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cis-Dichloridobis{[(*S*)-*N*-(3,5-dioxa-4-phosphacyclohepta[2,1-*a*;3,4-*a*′]-dinaphthalen-4-yl]dibenz[*b*,*f*]azepin-κ*P*}-palladium(II) deuterochloroform disolvate

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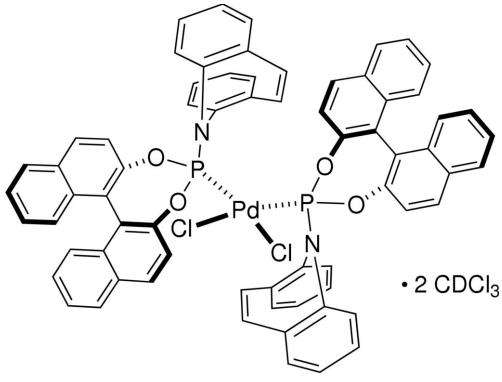
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.017 Å; some non-H atoms missing; disorder in main residue; R factor = 0.074; wR factor = 0.248; data-to-parameter ratio = 16.3.

The chiral bidentate P-olefin ligand (*S*)-(3,5-dioxa-4-phosphacyclohepta[2,1-*a*;3,4-*a*′]dinaphthalen-4-yl)dibenzo[*b*,*f*]azepin reacted with $PdCl_2(NCCH_3)_2$ to form the title compound, $[PdCl_2(C_{34}H_{22}NO_2P)_2]\cdot 2CDCl_3$. The Pd atom displays a distorted $PdCl_2P_2$ square-planar *cis* geometry. The title compound forms a three-dimensional hydrogen-bonded network from supramolecular right-handed helices assembled *via* C—H···Cl interactions, resulting in an array of channels along the *c* axis filled with deuterochloroform solvent molecules.

Related literature

For related literature, see: Deblon *et al.* (2003); Defieber *et al.* (2007); Maire *et al.* (2004); Van den Berg *et al.* (2000).



Experimental

Crystal data

$[PdCl_2(C_{34}H_{22}NO_2P)_2]\cdot 2CDCl_3$
M_r = 1431.03
Tetragonal, $P4_1$
a = 19.866 (2) Å
c = 17.497 (2) Å
V = 6905.2 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 293 (2) K
0.42 × 0.30 × 0.25 mm

Data collection

Rigaku AFC-7S Mercury diffractometer
Absorption correction: multi-scan Jacobson (1998)
T_{min} = 0.802, T_{max} = 0.891
(expected range = 0.761–0.845)
79958 measured reflections
14408 independent reflections
9124 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.248
S = 1.07
14408 reflections
886 parameters
593 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Absolute structure: Flack (1983), 5079 Friedel pairs
Flack parameter: 0.02 (4)

Table 1
Selected bond lengths (Å).

Pd1—P2	2.232 (2)	Pd1—Cl2	2.334 (2)
Pd1—P1	2.232 (2)	Pd1—Cl1	2.343 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2*S*—H2*S*···Cl1^{i}	0.97	2.47	3.358 (10)	151
C27—H27···Cl2^{ii}	0.93	2.82	3.741 (12)	172
C14—H14···Cl2^{ii}	0.93	2.84	3.613 (10)	142

Symmetry codes: (i) $-y+1, x, z-\frac{3}{4}$; (ii) $-y+1, x, z+\frac{1}{4}$.

Data collection: *CrystalClear* (Rigaku/MSC Inc., 2000); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC Inc., 2004); program(s) used to solve structure: *SHELXTL-NT* (Bruker, 1998); program(s) used to refine structure: *SHELXTL-NT*; molecular graphics: *SHELXTL-NT* and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXTL-NT* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2403).

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supplementary materials

Acta Cryst. (2007). E**63**, m1718-m1719 [**doi:10.1107/S1600536807023173**]

cis-Dichloridobis{[(*S*)-*N*-(3,5-dioxa-4-phosphacyclohepta[2,1-*a*;3,4-*a*']dinaphthalen-4-yl]dibenz[*b*,*f*]azepin-κ*P*}palladium(II) deuterochloroform disolvate

A. Briceño and R. Dorta

Comment

P-olefin ligands constitute a rather new entry in the development of efficient bidentate ligand systems for organometallic reactivity and catalysis. Grützmacher *et al.* developed a new class of P-olefin ligands based on the dibenzo[b,f]cycloheptene and dibenzo[b,f]azepin motifs, several of them chiral (Maire *et al.*, 2004). On the other hand, de Vries and Feringa (Van den Berg *et al.*, 2000) showed that chiral phosphoramidites are highly versatile monodentate ligands and so we anticipated that the dibenzo[b,f]azepin molecule could be readily used to form new chiral phosphoramidite-olefin ligands such as (I). Ligand (I) has very recently been disclosed to be an excellent ligand for the enantioselective formation of allylic amines from allylic alcohols (Defieber *et al.*, 2007).

Here, we report the crystal structure of a Pd(II) complex bearing the chiral ligand (I). The asymmetric unit of (II) consists of one metal complex molecule and three independent deuterocloroform molecules (two of which are half occupied). The Pd atom displays a distorted square planar environment, in which two ligands (I) are coordinated through their P atoms in a monodentade fashion and the remaining coordination sites are occupied by two chloride anions. The ligands are located in a *cis* configuration around the metal centre [P2—Pd1—P1 = 94.59 (7)°; Cl2—Pd—Cl1 = 89.24 (9)°]. The metal complex adopts pseudo-*C*2 symmetry about the Pd atom (Fig. 1). The naphtyl groups are twisted in both molecules as indicated by torsion angles of 50.39 (2)° for C1—C10—C11—C12 and 52.23 (2)° for C35—C44—C45—C46.

The crystal structure of (II) consists of a three-dimensional-hydrogen bonded network, which is self assembled *via* C—H$^{\cdots}$π and C—H$^{\cdots}$Cl interaction [C27$^{i\cdots}$CL2: 3.741 (12) Å and C14$^{i\cdots}$Cl2: 3.613 (10) Å; i= *x*, 1 − *y*, 1/4 + *z*]. The coordinated chloride (Cl2) ion causes the self-assembly of the metal complex to generate a right-handed helical arrangement along the fourfold screw axis (4_1). Neighbouring helices are assembled by weak van der Waals interactions to afford a 3-D hydrogen-bonded assembly. This organization yields a framework with one-dimensional channels running along the *c* axis, where the deuterochloroform molecules are allocated, and which are sustained into the channels by C—H$^{\cdots}$Cl hydrogen bonding to the chloride ligands of the complex [Cl1$^{\cdots}$C2S^{ii}: 3.358 Å; ii= *x*, 1 − *y*, −3/4 + *z*] (Fig 2).

Experimental

Two equivalents of ligand (I) reacted cleanly with $PdCl_2(NCCH_3)_2$ in toluene/acetonitrile solution to afford the title compound (II) in yields of over 90%. Single crystals of (II) were grown from a $CDCl_3$ solution layered with Et_2O. The crystals are unstable outside their mother-liquor, and a single-crystal was sealed in a Lindemann capillary for intensity collection, together with its mother-liquor.

Refinement

All H atoms bound to carbon were included in calculated positions (C—H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = 1.2Ueq(C).

The dibenzoazepine framgment containing the N2 atom was found disordered over two positions. The occupational parameters were determined to be 0.70 and 0.30. The non-hydrogen atoms of the six-menbered ring of this frament were refined with the atoms being forced into planarity. The deuterochloroform molecules were also found disordered. The disorder of the C2S molecule was modelled by two sets of chlorine positions with occupancies 0.60 and 0.40. The best model for the remaining solvent molecules was obtained by free refinement of its occupation factors, giving a partial occupation of approximatly 0.5 for both molecules. These occupations were fixed during the final refinements. All C—Cl distances were restrained to similar lenghts. The absolute structure was assigned from the known configuration of the starting binaphtol reagent used in the synthesis of ligand (I) and confirmed by refinement of the Flack (1983) absolute structure parameter.

Figures

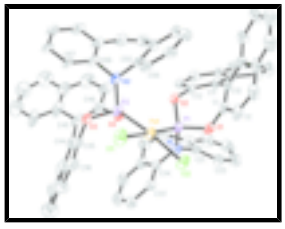

Fig. 1. Molecular structure of the main molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

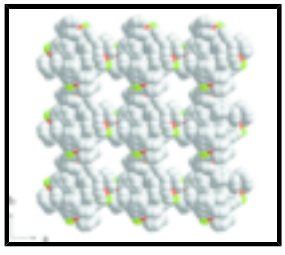

Fig. 2. View of the three-dimensional hydrogen bonded network observed in the crystal structure of (II), showing one-dimensional channels along the *c* axis.

Fig. 3. Reaction scheme.

Dichlorido- *cis*-bis[{(*S*)-(3,5-dioxa-4-phosphacyclohepta[2,1 – a;3,4–a']dinaphthalen-4-yl)dibenz[b,f]azepin}-\k*P*]palladium(II) deuterochloroform disolvate

Crystal data

$[PdCl_2(C_{34}H_{22}NO_2P)_2].2CDCl_3$	$Z = 4$
$M_r = 1431.03$	$F_{000} = 2896$
Tetragonal, $P4_1$	$D_x = 1.377$ Mg m^{-3}
Hall symbol: P 4w	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 19.866$ (2) Å	Cell parameters from 2378 reflections

b = 19.866 (2) Å
c = 17.497 (2) Å
α = 90º
β = 90º
γ = 90º
V = 6905.2 (13) Å^3

θ = 2.0–27.6º
μ = 0.67 mm^{-1}
T = 293 (2) K
Prism, yellow
0.42 × 0.30 × 0.25 mm

Data collection

Rigaku AFC7S Mercury diffractometer
Radiation source: normal-focus sealed tube
Monochromator: graphite
Detector resolution: 14.6306 pixels mm^{-1}
T = 293(2) K
ω scans
Absorption correction: Multi-scan Jacobson (1998)
T_{min} = 0.802, T_{max} = 0.891
79958 measured reflections

14408 independent reflections
9124 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 28.0º
θ_{min} = 1.5º
h = −24→21
k = −23→23
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.248
S = 1.07
14408 reflections
886 parameters
593 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
$w = 1/[\sigma^2(F_o^2) + (0.1446P)^2 + 3.0373P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}
Extinction correction: none
Absolute structure: Flack (1983), with 5079 Friedel pairs
Flack parameter: 0.02 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Pd1	0.46461 (3)	0.15920 (3)	0.07387 (5)	0.05517 (19)	
P2	0.51845 (11)	0.13326 (10)	0.18186 (12)	0.0550 (5)	
P1	0.54240 (10)	0.23245 (9)	0.03450 (12)	0.0515 (4)	
Cl2	0.39914 (12)	0.18825 (12)	−0.03184 (14)	0.0767 (6)	
Cl1	0.38606 (14)	0.07551 (13)	0.10470 (16)	0.0870 (8)	
O1	0.5486 (3)	0.2454 (3)	−0.0559 (3)	0.0584 (13)	
O2	0.6150 (2)	0.2024 (2)	0.0547 (3)	0.0511 (12)	
O4	0.5558 (3)	0.1988 (3)	0.2163 (3)	0.0591 (13)	
N1	0.5325 (3)	0.3100 (3)	0.0626 (4)	0.0546 (15)	
O3	0.4720 (3)	0.1113 (3)	0.2535 (3)	0.0614 (13)	
N2	0.5686 (4)	0.0687 (3)	0.1775 (4)	0.0601 (16)	
C1	0.5795 (5)	0.2007 (4)	−0.1058 (4)	0.060 (2)	
C2	0.5371 (5)	0.1683 (5)	−0.1609 (5)	0.073 (2)	
H2A	0.4910	0.1760	−0.1613	0.087*	
C3	0.5653 (6)	0.1268 (5)	−0.2116 (5)	0.077 (3)	
H3	0.5385	0.1092	−0.2503	0.093*	
C4	0.6333 (6)	0.1087 (5)	−0.2095 (5)	0.075 (2)	
C5	0.6571 (8)	0.0573 (6)	−0.2551 (7)	0.094 (3)	
H5	0.6287	0.0364	−0.2899	0.112*	
C6	0.7220 (10)	0.0376 (8)	−0.2491 (8)	0.118 (5)	
H6	0.7376	0.0028	−0.2800	0.141*	
C7	0.7696 (7)	0.0704 (6)	−0.1936 (7)	0.101 (4)	
H7	0.8145	0.0573	−0.1908	0.121*	
C8	0.7454 (6)	0.1190 (5)	−0.1486 (6)	0.081 (3)	
H8	0.7739	0.1397	−0.1136	0.098*	
C9	0.6768 (5)	0.1399 (4)	−0.1532 (5)	0.063 (2)	
C10	0.6474 (4)	0.1920 (4)	−0.1040 (4)	0.0544 (18)	
C11	0.6907 (4)	0.2292 (4)	−0.0508 (4)	0.0519 (17)	
C12	0.6748 (4)	0.2308 (4)	0.0278 (5)	0.0525 (17)	
C13	0.7195 (4)	0.2582 (4)	0.0825 (5)	0.0581 (18)	
H13	0.7101	0.2547	0.1344	0.070*	
C14	0.7761 (4)	0.2895 (4)	0.0584 (6)	0.068 (2)	
H14	0.8055	0.3067	0.0949	0.082*	
C15	0.7926 (4)	0.2973 (4)	−0.0205 (5)	0.0586 (19)	
C16	0.8480 (5)	0.3376 (6)	−0.0460 (7)	0.088 (3)	
H16	0.8754	0.3586	−0.0101	0.105*	
C17	0.8612 (5)	0.3456 (6)	−0.1210 (7)	0.089 (3)	
H17	0.8984	0.3702	−0.1372	0.106*	
C18	0.8180 (6)	0.3163 (6)	−0.1731 (7)	0.093 (3)	
H18	0.8261	0.3228	−0.2249	0.112*	
C19	0.7646 (5)	0.2785 (5)	−0.1523 (5)	0.077 (3)	
H19	0.7380	0.2589	−0.1898	0.092*	

C20	0.7485 (4)	0.2680 (4)	−0.0745 (5)	0.0599 (19)
C21	0.5706 (4)	0.3648 (4)	0.0274 (6)	0.062 (2)
C22	0.5534 (5)	0.3884 (5)	−0.0429 (6)	0.074 (2)
H22	0.5168	0.3706	−0.0690	0.089*
C23	0.5926 (7)	0.4409 (6)	−0.0759 (8)	0.103 (4)
H23	0.5805	0.4615	−0.1216	0.123*
C24	0.6528 (7)	0.4604 (7)	−0.0339 (9)	0.109 (4)
H24	0.6848	0.4874	−0.0577	0.131*
C25	0.6625 (7)	0.4415 (7)	0.0337 (9)	0.103 (4)
H25	0.6987	0.4599	0.0602	0.124*
C26	0.6224 (5)	0.3950 (4)	0.0710 (8)	0.083 (3)
C27	0.6332 (6)	0.3784 (5)	0.1545 (8)	0.093 (4)
H27	0.6774	0.3786	0.1717	0.111*
C28	0.5873 (7)	0.3639 (5)	0.2047 (7)	0.083 (3)
H28	0.6020	0.3570	0.2545	0.100*
C29	0.5158 (5)	0.3573 (4)	0.1913 (6)	0.071 (2)
C30	0.4712 (8)	0.3774 (5)	0.2496 (6)	0.096 (4)
H30	0.4879	0.3960	0.2945	0.115*
C31	0.4029 (8)	0.3694 (6)	0.2403 (8)	0.100 (4)
H31	0.3739	0.3837	0.2788	0.119*
C32	0.3770 (5)	0.3413 (6)	0.1763 (6)	0.082 (3)
H32	0.3308	0.3351	0.1714	0.099*
C33	0.4200 (5)	0.3218 (5)	0.1183 (5)	0.070 (2)
H33	0.4021	0.3032	0.0739	0.083*
C34	0.4880 (4)	0.3293 (4)	0.1245 (5)	0.0581 (18)
C35	0.4326 (5)	0.1589 (5)	0.2915 (5)	0.071 (2)
C36	0.3637 (6)	0.1578 (6)	0.2814 (7)	0.086 (3)
H36	0.3439	0.1278	0.2473	0.104*
C37	0.3255 (6)	0.2014 (6)	0.3222 (9)	0.099 (3)
H37	0.2788	0.1995	0.3188	0.119*
C38	0.3572 (9)	0.2514 (7)	0.3714 (7)	0.110 (5)
C39	0.3148 (10)	0.3015 (9)	0.4059 (10)	0.132 (5)
H39	0.2687	0.3015	0.3966	0.158*
C40	0.3424 (10)	0.3484 (8)	0.4520 (10)	0.127 (5)
H40	0.3150	0.3792	0.4772	0.152*
C41	0.4097 (9)	0.3507 (8)	0.4613 (8)	0.118 (4)
H41	0.4279	0.3842	0.4920	0.142*
C42	0.4522 (8)	0.3057 (7)	0.4274 (8)	0.108 (4)
H42	0.4985	0.3100	0.4341	0.130*
C43	0.4255 (8)	0.2510 (6)	0.3809 (6)	0.091 (3)
C44	0.4646 (6)	0.2013 (4)	0.3422 (5)	0.077 (3)
C45	0.5384 (7)	0.1966 (5)	0.3536 (6)	0.084 (3)
C46	0.5826 (5)	0.1984 (4)	0.2924 (5)	0.070 (2)
C47	0.6528 (6)	0.2003 (5)	0.2969 (7)	0.086 (3)
H47	0.6793	0.2051	0.2535	0.103*
C48	0.6815 (7)	0.1945 (7)	0.3696 (8)	0.107 (4)
H48	0.7278	0.1981	0.3756	0.128*
C49	0.6400 (7)	0.1831 (6)	0.4341 (8)	0.101 (3)
C50	0.6698 (11)	0.1701 (9)	0.5084 (11)	0.142 (5)

H50	0.7160	0.1681	0.5172	0.171*	
C51	0.6216 (11)	0.1611 (10)	0.5647 (12)	0.149 (6)	
H51	0.6392	0.1514	0.6127	0.178*	
C52	0.5558 (10)	0.1637 (8)	0.5626 (11)	0.136 (5)	
H52	0.5295	0.1564	0.6058	0.163*	
C53	0.5293 (9)	0.1772 (7)	0.4950 (6)	0.115 (5)	
H53	0.4828	0.1806	0.4904	0.138*	
C54	0.5703 (8)	0.1869 (5)	0.4288 (6)	0.096 (4)	
C55	0.5962 (5)	0.0410 (5)	0.1050 (4)	0.058 (2)	0.695 (16)
C56	0.5506 (5)	0.0098 (5)	0.0564 (5)	0.065 (3)	0.695 (16)
H56	0.5062	0.0035	0.0718	0.078*	0.695 (16)
C57	0.5713 (6)	−0.0121 (5)	−0.0154 (5)	0.077 (3)	0.695 (16)
H57	0.5407	−0.0330	−0.0479	0.092*	0.695 (16)
C58	0.6376 (7)	−0.0028 (6)	−0.0384 (4)	0.085 (4)	0.695 (16)
H58	0.6514	−0.0174	−0.0864	0.102*	0.695 (16)
C59	0.6832 (6)	0.0285 (6)	0.0103 (5)	0.077 (3)	0.695 (16)
H59	0.7276	0.0347	−0.0051	0.092*	0.695 (16)
C60	0.6625 (5)	0.0503 (5)	0.0820 (5)	0.066 (3)	0.695 (16)
C61	0.7128 (8)	0.0813 (8)	0.1411 (12)	0.074 (3)	0.695 (16)
H61	0.7465	0.1079	0.1198	0.089*	0.695 (16)
C62	0.7154 (9)	0.0763 (9)	0.2117 (11)	0.077 (3)	0.695 (16)
H62	0.7501	0.0984	0.2373	0.092*	0.695 (16)
C63	0.6679 (10)	0.0385 (9)	0.2562 (9)	0.067 (3)	0.695 (16)
C64	0.6964 (7)	0.0089 (8)	0.3205 (10)	0.080 (3)	0.695 (16)
H64	0.7424	0.0127	0.3293	0.096*	0.695 (16)
C65	0.6562 (7)	−0.0265 (7)	0.3716 (8)	0.083 (3)	0.695 (16)
H65	0.6753	−0.0463	0.4146	0.099*	0.695 (16)
C66	0.5874 (7)	−0.0322 (7)	0.3585 (9)	0.074 (3)	0.695 (16)
H66	0.5605	−0.0559	0.3927	0.088*	0.695 (16)
C67	0.5589 (7)	−0.0026 (8)	0.2942 (11)	0.066 (3)	0.695 (16)
H67	0.5129	−0.0064	0.2854	0.079*	0.695 (16)
C68	0.5991 (10)	0.0327 (10)	0.2431 (9)	0.061 (3)	0.695 (16)
C55A	0.5723 (12)	0.0395 (13)	0.1022 (11)	0.062 (4)	0.305 (16)
C56A	0.5214 (11)	0.0042 (13)	0.0653 (13)	0.059 (4)	0.305 (16)
H56A	0.4794	−0.0002	0.0881	0.071*	0.305 (16)
C57A	0.5334 (14)	−0.0246 (12)	−0.0059 (14)	0.077 (5)	0.305 (16)
H57A	0.4994	−0.0483	−0.0306	0.092*	0.305 (16)
C58A	0.5962 (16)	−0.0181 (14)	−0.0400 (12)	0.084 (5)	0.305 (16)
H58A	0.6042	−0.0373	−0.0876	0.100*	0.305 (16)
C59A	0.6471 (13)	0.0173 (14)	−0.0031 (14)	0.078 (4)	0.305 (16)
H59A	0.6891	0.0217	−0.0260	0.094*	0.305 (16)
C60A	0.6351 (11)	0.0461 (13)	0.0680 (14)	0.067 (4)	0.305 (16)
C61A	0.6951 (19)	0.0703 (18)	0.109 (2)	0.069 (4)	0.305 (16)
H61A	0.7288	0.0923	0.0816	0.083*	0.305 (16)
C62A	0.7034 (19)	0.060 (2)	0.1941 (18)	0.073 (4)	0.305 (16)
H62A	0.7489	0.0623	0.2062	0.087*	0.305 (16)
C63A	0.664 (2)	0.048 (2)	0.266 (2)	0.069 (4)	0.305 (16)
C64A	0.6841 (15)	0.020 (2)	0.335 (2)	0.074 (4)	0.305 (16)
H64A	0.7288	0.0238	0.3506	0.089*	0.305 (16)

C65A	0.6376 (16)	−0.0119 (17)	0.3818 (18)	0.074 (5)	0.305 (16)
H65A	0.6511	−0.0301	0.4283	0.088*	0.305 (16)
C66A	0.5707 (15)	−0.0169 (17)	0.359 (2)	0.067 (5)	0.305 (16)
H66A	0.5396	−0.0385	0.3900	0.080*	0.305 (16)
C67A	0.5505 (19)	0.010 (2)	0.289 (3)	0.064 (4)	0.305 (16)
H67A	0.5058	0.0070	0.2741	0.077*	0.305 (16)
C68A	0.597 (3)	0.043 (2)	0.2429 (19)	0.060 (4)	0.305 (16)
C2S	0.9015 (4)	0.2278 (5)	−0.7074 (4)	0.141 (6)	
H2S	0.9000	0.2771	−0.7069	0.169*	
Cl6	0.8807 (9)	0.1984 (9)	−0.6167 (4)	0.205 (8)	0.60
Cl7	0.9823 (4)	0.2023 (10)	−0.7313 (11)	0.182 (7)	0.60
Cl8	0.8439 (6)	0.1985 (9)	−0.7743 (6)	0.171 (6)	0.60
Cl6A	0.8781 (16)	0.1686 (13)	−0.6377 (10)	0.213 (10)	0.40
Cl7A	0.9894 (4)	0.2278 (15)	−0.7147 (16)	0.168 (9)	0.40
Cl8A	0.8676 (13)	0.2024 (16)	−0.7954 (8)	0.195 (10)	0.40
C3S	0.9472 (8)	0.1616 (12)	−0.0093 (10)	0.178 (16)*	0.50
H3SA	0.9545	0.2078	0.0085	0.214*	0.50
Cl9	0.9319 (8)	0.1577 (8)	−0.1072 (9)	0.203 (5)*	0.50
Cl10	1.0123 (7)	0.1075 (7)	0.0173 (9)	0.191 (5)*	0.50
Cl11	0.8718 (8)	0.1281 (8)	0.0235 (9)	0.202 (5)*	0.50
C1S	0.8771 (9)	0.1196 (7)	−0.3571 (9)	0.174 (16)*	0.50
H1S	0.8296	0.1086	−0.3486	0.209*	0.50
Cl3	0.9285 (11)	0.0522 (7)	−0.3290 (10)	0.263 (8)*	0.50
Cl4	0.8997 (9)	0.1906 (7)	−0.3039 (8)	0.226 (6)*	0.50
Cl5	0.8915 (10)	0.1362 (8)	−0.4541 (7)	0.255 (8)*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0611 (4)	0.0510 (3)	0.0534 (3)	−0.0077 (2)	−0.0145 (3)	0.0077 (3)
P2	0.0676 (12)	0.0490 (10)	0.0485 (10)	−0.0017 (8)	−0.0124 (9)	0.0068 (8)
P1	0.0576 (11)	0.0479 (10)	0.0491 (10)	−0.0041 (8)	−0.0124 (8)	0.0043 (8)
Cl2	0.0803 (14)	0.0791 (14)	0.0707 (14)	−0.0126 (11)	−0.0330 (11)	0.0156 (11)
Cl1	0.0904 (16)	0.0877 (16)	0.0831 (16)	−0.0376 (13)	−0.0170 (13)	0.0199 (13)
O1	0.067 (3)	0.057 (3)	0.052 (3)	0.002 (2)	−0.019 (3)	0.002 (2)
O2	0.058 (3)	0.048 (3)	0.047 (3)	0.002 (2)	−0.007 (2)	0.006 (2)
O4	0.080 (4)	0.050 (3)	0.048 (3)	0.002 (2)	−0.019 (3)	0.003 (2)
N1	0.052 (3)	0.046 (3)	0.065 (4)	−0.008 (2)	−0.007 (3)	0.001 (3)
O3	0.075 (4)	0.056 (3)	0.053 (3)	0.007 (2)	−0.004 (3)	0.014 (2)
N2	0.075 (4)	0.053 (4)	0.053 (4)	0.001 (3)	−0.007 (3)	0.009 (3)
C1	0.082 (6)	0.059 (4)	0.038 (4)	−0.005 (4)	−0.017 (4)	0.003 (3)
C2	0.094 (6)	0.077 (6)	0.047 (5)	−0.026 (5)	−0.014 (4)	0.006 (4)
C3	0.115 (8)	0.070 (6)	0.047 (5)	−0.027 (5)	−0.007 (5)	−0.016 (4)
C4	0.105 (8)	0.080 (6)	0.039 (4)	−0.017 (5)	0.001 (5)	−0.001 (4)
C5	0.128 (10)	0.092 (7)	0.061 (6)	−0.018 (7)	0.006 (6)	−0.019 (6)
C6	0.171 (15)	0.111 (10)	0.071 (7)	−0.005 (9)	0.006 (9)	−0.032 (7)
C7	0.131 (9)	0.097 (8)	0.074 (7)	0.021 (7)	0.027 (7)	−0.020 (6)
C8	0.103 (8)	0.081 (6)	0.060 (5)	0.008 (5)	0.005 (5)	−0.010 (5)

C9	0.079 (5)	0.061 (5)	0.051 (5)	−0.003 (4)	−0.004 (4)	−0.007 (3)
C10	0.072 (5)	0.059 (4)	0.032 (3)	−0.005 (4)	−0.007 (3)	0.002 (3)
C11	0.062 (4)	0.050 (4)	0.044 (4)	−0.009 (3)	−0.009 (3)	0.001 (3)
C12	0.048 (4)	0.050 (4)	0.059 (5)	0.004 (3)	−0.012 (3)	0.002 (3)
C13	0.067 (5)	0.055 (4)	0.053 (4)	0.006 (3)	−0.013 (4)	−0.014 (4)
C14	0.064 (5)	0.058 (4)	0.083 (7)	−0.003 (4)	−0.016 (4)	−0.017 (4)
C15	0.051 (4)	0.057 (4)	0.067 (5)	−0.005 (3)	−0.011 (4)	−0.007 (4)
C16	0.060 (5)	0.101 (8)	0.102 (9)	−0.018 (5)	−0.016 (5)	−0.015 (6)
C17	0.071 (6)	0.117 (8)	0.077 (7)	−0.032 (6)	−0.006 (5)	0.014 (6)
C18	0.101 (7)	0.111 (8)	0.068 (6)	−0.028 (6)	0.007 (6)	0.014 (6)
C19	0.079 (6)	0.095 (7)	0.056 (5)	−0.018 (5)	0.000 (4)	0.006 (4)
C20	0.061 (5)	0.056 (4)	0.063 (5)	−0.008 (3)	0.000 (4)	0.003 (4)
C21	0.057 (5)	0.043 (4)	0.085 (6)	0.001 (3)	0.000 (4)	−0.006 (4)
C22	0.080 (6)	0.065 (5)	0.078 (6)	0.004 (4)	0.014 (5)	0.011 (5)
C23	0.125 (7)	0.093 (6)	0.091 (6)	0.008 (5)	0.026 (5)	0.012 (5)
C24	0.110 (6)	0.092 (6)	0.125 (7)	−0.021 (5)	0.038 (6)	−0.010 (6)
C25	0.095 (6)	0.097 (6)	0.117 (7)	−0.023 (5)	0.016 (5)	−0.020 (5)
C26	0.063 (5)	0.054 (4)	0.131 (9)	−0.014 (4)	0.020 (6)	−0.022 (6)
C27	0.081 (7)	0.077 (6)	0.121 (10)	0.008 (5)	−0.048 (7)	−0.042 (7)
C28	0.105 (8)	0.071 (6)	0.074 (6)	−0.004 (5)	−0.007 (6)	−0.018 (5)
C29	0.094 (7)	0.049 (4)	0.070 (6)	−0.001 (4)	−0.017 (5)	0.000 (4)
C30	0.165 (13)	0.068 (6)	0.054 (5)	0.012 (7)	0.011 (7)	−0.003 (4)
C31	0.125 (11)	0.091 (8)	0.082 (8)	0.011 (7)	0.026 (8)	0.016 (6)
C32	0.069 (6)	0.101 (7)	0.076 (7)	0.010 (5)	0.005 (5)	0.019 (6)
C33	0.069 (5)	0.082 (6)	0.058 (5)	0.009 (4)	−0.006 (4)	0.011 (4)
C34	0.060 (5)	0.061 (5)	0.053 (4)	0.002 (3)	0.000 (4)	0.006 (4)
C35	0.098 (7)	0.067 (5)	0.050 (5)	0.008 (5)	0.004 (5)	0.014 (4)
C36	0.089 (7)	0.089 (7)	0.081 (7)	0.001 (5)	−0.007 (6)	0.014 (6)
C37	0.083 (7)	0.107 (8)	0.109 (9)	0.011 (6)	0.021 (7)	−0.002 (8)
C38	0.161 (13)	0.101 (9)	0.067 (7)	0.045 (9)	0.031 (8)	0.016 (6)
C39	0.146 (8)	0.123 (7)	0.127 (8)	0.021 (6)	0.010 (6)	0.016 (6)
C40	0.150 (8)	0.115 (7)	0.114 (7)	0.009 (6)	0.025 (6)	−0.004 (6)
C41	0.157 (8)	0.106 (7)	0.091 (6)	0.000 (6)	0.010 (6)	−0.011 (5)
C42	0.149 (7)	0.097 (6)	0.079 (6)	0.003 (6)	0.006 (6)	0.012 (5)
C43	0.136 (11)	0.078 (7)	0.060 (6)	0.007 (6)	−0.003 (6)	0.014 (5)
C44	0.113 (8)	0.052 (5)	0.065 (6)	0.001 (4)	−0.001 (5)	0.011 (4)
C45	0.133 (9)	0.054 (5)	0.064 (6)	0.012 (5)	−0.021 (6)	0.010 (4)
C46	0.100 (7)	0.057 (5)	0.054 (5)	0.005 (4)	−0.015 (5)	−0.008 (4)
C47	0.086 (7)	0.077 (6)	0.096 (8)	0.005 (5)	−0.034 (6)	−0.010 (5)
C48	0.104 (9)	0.119 (10)	0.097 (9)	−0.013 (7)	−0.053 (8)	0.000 (7)
C49	0.116 (6)	0.101 (6)	0.086 (6)	−0.015 (5)	−0.040 (6)	0.017 (5)
C50	0.146 (8)	0.143 (8)	0.139 (8)	−0.019 (7)	−0.031 (7)	−0.006 (7)
C51	0.166 (9)	0.154 (8)	0.126 (8)	−0.013 (7)	−0.013 (7)	−0.002 (7)
C52	0.156 (8)	0.137 (8)	0.116 (8)	−0.019 (6)	−0.015 (7)	0.008 (6)
C53	0.193 (14)	0.110 (9)	0.040 (5)	−0.018 (9)	−0.010 (7)	0.017 (5)
C54	0.167 (12)	0.061 (5)	0.059 (6)	−0.026 (6)	−0.029 (7)	0.005 (4)
C55	0.069 (5)	0.045 (5)	0.059 (5)	0.015 (5)	−0.006 (4)	0.011 (4)
C56	0.077 (6)	0.047 (5)	0.072 (6)	0.005 (5)	−0.004 (5)	0.003 (4)
C57	0.098 (7)	0.060 (6)	0.074 (6)	0.026 (6)	−0.005 (6)	−0.008 (5)

C58	0.107 (8)	0.083 (7)	0.064 (6)	0.024 (7)	0.005 (5)	−0.012 (5)
C59	0.091 (7)	0.073 (7)	0.066 (5)	0.010 (6)	0.012 (5)	0.015 (5)
C60	0.075 (6)	0.059 (5)	0.064 (5)	0.008 (5)	−0.003 (4)	0.006 (5)
C61	0.074 (6)	0.056 (6)	0.093 (5)	−0.003 (5)	−0.002 (6)	−0.004 (6)
C62	0.081 (6)	0.058 (7)	0.092 (5)	−0.011 (5)	−0.021 (5)	−0.006 (5)
C63	0.075 (5)	0.048 (7)	0.080 (6)	0.006 (5)	−0.020 (4)	−0.001 (5)
C64	0.086 (7)	0.067 (8)	0.087 (7)	0.014 (6)	−0.033 (5)	−0.001 (6)
C65	0.105 (8)	0.072 (8)	0.071 (7)	0.039 (6)	−0.020 (6)	0.008 (6)
C66	0.096 (7)	0.056 (8)	0.069 (5)	0.036 (6)	−0.004 (6)	0.017 (5)
C67	0.086 (6)	0.040 (7)	0.071 (6)	0.021 (5)	−0.006 (5)	0.019 (5)
C68	0.075 (4)	0.043 (8)	0.065 (5)	0.009 (5)	−0.019 (4)	0.013 (5)
C55A	0.067 (5)	0.059 (5)	0.061 (5)	0.005 (4)	−0.003 (3)	0.002 (4)
C56A	0.074 (9)	0.047 (9)	0.056 (8)	0.008 (8)	−0.016 (7)	0.017 (7)
C57A	0.094 (9)	0.060 (9)	0.076 (9)	0.011 (9)	−0.016 (9)	−0.007 (7)
C58A	0.101 (10)	0.078 (10)	0.072 (9)	0.023 (9)	−0.007 (8)	−0.004 (8)
C59A	0.093 (8)	0.068 (8)	0.073 (7)	0.012 (8)	0.010 (6)	0.003 (7)
C60A	0.076 (7)	0.055 (7)	0.070 (7)	0.002 (7)	0.005 (5)	0.008 (6)
C61A	0.066 (7)	0.056 (9)	0.086 (6)	0.010 (8)	0.003 (7)	0.008 (8)
C62A	0.076 (7)	0.052 (10)	0.089 (7)	0.001 (8)	−0.012 (6)	0.006 (8)
C63A	0.079 (6)	0.051 (9)	0.078 (6)	0.007 (7)	−0.016 (5)	0.002 (7)
C64A	0.085 (8)	0.061 (10)	0.077 (8)	0.022 (8)	−0.018 (6)	−0.003 (8)
C65A	0.092 (10)	0.057 (10)	0.072 (8)	0.037 (9)	−0.017 (7)	0.012 (8)
C66A	0.085 (9)	0.045 (11)	0.070 (8)	0.035 (9)	−0.009 (8)	0.012 (8)
C67A	0.080 (7)	0.046 (11)	0.066 (8)	0.020 (8)	−0.008 (6)	0.013 (8)
C68A	0.077 (6)	0.033 (9)	0.069 (7)	0.009 (8)	−0.012 (5)	0.001 (7)
C2S	0.121 (11)	0.148 (13)	0.155 (15)	−0.028 (10)	−0.036 (10)	0.019 (11)
Cl6	0.224 (11)	0.248 (18)	0.143 (8)	−0.111 (13)	−0.047 (8)	0.046 (10)
Cl7	0.149 (8)	0.182 (15)	0.214 (17)	0.004 (7)	−0.039 (8)	0.025 (12)
Cl8	0.209 (11)	0.131 (8)	0.173 (9)	−0.040 (8)	−0.092 (9)	0.023 (8)
Cl6A	0.26 (2)	0.179 (19)	0.203 (18)	−0.047 (15)	−0.004 (17)	0.037 (15)
Cl7A	0.153 (12)	0.20 (2)	0.155 (12)	0.029 (11)	−0.047 (9)	−0.031 (13)
Cl8A	0.27 (2)	0.138 (12)	0.171 (14)	−0.101 (14)	−0.104 (15)	0.046 (11)

Geometric parameters (Å, °)

Pd1—P2	2.232 (2)	C40—C41	1.35 (2)
Pd1—P1	2.232 (2)	C40—H40	0.9300
Pd1—Cl2	2.334 (2)	C41—C42	1.36 (2)
Pd1—Cl1	2.343 (2)	C41—H41	0.9300
P2—O4	1.615 (6)	C42—C43	1.457 (19)
P2—O3	1.617 (6)	C42—H42	0.9300
P2—N2	1.625 (7)	C43—C44	1.426 (16)
P1—O2	1.601 (5)	C44—C45	1.483 (16)
P1—O1	1.608 (6)	C45—C46	1.385 (15)
P1—N1	1.630 (6)	C45—C54	1.473 (15)
O1—C1	1.387 (11)	C46—C47	1.397 (15)
O2—C12	1.397 (9)	C47—C48	1.398 (16)
O4—C46	1.434 (10)	C47—H47	0.9300
N1—C34	1.448 (11)	C48—C49	1.42 (2)

N1—C21	1.463 (10)	C48—H48	0.9300
O3—C35	1.396 (11)	C49—C54	1.391 (19)
N2—C68A	1.38 (3)	C49—C50	1.45 (2)
N2—C55A	1.441 (19)	C50—C51	1.38 (3)
N2—C68	1.481 (13)	C50—H50	0.9300
N2—C55	1.487 (10)	C51—C52	1.31 (2)
C1—C10	1.361 (12)	C51—H51	0.9300
C1—C2	1.432 (11)	C52—C53	1.32 (2)
C2—C3	1.334 (14)	C52—H52	0.9300
C2—H2A	0.9300	C53—C54	1.428 (19)
C3—C4	1.397 (15)	C53—H53	0.9300
C3—H3	0.9300	C55—C56	1.3900
C4—C5	1.379 (16)	C55—C60	1.3900
C4—C9	1.450 (12)	C56—C57	1.3900
C5—C6	1.35 (2)	C56—H56	0.9300
C5—H5	0.9300	C57—C58	1.3900
C6—C7	1.50 (2)	C57—H57	0.9300
C6—H6	0.9300	C58—C59	1.3900
C7—C8	1.335 (14)	C58—H58	0.9300
C7—H7	0.9300	C59—C60	1.3900
C8—C9	1.428 (15)	C59—H59	0.9300
C8—H8	0.9300	C60—C61	1.564 (18)
C9—C10	1.466 (12)	C61—C62	1.24 (3)
C10—C11	1.467 (10)	C61—H61	0.9300
C11—C12	1.411 (11)	C62—C63	1.44 (2)
C11—C20	1.444 (11)	C62—H62	0.9300
C12—C13	1.414 (10)	C63—C64	1.3900
C13—C14	1.352 (13)	C63—C68	1.3900
C13—H13	0.9300	C64—C65	1.3900
C14—C15	1.428 (13)	C64—H64	0.9300
C14—H14	0.9300	C65—C66	1.3900
C15—C20	1.415 (11)	C65—H65	0.9300
C15—C16	1.431 (13)	C66—C67	1.3900
C16—C17	1.348 (16)	C66—H66	0.9300
C16—H16	0.9300	C67—C68	1.3900
C17—C18	1.380 (16)	C67—H67	0.9300
C17—H17	0.9300	C55A—C56A	1.3900
C18—C19	1.350 (14)	C55A—C60A	1.3900
C18—H18	0.9300	C56A—C57A	1.3900
C19—C20	1.413 (13)	C56A—H56A	0.9300
C19—H19	0.9300	C57A—C58A	1.3900
C21—C22	1.361 (13)	C57A—H57A	0.9300
C21—C26	1.415 (14)	C58A—C59A	1.3900
C22—C23	1.424 (16)	C58A—H58A	0.9300
C22—H22	0.9300	C59A—C60A	1.3900
C23—C24	1.46 (2)	C59A—H59A	0.9300
C23—H23	0.9300	C60A—C61A	1.47 (4)
C24—C25	1.26 (2)	C61A—C62A	1.51 (5)
C24—H24	0.9300	C61A—H61A	0.9300

C25—C26	1.383 (16)	C62A—C63A	1.501 (5)
C25—H25	0.9300	C62A—H62A	0.9300
C26—C27	1.512 (19)	C63A—C64A	1.3900
C27—C28	1.298 (17)	C63A—C68A	1.3900
C27—H27	0.9300	C64A—C65A	1.3900
C28—C29	1.445 (16)	C64A—H64A	0.9300
C28—H28	0.9300	C65A—C66A	1.3900
C29—C34	1.408 (13)	C65A—H65A	0.9300
C29—C30	1.409 (16)	C66A—C67A	1.3900
C30—C31	1.375 (19)	C66A—H66A	0.9300
C30—H30	0.9300	C67A—C68A	1.3900
C31—C32	1.354 (18)	C67A—H67A	0.9300
C31—H31	0.9300	C2S—Cl7	1.734 (8)
C32—C33	1.381 (14)	C2S—Cl8	1.737 (8)
C32—H32	0.9300	C2S—Cl6	1.740 (8)
C33—C34	1.365 (12)	C2S—Cl7A	1.751 (8)
C33—H33	0.9300	C2S—Cl8A	1.755 (8)
C35—C44	1.379 (14)	C2S—Cl6A	1.756 (8)
C35—C36	1.381 (15)	C2S—H2S	0.9800
C36—C37	1.355 (17)	C3S—Cl11	1.736 (8)
C36—H36	0.9300	C3S—Cl9	1.741 (8)
C37—C38	1.46 (2)	C3S—Cl10	1.746 (8)
C37—H37	0.9300	C3S—H3SA	0.9800
C38—C43	1.37 (2)	C1S—Cl4	1.748 (8)
C38—C39	1.44 (2)	C1S—Cl5	1.751 (8)
C39—C40	1.35 (2)	C1S—Cl3	1.755 (8)
C39—H39	0.9300	C1S—H1S	0.9800
P2—Pd1—P1	94.59 (7)	C39—C40—H40	120.1
P2—Pd1—Cl2	174.48 (10)	C40—C41—C42	122.7 (16)
P1—Pd1—Cl2	88.86 (8)	C40—C41—H41	118.7
P2—Pd1—Cl1	87.73 (8)	C42—C41—H41	118.7
P1—Pd1—Cl1	174.22 (10)	C41—C42—C43	120.3 (16)
Cl2—Pd1—Cl1	89.24 (9)	C41—C42—H42	119.8
O4—P2—O3	101.0 (3)	C43—C42—H42	119.8
O4—P2—N2	111.8 (3)	C38—C43—C44	119.0 (12)
O3—P2—N2	100.0 (3)	C38—C43—C42	115.2 (13)
O4—P2—Pd1	110.5 (2)	C44—C43—C42	125.7 (14)
O3—P2—Pd1	116.4 (2)	C35—C44—C43	118.5 (11)
N2—P2—Pd1	115.9 (3)	C35—C44—C45	120.2 (9)
O2—P1—O1	102.0 (3)	C43—C44—C45	121.2 (10)
O2—P1—N1	113.3 (3)	C46—C45—C54	114.9 (11)
O1—P1—N1	99.0 (3)	C46—C45—C44	121.5 (9)
O2—P1—Pd1	108.2 (2)	C54—C45—C44	123.6 (11)
O1—P1—Pd1	117.5 (2)	C45—C46—C47	126.1 (9)
N1—P1—Pd1	116.1 (2)	C45—C46—O4	118.8 (9)
C1—O1—P1	123.4 (5)	C47—C46—O4	115.0 (9)
C12—O2—P1	122.8 (4)	C48—C47—C46	117.1 (12)
C46—O4—P2	120.9 (5)	C48—C47—H47	121.4
C34—N1—C21	115.7 (6)	C46—C47—H47	121.4

C34—N1—P1	123.3 (5)	C47—C48—C49	120.1 (12)
C21—N1—P1	120.9 (6)	C47—C48—H48	119.9
C35—O3—P2	120.4 (5)	C49—C48—H48	119.9
C68A—N2—C55A	126 (2)	C54—C49—C48	121.2 (11)
C55A—N2—C68	119.5 (15)	C54—C49—C50	118.5 (15)
C68A—N2—C55	115 (2)	C48—C49—C50	120.3 (14)
C68—N2—C55	109.3 (10)	C51—C50—C49	112.2 (17)
C68A—N2—P2	121 (2)	C51—C50—H50	123.9
C55A—N2—P2	113.0 (11)	C49—C50—H50	123.9
C68—N2—P2	126.5 (10)	C52—C51—C50	132 (2)
C55—N2—P2	124.0 (6)	C52—C51—H51	114.1
C10—C1—O1	120.3 (7)	C50—C51—H51	114.1
C10—C1—C2	122.7 (8)	C51—C52—C53	115.5 (19)
O1—C1—C2	116.8 (8)	C51—C52—H52	122.2
C3—C2—C1	118.6 (10)	C53—C52—H52	122.2
C3—C2—H2A	120.7	C52—C53—C54	121.7 (17)
C1—C2—H2A	120.7	C52—C53—H53	119.2
C2—C3—C4	123.2 (9)	C54—C53—H53	119.2
C2—C3—H3	118.4	C49—C54—C53	120.4 (11)
C4—C3—H3	118.4	C49—C54—C45	119.7 (12)
C5—C4—C3	120.5 (10)	C53—C54—C45	119.8 (13)
C5—C4—C9	120.3 (11)	C56—C55—C60	120.0
C3—C4—C9	118.9 (8)	C56—C55—N2	116.6 (6)
C6—C5—C4	119.8 (11)	C60—C55—N2	123.2 (6)
C6—C5—H5	120.1	C55—C56—C57	120.0
C4—C5—H5	120.1	C55—C56—H56	120.0
C5—C6—C7	121.6 (12)	C57—C56—H56	120.0
C5—C6—H6	119.2	C58—C57—C56	120.0
C7—C6—H6	119.2	C58—C57—H57	120.0
C8—C7—C6	117.8 (13)	C56—C57—H57	120.0
C8—C7—H7	121.1	C57—C58—C59	120.0
C6—C7—H7	121.1	C57—C58—H58	120.0
C7—C8—C9	121.5 (11)	C59—C58—H58	120.0
C7—C8—H8	119.3	C58—C59—C60	120.0
C9—C8—H8	119.3	C58—C59—H59	120.0
C8—C9—C4	118.8 (8)	C60—C59—H59	120.0
C8—C9—C10	123.6 (8)	C59—C60—C55	120.0
C4—C9—C10	117.6 (8)	C59—C60—C61	122.1 (8)
C1—C10—C9	118.1 (7)	C55—C60—C61	117.8 (8)
C1—C10—C11	122.1 (7)	C62—C61—C60	130.9 (15)
C9—C10—C11	119.6 (7)	C62—C61—H61	114.6
C12—C11—C20	116.5 (7)	C60—C61—H61	114.6
C12—C11—C10	120.0 (7)	C61—C62—C63	123.6 (16)
C20—C11—C10	123.5 (7)	C61—C62—H62	118.2
O2—C12—C11	120.6 (6)	C63—C62—H62	118.2
O2—C12—C13	117.5 (7)	C64—C63—C68	120.0
C11—C12—C13	121.8 (7)	C64—C63—C62	113.0 (14)
C14—C13—C12	119.3 (8)	C68—C63—C62	126.9 (14)
C14—C13—H13	120.4	C63—C64—C65	120.0

C12—C13—H13	120.4	C63—C64—H64	120.0
C13—C14—C15	122.8 (8)	C65—C64—H64	120.0
C13—C14—H14	118.6	C64—C65—C66	120.0
C15—C14—H14	118.6	C64—C65—H65	120.0
C20—C15—C14	117.3 (7)	C66—C65—H65	120.0
C20—C15—C16	119.9 (9)	C65—C66—C67	120.0
C14—C15—C16	122.6 (8)	C65—C66—H66	120.0
C17—C16—C15	121.2 (9)	C67—C66—H66	120.0
C17—C16—H16	119.4	C68—C67—C66	120.0
C15—C16—H16	119.4	C68—C67—H67	120.0
C16—C17—C18	118.2 (9)	C66—C67—H67	120.0
C16—C17—H17	120.9	C67—C68—C63	120.0
C18—C17—H17	120.9	C67—C68—N2	120.5 (12)
C19—C18—C17	123.0 (11)	C63—C68—N2	119.4 (12)
C19—C18—H18	118.5	C56A—C55A—C60A	120.0
C17—C18—H18	118.5	C56A—C55A—N2	126.3 (14)
C18—C19—C20	121.3 (10)	C60A—C55A—N2	113.6 (14)
C18—C19—H19	119.3	C57A—C56A—C55A	120.0
C20—C19—H19	119.3	C57A—C56A—H56A	120.0
C19—C20—C15	116.2 (8)	C55A—C56A—H56A	120.0
C19—C20—C11	122.4 (8)	C56A—C57A—C58A	120.0
C15—C20—C11	121.4 (8)	C56A—C57A—H57A	120.0
C22—C21—C26	121.6 (9)	C58A—C57A—H57A	120.0
C22—C21—N1	120.5 (8)	C59A—C58A—C57A	120.0
C26—C21—N1	117.8 (9)	C59A—C58A—H58A	120.0
C21—C22—C23	118.8 (11)	C57A—C58A—H58A	120.0
C21—C22—H22	120.6	C60A—C59A—C58A	120.0
C23—C22—H22	120.6	C60A—C59A—H59A	120.0
C22—C23—C24	116.0 (12)	C58A—C59A—H59A	120.0
C22—C23—H23	122.0	C59A—C60A—C55A	120.0
C24—C23—H23	122.0	C59A—C60A—C61A	115 (2)
C25—C24—C23	121.4 (12)	C55A—C60A—C61A	123 (2)
C25—C24—H24	119.3	C60A—C61A—C62A	121 (3)
C23—C24—H24	119.3	C60A—C61A—H61A	119.3
C24—C25—C26	123.8 (14)	C62A—C61A—H61A	119.3
C24—C25—H25	118.1	C63A—C62A—H62A	109.0
C26—C25—H25	118.1	C61A—C62A—H62A	109.0
C25—C26—C21	116.5 (13)	C64A—C63A—C68A	120.0
C25—C26—C27	121.4 (11)	C64A—C63A—C62A	130 (3)
C21—C26—C27	122.1 (9)	C68A—C63A—C62A	106 (3)
C28—C27—C26	127.1 (10)	C65A—C64A—C63A	120.0
C28—C27—H27	116.4	C65A—C64A—H64A	120.0
C26—C27—H27	116.5	C63A—C64A—H64A	120.0
C27—C28—C29	126.9 (11)	C64A—C65A—C66A	120.0
C27—C28—H28	116.5	C64A—C65A—H65A	120.0
C29—C28—H28	116.5	C66A—C65A—H65A	120.0
C34—C29—C30	117.8 (10)	C67A—C66A—C65A	120.0
C34—C29—C28	123.7 (9)	C67A—C66A—H66A	120.0
C30—C29—C28	118.4 (10)	C65A—C66A—H66A	120.0

C31—C30—C29	120.2 (11)	C68A—C67A—C66A	120.0
C31—C30—H30	119.9	C68A—C67A—H67A	120.0
C29—C30—H30	119.9	C66A—C67A—H67A	120.0
C32—C31—C30	121.3 (12)	N2—C68A—C67A	113 (3)
C32—C31—H31	119.3	N2—C68A—C63A	127 (3)
C30—C31—H31	119.3	C67A—C68A—C63A	120.0
C31—C32—C33	119.3 (11)	Cl7—C2S—Cl8	110.4 (6)
C31—C32—H32	120.4	Cl7—C2S—Cl6	110.0 (6)
C33—C32—H32	120.4	Cl8—C2S—Cl6	110.3 (5)
C34—C33—C32	121.5 (9)	Cl6—C2S—Cl7A	107.7 (16)
C34—C33—H33	119.2	Cl7A—C2S—Cl8A	108.6 (6)
C32—C33—H33	119.2	Cl7—C2S—Cl6A	102.5 (18)
C33—C34—C29	119.8 (9)	Cl7A—C2S—Cl6A	108.4 (6)
C33—C34—N1	121.0 (8)	Cl8A—C2S—Cl6A	108.4 (6)
C29—C34—N1	119.1 (7)	Cl7—C2S—H2S	108.7
C44—C35—C36	123.3 (10)	Cl8—C2S—H2S	108.7
C44—C35—O3	117.6 (9)	Cl6—C2S—H2S	108.7
C36—C35—O3	118.9 (9)	Cl7A—C2S—H2S	91.7
C37—C36—C35	118.5 (11)	Cl8A—C2S—H2S	106.4
C37—C36—H36	120.7	Cl6A—C2S—H2S	131.0
C35—C36—H36	120.7	Cl11—C3S—Cl9	99.1 (12)
C36—C37—C38	120.3 (12)	Cl11—C3S—Cl10	108.3 (13)
C36—C37—H37	119.9	Cl9—C3S—Cl10	111.3 (14)
C38—C37—H37	119.9	Cl11—C3S—H3SA	112.4
C43—C38—C39	122.3 (16)	Cl9—C3S—H3SA	112.4
C43—C38—C37	119.9 (11)	Cl10—C3S—H3SA	112.4
C39—C38—C37	117.8 (16)	Cl4—C1S—Cl5	108.8 (6)
C40—C39—C38	119.5 (18)	Cl4—C1S—Cl3	108.5 (6)
C40—C39—H39	120.3	Cl5—C1S—Cl3	108.7 (6)
C38—C39—H39	120.3	Cl4—C1S—H1S	110.3
C41—C40—C39	119.9 (17)	Cl5—C1S—H1S	110.3
C41—C40—H40	120.1	Cl3—C1S—H1S	110.3

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2S—H2S···Cl1^{i}	0.97	2.47	3.358 (10)	151
C27—H27···Cl2^{ii}	0.93	2.82	3.741 (12)	172
C14—H14···Cl2^{ii}	0.93	2.84	3.613 (10)	142

Symmetry codes: (i) −*y*+1, *x*, *z*−3/4; (ii) −*y*+1, *x*, *z*+1/4.

Fig. 1

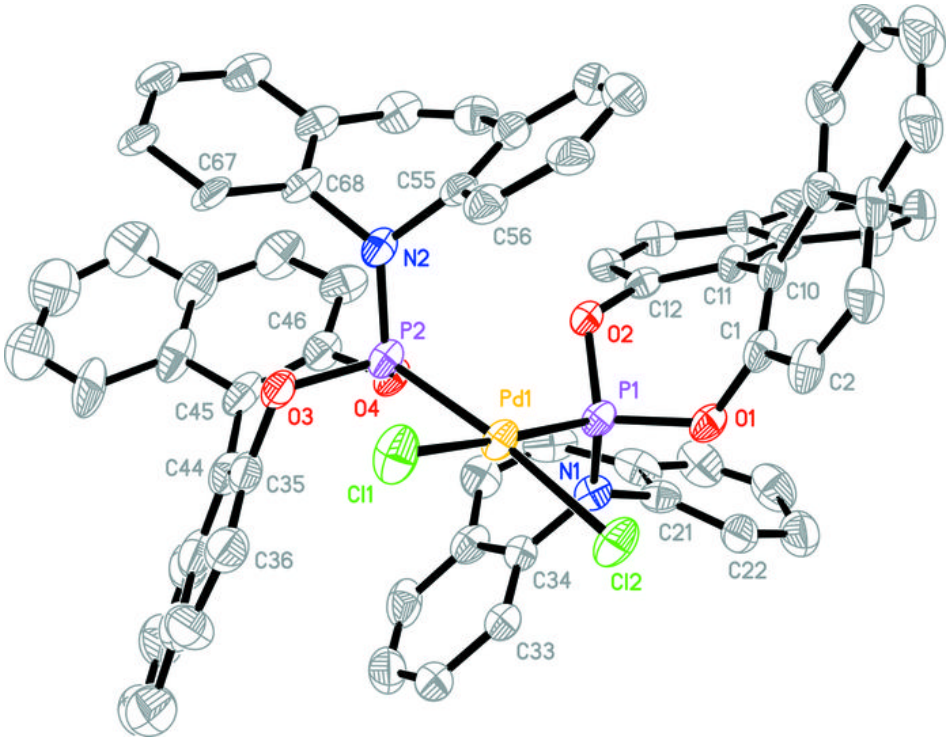
C67
C68
C55
C56
N2
C46
P2
C12
C11
C10
O2
C1
C2
C45
O3
O4
Pd1
P1
O1
C44
C35
Cl1
N1
C21
C22
C36
C34
Cl2
C33

Fig. 2

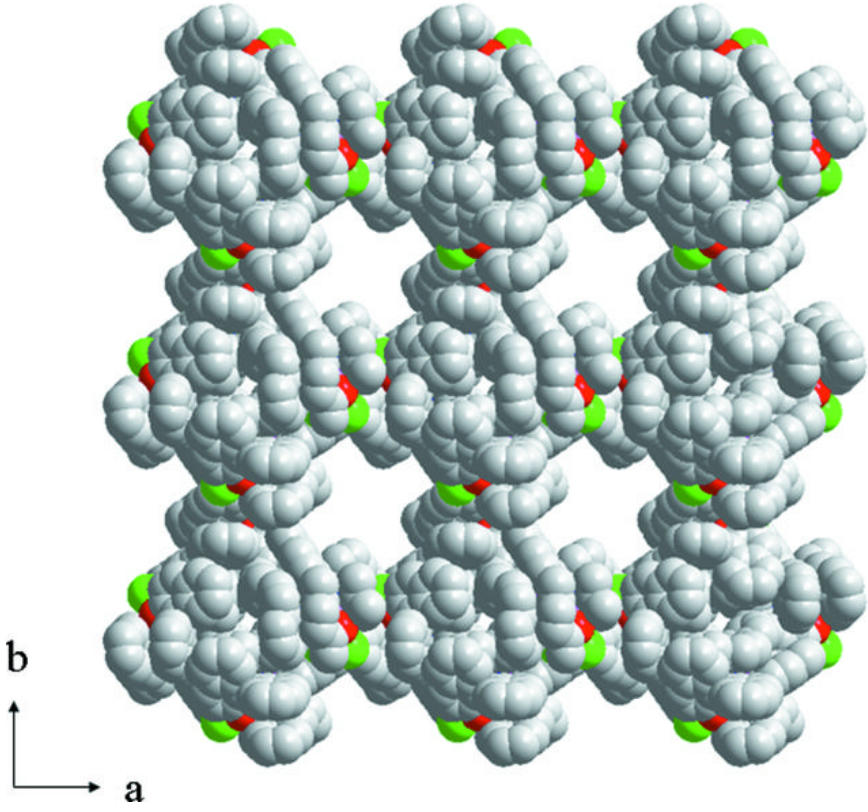

Fig. 3