metal-organic compounds

Bis{μ-1,4-bis[(diphenylphosphanyl)ethynyl]benzene-$\kappa^2 P$:P'}bis[trans-diiodidopalladium(II)] chloroform trisolvate

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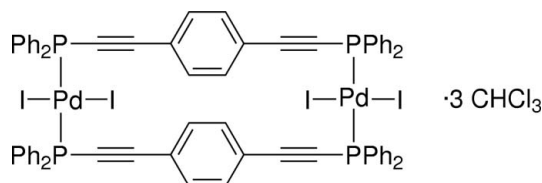Correspondence e-mail: alough@chem.utoronto.ca

The title compound, [Pd$_2$I$_4$(C$_{34}$H$_{24}$P$_2$)$_2$]·3CHCl$_3$, was obtained from the direct reaction of PdI$_2$ with 1,4-bis[(diphenylphosphanyl)ethynyl]benzene. The dimer complex molecule is centrosymmetric, with each Pd^{II} atom in a slightly distorted square-planar coordination geometry.

Related literature

Metal-containing rings are currently of interest as polymer precursors (Nguyen *et al.*, 1999; Herbert *et al.*, 2007). We have also determined the structure of a triclinic modification of the title compound (Baumgartner *et al.*, 2007). A related complex containing Pt^{II} atoms and chloride ligands was found to form a triangle (Baumgartner *et al.*, 2002).

Experimental

Crystal data

[Pd$_2$I$_4$(C$_{34}$H$_{24}$P$_2$)$_2$]·3CHCl$_3$	V = 7669.6 (7) Å^3
M_r = 2067.45	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 20.8960 (9) Å	μ = 2.52 mm^{-1}
b = 9.5920 (5) Å	T = 150 (1) K
c = 38.832 (2) Å	0.14 × 0.06 × 0.02 mm
β = 99.804 (3)°	

Data collection

Nonius KappaCCD diffractometer	19576 measured reflections
Absorption correction: multi-scan	6672 independent reflections
(*SORTAV*; Blessing, 1995)	3732 reflections with $I > 2\sigma(I)$
T_{min} = 0.827, T_{max} = 0.958	R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056	4 restraints
$wR(F^2)$ = 0.112	H-atom parameters constrained
S = 1.01	$\Delta\rho_{max}$ = 1.06 e Å^{-3}
6672 reflections	$\Delta\rho_{min}$ = −0.91 e Å^{-3}
411 parameters	

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supplementary materials

Bis{*μ*-1,4-bis[(diphenylphosphanyl)ethynyl]benzene-*κ²P:P'*}bis[*trans*-diiodidopalladium(II)] chloroform trisolvate

W. Y. Chan, T. Baumgartner, A. J. Lough and I. Manners

Comment

The bidentate phosphane ligand used in this work has been previously found to form a triangle when reacted with $PtCl_2$ (Baumgartner *et al.*, 2002). In this work, we report the formation of a rectangle using PdI_2 as the metal source. The Pd centres in the title compound exhibit a slightly distorted square planar geometry, where the P—Pd—I angles deviate from 90° by less than 3°.

Experimental

PdI_2 (11 mg, 0.031 mmol) and 1,4-bis-[(diphenylphosphanyl)-ethynyl]-benzene (15 mg, 0.030 mmol) was added to $CDCl_3$ (0.6 ml). Crystals of the title compound was obtained after 2 days.

Refinement

All hydrogen atoms were placed in calculated positions with C—H = 0.95 or 1.00 Å and were included in the refinement in the riding-motion approximation with $U_{iso}(H) = 1.2U_{eq}(C)$. One of the $CHCl_3$ solvent molecules is disordered over a twofold rotation axis. For each $CHCl_3$ the three C—Cl distances were constrained to be equal with a standard uncertainty of 0.002Å [see: SADI command in *SHELXTL* (Sheldrick, 2001)]

Figures
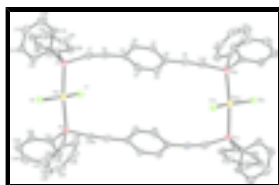
Fig. 1. The molecular structure with displacement ellipsoids drawn at the 30% probability level. Neither the chloroform solvent molecules nor H atoms are shown. Atoms labeled with the suffix 'a' are related by the symmetry operator (−*x* + 3/2,−*y* + 1/2,-*z* + 1).

Bis{μ-1,4-bis[(diphenylphosphanyl)ethynyl]benzene-*κ²P:P'*}bis[*trans*- diiodidopalladium(II)] chloroform trisolvate

Crystal data

[Pd$_2$I$_4$(C$_{34}$H$_{24}$P$_2$)$_2$]·3CHCl$_3$	$F_{000} = 3976$
$M_r = 2067.45$	$D_x = 1.790$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 19576 reflections

$a = 20.8960\ (9)$ Å

$b = 9.5920\ (5)$ Å

$c = 38.832\ (2)$ Å

$\beta = 99.804\ (3)^\circ$

$V = 7669.6\ (7)$ Å^3

$Z = 4$

$\theta = 2.6\text{–}25.0^\circ$

$\mu = 2.52$ mm^{-1}

$T = 150\ (1)$ K

Needle, colourless

$0.14 \times 0.06 \times 0.02$ mm

Data collection

Nonius KappaCCD diffractometer	6672 independent reflections
Radiation source: fine-focus sealed tube	3732 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.086$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 25.0^\circ$
$T = 150(2)$ K	$\theta_{min} = 2.6^\circ$
φ scans and ω scans with κ offsets	$h = -24 \rightarrow 24$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$k = -11 \rightarrow 11$
$T_{min} = 0.827$, $T_{max} = 0.958$	$l = -45 \rightarrow 46$
19576 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.112$	$w = 1/[\sigma^2(F_o^2) + (0.024P)^2 + 42.7375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.002$
6672 reflections	$\Delta\rho_{max} = 1.06$ e Å^{-3}
411 parameters	$\Delta\rho_{min} = -0.91$ e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
Pd1	0.67135 (3)	0.12571 (7)	0.633756 (18)	0.0337 (2)	
I1	0.72848 (3)	−0.04250 (7)	0.595490 (18)	0.0489 (2)	
I2	0.61881 (3)	0.24764 (6)	0.681462 (16)	0.04193 (19)	
P1	0.57291 (10)	0.0364 (2)	0.60639 (6)	0.0338 (6)	
P2	0.77171 (11)	0.2254 (2)	0.65447 (6)	0.0363 (6)	
C1	0.5693 (4)	0.0548 (9)	0.5615 (3)	0.044 (3)	
C2	0.5810 (4)	0.0764 (9)	0.5327 (2)	0.040 (2)	
C3	0.6019 (4)	0.1060 (10)	0.5002 (2)	0.040 (2)	
C4	0.6216 (5)	0.2398 (11)	0.4932 (3)	0.061 (3)	
H4A	0.6192	0.3123	0.5096	0.073*	
C5	0.6448 (5)	0.2686 (11)	0.4626 (3)	0.059 (3)	
H5A	0.6577	0.3608	0.4580	0.071*	
C6	0.6491 (4)	0.1649 (10)	0.4388 (2)	0.043 (2)	
C7	0.6299 (4)	0.0311 (9)	0.4456 (2)	0.043 (2)	
H7A	0.6329	−0.0411	0.4291	0.051*	
C8	0.6064 (4)	0.0008 (10)	0.4760 (2)	0.043 (2)	
H8A	0.5933	−0.0915	0.4803	0.052*	
C9	0.6710 (4)	0.1963 (9)	0.4067 (3)	0.045 (3)	
C10	0.6902 (4)	0.2313 (9)	0.3808 (2)	0.039 (2)	
C11	0.5605 (4)	−0.1473 (9)	0.6139 (2)	0.034 (2)	
C12	0.5926 (4)	−0.2073 (10)	0.6446 (2)	0.045 (3)	
H12A	0.6227	−0.1541	0.6604	0.054*	
C13	0.5805 (4)	−0.3451 (10)	0.6522 (3)	0.047 (3)	
H13A	0.6024	−0.3867	0.6731	0.056*	
C14	0.5369 (5)	−0.4205 (10)	0.6294 (3)	0.053 (3)	
H14A	0.5283	−0.5145	0.6348	0.063*	
C15	0.5047 (5)	−0.3619 (11)	0.5983 (3)	0.064 (3)	
H15A	0.4745	−0.4155	0.5826	0.077*	
C16	0.5172 (4)	−0.2262 (9)	0.5908 (3)	0.044 (3)	
H16A	0.4960	−0.1858	0.5695	0.053*	
C21	0.4980 (4)	0.1166 (9)	0.6141 (2)	0.034 (2)	
C22	0.4672 (4)	0.0657 (10)	0.6404 (2)	0.045 (3)	
H22A	0.4846	−0.0130	0.6536	0.054*	
C23	0.4114 (4)	0.1278 (11)	0.6476 (3)	0.051 (3)	
H23A	0.3904	0.0911	0.6655	0.061*	
C24	0.3862 (5)	0.2435 (11)	0.6288 (3)	0.061 (3)	
H24A	0.3479	0.2864	0.6339	0.073*	
C25	0.4161 (4)	0.2965 (9)	0.6030 (3)	0.057 (3)	
H25A	0.3988	0.3763	0.5903	0.069*	
C26	0.4718 (4)	0.2337 (9)	0.5956 (2)	0.045 (2)	
H26A	0.4924	0.2708	0.5776	0.053*	
C31	0.7750 (4)	0.3889 (10)	0.6778 (3)	0.041 (2)	
C32	0.8069 (5)	0.4039 (11)	0.7117 (3)	0.055 (3)	
H32A	0.8268	0.3249	0.7238	0.066*	
C33	0.8103 (5)	0.5302 (14)	0.7282 (3)	0.076 (4)	

H33A	0.8324	0.5386	0.7517	0.092*	
C34	0.7823 (5)	0.6428 (14)	0.7111 (4)	0.080 (4)	
H34A	0.7843	0.7297	0.7230	0.096*	
C35	0.7511 (5)	0.6362 (12)	0.6771 (4)	0.068 (4)	
H35A	0.7321	0.7168	0.6654	0.082*	
C36	0.7483 (4)	0.5061 (11)	0.6601 (3)	0.053 (3)	
H36A	0.7279	0.4988	0.6364	0.063*	
C41	0.8270 (4)	0.1125 (9)	0.6827 (2)	0.038 (2)	
C42	0.8062 (5)	0.0596 (10)	0.7121 (3)	0.056 (3)	
H42A	0.7633	0.0772	0.7160	0.067*	
C43	0.8482 (6)	−0.0186 (11)	0.7354 (3)	0.067 (3)	
H43A	0.8343	−0.0508	0.7561	0.080*	
C44	0.9086 (6)	−0.0514 (11)	0.7300 (3)	0.071 (3)	
H44A	0.9364	−0.1086	0.7461	0.085*	
C45	0.9286 (5)	0.0010 (11)	0.7005 (3)	0.066 (3)	
H45A	0.9710	−0.0205	0.6963	0.080*	
C46	0.8881 (5)	0.0846 (10)	0.6767 (3)	0.053 (3)	
H46A	0.9029	0.1214	0.6567	0.064*	
Cl1	0.56608 (16)	0.6246 (3)	0.51296 (10)	0.0983 (11)	
Cl2	0.7013 (2)	0.6345 (6)	0.5168 (2)	0.255 (4)	
Cl3	0.6484 (3)	0.4900 (7)	0.56931 (13)	0.206 (3)	
C1S	0.63949 (19)	0.6309 (12)	0.5412 (2)	0.150 (8)	
H1SA	0.6412	0.7185	0.5553	0.180*	
Cl5	1.03790 (18)	−0.6728 (4)	0.72255 (9)	0.1068 (12)	
Cl4	1.0000	−0.4169 (5)	0.7500	0.125 (2)	
C2S	0.9795 (9)	−0.5888 (12)	0.7413 (6)	0.110 (13)	0.50
H2SA	0.9404	−0.5888	0.7242	0.132*	0.50

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0336 (4)	0.0372 (4)	0.0304 (4)	−0.0009 (3)	0.0059 (3)	−0.0015 (4)
I1	0.0401 (4)	0.0537 (4)	0.0545 (5)	0.0011 (3)	0.0122 (3)	−0.0148 (4)
I2	0.0428 (4)	0.0494 (4)	0.0349 (4)	−0.0031 (3)	0.0102 (3)	−0.0075 (3)
P1	0.0339 (13)	0.0402 (15)	0.0282 (15)	−0.0027 (11)	0.0075 (11)	−0.0005 (12)
P2	0.0398 (14)	0.0423 (16)	0.0275 (14)	−0.0021 (12)	0.0076 (12)	0.0013 (12)
C1	0.045 (6)	0.042 (6)	0.048 (7)	−0.003 (5)	0.015 (5)	−0.009 (5)
C2	0.043 (6)	0.041 (6)	0.037 (6)	−0.012 (5)	0.009 (5)	0.003 (5)
C3	0.051 (6)	0.044 (6)	0.027 (6)	−0.006 (5)	0.012 (5)	0.001 (5)
C4	0.101 (9)	0.050 (7)	0.036 (7)	−0.014 (6)	0.024 (6)	−0.004 (5)
C5	0.081 (8)	0.058 (7)	0.044 (7)	−0.025 (6)	0.023 (6)	−0.002 (6)
C6	0.050 (6)	0.044 (6)	0.036 (6)	−0.009 (5)	0.009 (5)	−0.001 (5)
C7	0.051 (6)	0.044 (6)	0.033 (6)	−0.014 (5)	0.009 (5)	−0.001 (5)
C8	0.047 (6)	0.045 (6)	0.038 (6)	−0.008 (5)	0.010 (5)	0.007 (5)
C9	0.039 (6)	0.051 (6)	0.045 (7)	−0.012 (5)	0.009 (5)	0.006 (5)
C10	0.037 (5)	0.040 (6)	0.042 (6)	−0.016 (5)	0.010 (5)	−0.001 (5)
C11	0.032 (5)	0.041 (6)	0.033 (6)	−0.002 (5)	0.012 (5)	−0.003 (5)
C12	0.054 (6)	0.048 (7)	0.036 (7)	−0.011 (5)	0.012 (5)	0.000 (5)

C13	0.038 (6)	0.047 (7)	0.052 (7)	0.000 (5)	0.002 (5)	0.004 (6)
C14	0.062 (7)	0.033 (6)	0.065 (8)	−0.005 (5)	0.018 (6)	0.012 (6)
C15	0.068 (7)	0.043 (7)	0.079 (10)	−0.021 (6)	0.007 (7)	−0.009 (7)
C16	0.043 (6)	0.033 (6)	0.054 (7)	−0.009 (5)	0.000 (5)	0.001 (5)
C21	0.034 (5)	0.034 (6)	0.035 (6)	0.000 (4)	0.006 (5)	0.001 (5)
C22	0.045 (6)	0.055 (6)	0.040 (6)	−0.002 (5)	0.023 (5)	−0.004 (5)
C23	0.050 (6)	0.060 (7)	0.043 (7)	−0.003 (6)	0.009 (5)	−0.005 (6)
C24	0.038 (6)	0.056 (7)	0.089 (9)	−0.005 (6)	0.012 (6)	−0.018 (7)
C25	0.035 (6)	0.034 (6)	0.102 (10)	0.006 (5)	0.006 (6)	0.006 (6)
C26	0.044 (6)	0.039 (6)	0.051 (7)	−0.009 (5)	0.010 (5)	−0.001 (5)
C31	0.040 (6)	0.054 (7)	0.029 (6)	−0.014 (5)	0.003 (5)	0.001 (5)
C32	0.059 (7)	0.048 (7)	0.061 (8)	0.000 (5)	0.017 (6)	−0.014 (6)
C33	0.067 (8)	0.091 (10)	0.071 (10)	0.005 (8)	0.012 (7)	−0.029 (9)
C34	0.047 (8)	0.072 (10)	0.126 (14)	−0.017 (7)	0.026 (8)	−0.049 (10)
C35	0.047 (7)	0.052 (8)	0.111 (12)	−0.007 (6)	0.025 (8)	0.002 (8)
C36	0.046 (6)	0.053 (7)	0.059 (8)	−0.011 (6)	0.006 (6)	0.001 (6)
C41	0.036 (5)	0.039 (6)	0.037 (6)	−0.001 (5)	−0.001 (5)	0.001 (5)
C42	0.052 (6)	0.064 (7)	0.049 (7)	−0.005 (6)	0.004 (6)	0.024 (6)
C43	0.071 (8)	0.064 (8)	0.056 (8)	−0.009 (7)	−0.017 (7)	0.032 (6)
C44	0.064 (8)	0.058 (8)	0.080 (10)	0.004 (7)	−0.021 (7)	0.023 (7)
C45	0.050 (7)	0.074 (9)	0.073 (9)	0.026 (6)	0.005 (7)	0.000 (7)
C46	0.049 (6)	0.062 (7)	0.048 (7)	0.008 (6)	0.005 (6)	−0.002 (6)
Cl1	0.106 (3)	0.084 (2)	0.103 (3)	−0.009 (2)	0.012 (2)	−0.011 (2)
Cl2	0.114 (4)	0.210 (6)	0.467 (11)	−0.044 (4)	0.127 (5)	−0.152 (7)
Cl3	0.205 (5)	0.282 (7)	0.101 (4)	0.136 (5)	−0.064 (4)	−0.085 (4)
C1S	0.059 (9)	0.165 (16)	0.21 (2)	−0.002 (10)	−0.009 (11)	−0.137 (15)
Cl5	0.127 (3)	0.115 (3)	0.084 (3)	0.009 (2)	0.033 (2)	−0.017 (2)
Cl4	0.150 (5)	0.084 (4)	0.159 (6)	0.000	0.081 (4)	0.000
C2S	0.042 (18)	0.12 (2)	0.18 (4)	0.011 (13)	0.05 (2)	0.10 (2)

Geometric parameters (Å, °)

Pd1—P1	2.313 (2)	C23—C24	1.383 (13)
Pd1—P2	2.321 (2)	C23—H23A	0.9500
Pd1—I2	2.5880 (9)	C24—C25	1.365 (13)
Pd1—I1	2.6155 (9)	C24—H24A	0.9500
P1—C1	1.743 (10)	C25—C26	1.384 (11)
P1—C11	1.812 (9)	C25—H25A	0.9500
P1—C21	1.813 (8)	C26—H26A	0.9500
P2—C10[i]	1.749 (9)	C31—C32	1.379 (12)
P2—C31	1.806 (10)	C31—C36	1.384 (12)
P2—C41	1.809 (9)	C32—C33	1.367 (13)
C1—C2	1.202 (11)	C32—H32A	0.9500
C2—C3	1.434 (12)	C33—C34	1.349 (16)
C3—C4	1.388 (12)	C33—H33A	0.9500
C3—C8	1.393 (11)	C34—C35	1.370 (15)
C4—C5	1.388 (12)	C34—H34A	0.9500
C4—H4A	0.9500	C35—C36	1.408 (13)
C5—C6	1.371 (12)	C35—H35A	0.9500

C5—H5A	0.9500	C36—H36A	0.9500
C6—C7	1.384 (11)	C41—C46	1.364 (11)
C6—C9	1.430 (12)	C41—C42	1.386 (12)
C7—C8	1.384 (11)	C42—C43	1.373 (12)
C7—H7A	0.9500	C42—H42A	0.9500
C8—H8A	0.9500	C43—C44	1.351 (13)
C9—C10	1.191 (11)	C43—H43A	0.9500
C10—P2^{i}	1.749 (9)	C44—C45	1.381 (14)
C11—C16	1.386 (11)	C44—H44A	0.9500
C11—C12	1.390 (11)	C45—C46	1.396 (13)
C12—C13	1.386 (12)	C45—H45A	0.9500
C12—H12A	0.9500	C46—H46A	0.9500
C13—C14	1.363 (12)	Cl1—C1S	1.728 (5)
C13—H13A	0.9500	Cl2—C1S	1.728 (5)
C14—C15	1.396 (13)	Cl3—C1S	1.728 (5)
C14—H14A	0.9500	C1S—H1SA	1.0000
C15—C16	1.370 (12)	Cl5—C2S^{ii}	1.71 (2)
C15—H15A	0.9500	Cl5—C2S	1.723 (11)
C16—H16A	0.9500	Cl4—C2S	1.723 (11)
C21—C22	1.384 (11)	Cl4—C2S^{ii}	1.723 (11)
C21—C26	1.395 (11)	C2S—C2S^{ii}	1.00 (4)
C22—C23	1.379 (11)	C2S—Cl5^{ii}	1.71 (2)
C22—H22A	0.9500	C2S—H2SA	0.9600
P1—Pd1—P2	172.93 (9)	C22—C23—H23A	120.1
P1—Pd1—I2	92.51 (6)	C24—C23—H23A	120.1
P2—Pd1—I2	91.96 (6)	C25—C24—C23	120.3 (9)
P1—Pd1—I1	88.09 (6)	C25—C24—H24A	119.8
P2—Pd1—I1	88.75 (6)	C23—C24—H24A	119.8
I2—Pd1—I1	167.36 (4)	C24—C25—C26	119.9 (10)
C1—P1—C11	106.1 (4)	C24—C25—H25A	120.1
C1—P1—C21	103.4 (4)	C26—C25—H25A	120.1
C11—P1—C21	103.6 (4)	C25—C26—C21	120.8 (9)
C1—P1—Pd1	107.8 (3)	C25—C26—H26A	119.6
C11—P1—Pd1	115.3 (3)	C21—C26—H26A	119.6
C21—P1—Pd1	119.5 (3)	C32—C31—C36	118.5 (10)
C10^{i}—P2—C31	101.7 (4)	C32—C31—P2	122.7 (8)
C10^{i}—P2—C41	106.7 (4)	C36—C31—P2	118.6 (8)
C31—P2—C41	104.5 (4)	C33—C32—C31	121.3 (11)
C10^{i}—P2—Pd1	109.3 (3)	C33—C32—H32A	119.4
C31—P2—Pd1	119.1 (3)	C31—C32—H32A	119.4
C41—P2—Pd1	114.2 (3)	C34—C33—C32	119.6 (12)
C2—C1—P1	165.4 (9)	C34—C33—H33A	120.2
C1—C2—C3	173.7 (10)	C32—C33—H33A	120.2
C4—C3—C8	118.9 (8)	C33—C34—C35	122.2 (13)
C4—C3—C2	120.0 (8)	C33—C34—H34A	118.9
C8—C3—C2	121.1 (8)	C35—C34—H34A	118.9
C3—C4—C5	120.6 (9)	C34—C35—C36	117.9 (12)

C3—C4—H4A	119.7	C34—C35—H35A	121.0
C5—C4—H4A	119.7	C36—C35—H35A	121.0
C6—C5—C4	120.4 (9)	C31—C36—C35	120.4 (10)
C6—C5—H5A	119.8	C31—C36—H36A	119.8
C4—C5—H5A	119.8	C35—C36—H36A	119.8
C5—C6—C7	119.3 (9)	C46—C41—C42	120.2 (9)
C5—C6—C9	120.1 (9)	C46—C41—P2	122.3 (7)
C7—C6—C9	120.5 (9)	C42—C41—P2	117.4 (7)
C6—C7—C8	120.9 (9)	C43—C42—C41	119.3 (10)
C6—C7—H7A	119.5	C43—C42—H42A	120.3
C8—C7—H7A	119.5	C41—C42—H42A	120.3
C7—C8—C3	119.8 (8)	C44—C43—C42	122.2 (11)
C7—C8—H8A	120.1	C44—C43—H43A	118.9
C3—C8—H8A	120.1	C42—C43—H43A	118.9
C10—C9—C6	175.6 (10)	C43—C44—C45	117.9 (10)
C9—C10—P2^{i}	172.5 (9)	C43—C44—H44A	121.0
C16—C11—C12	119.8 (8)	C45—C44—H44A	121.0
C16—C11—P1	121.5 (7)	C44—C45—C46	121.6 (10)
C12—C11—P1	118.7 (7)	C44—C45—H45A	119.2
C13—C12—C11	119.9 (9)	C46—C45—H45A	119.2
C13—C12—H12A	120.1	C41—C46—C45	118.7 (10)
C11—C12—H12A	120.1	C41—C46—H46A	120.6
C14—C13—C12	119.6 (10)	C45—C46—H46A	120.6
C14—C13—H13A	120.2	Cl2—C1S—Cl1	108.6 (5)
C12—C13—H13A	120.2	Cl2—C1S—Cl3	110.6 (6)
C13—C14—C15	121.1 (9)	Cl1—C1S—Cl3	111.2 (5)
C13—C14—H14A	119.4	Cl2—C1S—H1SA	108.8
C15—C14—H14A	119.4	Cl1—C1S—H1SA	108.8
C16—C15—C14	119.1 (10)	Cl3—C1S—H1SA	108.8
C16—C15—H15A	120.5	C2S^{ii}—Cl5—C2S	33.7 (14)
C14—C15—H15A	120.5	C2S—Cl4—C2S^{ii}	33.6 (15)
C15—C16—C11	120.5 (10)	C2S^{ii}—C2S—Cl5^{ii}	74 (2)
C15—C16—H16A	119.8	C2S^{ii}—C2S—Cl4	73.2 (7)
C11—C16—H16A	119.8	Cl5^{ii}—C2S—Cl4	111.7 (10)
C22—C21—C26	118.2 (8)	C2S^{ii}—C2S—Cl5	72.5 (17)
C22—C21—P1	119.7 (7)	Cl5^{ii}—C2S—Cl5	113.0 (10)
C26—C21—P1	122.0 (7)	Cl4—C2S—Cl5	111.2 (10)
C23—C22—C21	121.0 (9)	C2S^{ii}—C2S—H2SA	179.2
C23—C22—H22A	119.5	Cl5^{ii}—C2S—H2SA	106.9
C21—C22—H22A	119.5	Cl4—C2S—H2SA	106.8
C22—C23—C24	119.8 (9)	Cl5—C2S—H2SA	106.8

Symmetry codes: (i) −x+3/2, −y+1/2, −z+1; (ii) −x+2, y, −z+3/2.

Fig. 1