IUCrData

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Di-μ-chlorido-bis({4-[bis(trimethylsilyl)amino]-6-chloro-2,2,8,8-tetramethyl-5,7-bis(trimethylsilyl)-3,5,7-triaza-4,6-diphospha-2,8-disilanon-3-en-4-ido-κ^2P,P'}palladium(II)) diethyl ether disolvate

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Keywords: crystal structure; dinuclear palladium complex; chloride-bridged complex; metal catalyzed coupling; phosphorous ligand.

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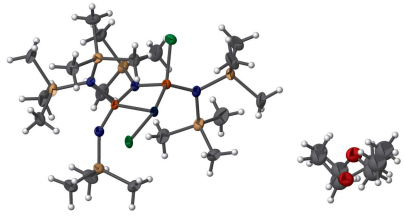
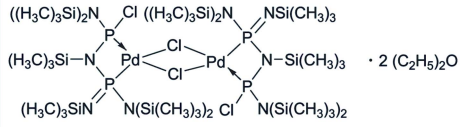
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $[Pd_2(C_{18}H_{54}Cl_2N_4P_2Si_6)_2Cl_2]\cdot 2C_4H_{10}O$, features a dinuclear chloride-bridged palladium complex bearing two equivalents of the novel monoanionic mixed valent (λ^3-P)—N—(λ^5-P) ligand. A metal catalyzed coupling of two aminoiminophosphines and a shift of one chlorine from the metal to the phosphorus results in the (λ^3-P)—N—(λ^5-P) ligand. The molecule contains a planar bimetallic Pd_2Cl_2 core with a crystallographic centre of inversion at the mid-point of the Pd···Pd line. The Pd atoms are in a distorted square-planar arrangement, where the P/Pd/P and Cl/Pd/Cl planes are twisted with respect to each other by a dihedral angle of 7.57 (4)°. The P—Pd—P bite angle is 71.380 (18)°. Intramolecular C—H···Cl interactions are observed. In the crystal, the diethyl ether solvent molecule is disordered over two sites, with an occupancy ratio of 0.788 (5):0.212 (5).

3D view



Chemical scheme



Structure description

Low-valent phosphorous compounds, such as aminoiminophosphines $R_2N—P{=}NR$, are versatile ligands. Due to their unsaturated P=N double bond they can undergo [2 + 2] cycloaddition to yield the cyclodiphosphadiazanes (Niecke *et al.*, 1976). In the presence of dichlorido(1,5-cyclooctadiene)palladium(II), two N—P=N molecules undergo a [2 + 1] cycloaddition which results in a kind of azadiphosphiridine or, respectively, a mono-anionic six-electron-donor ligand (Scherer & Brück, 1987). In the course of coordination to dichlorido(1,5-cyclooctadiene)palladium(II), one chlorine is transferred from the metal centre to the P^{III} atom. As a consequence, two palladium cores dimerize by building a dinuclear chloride-bridged palladium complex with two equivalents of the

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3C···Cl1^{i}	0.98	2.72	3.564 (3)	144
C14—H14C···Cl2	0.98	2.67	3.585 (2)	156

Symmetry code: (i) $-x+1, -y+1, -z+1$.

monoanionic mixed-valent (λ^3-P)—N—(λ^5-P) ligand. The molecule contains a planar bimetallic Pd_2Cl_2 core with a crystallographic centre of inversion at the mid-point of Pd···Pd line (Fig. 1). The Pd—Cl bond lengths are 2.4296 (5) and 2.4815 (4) Å, which are similar to other dinuclear chloride-bridged palladium(II) complexes (average Pd—Cl = 2.432 Å for Pd—Cl—Pd > 85°; Orpen *et al.*, 1989). Due to the large Pd···Pd distance of 3.521 Å there is no metal–metal interaction. The Pd atoms are in a distorted square-planar arrangement where the P1/Pd1/P2 and Cl1/Pd1/Cl1^{i} planes [symmetry code: (i) $-x+1, -y+1, -z+1$] are twisted with respect to each other by an angle of 7.57 (4)°. The mixed valent (λ^3-P)—N—(λ^5-P) ligand is remarkable, with the ability to form a four-membered metallacycle that is nearly planar, with a P2—N2—P1—Pd1 torsion angle of 6.45 (7)°. The different Pd—P distances are 2.1947 (5) (λ^3-P—Pd) and 2.2294 (5) Å (λ^5-P—Pd). Compared to a λ^3-P–metal bond, the λ^5-P–metal bond length is elongated. A longer λ^5-P–metal bond compared to the λ^3-P–metal bond length was also observed in a Pd complex bearing a mixed-valent (λ^3-P)—N—(λ^5-P) ligand (du Mont *et al.*, 2009)

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Pd_2(C_{18}H_{54}Cl_2N_4P_2Si_6)_2Cl_2]\cdot 2C_4H_{10}O$
M_r	1617.10
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	12.7331 (3), 12.9694 (3), 14.6354 (3)
α, β, γ (°)	104.1592 (6), 101.3576 (6), 112.6871 (6)
V (Å^3)	2044.32 (8)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.86
Crystal size (mm)	0.42 × 0.40 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.77, 0.86
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	40794, 9871, 8984
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.076, 1.04
No. of reflections	9871
No. of parameters	367
No. of restraints	29
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.95, −0.92

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008), and *publCIF* (Westrip, 2010).

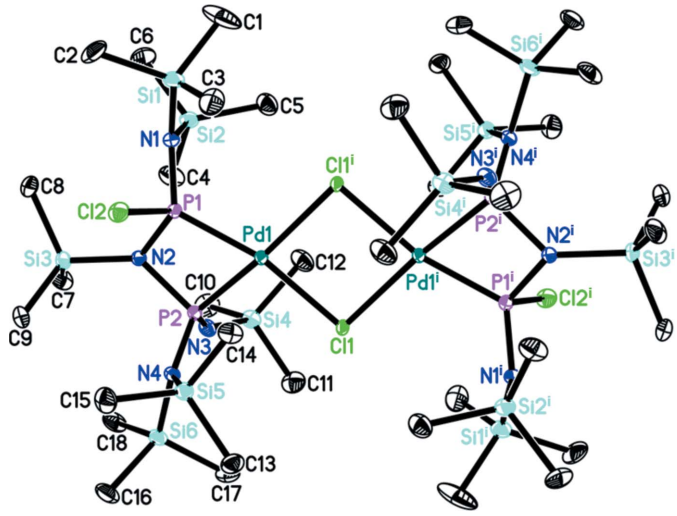


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level. H atoms and disordered solvent have been omitted for clarity. [Symmetry code: (i) $-x+1, -y+1, -z+1$.]

The P1—Pd1—P2 bite angle is 71.380 (18)°. For the (λ^5-P)═N double bond (N3—P2), a value of 1.5346 (17) Å was observed. All other P—N bond distances range from 1.6519 (16) (P1—N1) to 1.7772 (16) Å (P2—N2). Compared to the calculated P—N single-bond distance (1.82 Å; Pyykkö, 2015) they are obviously shorter and display some multiple bonding character. The calculated P═N double bond length is 1.62 Å (Pyykkö, 2015). This might be explained by being a consequence of hyperconjugation in the alternating P—N ligand backbone. Similar but mononuclear iron and platinum complexes with mixed-valent (λ^3-P)—N—(λ^5-P) ligands have been reported (du Mont *et al.*, 2009; Jones *et al.*, 2015). Intramolecular C—H···Cl interactions are observed (Table 1). The crystal of the title compound contains a diethyl ether solvent molecule which is disordered over two sites with an occupancy ratio of 0.788 (5):0.212 (5).

Synthesis and crystallization

A pale-yellow solution of 1.27 mmol of the aminoiminophosphine $(TMS)_2N—P{=}N(TMS)$ in 5 ml toluene was added dropwise to a bright-yellow suspension of 0.58 mmol dichlorido(1,5-cyclooctadiene)palladium(II) in 15 ml toluene. After stirring for 10 min at room temperature, the yellow solution became clear. It was stirred for another 12 h before the solvent was reduced in a vacuum. The resulting yellow solution was filtered. After adding diethyl ether, it was cooled to 233 K to obtain yellow crystals of the title compound that were suitable for X-ray analysis (yield 77%; m.p. decomposition above 347 K). Analysis calculated for $C_{36}H_{108}Cl_4N_8P_4Pd_2Si_{12}$: C 29.44, H 7.41, N 7.63, P 8.43%; found: C 29.24, H

7.66, N 7.07, P 8.51%. ^{1}H NMR (400 MHz, C_6D_6): δ 0.44 (*s*, 24H, 2 × TMS), 0.57 (*s*, 24H, 2 × TMS), 0.69 (*s*, 12H, TMS), 0.74 (*s*, 12H, TMS). ^{13}C (100.6 MHz, C_6D_6): δ 3.8 (TMS), 5.7 (TMS), 6.3 (TMS), 7.5 (TMS). ^{31}P (121 MHz, C_6D_6): δ −88.52 (*s*), −28.06 (*br*, *s*). ^{29}Si (79.5 MHz, C_6D_6): δ 8.32 (2 × N-TMS), 11.34 (4 × N-TMS). MS CI (*m*/*z*) 1466 $[M + 2]^+$, 1275 $[C_{30}H_{90}N_7P_3Pd_2Si_{10}]^+$, 1158 $[C_{27}H_{81}N_6P_3Pd_2Si_9 + 3H]^+$. IR ATR ($cm^{-1}$) 2896.9 (*w*), 2957.2 (*w*).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The diethyl ether molecule is disordered over two sites with refined occupancies 0.788 (5):0.212 (5). DFIX and DANG instructions were used to improve the geometry of the disordered solvent molecule. Additionally, the anisotropic displacement parameters of O1*A* and O1*B* were restrained to be equal (SIMU). Atoms C19*A*, C19*B* C20*A*, C20*B*, C21*A*, C21*B*, C22*A* and C22*B* were refined isotropically. A SIMU instruction was also applied for these atoms.

Acknowledgements

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References

Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jones, P. G., Daniliuc, C., du Mont, W.-W. & Goers, C. (2015). Private communication (refcode BUCGIR). CCDC, Cambridge, England.
Mont, W.-W. du, Lungu, D., Daniliuc, C., Jones, P. G., Nyulaszi, L., Benko, Z. & Bartsch, R. (2009). *Eur. J. Inorg. Chem.* pp. 2901–2905.
Niecke, E., Flick, W. & Pohl, S. (1976). *Angew. Chem.* **88**, 305–306.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–S83.
Pyykkö, P. (2015). *J. Phys. Chem. A*, **119**, 2326–2337.
Scherer, O. J. & Brück, T. (1987). *Angew. Chem.* **99**, 59–61.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

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Di-μ-chlorido-bis({4-[bis(trimethylsilyl)amino]-6-chloro-2,2,8,8-tetramethyl-5,7-bis(trimethylsilyl)-3,5,7-triaza-4,6-diphospha-2,8-disilanon-3-en-4-ido-κ^2P,P'}palladium(II)) diethyl ether disolvate

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Crystal data

$[Pd_2(C_{18}H_{54}Cl_2N_4P_2Si_6)_2Cl_2]\cdot 2C_4H_{10}O$
M_r = 1617.10
Triclinic, $P\overline{1}$
a = 12.7331 (3) Å
b = 12.9694 (3) Å
c = 14.6354 (3) Å
α = 104.1592 (6)°
β = 101.3576 (6)°
γ = 112.6871 (6)°
V = 2044.32 (8) Å^3
Z = 1
$F(000)$ = 852
D_x = 1.314 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9897 reflections
θ = 2.5–29.0°
μ = 0.86 mm^{-1}
T = 150 K
Prism, yellow
0.42 × 0.40 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.77, T_{max} = 0.86
40794 measured reflections
9871 independent reflections
8984 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 28.0°, θ_{min} = 1.5°
h = −16→16
k = −17→17
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.076
S = 1.04
9871 reflections
367 parameters
29 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 2.0017P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.92 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6444 (3)	0.1775 (4)	0.2536 (4)	0.0864 (14)	
H1A	0.6442	0.1971	0.3226	0.130*	
H1B	0.7235	0.1847	0.2523	0.130*	
H1C	0.5825	0.0954	0.2142	0.130*	
C2	0.6250 (3)	0.2550 (3)	0.0734 (2)	0.0517 (7)	
H2A	0.5583	0.1781	0.0276	0.078*	
H2B	0.7017	0.2535	0.0751	0.078*	
H2C	0.6217	0.3190	0.0506	0.078*	
C3	0.7322 (2)	0.4325 (3)	0.2872 (2)	0.0539 (7)	
H3A	0.7625	0.4829	0.2489	0.081*	
H3B	0.7983	0.4239	0.3262	0.081*	
H3C	0.6988	0.4697	0.3323	0.081*	
C4	0.1995 (2)	0.0972 (2)	0.1565 (2)	0.0443 (6)	
H4A	0.1965	0.1676	0.1972	0.066*	
H4B	0.1485	0.0265	0.1687	0.066*	
H4C	0.1705	0.0858	0.0859	0.066*	
C5	0.3946 (2)	0.1077 (2)	0.3154 (2)	0.0433 (6)	
H5A	0.4650	0.0928	0.3264	0.065*	
H5B	0.3262	0.0417	0.3191	0.065*	
H5C	0.4128	0.1826	0.3666	0.065*	
C6	0.3559 (3)	−0.0021 (3)	0.0910 (2)	0.0583 (8)	
H6A	0.3386	0.0099	0.0272	0.087*	
H6B	0.2941	−0.0795	0.0855	0.087*	
H6C	0.4351	−0.0006	0.1080	0.087*	
C7	0.0637 (2)	0.2099 (2)	−0.0030 (2)	0.0447 (6)	
H7A	0.0499	0.1267	−0.0250	0.067*	
H7B	0.0159	0.2226	−0.0564	0.067*	
H7C	0.0397	0.2270	0.0564	0.067*	
C8	0.2971 (3)	0.2308 (3)	−0.04103 (17)	0.0472 (7)	
H8A	0.3813	0.2862	−0.0280	0.071*	
H8B	0.2536	0.1990	−0.1127	0.071*	
H8C	0.2936	0.1648	−0.0183	0.071*	
C9	0.2465 (2)	0.4447 (2)	−0.00733 (17)	0.0392 (5)	
H9A	0.2120	0.4890	0.0302	0.059*	
H9B	0.2056	0.4191	−0.0789	0.059*	
H9C	0.3325	0.4963	0.0085	0.059*	
C10	−0.0606 (2)	0.1388 (2)	0.2237 (2)	0.0481 (6)	
H10A	−0.1082	0.1654	0.1831	0.072*	
H10B	−0.1109	0.0892	0.2544	0.072*	

H10C	−0.0320	0.0919	0.1815	0.072*
C11	0.0147 (3)	0.3501 (3)	0.4110 (2)	0.0465 (6)
H11A	0.0827	0.4211	0.4631	0.070*
H11B	−0.0296	0.2958	0.4416	0.070*
H11C	−0.0392	0.3741	0.3741	0.070*
C12	0.1624 (3)	0.2200 (3)	0.3995 (2)	0.0469 (6)
H12A	0.1869	0.1699	0.3557	0.070*
H12B	0.1138	0.1734	0.4327	0.070*
H12C	0.2342	0.2892	0.4497	0.070*
C13	0.4009 (2)	0.8176 (2)	0.39408 (18)	0.0376 (5)
H13A	0.3302	0.8261	0.3640	0.056*
H13B	0.4730	0.8947	0.4175	0.056*
H13C	0.3907	0.7914	0.4505	0.056*
C14	0.55987 (19)	0.70951 (19)	0.36919 (19)	0.0343 (5)
H14A	0.5512	0.6912	0.4292	0.051*
H14B	0.6264	0.7896	0.3883	0.051*
H14C	0.5768	0.6506	0.3269	0.051*
C15	0.4489 (2)	0.7538 (2)	0.1936 (2)	0.0420 (6)
H15A	0.4773	0.7035	0.1548	0.063*
H15B	0.5109	0.8373	0.2200	0.063*
H15C	0.3750	0.7466	0.1507	0.063*
C16	0.1596 (2)	0.6979 (2)	0.1776 (2)	0.0449 (6)
H16A	0.2305	0.7723	0.2221	0.067*
H16B	0.0875	0.7100	0.1711	0.067*
H16C	0.1687	0.6744	0.1119	0.067*
C17	0.1115 (2)	0.6141 (2)	0.3497 (2)	0.0405 (5)
H17A	0.1503	0.5863	0.3961	0.061*
H17B	0.0243	0.5749	0.3365	0.061*
H17C	0.1426	0.7009	0.3792	0.061*
C18	0.0096 (2)	0.4459 (2)	0.1325 (2)	0.0436 (6)
H18A	0.0295	0.4246	0.0715	0.065*
H18B	−0.0568	0.4665	0.1189	0.065*
H18C	−0.0145	0.3779	0.1554	0.065*
Cl1	0.39377 (4)	0.54640 (5)	0.51614 (3)	0.02502 (10)
Cl2	0.54536 (5)	0.48760 (5)	0.15850 (4)	0.03183 (11)
N1	0.46063 (15)	0.26090 (15)	0.19051 (12)	0.0242 (3)
N2	0.30091 (14)	0.36004 (14)	0.15804 (12)	0.0208 (3)
N3	0.14924 (14)	0.36071 (15)	0.26758 (13)	0.0252 (3)
N4	0.28301 (14)	0.56547 (14)	0.25223 (12)	0.0210 (3)
P1	0.43459 (4)	0.37814 (4)	0.21907 (3)	0.01973 (10)
P2	0.27309 (4)	0.43630 (4)	0.26184 (3)	0.01800 (9)
Pd1	0.43737 (2)	0.45924 (2)	0.37046 (2)	0.01843 (5)
Si1	0.61214 (6)	0.28225 (6)	0.20011 (5)	0.03514 (14)
Si2	0.35607 (6)	0.11934 (5)	0.19020 (5)	0.03079 (13)
Si3	0.22635 (6)	0.31142 (5)	0.02732 (4)	0.02928 (13)
Si4	0.07178 (5)	0.27242 (5)	0.32387 (5)	0.02940 (13)
Si5	0.41760 (5)	0.70410 (5)	0.29902 (4)	0.02496 (11)
Si6	0.14410 (5)	0.57669 (5)	0.23102 (5)	0.02895 (13)

O1A	0.9440 (2)	0.1275 (2)	0.5576 (2)	0.0579 (8)	0.788 (5)
C19A	1.1344 (4)	0.2281 (5)	0.6796 (4)	0.0839 (14)*	0.788 (5)
H19A	1.1836	0.2323	0.7423	0.126*	0.788 (5)
H19B	1.1707	0.2137	0.6277	0.126*	0.788 (5)
H19C	1.1303	0.3037	0.6889	0.126*	0.788 (5)
C20A	1.0105 (4)	0.1284 (5)	0.6491 (3)	0.0791 (13)*	0.788 (5)
H20A	1.0131	0.0513	0.6394	0.095*	0.788 (5)
H20B	0.9726	0.1418	0.7008	0.095*	0.788 (5)
C21A	0.8231 (3)	0.0430 (4)	0.5182 (3)	0.0676 (11)*	0.788 (5)
H21A	0.7833	0.0527	0.5691	0.081*	0.788 (5)
H21B	0.8173	−0.0383	0.5007	0.081*	0.788 (5)
C22A	0.7602 (5)	0.0556 (5)	0.4282 (3)	0.0604 (11)*	0.788 (5)
H22A	0.6756	−0.0048	0.4022	0.091*	0.788 (5)
H22B	0.7647	0.1355	0.4456	0.091*	0.788 (5)
H22C	0.7986	0.0446	0.3772	0.091*	0.788 (5)
O1B	0.8894 (7)	0.1045 (8)	0.6139 (6)	0.050 (2)	0.212 (5)
C19B	1.0964 (9)	0.2188 (16)	0.7046 (13)	0.080 (2)*	0.212 (5)
H19D	1.1774	0.2298	0.7061	0.121*	0.212 (5)
H19E	1.0905	0.2919	0.7045	0.121*	0.212 (5)
H19F	1.0809	0.2016	0.7635	0.121*	0.212 (5)
C20B	1.0053 (8)	0.1165 (16)	0.6123 (12)	0.0791 (18)*	0.212 (5)
H20C	1.0200	0.1327	0.5521	0.095*	0.212 (5)
H20D	1.0104	0.0420	0.6115	0.095*	0.212 (5)
C21B	0.7957 (10)	0.0114 (9)	0.5278 (9)	0.0730 (17)*	0.212 (5)
H21C	0.7248	−0.0318	0.5462	0.088*	0.212 (5)
H21D	0.8233	−0.0463	0.4977	0.088*	0.212 (5)
C22B	0.7628 (16)	0.0660 (17)	0.4551 (9)	0.060 (2)*	0.212 (5)
H22D	0.6982	0.0030	0.3951	0.090*	0.212 (5)
H22E	0.7352	0.1226	0.4854	0.090*	0.212 (5)
H22F	0.8334	0.1081	0.4371	0.090*	0.212 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.059 (2)	0.121 (3)	0.156 (4)	0.070 (2)	0.059 (2)	0.106 (3)
C2	0.0638 (18)	0.0589 (17)	0.0496 (16)	0.0372 (15)	0.0389 (14)	0.0152 (13)
C3	0.0295 (12)	0.083 (2)	0.0448 (15)	0.0278 (14)	0.0115 (11)	0.0131 (15)
C4	0.0347 (12)	0.0253 (11)	0.0618 (17)	0.0086 (10)	0.0045 (11)	0.0146 (11)
C5	0.0484 (15)	0.0459 (14)	0.0460 (15)	0.0238 (12)	0.0179 (12)	0.0272 (12)
C6	0.089 (2)	0.0374 (14)	0.0527 (17)	0.0402 (16)	0.0212 (16)	0.0061 (12)
C7	0.0362 (13)	0.0378 (13)	0.0383 (13)	0.0137 (11)	−0.0122 (10)	0.0029 (10)
C8	0.0655 (18)	0.0591 (16)	0.0202 (11)	0.0409 (15)	0.0076 (11)	0.0038 (11)
C9	0.0516 (14)	0.0488 (14)	0.0251 (11)	0.0294 (12)	0.0094 (10)	0.0176 (10)
C10	0.0288 (12)	0.0385 (13)	0.0619 (18)	0.0034 (10)	0.0159 (12)	0.0125 (12)
C11	0.0463 (15)	0.0494 (15)	0.0564 (17)	0.0237 (12)	0.0319 (13)	0.0240 (13)
C12	0.0488 (15)	0.0506 (15)	0.0573 (17)	0.0263 (13)	0.0209 (13)	0.0361 (14)
C13	0.0409 (13)	0.0274 (11)	0.0372 (13)	0.0159 (10)	0.0079 (10)	0.0028 (9)
C14	0.0248 (10)	0.0249 (10)	0.0442 (13)	0.0077 (8)	0.0057 (9)	0.0082 (9)

C15	0.0477 (14)	0.0385 (13)	0.0419 (14)	0.0144 (11)	0.0189 (11)	0.0231 (11)
C16	0.0480 (15)	0.0469 (14)	0.0500 (15)	0.0319 (12)	0.0073 (12)	0.0238 (12)
C17	0.0391 (13)	0.0427 (13)	0.0527 (15)	0.0274 (11)	0.0226 (11)	0.0175 (11)
C18	0.0288 (11)	0.0446 (14)	0.0500 (15)	0.0193 (11)	−0.0045 (10)	0.0144 (12)
Cl1	0.0260 (2)	0.0423 (3)	0.01267 (19)	0.0246 (2)	0.00442 (16)	0.00584 (18)
Cl2	0.0334 (3)	0.0344 (3)	0.0361 (3)	0.0172 (2)	0.0200 (2)	0.0163 (2)
N1	0.0291 (8)	0.0277 (8)	0.0219 (8)	0.0191 (7)	0.0090 (7)	0.0081 (7)
N2	0.0214 (7)	0.0216 (7)	0.0175 (7)	0.0115 (6)	0.0028 (6)	0.0040 (6)
N3	0.0187 (8)	0.0297 (9)	0.0283 (9)	0.0109 (7)	0.0066 (7)	0.0130 (7)
N4	0.0220 (8)	0.0217 (7)	0.0210 (8)	0.0122 (6)	0.0050 (6)	0.0082 (6)
P1	0.0209 (2)	0.0231 (2)	0.0164 (2)	0.01211 (19)	0.00553 (17)	0.00576 (18)
P2	0.0167 (2)	0.0206 (2)	0.0167 (2)	0.00981 (18)	0.00340 (17)	0.00615 (17)
Pd1	0.01793 (7)	0.02364 (8)	0.01437 (7)	0.01211 (6)	0.00337 (5)	0.00495 (5)
Si1	0.0382 (3)	0.0500 (4)	0.0408 (4)	0.0334 (3)	0.0227 (3)	0.0243 (3)
Si2	0.0376 (3)	0.0239 (3)	0.0316 (3)	0.0180 (3)	0.0078 (3)	0.0071 (2)
Si3	0.0357 (3)	0.0322 (3)	0.0163 (3)	0.0189 (3)	0.0001 (2)	0.0037 (2)
Si4	0.0228 (3)	0.0302 (3)	0.0377 (3)	0.0109 (2)	0.0124 (2)	0.0161 (3)
Si5	0.0267 (3)	0.0214 (3)	0.0260 (3)	0.0107 (2)	0.0076 (2)	0.0083 (2)
Si6	0.0261 (3)	0.0314 (3)	0.0330 (3)	0.0189 (2)	0.0044 (2)	0.0118 (2)
O1A	0.0549 (16)	0.0586 (16)	0.0620 (18)	0.0237 (13)	0.0201 (13)	0.0273 (13)
O1B	0.058 (5)	0.046 (4)	0.053 (5)	0.027 (4)	0.027 (4)	0.017 (4)

Geometric parameters (Å, º)

C1—Si1	1.858 (3)	C15—H15C	0.9800
C1—H1A	0.9800	C16—Si6	1.882 (3)
C1—H1B	0.9800	C16—H16A	0.9800
C1—H1C	0.9800	C16—H16B	0.9800
C2—Si1	1.853 (3)	C16—H16C	0.9800
C2—H2A	0.9800	C17—Si6	1.858 (3)
C2—H2B	0.9800	C17—H17A	0.9800
C2—H2C	0.9800	C17—H17B	0.9800
C3—Si1	1.865 (3)	C17—H17C	0.9800
C3—H3A	0.9800	C18—Si6	1.865 (3)
C3—H3B	0.9800	C18—H18A	0.9800
C3—H3C	0.9800	C18—H18B	0.9800
C4—Si2	1.844 (3)	C18—H18C	0.9800
C4—H4A	0.9800	Cl1—Pd1	2.4296 (5)
C4—H4B	0.9800	Cl1—Pd1^{i}	2.4815 (4)
C4—H4C	0.9800	Cl2—P1	2.0867 (7)
C5—Si2	1.860 (3)	N1—P1	1.6519 (16)
C5—H5A	0.9800	N1—Si2	1.8032 (19)
C5—H5B	0.9800	N1—Si1	1.8112 (18)
C5—H5C	0.9800	N2—P1	1.6556 (16)
C6—Si2	1.862 (3)	N2—P2	1.7772 (16)
C6—H6A	0.9800	N2—Si3	1.7865 (17)
C6—H6B	0.9800	N3—P2	1.5346 (17)
C6—H6C	0.9800	N3—Si4	1.6950 (18)

C7—Si3	1.862 (3)	N4—P2	1.6736 (16)
C7—H7A	0.9800	N4—Si5	1.7881 (17)
C7—H7B	0.9800	N4—Si6	1.8029 (16)
C7—H7C	0.9800	P1—Pd1	2.1947 (5)
C8—Si3	1.864 (3)	P2—Pd1	2.2294 (5)
C8—H8A	0.9800	Pd1—Cl1^{i}	2.4815 (4)
C8—H8B	0.9800	O1A—C21A	1.394 (3)
C8—H8C	0.9800	O1A—C20A	1.430 (3)
C9—Si3	1.857 (2)	C19A—C20A	1.492 (4)
C9—H9A	0.9800	C19A—H19A	0.9800
C9—H9B	0.9800	C19A—H19B	0.9800
C9—H9C	0.9800	C19A—H19C	0.9800
C10—Si4	1.877 (3)	C20A—H20A	0.9900
C10—H10A	0.9800	C20A—H20B	0.9900
C10—H10B	0.9800	C21A—C22A	1.482 (3)
C10—H10C	0.9800	C21A—H21A	0.9900
C11—Si4	1.863 (3)	C21A—H21B	0.9900
C11—H11A	0.9800	C22A—H22A	0.9800
C11—H11B	0.9800	C22A—H22B	0.9800
C11—H11C	0.9800	C22A—H22C	0.9800
C12—Si4	1.868 (3)	O1B—C21B	1.422 (4)
C12—H12A	0.9800	O1B—C20B	1.429 (4)
C12—H12B	0.9800	C19B—C20B	1.500 (4)
C12—H12C	0.9800	C19B—H19D	0.9800
C13—Si5	1.872 (2)	C19B—H19E	0.9800
C13—H13A	0.9800	C19B—H19F	0.9800
C13—H13B	0.9800	C20B—H20C	0.9900
C13—H13C	0.9800	C20B—H20D	0.9900
C14—Si5	1.864 (2)	C21B—C22B	1.494 (4)
C14—H14A	0.9800	C21B—H21C	0.9900
C14—H14B	0.9800	C21B—H21D	0.9900
C14—H14C	0.9800	C22B—H22D	0.9800
C15—Si5	1.867 (2)	C22B—H22E	0.9800
C15—H15A	0.9800	C22B—H22F	0.9800
C15—H15B	0.9800		

Si1—C1—H1A	109.5	P2—N3—Si4	146.58 (11)
Si1—C1—H1B	109.5	P2—N4—Si5	125.76 (9)
H1A—C1—H1B	109.5	P2—N4—Si6	115.33 (9)
Si1—C1—H1C	109.5	Si5—N4—Si6	115.66 (9)
H1A—C1—H1C	109.5	N1—P1—N2	116.97 (8)
H1B—C1—H1C	109.5	N1—P1—Cl2	101.47 (7)
Si1—C2—H2A	109.5	N2—P1—Cl2	101.22 (6)
Si1—C2—H2B	109.5	N1—P1—Pd1	120.46 (6)
H2A—C2—H2B	109.5	N2—P1—Pd1	97.69 (6)
Si1—C2—H2C	109.5	Cl2—P1—Pd1	118.18 (3)
H2A—C2—H2C	109.5	N1—P1—P2	140.56 (7)
H2B—C2—H2C	109.5	Cl2—P1—P2	114.75 (3)

Si1—C3—H3A	109.5	Pd1—P1—P2	54.935 (15)
Si1—C3—H3B	109.5	N3—P2—N4	110.53 (9)
H3A—C3—H3B	109.5	N3—P2—N2	111.64 (9)
Si1—C3—H3C	109.5	N4—P2—N2	108.49 (8)
H3A—C3—H3C	109.5	N3—P2—Pd1	118.85 (7)
H3B—C3—H3C	109.5	N4—P2—Pd1	112.87 (6)
Si2—C4—H4A	109.5	N2—P2—Pd1	92.90 (5)
Si2—C4—H4B	109.5	N3—P2—P1	131.03 (7)
H4A—C4—H4B	109.5	N4—P2—P1	116.17 (6)
Si2—C4—H4C	109.5	Pd1—P2—P1	53.685 (15)
H4A—C4—H4C	109.5	P1—Pd1—P2	71.380 (18)
H4B—C4—H4C	109.5	P1—Pd1—Cl1	165.791 (17)
Si2—C5—H5A	109.5	P2—Pd1—Cl1	94.413 (16)
Si2—C5—H5B	109.5	P1—Pd1—Cl1^{i}	105.637 (16)
H5A—C5—H5B	109.5	P2—Pd1—Cl1^{i}	171.920 (18)
Si2—C5—H5C	109.5	Cl1—Pd1—Cl1^{i}	88.411 (15)
H5A—C5—H5C	109.5	N1—Si1—C2	108.05 (12)
H5B—C5—H5C	109.5	N1—Si1—C1	110.50 (12)
Si2—C6—H6A	109.5	C2—Si1—C1	110.16 (18)
Si2—C6—H6B	109.5	N1—Si1—C3	114.01 (10)
H6A—C6—H6B	109.5	C2—Si1—C3	110.42 (14)
Si2—C6—H6C	109.5	C1—Si1—C3	103.65 (19)
H6A—C6—H6C	109.5	N1—Si2—C4	112.48 (10)
H6B—C6—H6C	109.5	N1—Si2—C5	110.76 (10)
Si3—C7—H7A	109.5	C4—Si2—C5	107.33 (13)
Si3—C7—H7B	109.5	N1—Si2—C6	108.33 (12)
H7A—C7—H7B	109.5	C4—Si2—C6	105.24 (15)
Si3—C7—H7C	109.5	C5—Si2—C6	112.64 (13)
H7A—C7—H7C	109.5	N2—Si3—C9	108.72 (10)
H7B—C7—H7C	109.5	N2—Si3—C7	109.53 (11)
Si3—C8—H8A	109.5	C9—Si3—C7	110.83 (12)
Si3—C8—H8B	109.5	N2—Si3—C8	108.61 (10)
H8A—C8—H8B	109.5	C9—Si3—C8	109.09 (13)
Si3—C8—H8C	109.5	C7—Si3—C8	110.02 (13)
H8A—C8—H8C	109.5	N3—Si4—C11	112.26 (11)
H8B—C8—H8C	109.5	N3—Si4—C12	113.80 (11)
Si3—C9—H9A	109.5	C11—Si4—C12	105.73 (14)
Si3—C9—H9B	109.5	N3—Si4—C10	107.63 (11)
H9A—C9—H9B	109.5	C11—Si4—C10	108.40 (13)
Si3—C9—H9C	109.5	C12—Si4—C10	108.88 (14)
H9A—C9—H9C	109.5	N4—Si5—C14	118.23 (9)
H9B—C9—H9C	109.5	N4—Si5—C15	109.61 (10)
Si4—C10—H10A	109.5	C14—Si5—C15	105.73 (12)
Si4—C10—H10B	109.5	N4—Si5—C13	109.35 (10)
H10A—C10—H10B	109.5	C14—Si5—C13	101.96 (11)
Si4—C10—H10C	109.5	C15—Si5—C13	111.74 (12)
H10A—C10—H10C	109.5	N4—Si6—C17	110.54 (10)
H10B—C10—H10C	109.5	N4—Si6—C18	116.19 (10)

Si4—C11—H11A	109.5	C17—Si6—C18	110.40 (13)
Si4—C11—H11B	109.5	N4—Si6—C16	108.40 (10)
H11A—C11—H11B	109.5	C17—Si6—C16	109.50 (12)
Si4—C11—H11C	109.5	C18—Si6—C16	101.29 (12)
H11A—C11—H11C	109.5	C21A—O1A—C20A	115.6 (3)
H11B—C11—H11C	109.5	C20A—C19A—H19A	109.5
Si4—C12—H12A	109.5	C20A—C19A—H19B	109.5
Si4—C12—H12B	109.5	H19A—C19A—H19B	109.5
H12A—C12—H12B	109.5	C20A—C19A—H19C	109.5
Si4—C12—H12C	109.5	H19A—C19A—H19C	109.5
H12A—C12—H12C	109.5	H19B—C19A—H19C	109.5
H12B—C12—H12C	109.5	O1A—C20A—C19A	106.5 (4)
Si5—C13—H13A	109.5	O1A—C20A—H20A	110.4
Si5—C13—H13B	109.5	C19A—C20A—H20A	110.4
H13A—C13—H13B	109.5	O1A—C20A—H20B	110.4
Si5—C13—H13C	109.5	C19A—C20A—H20B	110.4
H13A—C13—H13C	109.5	H20A—C20A—H20B	108.6
H13B—C13—H13C	109.5	O1A—C21A—C22A	111.4 (4)
Si5—C14—H14A	109.5	O1A—C21A—H21A	109.4
Si5—C14—H14B	109.5	C22A—C21A—H21A	109.4
H14A—C14—H14B	109.5	O1A—C21A—H21B	109.4
Si5—C14—H14C	109.5	C22A—C21A—H21B	109.4
H14A—C14—H14C	109.5	H21A—C21A—H21B	108.0
H14B—C14—H14C	109.5	C21A—C22A—H22A	109.5
Si5—C15—H15A	109.5	C21A—C22A—H22B	109.5
Si5—C15—H15B	109.5	H22A—C22A—H22B	109.5
H15A—C15—H15B	109.5	C21A—C22A—H22C	109.5
Si5—C15—H15C	109.5	H22A—C22A—H22C	109.5
H15A—C15—H15C	109.5	H22B—C22A—H22C	109.5
H15B—C15—H15C	109.5	C21B—O1B—C20B	111.9 (4)
Si6—C16—H16A	109.5	C20B—C19B—H19D	109.5
Si6—C16—H16B	109.5	C20B—C19B—H19E	109.5
H16A—C16—H16B	109.5	H19D—C19B—H19E	109.5
Si6—C16—H16C	109.5	C20B—C19B—H19F	109.5
H16A—C16—H16C	109.5	H19D—C19B—H19F	109.5
H16B—C16—H16C	109.5	H19E—C19B—H19F	109.5
Si6—C17—H17A	109.5	O1B—C20B—C19B	107.1 (4)
Si6—C17—H17B	109.5	O1B—C20B—H20C	110.3
H17A—C17—H17B	109.5	C19B—C20B—H20C	110.3
Si6—C17—H17C	109.5	O1B—C20B—H20D	110.3
H17A—C17—H17C	109.5	C19B—C20B—H20D	110.3
H17B—C17—H17C	109.5	H20C—C20B—H20D	108.5
Si6—C18—H18A	109.5	O1B—C21B—C22B	108.3 (4)
Si6—C18—H18B	109.5	O1B—C21B—H21C	110.0
H18A—C18—H18B	109.5	C22B—C21B—H21C	110.0
Si6—C18—H18C	109.5	O1B—C21B—H21D	110.0
H18A—C18—H18C	109.5	C22B—C21B—H21D	110.0
H18B—C18—H18C	109.5	H21C—C21B—H21D	108.4

Pd1—Cl1—Pd1^{i}	91.589 (15)	C21B—C22B—H22D	109.5
P1—N1—Si2	120.40 (10)	C21B—C22B—H22E	109.5
P1—N1—Si1	119.78 (10)	H22D—C22B—H22E	109.5
Si2—N1—Si1	116.70 (9)	C21B—C22B—H22F	109.5
P1—N2—P2	97.45 (8)	H22D—C22B—H22F	109.5
P1—N2—Si3	130.40 (10)	H22E—C22B—H22F	109.5
P2—N2—Si3	130.69 (9)		
Si2—N1—P1—N2	−51.67 (14)	P1—N1—Si1—C1	139.3 (2)
Si1—N1—P1—N2	148.97 (10)	Si2—N1—Si1—C1	−20.8 (2)
Si2—N1—P1—Cl2	−160.70 (9)	P1—N1—Si1—C3	23.00 (17)
Si1—N1—P1—Cl2	39.95 (11)	Si2—N1—Si1—C3	−137.10 (13)
Si2—N1—P1—Pd1	66.59 (12)	P1—N1—Si2—C4	30.84 (16)
Si1—N1—P1—Pd1	−92.77 (11)	Si1—N1—Si2—C4	−169.18 (13)
Si2—N1—P1—P2	−3.89 (17)	P1—N1—Si2—C5	−89.26 (14)
Si1—N1—P1—P2	−163.24 (5)	Si1—N1—Si2—C5	70.71 (14)
P2—N2—P1—N1	136.44 (8)	P1—N1—Si2—C6	146.73 (14)
Si3—N2—P1—N1	−56.48 (15)	Si1—N1—Si2—C6	−53.29 (15)
P2—N2—P1—Cl2	−114.39 (6)	P1—N2—Si3—C9	−99.16 (15)
Si3—N2—P1—Cl2	52.69 (13)	P2—N2—Si3—C9	63.83 (16)
P2—N2—P1—Pd1	6.45 (7)	P1—N2—Si3—C7	139.60 (14)
Si3—N2—P1—Pd1	173.53 (11)	P2—N2—Si3—C7	−57.41 (16)
Si3—N2—P1—P2	167.07 (18)	P1—N2—Si3—C8	19.42 (18)
Si4—N3—P2—N4	−136.4 (2)	P2—N2—Si3—C8	−177.58 (14)
Si4—N3—P2—N2	102.7 (2)	P2—N3—Si4—C11	105.7 (2)
Si4—N3—P2—Pd1	−3.6 (3)	P2—N3—Si4—C12	−14.4 (3)
Si4—N3—P2—P1	61.8 (3)	P2—N3—Si4—C10	−135.1 (2)
Si5—N4—P2—N3	149.26 (11)	P2—N4—Si5—C14	−2.46 (17)
Si6—N4—P2—N3	−9.39 (13)	Si6—N4—Si5—C14	156.13 (11)
Si5—N4—P2—N2	−88.03 (12)	P2—N4—Si5—C15	118.75 (14)
Si6—N4—P2—N2	113.32 (10)	Si6—N4—Si5—C15	−82.67 (13)
Si5—N4—P2—Pd1	13.45 (13)	P2—N4—Si5—C13	−118.43 (13)
Si6—N4—P2—Pd1	−145.20 (7)	Si6—N4—Si5—C13	40.16 (13)
Si5—N4—P2—P1	−45.96 (13)	P2—N4—Si6—C17	80.04 (13)
Si6—N4—P2—P1	155.39 (7)	Si5—N4—Si6—C17	−80.83 (13)
P1—N2—P2—N3	−128.98 (9)	P2—N4—Si6—C18	−46.76 (15)
Si3—N2—P2—N3	64.00 (15)	Si5—N4—Si6—C18	152.37 (12)
P1—N2—P2—N4	108.99 (9)	P2—N4—Si6—C16	−159.94 (12)
Si3—N2—P2—N4	−58.03 (14)	Si5—N4—Si6—C16	39.19 (14)
P1—N2—P2—Pd1	−6.30 (7)	C21A—O1A—C20A—C19A	−177.5 (4)
Si3—N2—P2—Pd1	−173.32 (11)	C20A—O1A—C21A—C22A	175.5 (4)
Si3—N2—P2—P1	−167.02 (18)	C21B—O1B—C20B—C19B	178.5 (15)
P1—N1—Si1—C2	−100.16 (14)	C20B—O1B—C21B—C22B	−98.0 (17)
Si2—N1—Si1—C2	99.74 (14)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*C*···Cl1^{i}	0.98	2.72	3.564 (3)	144
C14—H14*C*···Cl2	0.98	2.67	3.585 (2)	156

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.