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4,4,6,6-Tetrachloro-2-[(2,4-dimethylphenyl)sulfanyl]-*N*-[4-(2,2,4,4-tetrachloro-1,3,5,7,11-pentaaza-2λ^5,4λ^5,6λ^5-triphosphaspiro[5.5]undeca-1,3,5-trien-7-yl)butyl]-1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphinin-2-amine

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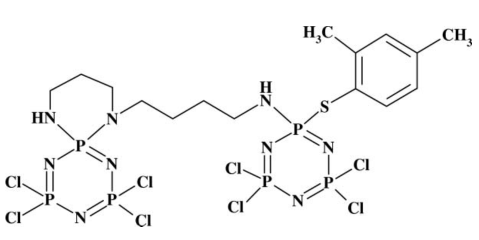
Key indicators: single-crystal X-ray study; *T* = 120 K; mean σ(C–C) = 0.006 Å; disorder in main residue; *R* factor = 0.043; *wR* factor = 0.107; data-to-parameter ratio = 20.7.

Reaction of the spermidine-bridged cyclophosphazene compound ($N_3P_3Cl_5$)spd($N_3P_3Cl_4$) (spd = spermidine residue) with 2,4-dimethylthiophenol results in substitution of the P—Cl bond at the bridgehead >P(NHR)Cl group to give the title compound, $C_{15}H_{25}C_{l8}N_9P_6S$. An N—H···N hydrogen bond helps to establish the packing.

Related literature

For background chemistry, see: Labarre *et al.* (1984); Beşli *et al.* (2004); Guerch *et al.* (1984); Kılıç *et al.* (1991); Cameron *et al.* (1989); İbişoğlu (2007); Guerch & Labarre (1989).



Experimental

Crystal data

$C_{15}H_{25}Cl_8N_9P_6S$
M_r = 832.92
Orthorhombic, $P2_12_12_1$
a = 8.5829 (17) Å
b = 15.587 (3) Å
c = 25.277 (5) Å
V = 3381.6 (12) Å^3
Z = 4
Mo *K*α radiation
μ = 1.04 mm^{-1}
T = 120 (2) K
0.48 × 0.20 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.539, T_{max} = 0.835
20485 measured reflections
7560 independent reflections
6007 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 0.99
7560 reflections
366 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), 3211 Friedel pairs
Flack parameter: 0.10 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*N*···N9^{i}	0.77 (4)	2.26 (4)	3.009 (4)	164 (3)

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

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2491).

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supplementary materials

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4,4,6,6-Tetrachloro-2-[(2,4-dimethylphenyl)sulfanyl]-*N*-[4-(2,2,4,4-tetrachloro-1,3,5,7,11-pentaaza-2λ^5,4λ^5,6λ^5-triphosphaspiro[5.5]undeca-1,3,5-trien-7-yl)butyl]-1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphinin-2-amine

S. J. Coles, D. B. Davies, M. B. Hursthouse, H. Ibisoglu, A. Kiliç and R. A. Shaw

Comment

The reaction of biogenic spermidine with cyclophosphazene, $N_3P_3Cl_6$, in aprotic solvents gives a spermidine-bridged cyclophosphazene compound, $(N_3P_3Cl_5)spd(N_3P_3Cl_4)$, (spd = trifunctional spermidine residue), (Labarre *et al.*, 1984; Guerch *et al.*, 1984; Kılıç *et al.*, 1991) and in protic solvents such as $CHCl_3$ proceeds cleanly to yield a spiro-*cis*-ansa spermidine derivative, 2,2,6-trichloro-1,3,5,7,11,16-hexaaza-4,6-diphosphatricyclohexadeca-2,4,6-triene (Guerch & Labarre, 1989; Cameron *et al.*,1989). We have investigated previously the reactions of the spiro-*cis*-ansa compound with primary and secondary amines, mono- and difunctional alcohols (*e.g.* $PhNH_2$, Pr^nNH_2, HNC_4H_8, $HNMe_2$, MeOH, and $HOCH_2(CF_2)_2CH_2OH$) (Beşli *et al.*, 2004; İbişoğlu, 2007).

In this work reaction of the spermidine-bridged cyclophosphazene with 2,4-dimethylthiophenol gave a novel cyclophosphazene-thiolate derivative, (I), (Fig. 1), in which the bridgehead P—Cl bond was replaced with a thiolate group. Thus the S*R* reagent has attacked the PCl(NHR) group, rather than the more electrophilic PCl_2 moiety, which is an attack not on the phosphorus centre, but on the hydrogen, giving the product *via* a proton abstraction/chloride elimination reaction.

In the cyclophosphazene rings of (I) the P—N bond lengths are in the range 1.554 (3)–1.632 (3) Å but separate into two distinct groups for each ring; the lengths of the four P—N bonds involving the >PCl_2 moiety in both rings are in a narrow range [1.554 (3)–1.585 (3) Å] with averages of *ca*. 1.57(±0.01) Å compared to the two P—N bond lengths involving the >P(N)(*X*) moiety [*X* =SPh or NH(bridge)], which are in the range 1.610 (3)–1.632 (3)Å and average *ca*. 1.62(±0.01) Å. The increase in bond length involving the >P(N)(*X*) moiety depend on electron release from either the SPh or the NH(bridge) atoms to the phosphazene rings. The donation of electrons from the >P(N)(*X*) moiety to the phosphazene ring is also manifested by the decrease in endocyclic N—P(N)(*X*)—N bond angles for the two rings [*X* = SPh, 114.6 (2)°; *X* = NH(bridge), 110.7 (2)°] compared to those found for the N—P(Cl_2)-N, 119.9(±0.3)°.

The effect of the electron-donating substituents is also reflected in differences in the P···P distances across the ring, even for rings that are nominally planar. It is found that the P···P distances involving the P(N)(*X*) moiety are 2.781 (6)/2.791 (6) Å (*X* = SPh) and 2.816 (5)/2.828 (6) Å (*X* = NH(bridge) are significantly greater than those involving the >PCl_2 moiety, 2.71–2.72 Å.

An N—H···N hydrogen bond (Table 1) helps to establish the packing.

Experimental

A solution of $(N_3P_3Cl_5)spd(N_3P_3Cl_4)$ (Labarre *et al.*, 1984, Kılıç *et al.* 1991) (1 g, 1.367 mmol) in 10 ml dry THF was added dropwise to a stirred solution of triethylamine (1.38 g, 13.63 mmol) in 8 ml dry THF at 195 K. Then, 2,4-dimethylthiophenol

(1.88 g, 13,6 mmol) in 10 ml dry THF was added and the reaction mixture was stirred under an atmosphere of argon at room temperature for 5 days. Triethylamine hydrochloride was filtered off and the solvent removed under reduced pressure at 303 K. One product,4,4,6,6-tetrachloro-2-[(2,4-dimethylphenyl)thio]-*N*-[4-(2,2,4,4-tetrachloro-1,3,5,7,11-pentaaza-2λ^5,4λ^5,6λ^5-triphosphaspiro[5.5]undeca-1,3,5-trien-7-yl)butyl]-1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphinin-2-amine, I, was detected by TLC using dichloromethane as mobile phase [R_f = 0.49] and was separated by column chromatography on silica gel using $CHCl_3$ as eluant. Colourless rods of (I) were re-crystallized from dichloromethane-*n*-hexane (1:1 *v*/*v*) (m.p. 405–406 K, 0.74 g, yield 65%).

Elemental analysis calcd (%) for $C_{15}H_{25}Cl_8N_9P_6S$;C, 21.63; H, 3.03; N, 15.13%; *found:* C, 21.36; H, 3.31; N, 15.22%; MS $(M+H)^+$,833M^+; 832.^{1}H NMR at 298 K in $CDCl_3$: δ 2.3 (s, 3H, CH_3); δ 2.5 (s, 3H, CH_3); δ 7.45 (q, 1H, CH-6); δ 7.07 (s, 1H, CH-3); δ 6.97 (d, 1H, CH-5); δ 3.3–2.8 p.p.m. (m, 8H)(4x N—CH_2); δ 1.6–1.8 (m, 6H)(3x C—CH_2); δ 3,0 (2*H*, 2x NH). The proton decoupled ^{31}P NMR spectrum at 298 K in $CDCl_3$ has two different spin A_2X spin systems: >*P*(Nspiro) cyclophosphazene ring; (δ*P*(Nspiro)(P_1), 10.7 p.p.m. (triplet); δPCl_2 (P_2), 22.2 p.p.m. (doublet), ^{2}J(P1P2) 40.3 Hz: >P(SC_8H_9) cyclophosphazene ring; δP($NHSC_8H_9$)(P_3), 30.7 p.p.m. (triplet); δPCl_2 (P4), 20.3 p.p.m. (doublet), ^{2}J(P3P4) 19.3 Hz.

Refinement

The C-bound hydrogen atoms and H6N were fixed in idealized positions [0.88 (NH), 0.98 (CH_3), 0.99 Å (CH_2) & 0.95 Å (CH)] and refined using the riding model with U_{iso}(H) either set to 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C). Atom H4N was located in a difference map and freely refined. The C14 methylene group exhibits positional disorder within its ring system and has been modelled as two discrete sites with an occupancy ratio of 65:35. This disorder results in large anisotropic displacement parameters for this atom and those connected to it.

Figures

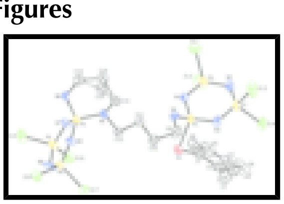


Fig. 1. View of (I) (50% probability displacement ellipsoids; H atoms omitted for clarity).

4,4,6,6-Tetrachloro-2-[(2,4-dimethylphenyl)sulfanyl]-*N*-[4-(2,2,4,4-\ tetrachloro-1,3,5,7,11-pentaaza-2λ^5,4λ^5,6λ^5-\ triphosphaspiro[5.5]undeca-1,3,5-trien-7-yl)butyl]-\ 1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphinin-2-amine

Crystal data

$C_{15}H_{25}Cl_8N_9P_6S$	F_{000} = 1680
M_r = 832.92	D_x = 1.636 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 18434 reflections

a = 8.5829 (17) Å	θ = 2.9–27.5°
b = 15.587 (3) Å	μ = 1.04 mm^{-1}
c = 25.277 (5) Å	T = 120 (2) K
V = 3381.6 (12) Å^3	Rod, colourless
Z = 4	0.48 × 0.20 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.082
φ and ω scans	θ_{max} = 27.5°
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	θ_{min} = 3.1°
T_{min} = 0.539, T_{max} = 0.835	h = −11→11
20485 measured reflections	k = −20→20
7560 independent reflections	l = −31→32
6007 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.107	$(\Delta/\sigma)_{max}$ = 0.010
S = 0.99	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
7560 reflections	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
366 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 3211 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.10 (7)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.7799 (5)	0.2378 (2)	0.17416 (15)	0.0340 (9)	
C2	0.6726 (5)	0.2176 (3)	0.13485 (16)	0.0385 (10)	
C3	0.7270 (6)	0.2077 (3)	0.08372 (18)	0.0504 (12)	
H3	0.6553	0.1926	0.0566	0.061*	

C4	0.8822 (7)	0.2192 (3)	0.0706 (2)	0.0563 (14)	
C5	0.9872 (6)	0.2373 (3)	0.1105 (2)	0.0579 (14)	
H5	1.0947	0.2436	0.1024	0.069*	
C6	0.9363 (5)	0.2464 (3)	0.1628 (2)	0.0468 (11)	
H6	1.0088	0.2584	0.1903	0.056*	
C7	0.5021 (6)	0.2092 (3)	0.1452 (2)	0.0607 (14)	
H7A	0.4483	0.1949	0.1122	0.091*	
H7B	0.4618	0.2636	0.1590	0.091*	
H7C	0.4844	0.1637	0.1713	0.091*	
C8	0.9353 (9)	0.2165 (3)	0.0138 (2)	0.095 (3)	
H8A	1.0482	0.2254	0.0122	0.142*	
H8B	0.8826	0.2619	−0.0063	0.142*	
H8C	0.9094	0.1606	−0.0016	0.142*	
C9	0.5008 (4)	0.1678 (3)	0.33785 (15)	0.0372 (9)	
H9A	0.4487	0.1147	0.3502	0.045*	
H9B	0.4545	0.1838	0.3034	0.045*	
C10	0.4714 (4)	0.2383 (3)	0.37703 (14)	0.0333 (9)	
H10A	0.3609	0.2560	0.3744	0.040*	
H10B	0.5363	0.2884	0.3673	0.040*	
C11	0.5061 (4)	0.2141 (2)	0.43411 (14)	0.0271 (8)	
H11A	0.4547	0.1589	0.4425	0.032*	
H11B	0.6198	0.2063	0.4385	0.032*	
C12	0.4492 (4)	0.2824 (2)	0.47241 (14)	0.0270 (8)	
H12A	0.4948	0.3384	0.4623	0.032*	
H12B	0.3345	0.2874	0.4695	0.032*	
C13	0.4204 (4)	0.1851 (2)	0.54963 (15)	0.0310 (8)	
H13A	0.3142	0.1804	0.5346	0.037*	
H13B	0.4809	0.1358	0.5360	0.037*	
C14'	0.4070 (17)	0.1747 (8)	0.6071 (5)	0.034 (4)	0.341 (13)
H14A	0.3043	0.1973	0.6182	0.041*	0.341 (13)
H14B	0.4080	0.1125	0.6151	0.041*	0.341 (13)
C14	0.5145 (9)	0.1547 (4)	0.5968 (2)	0.033 (2)	0.659 (13)
H14C	0.4664	0.1016	0.6108	0.039*	0.659 (13)
H14D	0.6209	0.1400	0.5846	0.039*	0.659 (13)
C15	0.5264 (5)	0.2159 (3)	0.63906 (18)	0.0445 (10)	
H15A	0.6221	0.1805	0.6376	0.053*	
H15B	0.4908	0.2169	0.6763	0.053*	
N1	0.9248 (3)	0.11118 (19)	0.28052 (12)	0.0283 (7)	
N2	0.9007 (4)	−0.0284 (2)	0.21604 (15)	0.0443 (9)	
N3	0.6461 (3)	0.0696 (2)	0.23679 (12)	0.0312 (7)	
N4	0.6684 (4)	0.1507 (2)	0.33068 (12)	0.0295 (7)	
N5	0.4910 (3)	0.26331 (18)	0.52811 (11)	0.0277 (7)	
N6	0.5661 (4)	0.3034 (2)	0.62336 (12)	0.0308 (7)	
H6N	0.6196	0.3349	0.6458	0.037*	
N7	0.6552 (3)	0.40436 (18)	0.54310 (13)	0.0315 (7)	
N8	0.4825 (4)	0.54976 (18)	0.53518 (15)	0.0393 (8)	
N9	0.3597 (3)	0.40272 (18)	0.57517 (13)	0.0291 (7)	
P1	0.74388 (10)	0.13582 (6)	0.27261 (4)	0.0250 (2)	
P2	1.00284 (12)	0.03262 (6)	0.25224 (4)	0.0326 (2)	

P3	0.72065 (12)	−0.01195 (6)	0.21149 (4)	0.0334 (2)
P4	0.51752 (10)	0.34622 (5)	0.56690 (4)	0.0243 (2)
P5	0.64364 (10)	0.50149 (6)	0.52941 (4)	0.0304 (2)
P6	0.33674 (10)	0.49668 (6)	0.55497 (4)	0.0271 (2)
S1	0.72088 (12)	0.25711 (6)	0.24069 (4)	0.0352 (2)
Cl1	1.11015 (15)	−0.03697 (8)	0.30779 (5)	0.0569 (3)
Cl2	1.18636 (13)	0.07085 (8)	0.20992 (6)	0.0611 (3)
Cl3	0.61143 (17)	−0.11661 (8)	0.23825 (5)	0.0639 (4)
Cl4	0.66310 (15)	−0.01667 (7)	0.13493 (4)	0.0517 (3)
Cl5	0.80436 (11)	0.56671 (7)	0.56962 (5)	0.0464 (3)
Cl6	0.71814 (13)	0.52044 (7)	0.45493 (4)	0.0490 (3)
Cl7	0.17627 (11)	0.49868 (6)	0.49709 (4)	0.0395 (2)
Cl8	0.22764 (12)	0.56464 (6)	0.61084 (4)	0.0415 (2)
H4N	0.722 (4)	0.129 (2)	0.3513 (16)	0.020 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.051 (2)	0.0225 (19)	0.028 (2)	0.0072 (16)	0.0079 (18)	0.0050 (16)
C2	0.055 (3)	0.032 (2)	0.029 (2)	0.0059 (18)	0.0015 (19)	0.0086 (17)
C3	0.090 (4)	0.032 (2)	0.029 (2)	0.008 (2)	−0.001 (2)	0.0071 (18)
C4	0.103 (4)	0.021 (2)	0.045 (3)	−0.003 (2)	0.029 (3)	0.009 (2)
C5	0.068 (3)	0.034 (3)	0.071 (4)	−0.005 (2)	0.037 (3)	0.005 (2)
C6	0.053 (3)	0.030 (2)	0.057 (3)	−0.0063 (19)	0.007 (2)	0.003 (2)
C7	0.054 (3)	0.076 (4)	0.051 (3)	0.015 (3)	−0.010 (3)	0.004 (3)
C8	0.188 (7)	0.038 (3)	0.058 (4)	0.003 (4)	0.064 (5)	0.004 (3)
C9	0.027 (2)	0.059 (3)	0.026 (2)	−0.0028 (18)	0.0038 (16)	−0.0086 (19)
C10	0.032 (2)	0.042 (2)	0.025 (2)	0.0060 (16)	0.0009 (15)	−0.0065 (17)
C11	0.0256 (17)	0.0287 (19)	0.027 (2)	−0.0005 (14)	0.0036 (15)	0.0010 (16)
C12	0.0308 (18)	0.029 (2)	0.0209 (19)	0.0035 (14)	−0.0021 (14)	0.0002 (15)
C13	0.036 (2)	0.0249 (19)	0.032 (2)	−0.0008 (15)	0.0017 (16)	0.0012 (16)
C14'	0.044 (10)	0.034 (7)	0.025 (6)	−0.002 (5)	0.008 (5)	−0.003 (5)
C14	0.039 (5)	0.031 (3)	0.028 (3)	0.001 (3)	0.008 (3)	0.004 (3)
C15	0.066 (3)	0.034 (2)	0.034 (2)	−0.006 (2)	−0.011 (2)	0.0093 (19)
N1	0.0297 (15)	0.0263 (16)	0.0288 (17)	0.0001 (12)	−0.0021 (13)	−0.0043 (14)
N2	0.050 (2)	0.038 (2)	0.045 (2)	0.0053 (15)	−0.0034 (17)	−0.0168 (17)
N3	0.0316 (15)	0.0353 (18)	0.0270 (17)	−0.0037 (13)	0.0000 (13)	−0.0064 (14)
N4	0.0309 (17)	0.040 (2)	0.0173 (17)	0.0083 (14)	−0.0009 (13)	−0.0024 (14)
N5	0.0423 (17)	0.0206 (15)	0.0203 (16)	0.0012 (13)	0.0018 (13)	0.0011 (12)
N6	0.0408 (18)	0.0281 (17)	0.0237 (17)	−0.0022 (13)	−0.0059 (14)	−0.0020 (13)
N7	0.0306 (16)	0.0215 (15)	0.043 (2)	0.0029 (12)	0.0067 (14)	0.0004 (14)
N8	0.0343 (17)	0.0204 (16)	0.063 (2)	0.0056 (13)	0.0067 (16)	0.0076 (16)
N9	0.0302 (15)	0.0226 (16)	0.0347 (19)	0.0032 (12)	0.0022 (13)	0.0042 (14)
P1	0.0293 (5)	0.0253 (5)	0.0203 (5)	−0.0001 (3)	0.0018 (3)	−0.0038 (4)
P2	0.0363 (5)	0.0306 (5)	0.0308 (5)	0.0054 (4)	0.0038 (4)	−0.0049 (4)
P3	0.0471 (6)	0.0279 (5)	0.0253 (5)	−0.0067 (4)	0.0005 (4)	−0.0061 (4)
P4	0.0282 (4)	0.0207 (4)	0.0238 (5)	0.0015 (3)	0.0015 (4)	−0.0011 (4)
P5	0.0289 (5)	0.0238 (5)	0.0386 (6)	−0.0009 (4)	0.0064 (4)	0.0009 (4)

P6	0.0277 (4)	0.0225 (5)	0.0310 (5)	0.0028 (4)	0.0011 (4)	−0.0005 (4)
S1	0.0508 (5)	0.0262 (5)	0.0285 (5)	0.0066 (4)	0.0029 (4)	−0.0005 (4)
Cl1	0.0682 (8)	0.0498 (7)	0.0527 (7)	0.0245 (6)	−0.0079 (6)	0.0022 (6)
Cl2	0.0467 (6)	0.0669 (8)	0.0695 (9)	0.0057 (6)	0.0270 (6)	−0.0005 (7)
Cl3	0.0988 (10)	0.0427 (7)	0.0501 (8)	−0.0335 (6)	0.0029 (7)	−0.0012 (6)
Cl4	0.0788 (8)	0.0493 (7)	0.0268 (5)	0.0018 (6)	−0.0056 (5)	−0.0129 (5)
Cl5	0.0415 (5)	0.0398 (6)	0.0578 (7)	−0.0148 (4)	0.0028 (5)	−0.0044 (5)
Cl6	0.0599 (6)	0.0455 (6)	0.0416 (6)	0.0046 (5)	0.0146 (5)	0.0103 (5)
Cl7	0.0442 (5)	0.0385 (6)	0.0358 (5)	0.0040 (4)	−0.0094 (4)	0.0021 (5)
Cl8	0.0455 (6)	0.0374 (5)	0.0415 (6)	0.0103 (4)	0.0030 (4)	−0.0115 (5)

Geometric parameters (Å, °)

C1—C2	1.391 (6)	C14'—C15	1.456 (13)
C1—C6	1.379 (6)	C14'—H14A	0.9900
C1—S1	1.782 (4)	C14'—H14B	0.9900
C2—C3	1.383 (6)	C14—C15	1.436 (7)
C2—C7	1.493 (7)	C14—H14C	0.9900
C3—C4	1.385 (8)	C14—H14D	0.9900
C3—H3	0.9500	C15—N6	1.460 (5)
C4—C5	1.381 (8)	C15—H15A	0.9900
C4—C8	1.508 (7)	C15—H15B	0.9900
C5—C6	1.401 (7)	N1—P2	1.568 (3)
C5—H5	0.9500	N1—P1	1.612 (3)
C6—H6	0.9500	N2—P3	1.571 (4)
C7—H7A	0.9800	N2—P2	1.585 (4)
C7—H7B	0.9800	N3—P3	1.561 (3)
C7—H7C	0.9800	N3—P1	1.609 (3)
C8—H8A	0.9800	N4—P1	1.621 (3)
C8—H8B	0.9800	N4—H4N	0.78 (4)
C8—H8C	0.9800	N5—P4	1.638 (3)
C9—N4	1.475 (5)	N6—P4	1.630 (3)
C9—C10	1.501 (5)	N6—H6N	0.8800
C9—H9A	0.9900	N7—P5	1.556 (3)
C9—H9B	0.9900	N7—P4	1.606 (3)
C10—C11	1.521 (5)	N8—P5	1.581 (3)
C10—H10A	0.9900	N8—P6	1.581 (3)
C10—H10B	0.9900	N9—P6	1.564 (3)
C11—C12	1.520 (5)	N9—P4	1.629 (3)
C11—H11A	0.9900	P1—S1	2.0651 (13)
C11—H11B	0.9900	P2—Cl2	1.9950 (15)
C12—N5	1.483 (4)	P2—Cl1	1.9991 (16)
C12—H12A	0.9900	P3—Cl4	1.9986 (15)
C12—H12B	0.9900	P3—Cl3	1.9995 (15)
C13—N5	1.466 (5)	P5—Cl5	1.9924 (14)
C13—C14'	1.466 (12)	P5—Cl6	2.0100 (15)
C13—C14	1.516 (7)	P6—Cl8	1.9984 (13)
C13—H13A	0.9900	P6—Cl7	2.0097 (13)
C13—H13B	0.9900		

C2—C1—C6	121.2 (4)	H14A—C14'—H14B	107.3
C2—C1—S1	121.6 (3)	C15—C14—C13	114.6 (5)
C6—C1—S1	117.1 (4)	C15—C14—H14C	108.6
C3—C2—C1	118.1 (4)	C13—C14—H14C	108.6
C3—C2—C7	119.0 (5)	C15—C14—H14D	108.6
C1—C2—C7	122.9 (4)	C13—C14—H14D	108.6
C2—C3—C4	122.3 (5)	H14C—C14—H14D	107.6
C2—C3—H3	118.9	C14—C15—N6	115.7 (4)
C4—C3—H3	118.9	C14—C15—C14'	40.8 (5)
C5—C4—C3	118.6 (5)	N6—C15—C14'	115.2 (6)
C5—C4—C8	120.3 (6)	C14—C15—H15A	70.0
C3—C4—C8	121.0 (6)	N6—C15—H15A	108.5
C4—C5—C6	120.5 (5)	C14'—C15—H15A	108.5
C4—C5—H5	119.8	C14—C15—H15B	134.0
C6—C5—H5	119.8	N6—C15—H15B	108.5
C1—C6—C5	119.3 (5)	C14'—C15—H15B	108.5
C1—C6—H6	120.3	H15A—C15—H15B	107.5
C5—C6—H6	120.3	P2—N1—P1	122.7 (2)
C2—C7—H7A	109.5	P3—N2—P2	119.2 (2)
C2—C7—H7B	109.5	P3—N3—P1	122.6 (2)
H7A—C7—H7B	109.5	C9—N4—P1	121.8 (3)
C2—C7—H7C	109.5	C9—N4—H4N	125 (3)
H7A—C7—H7C	109.5	P1—N4—H4N	108 (3)
H7B—C7—H7C	109.5	C13—N5—C12	114.8 (3)
C4—C8—H8A	109.5	C13—N5—P4	119.4 (2)
C4—C8—H8B	109.5	C12—N5—P4	116.3 (2)
H8A—C8—H8B	109.5	C15—N6—P4	124.2 (3)
C4—C8—H8C	109.5	C15—N6—H6N	117.9
H8A—C8—H8C	109.5	P4—N6—H6N	117.9
H8B—C8—H8C	109.5	P5—N7—P4	125.84 (18)
N4—C9—C10	112.1 (3)	P5—N8—P6	118.17 (19)
N4—C9—H9A	109.2	P6—N9—P4	124.70 (19)
C10—C9—H9A	109.2	N3—P1—N1	114.83 (16)
N4—C9—H9B	109.2	N3—P1—N4	113.10 (17)
C10—C9—H9B	109.2	N1—P1—N4	107.87 (18)
H9A—C9—H9B	107.9	N3—P1—S1	108.49 (13)
C9—C10—C11	114.3 (3)	N1—P1—S1	111.01 (12)
C9—C10—H10A	108.7	N4—P1—S1	100.64 (13)
C11—C10—H10A	108.7	N1—P2—N2	119.74 (18)
C9—C10—H10B	108.7	N1—P2—Cl2	110.37 (13)
C11—C10—H10B	108.7	N2—P2—Cl2	107.86 (16)
H10A—C10—H10B	107.6	N1—P2—Cl1	107.48 (13)
C12—C11—C10	111.6 (3)	N2—P2—Cl1	109.54 (15)
C12—C11—H11A	109.3	Cl2—P2—Cl1	100.08 (7)
C10—C11—H11A	109.3	N3—P3—N2	120.47 (18)
C12—C11—H11B	109.3	N3—P3—Cl4	108.99 (13)
C10—C11—H11B	109.3	N2—P3—Cl4	107.95 (15)
H11A—C11—H11B	108.0	N3—P3—Cl3	109.50 (13)
N5—C12—C11	112.7 (3)	N2—P3—Cl3	107.62 (16)

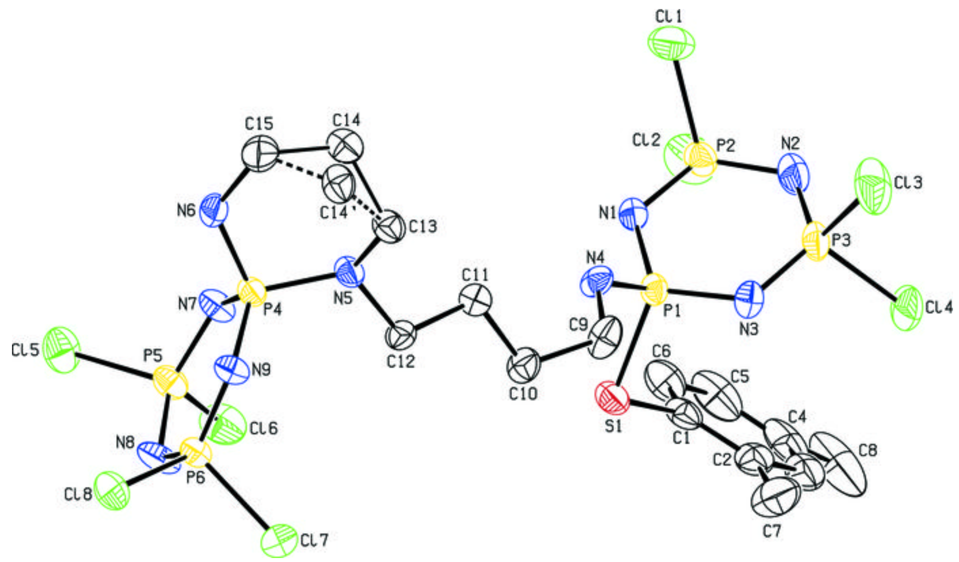
N5—C12—H12A	109.0	Cl4—P3—Cl3	100.47 (7)
C11—C12—H12A	109.0	N7—P4—N9	110.77 (15)
N5—C12—H12B	109.0	N7—P4—N6	111.80 (17)
C11—C12—H12B	109.0	N9—P4—N6	108.77 (17)
H12A—C12—H12B	107.8	N7—P4—N5	108.85 (16)
N5—C13—C14'	119.5 (6)	N9—P4—N5	112.82 (16)
N5—C13—C14	109.3 (4)	N6—P4—N5	103.67 (15)
C14'—C13—C14	39.5 (5)	N7—P5—N8	119.88 (16)
N5—C13—H13A	107.4	N7—P5—Cl5	109.82 (13)
C14'—C13—H13A	107.4	N8—P5—Cl5	108.40 (14)
C14—C13—H13A	140.0	N7—P5—Cl6	109.32 (13)
N5—C13—H13B	107.4	N8—P5—Cl6	107.14 (15)
C14'—C13—H13B	107.4	Cl5—P5—Cl6	100.52 (6)
C14—C13—H13B	76.0	N9—P6—N8	119.59 (15)
H13A—C13—H13B	107.0	N9—P6—Cl8	108.95 (13)
C15—C14'—C13	116.5 (9)	N8—P6—Cl8	108.47 (14)
C15—C14'—H14A	108.2	N9—P6—Cl7	109.80 (13)
C13—C14'—H14A	108.2	N8—P6—Cl7	107.69 (15)
C15—C14'—H14B	108.2	Cl8—P6—Cl7	100.67 (6)
C13—C14'—H14B	108.2	C1—S1—P1	100.74 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4N···N9^{i}	0.77 (4)	2.26 (4)	3.009 (4)	164 (3)

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

Fig. 1


Cl1
C15
C14
Cl2
P2
N2
Cl3
N6
C14
N1
C13
P3
N4
C11
N7
P4
N5
P1
N3
Cl4
Cl5
P5
C9
N9
C12
C10
C6
C5
S1
C1
C4
Cl6
N8
P6
C2
C8
Cl8
C7
Cl7