding model with $d(\text{C}-\text{H}) = 0.95 \text{ \AA}$ for aromatic H atoms and $d(\text{C}-\text{H}) = 0.99 \text{ \AA}$ for the CH_2 group and assigned $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The highest peak of 2.05 e \AA^{-3} is located at 1.69 \AA from I1. The deepest hole of -0.89 e \AA^{-3} is located at 0.81 \AA from I1.

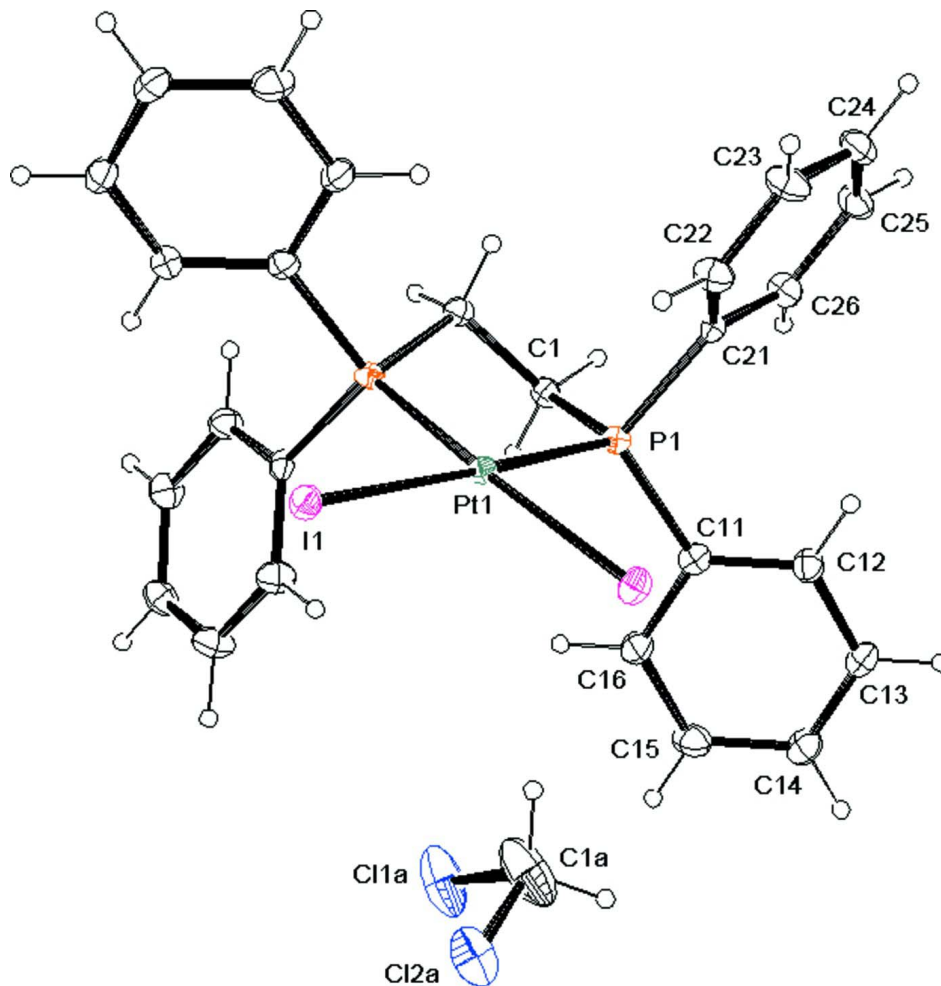


Figure 1

Molecular structure of the complex I, showing the atom-labelling scheme. Labelled atoms are related to unlabelled atoms by the symmetry code: $3/2 - x, 3/2 - y, z$. Non-H atoms are shown with 30% probability displacement ellipsoids and H atoms are shown as open circles.

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Crystal data

$[\text{PtI}_2(\text{C}_{26}\text{H}_{24}\text{P}_2)] \cdot 2\text{CH}_2\text{Cl}_2$
 $M_r = 1017.13$
 Orthorhombic, *Pccn*
 Hall symbol: $-P\ 2ab\ 2ac$
 $a = 12.7385 (2) \text{ \AA}$
 $b = 15.6542 (3) \text{ \AA}$
 $c = 16.7194 (3) \text{ \AA}$

$V = 3334.03 (10) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 1912$
 $D_x = 2.026 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 67782 reflections
 $\theta = 2.8\text{--}25.7^\circ$

$\mu = 6.49 \text{ mm}^{-1}$
 $T = 113 \text{ K}$

Needle, yellow
 $0.09 \times 0.08 \times 0.05 \text{ mm}$

Data collection

Nonius Kappa CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 $1.2^\circ \varphi$ scans, and ω
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.616$, $T_{\max} = 0.737$

67782 measured reflections
 3156 independent reflections
 2527 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.082$
 $\theta_{\max} = 25.7^\circ$, $\theta_{\min} = 2.8^\circ$
 $h = -15 \rightarrow 15$
 $k = -19 \rightarrow 19$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.068$
 $S = 1.12$
 3156 reflections
 168 parameters
 0 restraints
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 6.3267P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 2.05 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.90 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Half sphere of data collected using COLLECT strategy (Nonius, 2000). Crystal to detector distance = 30 mm; combination of φ and ω scans of 1.2° , 30 s per $^\circ$, 2 iterations.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.7500	0.7500	0.212727 (15)	0.02157 (9)
I1	0.81688 (3)	0.85922 (2)	0.322442 (19)	0.03005 (11)
Cl1A	0.3632 (2)	0.84723 (12)	0.26536 (13)	0.0849 (7)
Cl2A	0.41394 (18)	0.79969 (12)	0.42948 (12)	0.0718 (6)
P1	0.69591 (10)	0.66250 (8)	0.11491 (7)	0.0234 (3)
C1	0.6997 (4)	0.7235 (3)	0.0217 (3)	0.0259 (11)
H1A	0.6991	0.6843	-0.0248	0.031*
H1B	0.6377	0.7614	0.0180	0.031*
C11	0.5636 (4)	0.6218 (3)	0.1242 (3)	0.0263 (11)
C12	0.5399 (4)	0.5360 (3)	0.1280 (3)	0.0319 (12)
H12	0.5945	0.4950	0.1229	0.038*
C13	0.4379 (4)	0.5088 (4)	0.1390 (3)	0.0356 (13)

H13	0.4231	0.4494	0.1416	0.043*
C14	0.3577 (4)	0.5667 (4)	0.1463 (3)	0.0377 (13)
H14	0.2877	0.5477	0.1546	0.045*
C15	0.3796 (5)	0.6530 (4)	0.1414 (3)	0.0422 (14)
H15	0.3243	0.6935	0.1454	0.051*
C16	0.4827 (4)	0.6808 (4)	0.1307 (3)	0.0367 (13)
H16	0.4975	0.7402	0.1278	0.044*
C21	0.7835 (4)	0.5730 (3)	0.0982 (3)	0.0245 (11)
C22	0.8669 (4)	0.5580 (4)	0.1500 (3)	0.0363 (13)
H22	0.8767	0.5937	0.1953	0.044*
C23	0.9358 (5)	0.4916 (4)	0.1358 (3)	0.0424 (15)
H23	0.9921	0.4812	0.1718	0.051*
C24	0.9230 (5)	0.4405 (4)	0.0696 (3)	0.0385 (14)
H24	0.9713	0.3956	0.0596	0.046*
C25	0.8405 (4)	0.4540 (4)	0.0178 (3)	0.0394 (14)
H25	0.8314	0.4178	-0.0273	0.047*
C26	0.7706 (4)	0.5204 (4)	0.0315 (3)	0.0338 (13)
H26	0.7142	0.5301	-0.0045	0.041*
C1A	0.4117 (8)	0.7672 (5)	0.3296 (5)	0.089 (3)
H1A1	0.3671	0.7156	0.3246	0.106*
H1A2	0.4837	0.7516	0.3128	0.106*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02113 (14)	0.02447 (15)	0.01910 (14)	-0.00048 (12)	0.000	0.000
I1	0.03385 (19)	0.03157 (19)	0.02474 (18)	-0.00589 (15)	0.00190 (14)	-0.00346 (14)
Cl1A	0.1160 (18)	0.0525 (11)	0.0860 (14)	0.0233 (11)	-0.0591 (14)	-0.0198 (10)
Cl2A	0.0926 (15)	0.0549 (11)	0.0679 (12)	0.0121 (11)	-0.0133 (11)	-0.0114 (9)
P1	0.0227 (7)	0.0264 (7)	0.0211 (6)	0.0015 (5)	-0.0011 (5)	0.0007 (5)
C1	0.028 (3)	0.028 (3)	0.022 (2)	0.002 (2)	-0.003 (2)	-0.004 (2)
C11	0.028 (3)	0.032 (3)	0.019 (2)	-0.001 (2)	-0.003 (2)	0.002 (2)
C12	0.032 (3)	0.028 (3)	0.036 (3)	0.000 (2)	-0.002 (2)	0.002 (2)
C13	0.033 (3)	0.035 (3)	0.039 (3)	-0.009 (3)	0.002 (3)	0.002 (3)
C14	0.033 (3)	0.043 (4)	0.037 (3)	-0.010 (3)	-0.005 (2)	-0.003 (3)
C15	0.030 (3)	0.050 (4)	0.047 (3)	0.004 (3)	0.000 (3)	-0.009 (3)
C16	0.027 (3)	0.034 (3)	0.049 (3)	-0.004 (2)	-0.001 (3)	-0.003 (3)
C21	0.024 (2)	0.020 (2)	0.029 (3)	-0.004 (2)	0.005 (2)	0.001 (2)
C22	0.035 (3)	0.045 (3)	0.029 (3)	0.004 (3)	-0.003 (2)	-0.007 (2)
C23	0.036 (3)	0.050 (4)	0.041 (3)	0.016 (3)	-0.005 (3)	-0.003 (3)
C24	0.040 (3)	0.033 (3)	0.043 (3)	0.013 (3)	0.004 (3)	-0.001 (3)
C25	0.042 (3)	0.036 (3)	0.040 (3)	0.006 (3)	0.003 (3)	-0.009 (3)
C26	0.034 (3)	0.036 (3)	0.032 (3)	0.002 (2)	-0.005 (2)	-0.004 (2)
C1A	0.116 (8)	0.072 (6)	0.078 (6)	0.046 (5)	-0.045 (6)	-0.023 (4)

Geometric parameters (Å, °)

Pt1—P1	2.2418 (13)	C14—C15	1.382 (8)
Pt1—P1 ⁱ	2.2418 (13)	C14—H14	0.9500
Pt1—I1	2.6484 (4)	C15—C16	1.395 (8)
Pt1—I1 ⁱ	2.6484 (4)	C15—H15	0.9500
C11A—C1A	1.762 (8)	C16—H16	0.9500
C12A—C1A	1.747 (8)	C21—C22	1.391 (7)
P1—C1	1.829 (5)	C21—C26	1.396 (7)
P1—C11	1.808 (5)	C22—C23	1.381 (8)
P1—C21	1.813 (5)	C22—H22	0.9500
C1—C1 ⁱ	1.526 (10)	C23—C24	1.374 (8)
C1—H1A	0.9900	C23—H23	0.9500
C1—H1B	0.9900	C24—C25	1.378 (8)
C11—C12	1.379 (7)	C24—H24	0.9500
C11—C16	1.389 (7)	C25—C26	1.388 (7)
C12—C13	1.379 (7)	C25—H25	0.9500
C12—H12	0.9500	C26—H26	0.9500
C13—C14	1.372 (8)	C1A—H1A1	0.9900
C13—H13	0.9500	C1A—H1A2	0.9900
P1—Pt1—P1 ⁱ	86.31 (6)	C14—C15—C16	120.2 (6)
P1—Pt1—I1	176.99 (3)	C14—C15—H15	119.9
P1 ⁱ —Pt1—I1	90.69 (3)	C16—C15—H15	119.9
P1—Pt1—I1 ⁱ	90.69 (3)	C11—C16—C15	120.1 (5)
P1 ⁱ —Pt1—I1 ⁱ	176.99 (3)	C11—C16—H16	120.0
I1—Pt1—I1 ⁱ	92.321 (16)	C15—C16—H16	120.0
C11—P1—C21	108.3 (2)	C22—C21—C26	119.2 (5)
C11—P1—C1	106.4 (2)	C22—C21—P1	120.3 (4)
C21—P1—C1	104.8 (2)	C26—C21—P1	120.4 (4)
C11—P1—Pt1	116.04 (16)	C23—C22—C21	120.3 (5)
C21—P1—Pt1	113.30 (17)	C23—C22—H22	119.8
C1—P1—Pt1	107.13 (17)	C21—C22—H22	119.8
C1 ⁱ —C1—P1	107.8 (3)	C24—C23—C22	120.1 (5)
C1 ⁱ —C1—H1A	110.1	C24—C23—H23	120.0
P1—C1—H1A	110.1	C22—C23—H23	120.0
C1 ⁱ —C1—H1B	110.1	C23—C24—C25	120.5 (5)
P1—C1—H1B	110.1	C23—C24—H24	119.7
H1A—C1—H1B	108.5	C25—C24—H24	119.7
C12—C11—C16	118.8 (5)	C24—C25—C26	119.9 (5)
C12—C11—P1	123.5 (4)	C24—C25—H25	120.0
C16—C11—P1	117.7 (4)	C26—C25—H25	120.0
C11—C12—C13	120.9 (5)	C25—C26—C21	119.9 (5)
C11—C12—H12	119.6	C25—C26—H26	120.0
C13—C12—H12	119.6	C21—C26—H26	120.0
C14—C13—C12	120.7 (5)	C12A—C1A—C11A	112.4 (4)
C14—C13—H13	119.7	C12A—C1A—H1A1	109.1
C12—C13—H13	119.7	C11A—C1A—H1A1	109.1

C13—C14—C15	119.4 (5)	C12A—C1A—H1A2	109.1
C13—C14—H14	120.3	C11A—C1A—H1A2	109.1
C15—C14—H14	120.3	H1A1—C1A—H1A2	107.9
P1 ⁱ —Pt1—P1—C11	-131.36 (19)	C11—C12—C13—C14	0.2 (8)
I1—Pt1—P1—C11	-128.4 (6)	C12—C13—C14—C15	0.8 (8)
I1 ⁱ —Pt1—P1—C11	48.80 (18)	C13—C14—C15—C16	-1.2 (9)
P1 ⁱ —Pt1—P1—C21	102.38 (18)	C12—C11—C16—C15	0.4 (8)
I1—Pt1—P1—C21	105.3 (6)	P1—C11—C16—C15	-177.3 (4)
I1 ⁱ —Pt1—P1—C21	-77.47 (17)	C14—C15—C16—C11	0.6 (9)
P1 ⁱ —Pt1—P1—C1	-12.74 (17)	C11—P1—C21—C22	-122.6 (4)
I1—Pt1—P1—C1	-9.8 (7)	C1—P1—C21—C22	124.2 (4)
I1 ⁱ —Pt1—P1—C1	167.41 (18)	Pt1—P1—C21—C22	7.7 (5)
C11—P1—C1—C1 ⁱ	166.3 (4)	C11—P1—C21—C26	60.1 (5)
C21—P1—C1—C1 ⁱ	-79.1 (4)	C1—P1—C21—C26	-53.1 (5)
Pt1—P1—C1—C1 ⁱ	41.6 (5)	Pt1—P1—C21—C26	-169.6 (4)
C21—P1—C11—C12	7.6 (5)	C26—C21—C22—C23	-0.5 (8)
C1—P1—C11—C12	119.9 (4)	P1—C21—C22—C23	-177.8 (4)
Pt1—P1—C11—C12	-121.1 (4)	C21—C22—C23—C24	0.9 (9)
C21—P1—C11—C16	-174.8 (4)	C22—C23—C24—C25	-1.2 (9)
C1—P1—C11—C16	-62.5 (5)	C23—C24—C25—C26	1.1 (9)
Pt1—P1—C11—C16	56.5 (4)	C24—C25—C26—C21	-0.6 (8)
C16—C11—C12—C13	-0.8 (8)	C22—C21—C26—C25	0.3 (8)
P1—C11—C12—C13	176.8 (4)	P1—C21—C26—C25	177.6 (4)

Symmetry code: (i) $-x+3/2, -y+3/2, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A \cdots I1 ⁱⁱ	0.99	3.04	3.873 (5)	143

Symmetry code: (ii) $x, -y+3/2, z-1/2$.