Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1,3,3-Tetraphenyl-1,2,3-triphosphenium tetrachloroaluminate dichloromethane solvate

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Received 5 January 2005
Accepted 7 January 2005
Online 15 January 2005

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.075
Data-to-parameter ratio = 23.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

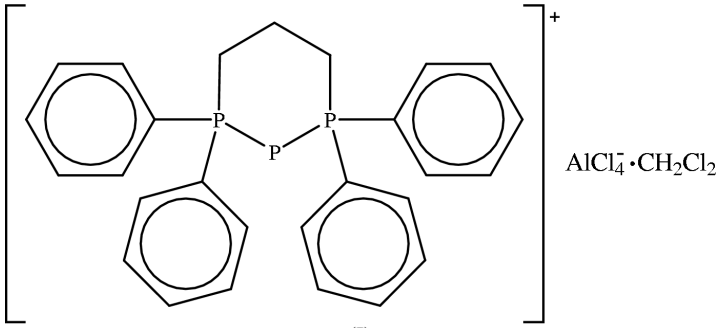
The title compound, $(C_{27}H_{26}P_3)[AlCl_4]\cdot CH_2Cl_2$, was isolated from a mixture containing the triphosphenium ion and its protonated derivative. The central cation ring is non-planar, as in the analogous hexachlorostannate (though the structures are not isomorphous), and the P—P distances are intermediate between those typical for single and double bonds.

Comment

The cyclic triphosphenium cation was formed as its chloride salt by a reaction between 1,3-bis(diphenylphosphino)-propane (dppp) and PCl_3 as shown below:

$$3Ph_2P(CH_2)_3PPh_2 + 2PCl_3 \rightarrow 2[C_{17}H_{26}P_3]^+\cdot Cl^- + [Ph_2P(Cl)(CH_2)_3P(Cl)Ph_2]^{2+}\cdot 2Cl^-$$

A 2:1 mixture of $AlCl_3$ and ^{t}BuCl was then added, both to protonate the cation and to complex the Cl^- ion as the tetrachloroaluminate(III) (Lochschmidt & Schmidpeter, 1985; Schmidpeter *et al.*, 1985; Burton *et al.*, 2005). While clear NMR evidence for protonation was obtained, the crystals isolated from the solution were found to be from the title compound, (I), *i.e.* the unprotonated ring cation as its tetrachloroaluminate(III) salt. This cation has been structurally characterized previously using X-ray crystallography as the hexachlorostannate(IV) salt (Boon *et al.*, 2000). Selected bond distances and angles for the cations in the two structures are listed in Table 2.



(I)

Despite the close relationship between the hexachlorostannate(IV) and (I), the structures reported are very different, with the former in the space group $I4/m$ and the tetrachloroaluminate in $P\overline{1}$.

In both the hexachlorostannate(IV) salt and (I) (Fig. 1), the six-membered cyclic triphosphenium ring is non-planar, as expected. However, in the hexachlorostannate, both P—P bond lengths are identical at 2.132 (1) Å since the cation

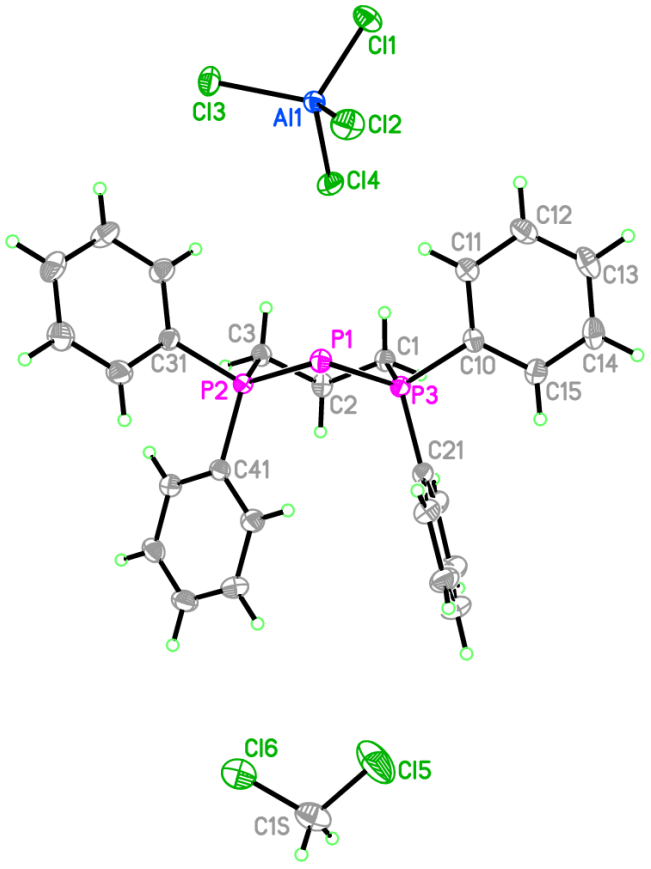


Figure 1
View of (I) with selected atoms labelled. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

occupies a position on a mirror plane, whereas in (I) there is a slight asymmetry in these distances [P1—P2 = 2.1259 (5) Å and P1—P3 = 2.1310 (5) Å]. In both structures, the P—P distance is clearly intermediate between the normal values for P—P single (2.20–2.25 Å) and P═P double bonds (2.00–2.05 Å; Schmidpeter *et al.*, 1983). These P—P bond lengths are comparable to those in the analogous five-membered ring compound [2.122 (1) and 2.128 (2) Å; Schmidpeter *et al.*, 1982], to those in the related (planar) five-membered ring with a benzene backbone [2.124 (1) and 2.122 (1) Å; Barnham *et al.*, 2001] and to those in a neutral six-membered ring compound containing three linked phosphorus atoms [2.134 (1) Å; Karsch *et al.*, 1995]. The P2—P1—P3 bond angle is slightly smaller in (I) at 95.58 (2)° compared with 96.44 (6)° in the hexachlorostannate, possibly reflecting crystal packing effects.

In (I), the charge is balanced by an isolated tetrahedral $[AlCl_4]^-$ anion, with Al—Cl distances between 2.1299 (5) and 2.1446 (5) Å [average 2.1361 (5) Å], and bond angles around the central Al atom between 108.22 (2) and 110.98 (2)° [average 109.47 (2)°]. There is also a dichloromethane solvent molecule in the structure, which is ordered and fully occupied (as is the $[AlCl_4]^-$), which is presumably due to the presence of weak C—H···Cl and Cl···Cl interactions (Table 1), though the magnitude of the anisotropic displacement parameters indicates that there is slightly increased motion compared with the rest of the structure.

Experimental

1,3-Bis(diphenylphosphino)propane (dppp) (0.503 g, 1.22 mmol) was dissolved in CH_2Cl_2 and PCl_3 (0.11 ml, 1.26 mmol) was added. The solution was stirred overnight; its ^{31}P NMR spectrum then showed that the six-membered ring cyclic triphosphenium cation had formed as its chloride salt [δ ^{31}P 23.1 (*d*, (2P), −209.4 (*t*, 1P; $^1J_{PP}$ = 424.8 Hz) (Burton *et al.*, 2005)]. $AlCl_3$ (0.344 g, 2.58 mmol) and ^{t}BuCl (0.14 ml, 1.29 mmol) were placed in a Schlenk tube, and the above solution was added to the mixture, with stirring. After overnight stirring, the $^{31}P\{^1H\}$ NMR spectrum showed formation of the protonated derivative [δ ^{31}P 13.8 (*d*, 2P), −156.1 (*t*, 1P; $^1J_{PP}$ = 226.0 Hz) (Burton *et al.*, 2005)]. This was confirmed by recording the proton-coupled spectrum, enabling $^1J_{PH}$ to be evaluated as 223.0 Hz. After filtration, the solution still showed the presence of the unprotonated ring cation. On cooling in a refrigerator, crystals of the title compound appeared after four weeks; the unprotonated ring was still present in the ^{31}P NMR spectrum of the filtrate.

Crystal data

$(C_{27}H_{26}P_3)[AlCl_4]\cdot CH_2Cl_2$
M_r = 697.09
Triclinic, $P\overline{1}$
a = 9.4446 (1) Å
b = 12.4158 (1) Å
c = 15.0802 (2) Å
α = 72.645 (1)°
β = 78.207 (1)°
γ = 77.390 (1)°
V = 1628.61 (3) Å^3
Z = 2
D_x = 1.422 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5459 reflections
θ = 2.5–28.3°
μ = 0.72 mm^{-1}
T = 120 (2) K
Block, colourless
0.20 × 0.11 × 0.07 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer
ω scans
Absorption correction: by integration (*XPREP*/*SHELXTL*; Sheldrick, 1997*a*)
T_{min} = 0.869, T_{max} = 0.951
15853 measured reflections
8053 independent reflections
7277 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 28.3°
$h = -11 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.075
S = 1.03
8053 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.9727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.97 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*A*···Cl4	0.99	2.89	3.8275 (14)	158
C3—H3*A*···Cl3^{i}	0.99	2.90	3.6609 (14)	134
C3—H3*B*···Cl4	0.99	2.90	3.8317 (14)	158
C36—H36···Cl1^{ii}	0.95	2.85	3.7275 (16)	155
C1S—H1*SA*···Cl1^{iii}	0.99	2.85	3.702 (2)	145

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, y-1, z$; (iii) $x-1, y-1, z$.

Table 2
Comparison of selected bond distances (Å) and angles (°) for the cation in the title compound as its $[SnCl_6]^{2-}$ (Boon *et al.*, 2000) and $[AlCl_4]^-$ salts.

	$[SnCl_6]^{2-}$	$[AlCl_4]^-$
P1—P2	2.132 (1)	2.1259 (5)
P1—P3	2.132 (1)	2.1310 (5)
P2—C3	1.815 (3)	1.8146 (13)
P3—C1	1.815 (3)	1.8143 (13)
C1—C2	1.535 (4)	1.5356 (18)
C2—C3	1.535 (4)	1.5362 (18)
P2—C31	1.810 (3)	1.8025 (13)
P2—C41	1.815 (3)	1.8109 (13)
P3—C10	1.815 (3)	1.8063 (13)
P3—C21	1.810 (3)	1.8060 (14)
P2—P1—P3	96.44(6)	95.579 (18)
P1—P2—C3	113.2 (1)	113.68 (4)
P2—C3—C2	113.4 (2)	112.69 (9)
C3—C2—C1	113.5 (4)	113.29 (11)
C31—P2—C41	105.9 (1)	107.16 (6)
C41—P2—C3	110.2 (1)	106.01 (6)
C31—P2—P1	103.4 (1)	104.73 (5)
C21—P3—C1	110.2 (1)	108.17 (6)
C10—P3—P1	103.4 (1)	104.71 (5)
C21—P3—C10	105.9 (1)	107.44 (6)

All H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined using a riding model, with $U_{iso} = 1.2U_{eq}$(C). The largest residual electron-density peak is located 0.88 Å from atom Cl5, as is the deepest hole.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*b*); molecular graphics: *SHELXTL* (Sheldrick, 1997*a*); software used to prepare material for publication: *SHELXTL*.

We thank Dr A. Kenwright for recording some of the NMR spectra, The Royal Society for an award under their Developing World study programme (to RMKD), and the EPSRC for a studentship (to ALT).

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supporting information

Acta Cryst. (2005). E**61**, m296–m298 [https://doi.org/10.1107/S1600536805000656]

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$M_r = 697.09$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.4446$ (1) Å
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$c = 15.0802$ (2) Å
$\alpha = 72.645$ (1)°
$\beta = 78.207$ (1)°
$\gamma = 77.390$ (1)°
$V = 1628.61$ (3) Å^3
$Z = 2$
$F(000) = 712$
$D_x = 1.422$ Mg m^{-3}
Melting point: Not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5459 reflections
$\theta = 2.5$–$28.3°$
$\mu = 0.72$ mm^{-1}
$T = 120$ K
Block, colourless
0.2 × 0.11 × 0.07 mm

Data collection

Bruker SMART CCD 6K area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω scans
Absorption correction: integration (XPREP/SHELXTL; Sheldrick, 1997a)
$T_{min} = 0.869$, $T_{max} = 0.951$
15853 measured reflections
8053 independent reflections
7277 reflections with $I > 2\sigma(I)$
$R_{int} = 0.010$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.7°$
$h = -11\rightarrow12$
$k = -16\rightarrow16$
$l = -20\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.075$
$S = 1.03$
8053 reflections
343 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.9727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.42$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

Special details

Experimental. The data collection nominally covered full sphere of reciprocal Space, by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (25 s exposure) covering 0.3° in ω. Crystal to detector distance 4.51 cm.

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}
P1	0.56045 (4)	0.45930 (3)	0.18324 (2)	0.01808 (7)
P2	0.51313 (4)	0.36113 (3)	0.32444 (2)	0.01560 (7)
P3	0.34265 (4)	0.55051 (3)	0.17372 (2)	0.01600 (7)
C1	0.25194 (15)	0.59411 (11)	0.27878 (9)	0.0188 (2)
H1A	0.3042	0.6505	0.2872	0.023*
H1B	0.1507	0.6330	0.2694	0.023*
C2	0.24455 (14)	0.49574 (12)	0.36912 (9)	0.0196 (2)
H2A	0.2020	0.4352	0.3587	0.023*
H2B	0.1781	0.5247	0.4200	0.023*
C3	0.39488 (14)	0.44310 (11)	0.40097 (9)	0.0185 (2)
H3A	0.3803	0.3925	0.4656	0.022*
H3B	0.4439	0.5052	0.4026	0.022*
C10	0.36384 (15)	0.67999 (11)	0.08106 (9)	0.0188 (2)
C11	0.46202 (16)	0.74660 (12)	0.08618 (10)	0.0247 (3)
H11	0.5156	0.7236	0.1375	0.030*
C12	0.48110 (18)	0.84668 (13)	0.01596 (11)	0.0300 (3)
H12	0.5481	0.8919	0.0192	0.036*
C13	0.40217 (19)	0.88063 (13)	−0.05913 (11)	0.0301 (3)
H13	0.4152	0.9492	−0.1070	0.036*
C14	0.30497 (19)	0.81480 (14)	−0.06422 (11)	0.0303 (3)
H14	0.2513	0.8383	−0.1156	0.036*
C15	0.28528 (17)	0.71412 (13)	0.00569 (10)	0.0251 (3)
H15	0.2186	0.6689	0.0020	0.030*
C21	0.22155 (14)	0.47883 (11)	0.14163 (9)	0.0190 (2)
C22	0.27765 (16)	0.42033 (13)	0.07254 (10)	0.0244 (3)
H22	0.3771	0.4191	0.0438	0.029*
C23	0.18856 (19)	0.36406 (14)	0.04587 (12)	0.0315 (3)
H23	0.2261	0.3256	−0.0020	0.038*
C24	0.04398 (19)	0.36422 (16)	0.08953 (13)	0.0363 (4)
H24	−0.0167	0.3245	0.0722	0.044*
C25	−0.01191 (18)	0.42155 (16)	0.15767 (13)	0.0357 (4)

H25	−0.1110	0.4212	0.1870	0.043*
C26	0.07568 (16)	0.48016 (13)	0.18395 (11)	0.0262 (3)
H26	0.0363	0.5207	0.2303	0.031*
C31	0.68874 (14)	0.31293 (11)	0.36512 (9)	0.0185 (2)
C32	0.75089 (16)	0.38556 (13)	0.39541 (10)	0.0241 (3)
H32	0.6991	0.4600	0.3966	0.029*
C33	0.88822 (17)	0.34893 (14)	0.42368 (12)	0.0294 (3)
H33	0.9298	0.3979	0.4452	0.035*
C34	0.96503 (18)	0.24101 (15)	0.42064 (14)	0.0362 (4)
H34	1.0589	0.2160	0.4403	0.043*
C35	0.90506 (19)	0.16953 (14)	0.38887 (16)	0.0404 (4)
H35	0.9586	0.0961	0.3859	0.049*
C36	0.76683 (17)	0.20506 (13)	0.36133 (13)	0.0295 (3)
H36	0.7257	0.1558	0.3399	0.035*
C41	0.43240 (14)	0.23518 (11)	0.34279 (9)	0.0177 (2)
C42	0.40349 (15)	0.16479 (12)	0.43380 (10)	0.0212 (3)
H42	0.4267	0.1827	0.4856	0.025*
C43	0.34060 (16)	0.06847 (12)	0.44799 (11)	0.0242 (3)
H43	0.3209	0.0204	0.5097	0.029*
C44	0.30660 (16)	0.04240 (12)	0.37240 (11)	0.0264 (3)
H44	0.2638	−0.0236	0.3825	0.032*
C45	0.33482 (17)	0.11221 (13)	0.28198 (11)	0.0273 (3)
H45	0.3107	0.0943	0.2305	0.033*
C46	0.39858 (16)	0.20850 (12)	0.26699 (10)	0.0226 (3)
H46	0.4190	0.2559	0.2051	0.027*
Al1	0.73228 (5)	0.77435 (3)	0.30260 (3)	0.01928 (9)
Cl1	0.72424 (5)	0.95008 (3)	0.29338 (3)	0.03079 (9)
Cl2	0.81252 (5)	0.74412 (4)	0.16798 (3)	0.03431 (9)
Cl3	0.87542 (4)	0.66723 (3)	0.39908 (3)	0.02840 (8)
Cl4	0.51639 (4)	0.73263 (3)	0.35352 (3)	0.02857 (8)
C1S	0.0779 (2)	−0.05980 (15)	0.13328 (14)	0.0401 (4)
H1SA	−0.0297	−0.0569	0.1489	0.048*
H1SB	0.1181	−0.1200	0.0998	0.048*
Cl1S	0.11409 (6)	0.07262 (4)	0.05784 (5)	0.06626 (18)
Cl2S	0.15236 (8)	−0.09790 (4)	0.23782 (5)	0.05955 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01489 (15)	0.02038 (16)	0.01723 (15)	−0.00291 (12)	−0.00128 (12)	−0.00317 (12)
P2	0.01491 (15)	0.01548 (14)	0.01654 (15)	−0.00244 (11)	−0.00321 (11)	−0.00397 (11)
P3	0.01514 (15)	0.01713 (15)	0.01526 (15)	−0.00306 (11)	−0.00257 (11)	−0.00317 (12)
C1	0.0188 (6)	0.0186 (6)	0.0181 (6)	−0.0005 (5)	−0.0025 (5)	−0.0054 (5)
C2	0.0175 (6)	0.0220 (6)	0.0163 (6)	−0.0007 (5)	−0.0005 (5)	−0.0042 (5)
C3	0.0199 (6)	0.0188 (6)	0.0163 (6)	−0.0016 (5)	−0.0030 (5)	−0.0048 (5)
C10	0.0189 (6)	0.0181 (6)	0.0173 (6)	−0.0018 (5)	−0.0022 (5)	−0.0028 (5)
C11	0.0265 (7)	0.0218 (6)	0.0256 (7)	−0.0065 (5)	−0.0069 (6)	−0.0019 (5)
C12	0.0330 (8)	0.0223 (7)	0.0328 (8)	−0.0106 (6)	−0.0039 (6)	−0.0010 (6)

C13	0.0376 (8)	0.0218 (7)	0.0232 (7)	−0.0041 (6)	0.0001 (6)	0.0017 (5)
C14	0.0367 (8)	0.0290 (7)	0.0204 (7)	−0.0019 (6)	−0.0080 (6)	0.0004 (6)
C15	0.0272 (7)	0.0258 (7)	0.0219 (7)	−0.0049 (6)	−0.0079 (5)	−0.0024 (5)
C21	0.0181 (6)	0.0196 (6)	0.0192 (6)	−0.0045 (5)	−0.0053 (5)	−0.0025 (5)
C22	0.0225 (7)	0.0283 (7)	0.0242 (7)	−0.0045 (5)	−0.0045 (5)	−0.0091 (6)
C23	0.0345 (8)	0.0338 (8)	0.0336 (8)	−0.0052 (6)	−0.0123 (7)	−0.0155 (7)
C24	0.0321 (8)	0.0397 (9)	0.0459 (10)	−0.0126 (7)	−0.0164 (7)	−0.0128 (8)
C25	0.0218 (7)	0.0480 (10)	0.0409 (9)	−0.0139 (7)	−0.0041 (6)	−0.0116 (8)
C26	0.0190 (6)	0.0335 (7)	0.0268 (7)	−0.0058 (6)	−0.0029 (5)	−0.0084 (6)
C31	0.0164 (6)	0.0194 (6)	0.0191 (6)	−0.0023 (5)	−0.0040 (5)	−0.0036 (5)
C32	0.0200 (6)	0.0266 (7)	0.0285 (7)	−0.0013 (5)	−0.0053 (5)	−0.0122 (6)
C33	0.0214 (7)	0.0386 (8)	0.0339 (8)	−0.0046 (6)	−0.0082 (6)	−0.0159 (7)
C34	0.0216 (7)	0.0386 (9)	0.0497 (10)	0.0014 (6)	−0.0161 (7)	−0.0109 (8)
C35	0.0261 (8)	0.0251 (8)	0.0709 (13)	0.0051 (6)	−0.0182 (8)	−0.0134 (8)
C36	0.0229 (7)	0.0211 (7)	0.0470 (9)	−0.0012 (5)	−0.0100 (6)	−0.0115 (6)
C41	0.0154 (6)	0.0162 (5)	0.0211 (6)	−0.0021 (4)	−0.0029 (5)	−0.0048 (5)
C42	0.0216 (6)	0.0209 (6)	0.0211 (6)	−0.0037 (5)	−0.0042 (5)	−0.0047 (5)
C43	0.0231 (7)	0.0196 (6)	0.0267 (7)	−0.0050 (5)	−0.0022 (5)	−0.0015 (5)
C44	0.0242 (7)	0.0201 (6)	0.0362 (8)	−0.0067 (5)	−0.0040 (6)	−0.0079 (6)
C45	0.0303 (7)	0.0268 (7)	0.0303 (7)	−0.0083 (6)	−0.0079 (6)	−0.0113 (6)
C46	0.0251 (7)	0.0223 (6)	0.0213 (6)	−0.0048 (5)	−0.0045 (5)	−0.0059 (5)
Al1	0.0219 (2)	0.01810 (18)	0.01827 (19)	−0.00393 (15)	−0.00434 (15)	−0.00416 (15)
Cl1	0.0405 (2)	0.01926 (15)	0.03274 (19)	−0.00635 (14)	−0.00286 (15)	−0.00794 (13)
Cl2	0.0443 (2)	0.0402 (2)	0.02303 (17)	−0.01149 (17)	−0.00072 (15)	−0.01501 (15)
Cl3	0.02176 (16)	0.03175 (18)	0.02578 (17)	−0.00138 (13)	−0.00626 (13)	0.00072 (13)
Cl4	0.02303 (16)	0.03265 (18)	0.03203 (18)	−0.00910 (14)	−0.00684 (14)	−0.00659 (14)
C1S	0.0458 (10)	0.0322 (8)	0.0433 (10)	−0.0166 (7)	0.0021 (8)	−0.0100 (7)
Cl1S	0.0487 (3)	0.0339 (2)	0.0981 (5)	−0.0129 (2)	−0.0053 (3)	0.0098 (3)
Cl2S	0.0791 (4)	0.0378 (2)	0.0722 (4)	−0.0077 (2)	−0.0342 (3)	−0.0163 (2)

Geometric parameters (Å, º)

P1—P2	2.1259 (5)	C24—H24	0.9500
P1—P3	2.1310 (5)	C25—C26	1.393 (2)
P2—C31	1.8025 (13)	C25—H25	0.9500
P2—C41	1.8109 (13)	C26—H26	0.9500
P2—C3	1.8146 (13)	C31—C36	1.3922 (19)
P3—C21	1.8060 (14)	C31—C32	1.3973 (19)
P3—C10	1.8063 (13)	C32—C33	1.387 (2)
P3—C1	1.8143 (13)	C32—H32	0.9500
C1—C2	1.5356 (18)	C33—C34	1.386 (2)
C1—H1A	0.9900	C33—H33	0.9500
C1—H1B	0.9900	C34—C35	1.386 (2)
C2—C3	1.5362 (18)	C34—H34	0.9500
C2—H2A	0.9900	C35—C36	1.390 (2)
C2—H2B	0.9900	C35—H35	0.9500
C3—H3A	0.9900	C36—H36	0.9500
C3—H3B	0.9900	C41—C46	1.3927 (19)

C10—C15	1.3935 (19)	C41—C42	1.3991 (19)
C10—C11	1.3971 (19)	C42—C43	1.3908 (19)
C11—C12	1.390 (2)	C42—H42	0.9500
C11—H11	0.9500	C43—C44	1.387 (2)
C12—C13	1.392 (2)	C43—H43	0.9500
C12—H12	0.9500	C44—C45	1.389 (2)
C13—C14	1.382 (2)	C44—H44	0.9500
C13—H13	0.9500	C45—C46	1.393 (2)
C14—C15	1.394 (2)	C45—H45	0.9500
C14—H14	0.9500	C46—H46	0.9500
C15—H15	0.9500	Al1—Cl1	2.1299 (5)
C21—C26	1.3938 (19)	Al1—Cl2	2.1302 (5)
C21—C22	1.3970 (19)	Al1—Cl3	2.1396 (5)
C22—C23	1.386 (2)	Al1—Cl4	2.1446 (5)
C22—H22	0.9500	Cl2S—C1S	1.751 (2)
C23—C24	1.389 (3)	C1S—Cl1S	1.7564 (18)
C23—H23	0.9500	C1S—H1SA	0.9900
C24—C25	1.375 (3)	C1S—H1SB	0.9900

P2—P1—P3	95.579 (18)	C25—C24—C23	120.47 (15)
C31—P2—C41	107.16 (6)	C25—C24—H24	119.8
C31—P2—C3	107.94 (6)	C23—C24—H24	119.8
C41—P2—C3	106.01 (6)	C24—C25—C26	120.47 (15)
C31—P2—P1	104.73 (5)	C24—C25—H25	119.8
C41—P2—P1	116.88 (5)	C26—C25—H25	119.8
C3—P2—P1	113.68 (4)	C25—C26—C21	119.43 (14)
C21—P3—C10	107.44 (6)	C25—C26—H26	120.3
C21—P3—C1	108.17 (6)	C21—C26—H26	120.3
C10—P3—C1	106.83 (6)	C36—C31—C32	119.68 (13)
C21—P3—P1	115.27 (5)	C36—C31—P2	119.79 (11)
C10—P3—P1	104.71 (5)	C32—C31—P2	120.44 (10)
C1—P3—P1	113.86 (5)	C33—C32—C31	119.96 (13)
C2—C1—P3	114.58 (9)	C33—C32—H32	120.0
C2—C1—H1A	108.6	C31—C32—H32	120.0
P3—C1—H1A	108.6	C34—C33—C32	120.16 (14)
C2—C1—H1B	108.6	C34—C33—H33	119.9
P3—C1—H1B	108.6	C32—C33—H33	119.9
H1A—C1—H1B	107.6	C35—C34—C33	120.08 (14)
C1—C2—C3	113.29 (11)	C35—C34—H34	120.0
C1—C2—H2A	108.9	C33—C34—H34	120.0
C3—C2—H2A	108.9	C34—C35—C36	120.15 (15)
C1—C2—H2B	108.9	C34—C35—H35	119.9
C3—C2—H2B	108.9	C36—C35—H35	119.9
H2A—C2—H2B	107.7	C35—C36—C31	119.95 (14)
C2—C3—P2	112.69 (9)	C35—C36—H36	120.0
C2—C3—H3A	109.1	C31—C36—H36	120.0
P2—C3—H3A	109.1	C46—C41—C42	120.02 (12)
C2—C3—H3B	109.1	C46—C41—P2	120.37 (10)

P2—C3—H3B	109.1	C42—C41—P2	119.61 (10)
H3A—C3—H3B	107.8	C43—C42—C41	119.62 (13)
C15—C10—C11	119.92 (13)	C43—C42—H42	120.2
C15—C10—P3	121.70 (11)	C41—C42—H42	120.2
C11—C10—P3	118.38 (10)	C44—C43—C42	120.20 (13)
C12—C11—C10	119.85 (14)	C44—C43—H43	119.9
C12—C11—H11	120.1	C42—C43—H43	119.9
C10—C11—H11	120.1	C43—C44—C45	120.34 (13)
C11—C12—C13	120.06 (14)	C43—C44—H44	119.8
C11—C12—H12	120.0	C45—C44—H44	119.8
C13—C12—H12	120.0	C44—C45—C46	119.88 (14)
C14—C13—C12	120.12 (14)	C44—C45—H45	120.1
C14—C13—H13	119.9	C46—C45—H45	120.1
C12—C13—H13	119.9	C41—C46—C45	119.93 (13)
C13—C14—C15	120.29 (14)	C41—C46—H46	120.0
C13—C14—H14	119.9	C45—C46—H46	120.0
C15—C14—H14	119.9	Cl1—Al1—Cl2	109.61 (2)
C10—C15—C14	119.76 (14)	Cl1—Al1—Cl3	110.55 (2)
C10—C15—H15	120.1	Cl2—Al1—Cl3	108.60 (2)
C14—C15—H15	120.1	Cl1—Al1—Cl4	108.87 (2)
C26—C21—C22	119.79 (13)	Cl2—Al1—Cl4	110.98 (2)
C26—C21—P3	121.92 (11)	Cl3—Al1—Cl4	108.22 (2)
C22—C21—P3	118.28 (10)	Cl2S—C1S—Cl1S	113.64 (11)
C23—C22—C21	120.18 (14)	Cl2S—C1S—H1SA	108.8
C23—C22—H22	119.9	Cl1S—C1S—H1SA	108.8
C21—C22—H22	119.9	Cl2S—C1S—H1SB	108.8
C22—C23—C24	119.64 (15)	Cl1S—C1S—H1SB	108.8
C22—C23—H23	120.2	H1SA—C1S—H1SB	107.7
C24—C23—H23	120.2		

P3—P1—P2—C31	−160.15 (5)	P3—C21—C22—C23	179.94 (12)
P3—P1—P2—C41	81.48 (5)	C21—C22—C23—C24	−1.3 (2)
P3—P1—P2—C3	−42.57 (5)	C22—C23—C24—C25	1.2 (3)
P2—P1—P3—C21	−85.47 (5)	C23—C24—C25—C26	−0.1 (3)
P2—P1—P3—C10	156.71 (5)	C24—C25—C26—C21	−1.0 (3)
P2—P1—P3—C1	40.39 (5)	C22—C21—C26—C25	0.9 (2)
C21—P3—C1—C2	73.30 (11)	P3—C21—C26—C25	−178.79 (12)
C10—P3—C1—C2	−171.31 (10)	C41—P2—C31—C36	29.28 (14)
P1—P3—C1—C2	−56.22 (11)	C3—P2—C31—C36	143.06 (12)
P3—C1—C2—C3	69.03 (13)	P1—P2—C31—C36	−95.50 (12)
C1—C2—C3—P2	−70.70 (13)	C41—P2—C31—C32	−154.40 (11)
C31—P2—C3—C2	176.15 (9)	C3—P2—C31—C32	−40.62 (13)
C41—P2—C3—C2	−69.30 (10)	P1—P2—C31—C32	80.82 (12)
P1—P2—C3—C2	60.45 (10)	C36—C31—C32—C33	−1.6 (2)
C21—P3—C10—C15	4.74 (14)	P2—C31—C32—C33	−177.89 (12)
C1—P3—C10—C15	−111.14 (12)	C31—C32—C33—C34	1.0 (2)
P1—P3—C10—C15	127.77 (11)	C32—C33—C34—C35	0.3 (3)
C21—P3—C10—C11	−175.25 (11)	C33—C34—C35—C36	−1.0 (3)

C1—P3—C10—C11	68.87 (12)	C34—C35—C36—C31	0.4 (3)
P1—P3—C10—C11	−52.21 (12)	C32—C31—C36—C35	0.9 (2)
C15—C10—C11—C12	−0.1 (2)	P2—C31—C36—C35	177.24 (14)
P3—C10—C11—C12	179.91 (12)	C31—P2—C41—C46	−119.56 (11)
C10—C11—C12—C13	0.2 (2)	C3—P2—C41—C46	125.36 (11)
C11—C12—C13—C14	−0.2 (2)	P1—P2—C41—C46	−2.51 (13)
C12—C13—C14—C15	0.0 (2)	C31—P2—C41—C42	60.71 (12)
C11—C10—C15—C14	−0.1 (2)	C3—P2—C41—C42	−54.37 (12)
P3—C10—C15—C14	179.92 (12)	P1—P2—C41—C42	177.76 (9)
C13—C14—C15—C10	0.1 (2)	C46—C41—C42—C43	−0.2 (2)
C10—P3—C21—C26	−104.38 (12)	P2—C41—C42—C43	179.58 (11)
C1—P3—C21—C26	10.61 (13)	C41—C42—C43—C44	−0.1 (2)
P1—P3—C21—C26	139.34 (11)	C42—C43—C44—C45	−0.1 (2)
C10—P3—C21—C22	75.91 (12)	C43—C44—C45—C46	0.5 (2)
C1—P3—C21—C22	−169.10 (11)	C42—C41—C46—C45	0.5 (2)
P1—P3—C21—C22	−40.37 (12)	P2—C41—C46—C45	−179.20 (11)
C26—C21—C22—C23	0.2 (2)	C44—C45—C46—C41	−0.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···Cl4	0.99	2.89	3.8275 (14)	158
C3—H3*A*···Cl3^{i}	0.99	2.90	3.6609 (14)	134
C3—H3*B*···Cl4	0.99	2.90	3.8317 (14)	158
C36—H36···Cl1^{ii}	0.95	2.85	3.7275 (16)	155
C1*S*—H1*SA*···Cl1^{iii}	0.99	2.85	3.702 (2)	145

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